DESIGN AND DEVELOPMENT OF HERITAGE MORTAR FOR RESTORATION

BY

MAPHOLE EMELLY LOKE

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Principal supervisor: Professor Kumar Pallav
External supervisor: Professor Giuseppe Cultrone

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Declaration

I, Maphole Emelly Loke, declare that the contents of this thesis represent my unaided work and that the thesis has not previously been submitted for academic examination towards any qualification. Furthermore, it expresses my own opinions, not necessarily those of the Cape Peninsula University of Technology or the Universidad de Granada.

Signed

18th December 2023
Date

Dedication

To nkhono *'Mats'eliso Loke*: Thank you for sacrificing all to ensure my dream of becoming a Doctor comes true. This work is your belated 80th birthday present from my heart to yours. Thank you for spending years of your life dedicating all to my academic success. I know the heavens shall reward you in ways my abilities fail. Long live my light in the darkness, my sister, friend, mother, father, grandparent, guardian, protector, prayer warrior, and partner. I could never have achieved this without your undeserved everlasting support, prayers, and cheering.

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Ke sa leboha hape Tlou!

Isiah 60: 22 ~ When the time is right, I, the Lord, shall make it happen.

Abstract

Historical buildings are a narrative of the countries' written and unwritten history. Their long-term existence ensures generational knowledge of past events, milestones, construction developments and evolution in materials, designs, concepts, and practices throughout the centuries. It is indisputable that heritage buildings' survival against human behaviour, natural disasters, and environmental and atmospheric attacks through the years has proven the use of durable materials for their construction. Inevitable decay due to long-term exposure to deterioration factors is a common problem for these monuments. This has resulted in a need to execute restoration works to reinstate the heritage structures to their original appearance, physical state, and strength for extended survival.

Several restoration projects in the past and recent times have been performed to reinstate the missing elements on heritage structures, masonry mortars in particular. Unfortunately, many of these restoration interventions on these structures have been erroneous and led to poor results from a restoration standpoint, especially regarding aesthetic criteria. The most common mistake in restoration work is the use of incompatible restoration materials due to a misunderstanding of the materials used on these historic wonders. This has resulted in a waste of resources due to consequential repeated restoration projects and the loss of original structural concepts in terms of appearance and integrity. These common mistakes can potentially threaten the historical significance of heritage structures. The problem extends further to designing and producing mortars that are compatible and their assessment for compatibility and durability.

To overcome the problems associated with material incompatibility, researchers and heritage restorers have discovered the applicability of analysing original mortars for their chemical, mineralogical, physical, and mechanical properties preceding the execution of restoration works (ICOMOS, Venice Charter, 1964). The experimental analysis of these mortars is believed to offer promising results for the long-term survival of restored historical masonries. To align the current study with the literature regarding sustainable and compatible restoration of historical mortars, two phases were examined. The initial phase of this study comprised an experimental analysis of the samples extracted from the masonry joints, the floor, plaster, and renders of the ancient colonial edifice in Cape Town, South Africa, Castle of Good Hope built in 1666. The building precincts on Robben Island were constructed between 1700s and 1800s. A total of nine representative samples were carefully extracted from the Castle of Good Hope, twelve from the Pre-primary school building and three samples from the Maximum-security prison on Robben Island for analysis. The sample size was decided based on material availability, considering the restriction associated with causing as little destruction as possible on historical structures.

Phase 1 of this doctoral thesis involved the determination of the aesthetic properties of the collected mortars using spectrophotometry, the chemical composition through X-ray fluorescence (XRF), the mineralogy using powder X-ray diffraction (PXRD) and thermogravimetry - differential scanning calorimetry (TGA-DSC), the microtexture by environmental scanning electron microscopy (ESEM) and the analysis of the porous system through mercury intrusion porosimetry (MIP). The original mortars from the Castle were found to be earth and hydraulic lime-based, with a porosity of between 21 and 38%. The Island building's mortars were mostly natural cement-based and hydraulic lime-based, highlighting possible previous restoration works using natural cement. These mortars portrayed a porosity lower than mortars from the Castle (18 to 30%). The raw materials used on these monuments include feldspar aggregates, possibly from the West Coast (Cape Town) and sub-hydraulic lime.

Phase 2 of this study involved the designand development of eight different mortar mixes following a unique procedure invented in this research work. The freshly mixed mortars were evaluated for consistency. In contrast, the hardened cubes of 40 × 40 × 40 mm and beams of 40 × 40 × 160 mm were evaluated for compatibility and durability using destructive (hygric tests, ageing tests through salt crystallisation and freeze-thaw cycles, compressive and centre point loading flexural strength tests) and a non-destructive techniques (ultrasound pulse velocity, UPV). The durability tests aimed to assess the new mortars' performance and verify their long-term existence in restoration practice. The old mortars properties were also assessed against the new ones, and restoration interventions made.

For restoration purposes, a hydrated lime-based mortar with a binder-to-aggregate ratio of 1:3 by weight, made of West Coast Sea sand and 5% seashell content with a porosity of 24% proved to be the most durable among the eight produced mortars. Meanwhile, the aggregates of similar sources with the addition of natural cement are proposed for earth mortars. The aesthetics of all the mortars were difficult to achieve, given the ageing factor of the original materials. Thus, the use of colour-enhancing pigments is recommended. A standardised guideline for producing compatible and durable mortars has been documented for restorers to execute the works on historic structures properly. This research confirmed that the compatibility and durability of heritage mortars depends not only on their performance, but also on their ability to match the properties of the existing materials with appropriate application techniques by skilled masons.

Keywords: historical mortar, design guideline, development procedure, mix design, compatible restoration

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Publications and Conferences

The findings of this study have been published in the following journals and presented and published in the mentioned conference proceedings.

Peer-reviewed journal articles and book chapters

1. Loke, M.E., Kumar, P., Cultrone, G. & Di Filippo, C. 2023. Investigating the standard design and production procedure of heritage mortars for compatible and durable masonry restoration, *Journal of Building Engineering* (revised manuscript under review).
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3. Loke, M.E., Kumar, P. & Cultrone, G. 2023. Challenges in characterisation and development of suitable historic repair mortars. *International Journal of Conservation Science*, ISSN:2067-533X. Vol. 14, Issue 3, July-September. DOI: 10.36868/IJCS.2023.03.02.
4. Loke, M. 2022. Sustainable restoration of local heritage structures: an engineering approach. *Civil Engineering= Siviele Ingenieurswese,* 30 (9), pp.36-37.

Conferences

1. Loke, M.E., Kumar, P. & Cultrone, G. 2024. Non-destructive testing of historic masonry of Robben Island – comparison of mechanical testing techniques for long-term conservation practice, *18th International Brick and Block Masonry Conference*. 21 – 24 July 2024, University of Birmingham, United Kingdom, Lecture Notes in Civil Engineering (Springer) and indexed in Scopus (Abstract accepted).

Other research and collaborations from this research

1. Participation in the Knowledge Exchange Webinar 5 for Sri Lanka, South Africa and Netherlands (SL-SA-NL) on Sustainable Conservation, 8 December 2023.
2. Participation and presentation of research at African World Heritage Fund Workshop on World Heritage Interpretation and Presentation in Africa, 19 – 24 October, 30 October – 2 November 2023, Twyfelfontein, Namibia.
3. Investigation of South African Parliament (National Assembly) mortars after fire disaster.
4. Presented the research at the SAHRA annual heritage week in September 2023 and 2022.
5. Participation in the Robben Island Museum Strategic Research Plan (2023-2028) to be integrated into the 4th IMP, 9-10 February 2023

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Nomenclature

Constants

A_b	Free water absorption
A_x	Pore interconnection degree
ΔE^*	Degree of colour difference
ΔL^*, Δa^*, Δb^*	Changes in L*, a* and b*
a*	Red/green chromatic coordinate
b	Beam width in mm
b*	Yellow/blue chromatic coordinate
b/a	Binder-to-aggregate ratio
B_0	Initial brucite content at time zero while using TGA/DSC analysis
B_x	Amount of brucite at the time x on TGA/DSC analysis
C*	Chroma
CI	Cementation Index
d	Height of the specimen in the place of rupture (mm)
D_i	Drying index
E_{dyn}	Dynamic modulus of elasticity
f	Flexural strength by centre loading test (MPa)
F	The maximum failure load in flexural strength calculation (N)
h^o	Hue angle
l	Beam sample span (mm)
L*	Lightness
M_H	Hydrostatic mass (g)
M_L	Mass of the sample saturated with water at atmospheric pressure (until constant mass is reached) (g)
M_{Mg}	Soluble magnesium
M_0	Initial mass of a sample (g)
M_s	The saturated mass (g)
M_t	Decreasing water weight content as a function of time
M_{24h}	Sample mass after 24 hr in water
P_o	Open porosity
P_{oMIP}	Open porosity by mercury intrusion porosimetry
P_0	Initial portlandite content at time zero while using TGA/DSC analysis
P_x	Amount of portlandite at the time x on TGA/DSC analysis
S	Saturation coefficient
t_f	The end time of the drying test
t_o	The initial time of the drying test
Vp	Longitudinal wave velocity

Vs	Shear wave velocity
X,Y,Z	Tristimulus values XYZ for 2° Standard Observer or X10, Y10, Z10 for 10° Supplementary Standard Observer of the specimen.
Xn, Yn, Zn	Tristimulus values XYZ for 2° Standard Observer or X10, Y10, Z10 for 10° Supplementary Standard Observer of a perfect reflecting diffuser.

Greek letter

ρ_a	Apparent density
ρ_r	Real (or skeletal) density
ν	Poisson's coefficient

Terms and concepts

AAS	Absorptive Atomic Spectroscopy
ASTM	American Society for Testing and Materials
B/A	Binder-to-aggregate ratio
BRE	Building Research Establishment
C-S-H	Calcium-Silicate-Hydrate
C&CI	Cement and concrete institute methods
CIC	Centro de Instumentación Científica (Centre for Scientific Instrumentation)
CIE	Commission Internationale de l'Eclairage (International Commission on Illumination)
CMP	Conservation Management Plan
CPUT	Cape Peninsula University of Technology
DOI	Digital Object Identifier
DSC	Differential Scanning Calorimetry
DTA	Differential Thermogravimetric Analysis
EDS	Energy Dispersive X-ray Spectroscopy
EDTA	Ethylenediaminetetraacetic Acid
EN	European standards of material testing
ESEM	Environmental scanning electron microscopy
FM	Fineness modulus
FTIR	Fourier Transform Infra-Red spectrometry
GDP	Gross Domestic Product
HI	Hydraulicity Index
IC	Ion Chromatography
ICCROM	International Centre for Study of the Preservation and Restoration of Cultural Property
ICP- OES	Inductively Coupled Plasma-Optical Emission Spectroscopy

ICR	Carbonation Degree Index
MDT	Minor Destructive Testing
MIP	Mercury intrusion porosimetry
MSDS	Material Safety Data Sheet
NDT	Non-Destructive Testing
NHL	Natural Hydraulic Lime
OPC	Ordinary Portland Cement
OSL	Optically stimulated luminescence
POM	Polarised optical microscopy
PSD	Pore size distribution
PXRD	Powder X-ray diffraction
REP	Research exchange program
RI	Robben Island
RILEM	International Union of Laboratories and Experts in Construction Materials, Systems and Structures
SABS	South African Bureau of Standards
SAHRA	South African Heritage Resources Agency
SANS	South African National Standards
SAP	Super absorbent polymers
SSA	Specific surface area
TEA	Triethanolamine
TGA	Thermogravimetric analysis
UGR	Universidad de Granada (University of Granada)
UNESCO	United Nations Educational, Scientific and Cultural Organisation
UPV	Ultrasonic Pulse Velocity
VOC	Dutch East India Company (Verenigde Oostindische Compagnie)
XRF	X-ray fluorescence

Chapter 1 Introduction

Mortars on heritage structures are susceptible to different deterioration factors and must be repaired and protected against such factors (Vukindu, 2021). The restoration and conservation of historic mortars are complex, involving many problems that need to be addressed (Caroselli *et al.,* 2021). Historic monuments were mainly built with lime mortars, which have been replaced with Portland cement-based binders in most restoration cases (Kang *et al*., 2019). Frequent restoration interventions on these structures use Portland cement, further exacerbating the decay of historical masonries where these mortars are used (Hawass, 1995). The Portland cement-based materials are unsuitable for the longstanding economic feasibility, sustainability, and authenticity of heritage structures (Kumar & Kumar, 2022). As a result of faulty repair work on historic structures, numerous valuable monuments and artifacts of significant historic significance have been lost. The main reason for the loss of these structures is the use of incompatible restoration materials, demonstrating a knowledge and practice gap (Feizolahbeigi, 2021).

This thesis focuses mainly on the key aspects and practices in the design and development of compatible restoration mortars for historic building masonries. It outlines the common restoration mistakes and the challenges facing the original historic mortar characterisation and offers valuable advice on sustainable and durable historic mortar repairs. Chapter 1 presents the background and problems related to historic mortar restoration, the aims and objectives, the questions to be addressed, and a thesis outline.

1.1 Research background and motivation

Buildings of historical significance have exceptional features (either architectural design or materials used) that distinguish them from modern structures (Hormes *et al.*, 2016). These features make the monuments stand out from the rest of the structures from recent eras, such as the marvellous designs by Antoni Gaudí in Barcelona (Spain) and other monuments such as the Colosseum in Rome (Italy), as depicted in Figure 1.1.

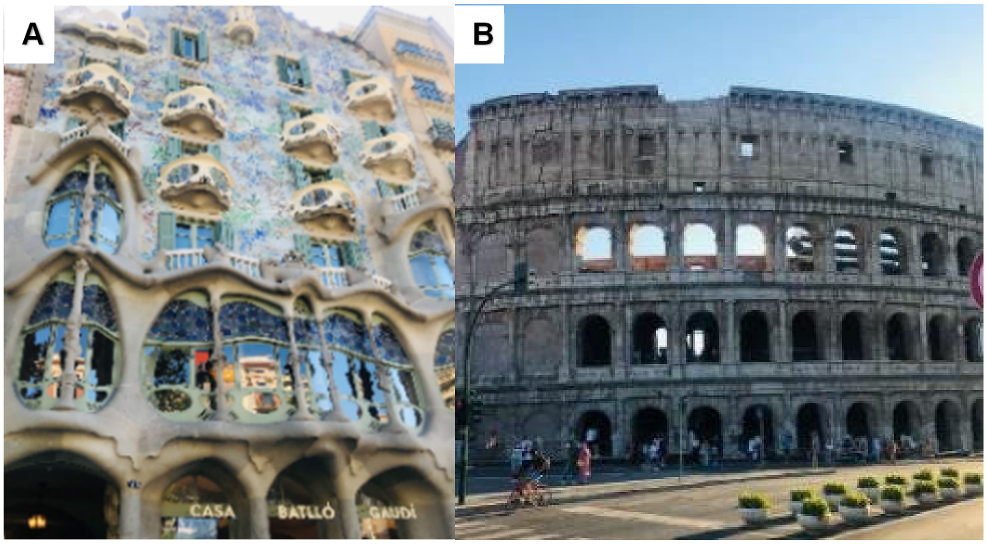


Figure 1.1 The UNESCO World heritage sites: a) Casa Batlló in Barcelona, Spain and b) the Colosseum in Rome, Italy

The most significant and oldest surviving monuments in the Western Cape (South Africa), are the Castle of Good Hope and the building precincts on Robben Island. These structures are under the protection of the South African Heritage Resources Agency (SAHRA) and the Department of Sports, Arts and Culture agency. SAHRA ensures that no unauthorised restoration and conservation works occur on these and other South African heritage buildings. And, if approved restoration is conducted, it is done to ensure compatibility while preserving the structure's authenticity for future generations. Heritage masonries were built using mortar components, many of which have been replaced with modern materials.

Even though SAHRA makes all efforts to ensure the existence of these structures, their mortars still decay because of inevitable ageing and other environmental factors. This threatens the heritage masonry’s existence as the mortar is regarded as one of the primary construction materials, especially its functioning and long-term existence (Salvadori, 1982, Holmes & Wingate, 1997). Most studies, especially in the United States of America and the United Kingdom, have focused on original heritage mortar analysis before commencing restoration projects. This results to longevity and enhances the structural service life of historic buildings (Park, 1988; Groot *et al*., 2007 and Van Domelen, 2009). The fundamental objective is to preserve and maintain the structure’s authenticity by matching the properties of the original materials to avoid disparity (Van Domelen, 2009). Much of the repair work done in the past on historical structures has been faulty (Arizzi, 2012). Portland cement is causing many problems (see Figure 1.2) faced by the restoration of historic mortars. This has caused regrettable and irreparable damages. The present state of the original appearance of these structures is a concern, not only in South Africa but also globally.

Figure 1.2 Faulty mortar repairs on some buildings on Robben Island

The restoration work conducted on these structures leaves many of the heritage buildings as safety hazards as these monumental structures undergo premature deterioration of such repairs (Al-Ostaz *et al.*, 2010 and Lukovic, 2016). According to Arito (2018), just half of the 215 case studies conducted in the United Kingdom by the Building Research Establishment (BRE) on the performance of concrete and mortar restoration materials were effective. He further highlighted that some researchers - Tilly (2006), Matthews (2007), Tilly and Jacobs (2007), Matthews and Morlidge (2008) studied 230 repair cases in Europe; of them, 20% displayed signs of failure within 5 years, 55% within 10 years, and 90% within 25 years of service. The buildings investigated were found to be more prone to cracking and debonding of repairs. Several research is undertaken in this regard to establish the root causes of early repair failure in these structures.

Ghezal and Assaf (2014) and Arito (2018) concluded that repair failures were caused by an incorrect diagnosis of the original cause of deterioration, incorrect and/or inappropriate design of intervention works and materials and inappropriate specifications and poor choice and selection of repair materials among others. The most favoured Portland cement-based mortars often fail to perform adequately because of variances in physical, chemical, mineralogical, mechanical properties, and overall behavior when applied to existing historic lime-based mortars. The use of modern binders (Portland cement-based) is said to be unsuitable as the materials with different properties are expected to perform together, both subjected to the same environmental stresses (differential thermal expansion, variances in water absorption, and water vapour transports among others), but with completely different reactions to these factors (Válek *et al*., 2019). This indicates a gap between the construction materials industry and the design of historic mortars. Although there is sufficient literature indicating the downsides of using materials with different properties on historic structures, no effort has been made to guide the industry in the design and manufacture of suitable restoration mortars. Binder production has mostly focused on cement-based binders for mass production, with less emphasis on lime-based binders for traditional masonries. This causes additional damage to the recovered surfaces and wastes resources because recurring repairs are always necessary (Van Domelen, 2009).

To overcome the early deterioration of historical mortar repairs, it is recommended that one fully studies the heritage mortars before commencement of restoration work. This is currently regarded as the most important and necessary element of the restoration process, as it is thought to be the only procedure that ensures compatibility of original and repair mortars while also allowing the longevity of the latter to be estimated at the outset (Papayianni & Stefanidou 2003; Válek *et al*., 2019 and Ponce-Antón *et al*., 2021). However, this practice has not been widely implemented in either South Africa or the Southern African region. Even after conducting material characterisation, Abdel-Mooty *et al.* (2009) emphasised that establishing authenticity during historic restoration is a difficult process involving the integration of existing/old and new elements. Other authors, like Acun and Aroglu (2006), Young (2008), and Clemente (2018), highlighted that selecting appropriate materials is also vital to avoid mixing elements with various qualities, which could induce alterations in the original structural authenticity. According to Loke (2020), utilising unsuitable materials causes premature cracking, degradation of existing lime-based mortars, color differences, and detachment from the original surface. In some instances, it causes irreparable damage to the structure that the intention was to preserve (Loke *et al*., 2020).

In providing solutions to this global problem, Arizzi (2012), Papayianni *et al*. (2019), Pintea and Manea (2019) and Lima *et al.* (2020) have investigated the use of several materials for ancient building restoration in Spain, Greece, Romania, and Portugal, respectively. Lime, shells, natural polymers such as starch, cactus extract, olive oil, rice paste jelly, and clay additions were among the materials investigated. The challenge is that the construction industry is not using any of these materials in restoration work. Since some materials are no longer used or are scarce, replacement materials have become necessary. Even so, these replacement materials perform poorly on heritage masonries, especially when new materials possess physical, mineralogical, chemical, and mechanical properties different from the original ones they are being merged with (Van

Domelen, 2009). Marini *et al.* (2018) discouraged the use of replacement materials, indicating that most of them have been shown to perform unsatisfactorily and could damage the original fabric of the structure if incorrectly selected. Proper and well-informed selection is key for optimum performance of replacement materials.

If the application of original material is impossible due to unavailability, replacements may be tolerated, but with the greatest caution. The replacement materials can be acceptable if they provide comparable or better performance and durability at a reduced cost, or if the original materials are of poor quality or cause damage to neighboring materials. However, the replacement material must physically and visually match the old, producing an appearance that fits the old/original material, design, color, and texture (Weeks & Grimmer, 1995 and Van Domelen, 2009). Park (1988) emphasises the importance of understanding the qualities of both the original and replacement materials for long-term restoration. He goes on to say that substitute materials should meet three key criteria before they are approved:

- They must be aesthetically and physically compatible with the historic materials, including color (which is frequently difficult to match), surface texture, surface reflectivity, and finish of the original material.
- Their physical attributes must be similar to those of the original materials.
- They must be installed in a way that tolerates variances and must meet some basic performance criteria over time.

From the findings by Ghezal and Assaf (2014) and Arito (2018) relating to premature deterioration of repair on heritage structures, it is important to address some of the factors, starting with the suitable selection of restoration mortars that are durable, economical, and technically sound. Based on the original mortars' properties, this study intended to design and develop suitable restoration mortars for heritage structures that shall last for centuries to come.

1.2 Research problem

There have been efforts to produce restoration materials for heritage buildings, such as aerial and hydraulic lime containing small amounts of cement (Arizzi, 2012). However, cement mortars compared to standard lime mortars, they have better compressive and flexural strengths, lower capillary porosity and water absorption, and are more resistant to water vapour transport. This becomes a significant challenge when executing restoration work on historic structures. The problem is compounded by common practice, which involves the use of replacement materials that often fail to perform adequately due to the differences in properties (Arizzi, 2012).

The European Standards (EN) and the American Society for Testing and Materials (ASTM) mainly focus on the procedures for characterising modern materials and less on ancient (lime-based) materials. However, the RILEM recommendations have made great advancements in providing guidelines and procedures for historic mortars. On the other hand, the South African National Standards (SANS) have not diverted necessary attention to analysing heritage mortars or heritage materials at large. While it is clear that considerable scientific effort has been focused on original cultural material characterisation, heritage restoration, and conservation, the positive outcomes of these efforts are yet to be proven.

From the literature studied, Abdel-Mooty *et al*. (2009), Arizzi (2012), Hughes *et al*. (2012), Aggelakopoulou *et al*. (2019), Válek *et al*. (2019) and Lima *et al*. (2020) developed numerous repair mortars with varying mixes and proportions of various binder kinds and components. Regardless, the selection of such proposed mortars for heritage structures is unclear and not well documented. There is a lack of outlined step-by-step design and production guidelines for future restoration practitioners and industry manufacturers for such materials (Coletti *et al*., 2016). The lack of knowledge makes it difficult to design and select compatible and sustainable restoration mortars. It also justifies the lack of readily available restoration material mixes that meet the mechanical, time-dependent, durability, and aesthetic requirements.

One cannot be certain that the selected mortar shall perform adequately due to a lack of guiding data, and this study failed to find evidence regarding the proposed mortars' applicability and durability. The details are lacking in some of the studies that provided material behaviour feedback. It remains questionable whether such mortars perform as intended; thus, an investigation into their applicability and durability is necessary after the design of repair mortars. This helps to confirm whether such repair mortars will work as intended. As far as can be ascertained, no research has been conducted regarding design methods and durability performance for different categories of repair mortars.

1.3 Research questions

The literature reviewed shows a need to characterise historic mortars before carrying out restoration works. The question of which material is correct for the restoration or renewal of the original fragments of the historical monuments remains unanswered (Subbotin, 2018). For the long-term existence of heritage structures, it is necessary to integrate research and the construction material industry while designing and developing compatible restoration mortars. There is also a gap between industries and research regarding historic mortar production and the need to ensure the compatibility and durability of the designed mortars.

This research work intended to address the following questions:

- What are the preliminary characterisation procedures for compatible restoration of heritage mortars?

- What are the design and development considerations for compatible restoration of heritage mortars?
- What are the critical performance requirements and tests for heritage restoration mortars?
- What are the procedures for ensuring the heritage repair mortars perform for the extended lifespan?

1.4 The specific need

Heritage building restoration is a present concern for many heritage authorities abroad, including SAHRA. This requires a multidisciplinary approach that includes complementing analytical techniques from archaeology, history, chemistry, mineralogy, geology, physics, and engineering, among other disciplines. The current study addresses numerous questions regarding the design of restoration mortars for historic monuments. It is intended to benefit the heritage authorities within South Africa and abroad and includes industries in terms of providing a guide for developing these mortars. In addition, it provides steps to the reassurance of the designed mortars' applicability, compatibility, and durability. Hence, financial sustainability results from once-off projects. This study contributes to improving the South African National Standards as a guide for mix design procedures and design and development processes for compatible long-term restoration mortars of heritage masonries.

1.5 Aims and objectives

A wide range of literature exists on historic mortar restoration. However, the mix design procedure, durability and compatibility checks on repair mortars have received limited attention. The main objectives of this research are set into two phases. Phase 1 identified, in detail, the properties of original heritage mortars compared to modern materials. Phase 2 focused on producing compatible and durable restoration mortars with the design mixes to be specified. This was accomplished by an extensive and critical review of the literature and comprehensive laboratory experimental investigations with the following objectives:

- To characterise mortars' chemical, physical and mineralogical properties from three eras (1600, 1700 and 1800's).
- To develop compatible ready-to-use mortars (from mix designs) for heritage mortar restoration based on the investigations performed on ancient/original mortars.
- To investigate and verify the compatibility and durability of the designed mortars in terms of their resistance to freeze-thaw and salt crystallisation cycles.
- To compare the original and new heritage mortar properties for the extended lifespan of heritage masonries.

1.6 Research context and significance

The design of compatible repair mortars is a vital tool in heritage masonry restoration. The current research sought to utilise the comprehensive property parameters to design and develop compatible mortars for preserving the heritage structural integrity while at the same time maintaining the authentic aesthetics of these unique structures. It aimed to guide the design of suitable heritage restoration mortars in conjunction with long-term performance evaluations and the creation of restoration materials that do not damage the original surface. The new materials will help to increase the use of replacement materials in heritage structures where there are no alternative means of restoring the mortars. This Doctoral thesis falls within civil engineering: structural and construction materials science. It also covers the geological aspect, mineralogy, and texture of the mortars. The outcomes of this applied research will be used as a guiding tool for heritage authorities during heritage mortar restoration. Research in this area is of great significance.

1.7 Scope and delimitation

This research was not a general investigation of all listed replacement materials and their qualities, but concentrated entirely on the process and methods for evaluating and selecting appropriate mortars for the three eras (1600, 1700, and 1800) materials based on the properties of the original materials. None of the materials from the literature were developed and tested since such materials possess different properties from those of the selected structures in this study. The compatibility design parameters outlined in the literature apply to the design of restoration mortars from different eras in different countries. It should be noted that the design and production process focused on optimisation and not on the production of all the mortar samples equivalent to the evaluated original mortars. This is mainly due to the practicality of applying only one mortar type on a masonry section. The study further investigated a procedure for developing restoration materials using high-end tests. In this study, only a limited number of analytical techniques were conducted based on their merits in heritage material characterisation, cost, and accessibility.

The study acknowledges that other substrate heritage materials (i.e. bricks, stones, paint) need investigation, design, and development as a restoration prerequisite since their behaviour impacts the masonry. These were not addressed, as they extend beyond the scope based on time and cost-related obligations. There is also a need to investigate the causes of decay on heritage mortars, the influence of climate change on mortar lifespan, and factors that contribute to long-term heritage restoration such as specification, environmental influences, curing procedures, and restoration workmanship, but these are beyond the current study's scope due to time and resources. No new mortars were applied to the heritage buildings during this research work due to the magnitude of undertaking such an exercise. A separate study to investigate the application of the proposed mortars and evaluate different application methods and conditions could be a valuable contribution to heritage conservation.

1.8 Assumptions

The current research aimed to design and develop mortars for the structural masonries from three eras in South Africa's Western Cape Province. This study hypothesised that an informed mix design approach could lead to an extended service life of heritage buildings. Other assumptions were made regarding the material used on heritage structures and modern materials based on the data collected.

The crucial assumption, supported by literature, is that heritage materials differ significantly from modern materials in terms of properties and performance. This being the case, the assumption was that no modern material (Portland cement-based) is suitable, without modifications for heritage mortar restoration. Without a doubt, such material shall fail to perform to its full potential, or it will damage the original surface. It was assumed that the modern materials that would be identified at the end of the study would need alterations to match the original materials' properties, thus ensuring compatibility.

Material design and production is not a generic concept. It was apparent in a study by Loke (2020) that the materials from different eras have properties that differ. Hence, their performance would not be similar. It was expected that the repair mortars for the three eras would differ in terms of properties, composition, and performance based on their categories (plaster, render, pointing).

1.9 Methodology

Firstly, a historical and contextual literature review was conducted in order to provide justification and context for this research. Following that, more literature was reviewed to identify the methods and factors used by conservation practitioners and other authors to evaluate and select replacement materials for historic structures in the modern day. A detailed methodology to achieve the set objectives was based on the characterisation and in-depth analysis of representative mortar samples collected from different parts of the Castle of Good Hope and the two oldest buildings on Robben Island (Primary school and maximum-security prison building), in South Africa. The sample size criteria included a collection of representative samples from the three-era buildings focusing on the floor, rendering, plastering and bonding/jointing mortars for a clear indication of material properties and design procedures for different mortars, with different performance requirements.

The experimental work was structured into two major phases: **Phase 1**- Mortar characterisation (original), **Phase 2A** - Design and development of repair mortars, and **Phase 2B** - Mortar characterisation (new), compatibility and durability analysis. More in detail, Phase 1 entailed investigating the original properties of the mortars. Phase 2 was divided into two subphases. Phase 2A further investigated the mix designs to choose the most suitable among various designs. The production of repair mortars for specific sections forms part of this sub-phase. The produced mortars using modern materials were modified with relative admixtures and/or property-enhancing materials to obtain optimum performance in terms of cracks, shrinkage, water resistance, water permeability, penetration, and weather resistance. Finally, the new materials were subjected to similar tests as the original materials for comparison checks, durability and compatibility tests as part of Phase 2B.

1.10 Thesis outline

This research entails a full investigation of the properties of historic mortars and their design and development of repair replacements. Various experiments were conducted to shed light on the problem stated and provide solutions for future restoration activities.

This thesis consists of 6 chapters as follows (summarised is Figure 1.3):

Chapter 1 – Introduction. It provides context and rationale for conducting mortar analysis prior to undertaking historic masonry restoration. The repercussions of eliminating this crucial process are also provided in this chapter, emphasising the need for this study and its contribution to the body of knowledge and heritage authorities. The aims and scope are also highlighted herein.

Chapter 2 – Literature review. Phase 1 - Historic mortar characterisation: Articles, books and internet sources on the analysis of original historic mortars during restoration works, the challenges faced during the characterisation process, and the solutions are elaborated in this chapter. Further gaps existing between the industrial and the research in terms of material analysis are identified with recommended solutions. **Phase 2 - Design and development, compatibility, and durability assessment of repair mortars.** The literature related to the design and development process of repair mortars for historical monuments, as well as the challenges associated with these processes, are outlined in this section. Since one of the objectives of this study is to ensure the long-term existence of the repair works, this section provides literature on assessing the durability of repair mortars.

Chapter 3 – Research methodology. The first section of this chapter describes the organised experimental process and equipment used to characterise samples from the Castle of Good Hope and buildings on Robben Island in terms of physical, mineralogical, chemical, and mechanical properties (*Phase 1*) using the RILEM recommendations and EN Standards. The second part focuses mainly on methods for developing new repair mortars and evaluating and verifying their compatibility and durability (*Phase 2A and 2B*).

Chapter 4 – Results. It outlines the results from visual investigations and laboratory experiments on the properties of the original materials and their subsequent repair materials (subdivided into two parts). The effects of additives to modify the mortar properties are shown in this section.

Chapter 5 – Discussion. It investigates the interpretation of the physical, mineralogical, chemical, and mechanical characteristics reported in chapter 4 compared to literature around historic mortar characterisation. It also provides a breakdown of the design mix formulation procedure for a proposition to industries while developing historic repair mortars.

Chapter 6 – Conclusions and recommendations for future research. This chapter concludes with the aspects to consider when designing and developing historic repair mortars. It also gives the general conclusions derived from the results obtained and relates them to the reviewed literature. Recommendations and suggestions are provided herein for lacking aspects in the literature. The chapter further outlined the methodology proposed to be incorporated into the South African National Standards (SANS) for improved restoration works on historic structures in the future.

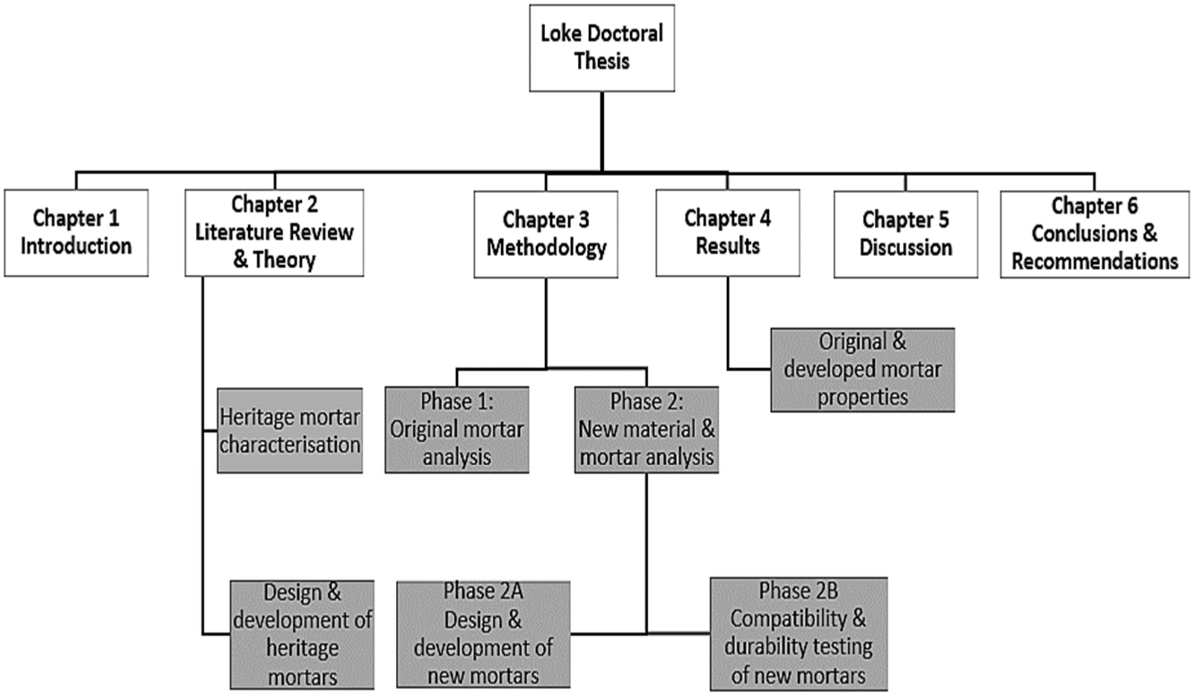


Figure 1.3 The layout of the thesis

Chapter 2 Literature review and theory: Heritage mortar characterisation

The use of proper mortars to reinstate historic masonry has a positive economic benefit, which is critical for restoration projects. This is accomplished because when a good mortar is used, no recurring repair is often required, therefore matching the existing surface qualities and delivering ideal performance. Matching existing properties is only attainable through mortar characterisation as a basis for the restoration process. This chapter discusses literature and theory on the benefits of characterising historic mortars, their characterisation procedure, challenges, and possible solutions.

2.1 Introduction

The analysis and characterisation of historic mortars have been carried out during the last decades by means of physical, mineralogical, chemical, and mechanical methods. Historic mortar characterisation is a well-studied topic that includes the assessment and investigation of hardened mortar qualities taken from existing historic building masonries and floors. Before conservation and restoration operations, it is critical to characterise historic and modern materials for research, documentation, or compatibility assessment (Hauková *et al.*, 2013). As Hauková et al. (2013) stated, knowing the properties of the original materials will allow one to forecast how the historic structure will react to the restoration materials. Original material characterisation is the only way to obtain such important information.

2.2 Lime mortars *versus* Portland cement mortars

Mortar is a type of artificial building material that consists of a binder and aggregates (mostly sand) mixed with water. Mortars are used in masonry to join stone ashlars and bricks as well as to protect both the outside (sacrificial renders) and interior (plaster) of masonry, including use for ornamentation. (Caroselli *et al.*, 2021 and Ergenç *et al.*, 2021). Hughes *et al*. (2012) indicate the functional categories of mortars to be as follows:

- Jointing mortars are used for setting units, adhesion, and bearing load. They have a structural function.
- Pointing mortar for water penetration protection and aesthetics.
- Exterior render (referred to as sacrificial and protective layers for substrate materials, i.e. bricks, stones) for water penetration protection and aesthetic and decorative covering. If the render is too porous, it allows the water to penetrate the wall and cause moth, and if it is too hard and impermeable, it prevents the wall from breathing.
- Interior plaster is used for aesthetic covering and as a substrate for decoration.
- Grout material is used for filling cavities in masonry to improve monolithic behaviour.

- Flooring mortar is applied as a supporting layer, levelling screed, and a substrate for tiles and mosaics.

According to Arizzi and Cultrone (2021), the major evolution of mortar components, production techniques, and application circumstances has occurred throughout time. The history of mortar usage in the construction industry dates back to Roman times, with lime-based mortars being used around the sixth millennium BC (Elsen, 2006). Although Portland cement-based mortar is the most prevalent building material in our cities today, its use on older structures is discouraged. This is owing to compatibility issues with its use on historic masonry. According to Arizzi (2012), lime is the sole substance capable of meeting the minimum historical mortar requirements for restorers, architects, and engineers. This is owing to variances in the properties and behaviour of the two binder types in terms of basic historical restoration requirements, as shown in Figure 2.1.

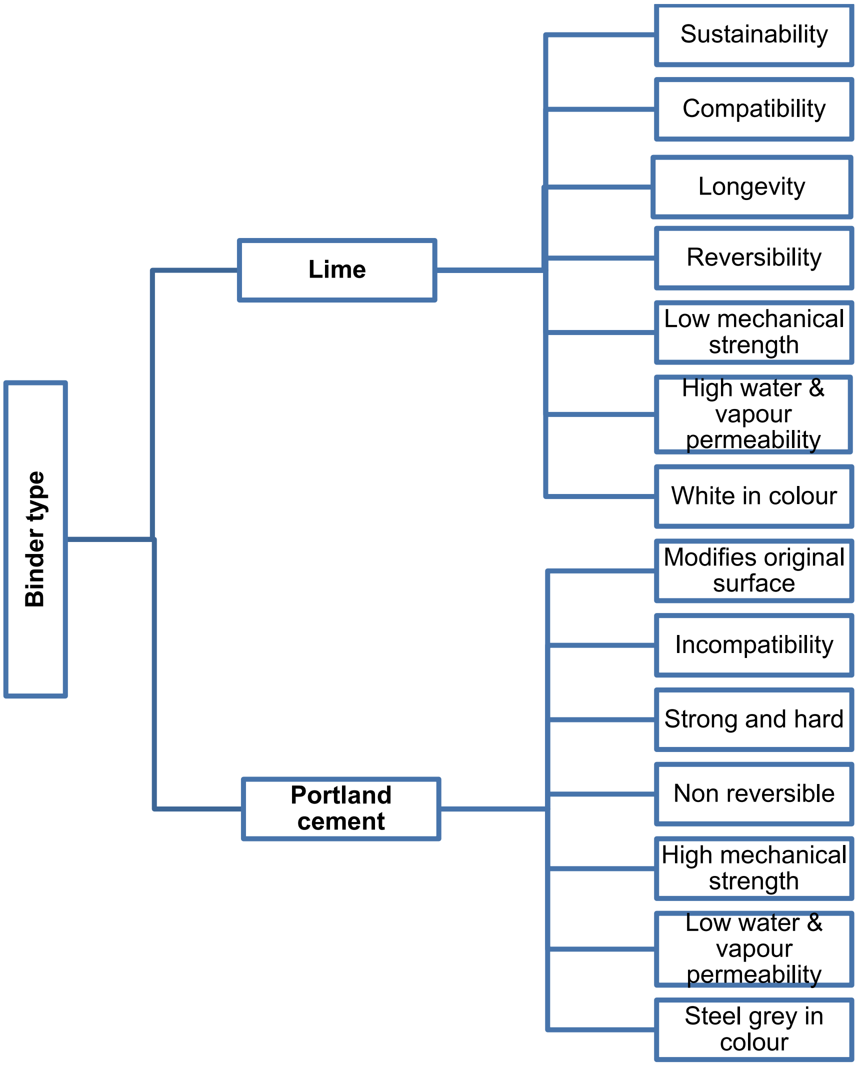


Figure 2.1 Lime-based mortar characteristics against Portland cement-based mortar characteristics (derived from Arizzi, 2012 and Palomo *et al.*, 2014)

A study by Elsen *et al.* (2010) provides a detailed summary of the evolution of binder materials over the years provided by Furlan and Bissegger (1975), as seen in Figure 2.2. According to Elsen *et al.* (2010), in 2010, natural hydraulic lime (NHL), a primary binder used for historic structures, was slowly facing extinction. These types of binder were only produced in limited Western Europe countries such as Belgium, Greece, Sweden, Switzerland, Germany, Portugal, United Kingdom, France, and Italy (Elsen *et al.*, 2010 and Elsen *et al.*, 2011).

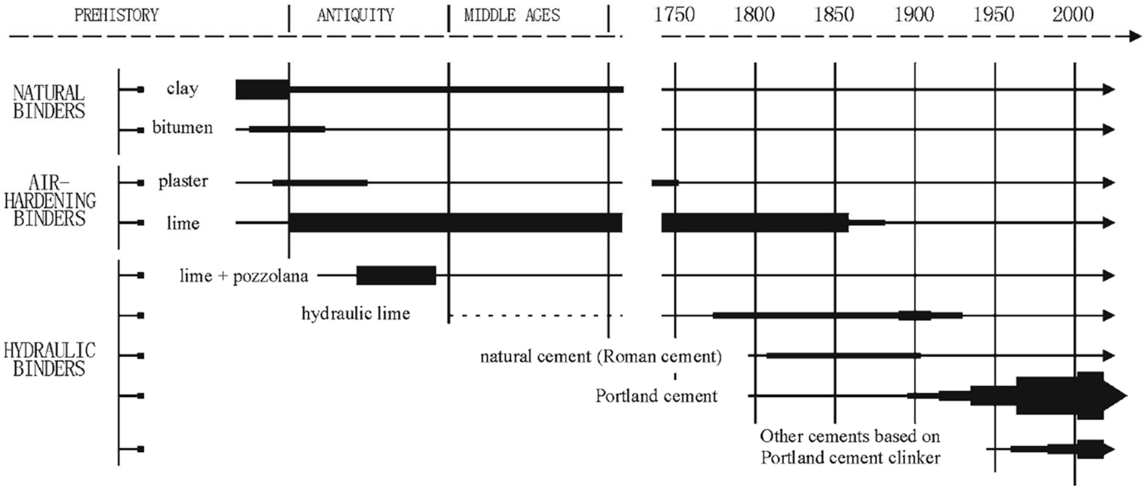


Figure 2.2 Introduction and use of different binder types from prehistory era to the 19th century. The arrows indicate the extent of binder use over a period (extracted from Elsen *et al.*, 2010)

Over time, the use of lime, as indicated by Elsen *et al.* (2010) decreased, leading to the use of Portland cement in the nineteenth century (Sanjurjo-Sánchez et al., 2010). Mortars used prior to the advent of Portland cement are hence referred to as 'historical' or 'ancient' mortars (Arandigoyen & Alvarez, 2007). The different types of binder and aggregates have an impact on mortar performance in masonry, any little changes to these components usually result in a significant difference in performance behavior. Hence, Portland cement was favoured over lime for its ability to attain various performance characteristics such as fast setting, higher strength, and hardness. Even though studies suggest that Portland cement is not a desirable option for historic buildings, it is suitable for most modern structures for which lime mortars are unsuitable. This complicates the study of historic mortars in the modern period, with calls for a multidisciplinary approach supported by complementing analytical and theoretical techniques from the domains of chemistry, mineralogy, physics, engineering, archaeology, architecture, historians, and historical practitioners (Feilden, 2003).

2.3 Mortar characterisation methods

When designing heritage mortars, it is necessary to understand which properties of existing materials must be evaluated. Figure 2.3 depicts the general approach, which involves numerous analytical techniques for determining various mortar properties. The physical, mechanical, mineralogical, and chemical properties of replacement materials have a considerable impact on their compatibility, functionality, and durability. Again, these qualities for the replacement and the original material should be known. In addition to studying the properties of the original and suggested replacement materials, historic restoration practitioners should examine the surrounding heritage structure's climatic conditions (dry or humid), as these will affect the performance of the applied mortar. This also limits the use of any one type of mortar anywhere in the world, as different historic structures require different types of mortar due to diverse climatic conditions. Assessing the climatic conditions of nearby heritage buildings is crucial to the design of durable and optimally performing mortars. (Van Domelen, 2009 and Hughes *et al*., 2012).

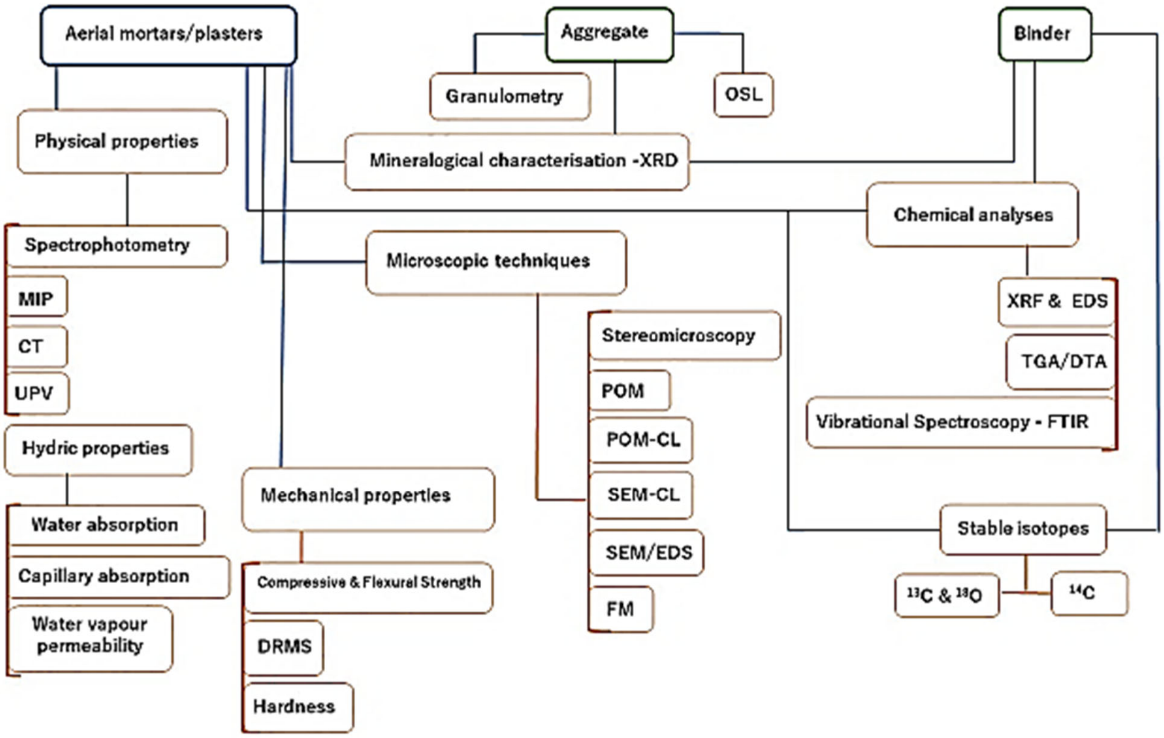


Figure 2.3 Sequence of tests to establish the composition of ancient mortar components and properties (extracted from Ergenç *et al*., 2021)

2.3.1 Aesthetic and physical properties

The appearance and texture of the original mortars are analysed using different methods to ascertain that the repair mortars match the original. This is the first factor to consider because authentic aesthetics are important in historic constructions (Schueremans *et al*., 2011). These features include deterioration processes and pore size distribution, which help determine the porosity, workability, lightness, and chromatism of mortars, as well as the hygric properties, which include water absorption, drying, capillary absorption, and water vapour permeability. It entails the use of environmental scanning electron microscopy (ESEM) coupled with energy dispersive spectroscopy (EDS) analysis. This technique helps in the differentiation of binder stages based on picture contrast. The analysis of physical properties can also identify important characteristics such as weathering compounds produced by decay processes.

2.3.2 Chemical properties

These include the identification of the type of binder, its quantity, the binder-to-aggregate ratio, and the materials' hydraulicity. The commonly used analytical technique for determining the chemical properties is X-ray fluorescence (XRF).

2.3.3 Mineralogical and petrological properties

These include the analysis of binder and aggregate mineralogy using polarised optical microscopy (POM) and powder X-ray diffraction (PXRD). The results of these analyses define the texture and the mineralogy of materials (Ngoma, 2009). Furthermore, ion chromatography (IC) detects and measures the soluble anions and cations in raw anhydrous mortars, such as chlorides (Cl-) and sodium (Na+). Other tests, such as thermogravimetry analysis (TGA) and Fourier transform infrared spectroscopy (FTIR), are used to analyse hydration levels and quantify portlandite/slaked lime ($Ca(OH)_2$) and calcite/limestone ($CaCO_3$). It is also recommended that a petrographic study of the mortar be performed in order to understand how it was prepared and detect potential production faults, such as the presence of lime lumps (Arizzi, 2012). The mineralogical study is key in determining the type of restoration mortar to use.

Ideally, the selected restoration mortar should have a similar mineralogical composition as the original mortars. It is worth noting that the presence of certain compounds during the mineralogical analysis may induce certain demands for restoration mortars for such mortars to meet the chemical compatibility requirements. For example, the addition of gypsum in small quantities, denoting its use as an additive to speed up the setting process, not acting as a binder, early carbonation of the restoration mortar could be necessary. This is required to prevent the attack of free lime by SO^- ions, towards the formation of gypsum. Furthermore, suppose original mortars demonstrate traces of calcium hydroxide during analysis. Similarly, rapid consumption of calcium hydroxide is required, necessitating the use of hydraulic lime or lime-pozzolan mortar. (Apostolopoulou *et al*., 2018).

2.3.4 Mechanical properties

This refers to the strength and stiffness properties of mortar, which are usually obtained through non-destructive testing (NDT) methods since the destructive methods require the use of large quantities of mortar from heritage buildings, which are not available. The commonly used techniques include the non-destructive ultrasound pulse velocity (UPV) test, which defines the mortar's compactness and a destructive uniaxial compressive strength (UCS) test, which determines the mechanical properties of historic mortars.

2.4 Characterisation challenges

While much work has been done to characterise historic mortars, some critical aspects have gotten insufficient attention (see Figure 2.4). The vast majority of academics have discussed the procerdure of analysing historical mortars, but none have established a systematic protocol for doing so. A review of the literature revealed no studies that provided instructions for a systematic process for characterising historic materials. In a few situations, researchers sought to develop a systematic approach for analysing heritage mortars. Nonetheless, recommendations for characterising historic cementing materials have received little attention thus far. This section discusses some of the challenges associated with characterising historic mortars, as well as potential solutions.

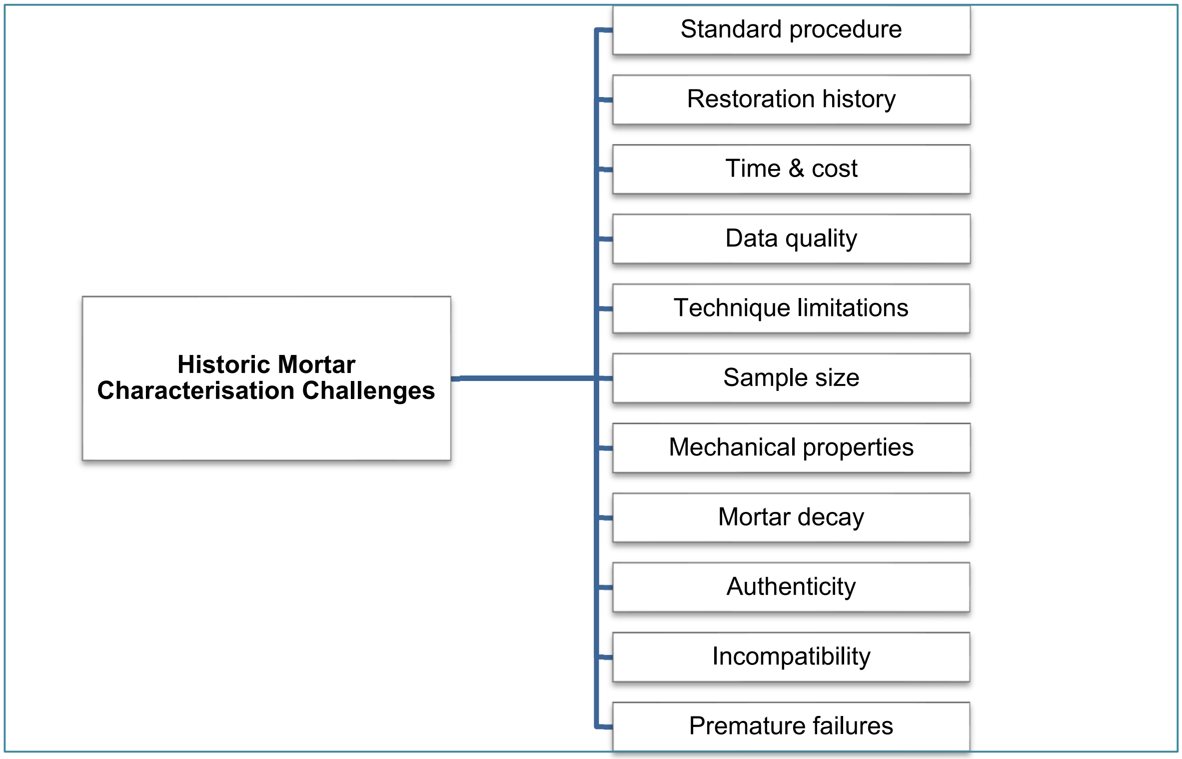


Figure 2.4 The challenges facing characterisation of heritage mortars

2.4.1 Standard protocol

Arizzi (2012) emphasises the importance of adhering to standards and using specific tests to investigate the components of the original mortar in order to ensure compatibility with repair mortars and achieve some degree of universality when characterising these commonly used construction materials (Monaco *et al.,* 2021). She noted that the lack of standards has led to the use of a wide range of procedures in different countries, resulting in variances in outcomes. Furthermore, Ngoma (2009); Loke *et al*. (2021), and Kumar and Kumar (2022) revealed a gap in the literature relating to the standard procedure for the analysis of historic mortars. They further suggest that a careful selection of the right methodology and implementing a standard procedure based on existing material analysis techniques will address the majority of restoration problems facing the construction industry in terms of appropriate repair works.

Arizzi (2012) pointed out the reference to the European (EN) and American Society for Testing and Materials (ASTM) standards for test methods, manufacture, and quality control of mortars. Previously, EN and ASTM standards were used to classify mortar based on its state: fresh or hardened, but not on its final function (structural, grouting, pointing, flooring, rendering, plastering, etc.), while also focusing primarily on industrial mortars as hydraulic binders (cement) rather than historic mortars (lime-based). The sole publications focusing on historic mortars were the RILEM (International Union of Laboratories and Experts in Construction Materials, Systems, and Structures) recommendations (Arizzi, 2012). Fortunately, the RILEM recommendations have made significant progress in recent years in creating systematic procedures for analysing and characterising historic mortars. The RILEM provides crucial technical committees such as Technical Committee 167-COM: Characterisation of historic mortars in relation to their repair, as well as hosting congresses (e.g., Historic Mortar Conference) and issuing recommendations. reports, and publications on historic mortars (Válek *et al*., 2019).

Furthermore, the European Committee for Standardisation has provided guidance in the field of cultural heritage conservation by publishing standards on terminology (EN 16572, 2015), sampling methodology (EN 16085, 2012), and various test methods (EN 15801, 2009; EN 15803, 2009; EN 16302, 2013 and EN 16322, 2013). Among these practical standards, EN 17187 (2020) elaborates on the methods to be used to characterize the mortars used in cultural heritage to specify their petrographic, mineralogical, chemical, physical, and mechanical qualities (Ergenç *et al.,* 2021). This is seen as a significant development in the description of historic mortar. Despite the fact that the EN and RILEM recommendations guide restorers, architects, and engineers on historic material characterization, Feizolahbeigi (2021) highlights the reliance of the scientific community (industries involved) on the ASTM standards for assessments of repair mortars. A gap still exists between the industries and knowledge in historic material analysis.

2.4.2 History of heritage conservation and restoration

Wallace (1865) undertook the first research of historic mortars in the nineteenth century (Ergenç *et al*., 2021).The study looked into the properties of historic mortars from 1500 to 3000 year old buildings in Egypt, Greece, Italy, and Cyprus. Jedrzejewska (1960) found inadequacies and a lack of documentation of original material information, such as the year of construction, the history of material restoration over time, and the restoration procedures used. The study identified difficulty in distinguishing between original and repair materials, especially when there is no physical difference between the new and old materials. Ergenç *et al*. (2021) reports on mortar dating techniques such as isotopic radiocarbon (^{14}C) and optically stimulated luminescence (OSL), which focus on analysing the content of the ^{14}C isotope in the binder matrix and the aggregates respectively.

2.4.3 Resources: cost and time

Because the primary purpose of historical mortar analysis is to ensure long-term compatibility, it is necessary to assess cost-effectiveness before moving on with scientific characterisation. According to Hauková *et al*. (2013), many of the analytical procedures reported by many researchers are too expensive (laboratory equipment and knowledge) to be used on a regular basis, particularly for conserving and restoring ancient buildings. It is recommended that existing analytical methods be standardised to produce cost-effective and time-effective characterisation methodologies and procedures. This will not only serve as a reputable source for restorers striving to repair heritage structures, but it will also play an important role in economic sustainability, particularly for developing countries.

2.4.4 Technical data accuracy

According to Hauková *et al.* (2013), some of the methodologies outlined and employed by numerous researchers rarely give useful information. Thus, one must evaluate the results/information obtained from a comprehensive variety of methodologies, as well as their significance or use in creating suitable replica mortars (Hughes *et al*., 2012). Some procedures give difficult-to-interpret data that may confuse the restoration process (Loke, 2020). For example, color analysis by the naked eye may differ depending on the researcher's viewpoint. Instead of the naked eye, accurate techniques such as spectrophotometry, colorimetry, or Munsell Soil Color Charts could be used to overcome this.

2.4.5 Usability

The primary goal of material characterisation is to obtain the necessary information for restoration using a small number and quantity of samples and as little time and money as possible (Schueremans *et al*., 2011). Architectural heritage research is generally multidisciplinary, with architects, engineers, geologists, chemists, and others providing optimal options for restoration interventions. For successful restoration operations, all essential specialist inputs must be included, providing useful inputs to the bigger scheme of restoration works.

2.4.6 Sample size constraints

Several authors studying ancient mortar properties stress the importance of proper sampling in accordance with the EN 16085 (2012) standard, as this phase will influence the results obtained (Loke *et al.,* 2023). The samples must meet the standards in terms of quantity, size, shape, and placement (Arizzi & Cultrone, 2021). Historic structures are protected by legislation due to their cultural significance, which means that, alterations are continually reviewed, with policies suggesting restrictions on proposed alterations. The goal is to do the least amount of harm feasible by collecting as few samples as possible to conduct the analysis (Chiari *et al*., 1996). Providing a concise approach (that takes into account the destruction constraints on historic mortar sample) as well as the study's aims before collecting the samples would also help alleviate unfounded disturbance on historical structures (Arizzi & Cultrone, 2021).

Heritage authorities only allow extraction of a limited amount of samples from historic structures, leading to non-standard sample sizes. This leads to high probability of errors in the obtained results (Monaco *et al*., 2021). It becomes a significant issue when doing a test with non-standard dimensions, which could have a detrimental impact on the analysis. When taking samples from the monument, non-destructive techniques such as portable XRF, ultrasonography, and colorimetry should be considered in order to preserve the structure's integrity. However, these techniques cannot supply some basic information, such as mineralogy or the texture of the mortars.

2.4.7 Mechanical properties evaluation

Inadequate test samples make it difficult to characterise the mechanical properties of mortars in existing masonry structures. This is mostly due to heritage authorities' reluctance to allow significant volumes of mortar to be taken from existing brickwork joints without endangering the structures (Benedetti & Pelà, 2012). Non-destructive testing procedures like as ultrasound pulse velocity (UPV) and rebound hardness tests, on the other hand, are frequently used to assess physical parameters that approximate mechanical properties without the requirement to sample the materials. In this respect, UPV measures the mortar compactness in accordance with the ASTM C597-16 (2016) standard and helps determine the Poisson’s ratio, Young, compressive and shear moduli by analysing longitudinal or P waves (V_p) and shear or S waves (V_s). The following expressions for the dynamic modulus of elasticity were used by Maras (2021) to evaluate both waves.

$$V_p = \sqrt{\frac{E_{dyn}(1-\nu)}{\rho(1+\nu)(1-2\nu)}} \tag{2.1}$$

$$V_s = \sqrt{\frac{E_{dyn}}{2\rho a(1+\nu)}} \tag{2.2}$$

Where:

V_p - longitudinal wave velocity

V_s - shear wave velocity

ρ_a – apparent density

E_{dyn} - dynamic modulus of elasticity

ν- Poisson's coefficient

2.4.8 Mortar decay

Mortars, the protective or sacrificial elements of masonry structures, are believed to be more susceptible to deterioration due to decay factors than bricks and stones in a building (Apostolopoulou *et al*., 2017). Sulphur oxides, for example, dissolve in water and produce acidic solutions. These chemical processes occur naturally; nevertheless, urbanisation has resulted in an alarming increase in their discharge into the atmosphere. Human activities such as the combustion of fossil fuels, such as petroleum, crude oil, and coal, contribute to the vast generation of acidic solutions that destroy mortars (Ngoma, 2009). According to Amoroso and Fassina (1983), European historic buildings are vulnerable to air pollutants such carbon dioxide, nitrogen oxides, ozone, sulfur oxides, and particle matter. These pollutants influence mortar composition and may result in inaccurate results. Because of these factors, conserving monuments has become a serious challenge because the monuments' durability is constantly jeopardised. External factors should also be considered by a restoration team. The literature proposes analysing the unexposed samples to overcome possible errors (Papayianni *et al.,* 2013).

2.4.9 Authenticity loss

When it comes to ancient mortar repair, material characterisation and authenticity are inextricably related. It should be highlighted that improper techniques, results, and interpretations in analysing original material will result in a misleading choice of new materials, resulting in premature failures in restoration operations. According to Abdel-Mooty *et al*. (2009), achieving authenticity during heritage restoration is a difficult exercise as it involves two materials of varying ages. Other authors, like Acun and Aroglu (2006), Young (2008), and Clemente (2018), highlighted that selecting appropriate materials is also critical to avoid the union of elements with various qualities, which could result in alterations to the original structural concept.

2.4.10 Using incompatible materials

Incompatible materials may be used on restoration projects if proper material characterisation was not performed or was completely neglected. Many restoration solutions use Ordinary

Portland Cement (OPC), according to research by Lanas and Alvarez (2003), Mitchell (2007), Martnez *et al.* (2013) and Marini *et al.* (2018). Although OPC mortars are thought to provide a rapid solution, they are said to cause long-term damage to the existing masonry fabric (Monaco *et al.*, 2021). Loke (2020) demonstrated that OPC-based plastic restorations are incompatible with masonry substrate materials because they entrap moisture and are relatively rigid. OPC repair materials are associated with low longevity, in addition to inflicting irreparable damage to the historical artifact intended to be conserved (Foster, 2010). The negative effect of incompatible OPC mortars is attributed to their physicochemical properties, which differ from the original building materials (Klimek & Grzegorczyk-Frańczak, 2021). Even after material characterisation, Abdel-Mooty *et al.* (2009) emphasised that achieving authenticity during heritage restoration is a complicated activity involving the union of existing/old and new elements.

2.4.11 Premature repair failures

Incorrect restoration work on heritage structures jeopardises their safety since premature deterioration might develop (Lukovic, 2016). This could be due to a poor choice of alternative binder or aggregates. For example, aggregates containing a high concentration of salts (mostly chlorides and sulfates) might cause salt crystallisation inside the mortars, causing premature deterioration (Zinn, 2005). The same is true for selecting the incorrect binder. The impermeable binder material creates a non-porous mortar that prevents moisture movement, which suffocates the building masonry and causes early repair problems. Frequently, the damage not only necessitates recurrent repairs but also raises the cost of future maintenance (Loke *et al.,* 2023).

2.5 Section summary

Protecting and properly repairing historical structures is an important exercise requiring caution. This is not only because of the rich history they represent but also because of their economic importance worldwide. Based on studies explored in this research, reverse engineering, which considers the original mortar characterisation prior to the restoration of heritage structures, is a key component in the design and development of historic repair mortars. However, this approach has not yet been implemented satisfactorily in Africa. It was observed that even though the literature strongly recommends this practice, it has not spread to the African continent, including South Africa. Some monuments' visible restoration mistakes can prove this. This is mainly due to a lack of documentation of the guiding literature associated with original heritage mortar characterisation and a better understanding of this activity's importance.

Chapter 2 Literature review and theory: Design and development of heritage mortars

Mortars contribute significantly to masonry protection and sustainability. However, they are complex, having different properties based on the raw materials utilised and several design parameters. Studies show that sourcing the correct replacements during restoration is challenging (Van Domelen, 2009). Hence, the development and restoration of mortars are considered areas of concern in restoring historic masonry. The current study addresses the prevailing complications facing the design procedure and the production of restoration mortars for historic structures.

2.6 Introduction

The design of mortars for restoration work is an essential stage in any conservation project relating to historic or modern structures. Several restoration projects of heritage buildings have shown that choosing restoration mortars for pre-existing masonry is challenging (Ngoma, 2009). It requires characteristics of the present materials and an in-depth analysis of the replacement ones for compatibility reasons. It is vital that the recommended materials, based on performance parameters and studied properties, are well understood. This will assist in achieving the compatibility of restoration mortars to produce long-term quality and durability of historic masonry.

2.7 Design requirements

The first stage in the design of any repair mortar is to define the basic specifications that the material must meet (Arizzi, 2012). Schueremans *et al.* (2011), basing their research on the Framework - RILEM TC-COM 167 (2003), outlined the mortar design requirements depicted in Figure 2.5 as the major factors to consider when deciding on repair mortars. This comprehensive procedure assures that the mortars meet all intended functions on a building masonry.

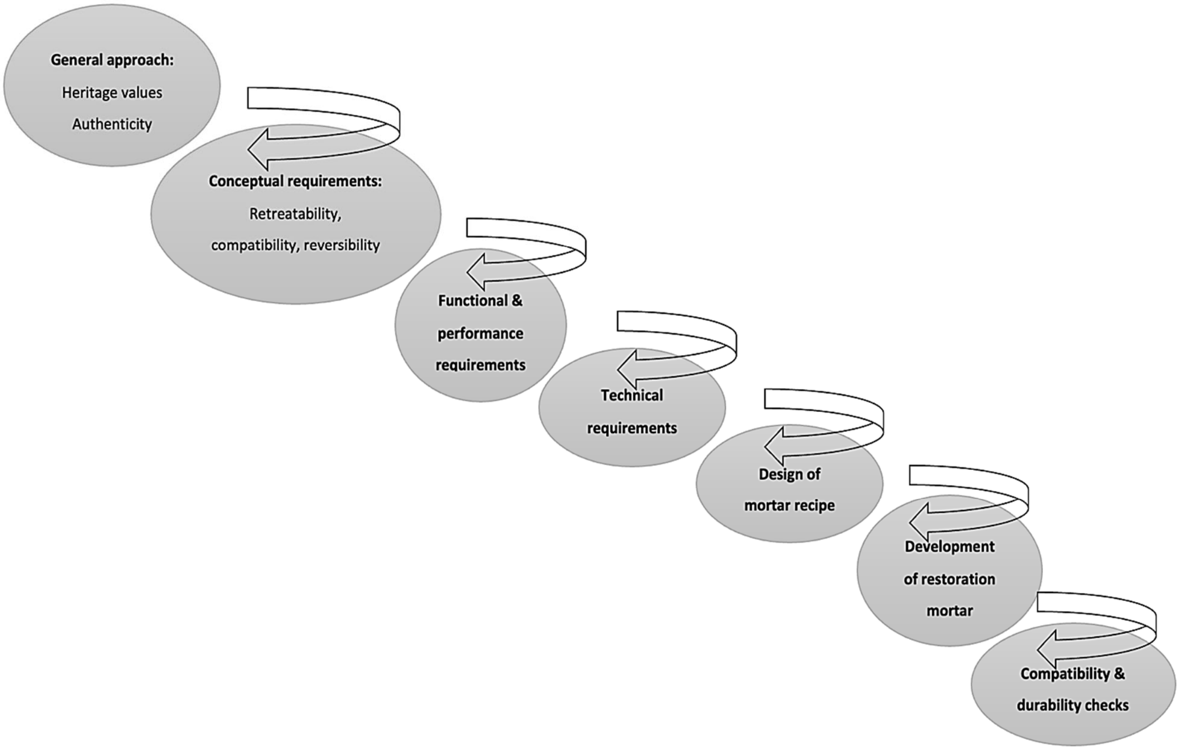


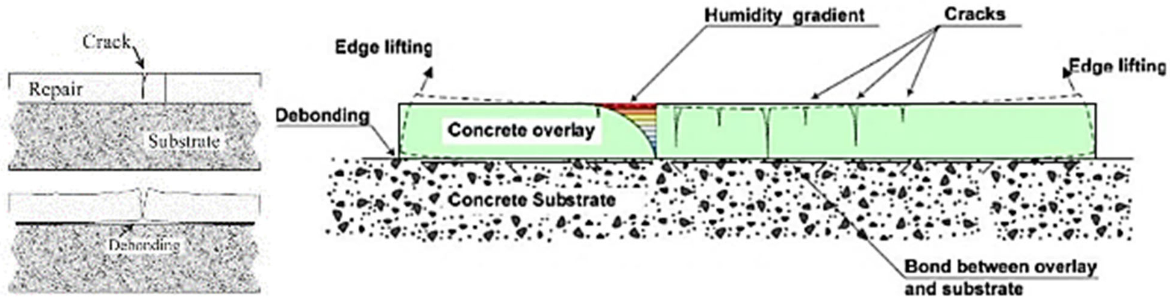
Figure 2.5 Design methodology for historical restoration mortars (extracted from Schueremans *et al.*, 2011 and RILEM TC-COM 167, 2003)

2.7.1 Integration with substrate materials

It is advisable to consider the properties of the substrate materials, such as stone, brick, concrete, etc., during the design of heritage restoration mortars. This is mainly because the behaviour of the mortar influences the behaviour of surrounding materials and, subsequently, the masonry. For example, one type of mortar may shrink by different degrees when combined with substrates with varying suction characteristics, necessitating the use of a mortar with high water retention capacity (Arizzi, 2012).

Cracking and shrinkage

It is believed that certain properties like shrinkage are very important factors to investigate during the design of heritage mortars. Studies indicate that shrinkage should be reduced to avoid cracking, delamination and corrosion of the reinforcement caused by aggressive products through chemical reactions (Carlswärd, 2006). In addition to shrinkage, the debonding of restoration materials from the original materials is another factor not to be overlooked during the design and development of heritage mortars. Debonding means "*the separation of the overlay from its substrate*" (Amba *et al.*, 2010 and Beushausen & Bester, 2016). This happens in most restoration projects, especially heritage structures where material compatibility was not initially assessed. According to Granju (2001), debonding is related to cracking as it usually occurs on free edges, joints, and fissures. It is caused by shear and tensile stresses induced by differential deformation of the overlay and the substrate. Most of the time, it is caused by various length variations in the overlay and substrate, as shown in Figure 2.6.



(a) (b)

Figure 2.6 (a) Cracking that leads to debonding from substrate material and (b) cracking and edge lifting of a bonded overlay exposed to shrinkage (extracted from Turatsinze *et al*., 2005 and Carlswärd, 2006)

2.7.2 Authenticity

Restoration of heritage buildings comes with high expectations of authenticity because these structures symbolise the country's past; thus, only minor changes are accepted (Subbotin, 2018). This is thought to be one of the most difficult features to execute because some of the original materials utilised no longer exist. The authenticity of masonry surface finishes is primarily dependent on the mortar, hence, it is critical to carefully study and develop restoration mortars that provide a finish and performance that do not compromise the heritage structure's originality.

2.7.3 Compatibility

Compatibility is the use of materials that do not negatively affect the properties of the original ones (Isebaert *et al*., 2014). Arizzi described compatibility as “*not causing any damage (in the broad sense, ranging from technical to aesthetical and historical) to the existing fabric and being as durable as possible under that condition.”* Teutonico *et al*. (1998) explain compatibility as "*the presented treatments of materials that will not have negative results when used together with the old*." Vukindu (2021) defines it as “*the balance of physical, chemical, and electrochemical properties and dimensions between the repair phase and the existing substrate phase of a repair system*.” These materials must have the same visual, physical, mechanical, and chemical qualities as the original (Schueremans *et al*., 2011). Compatibility is one of heritage restoration's most frequently used words (Rodrigues & Grossi, 2007).

According to Singh *et al*. (2014), the original material properties provide information on the binder used in mortar production as well as information on the aggregates. Compatibility is related to the use of appropriate materials and entails harmonisation by several parties involved in heritage conservation. This idea refers to aesthetic compatibility when materials, techniques, craftsmanship, and historical context are taken into account (Van Balen *et al*., 2005). Additionally, Válek *et al*. (2019) interpret the compatibility of historic materials as the ability of the new material (mostly the binder) to merge with the substrate. The authors do not necessarily refer to cement as an incompatible binder but rather indicate that any choice of binder should consider the compatibility of that binder with the substrate materials in general terms. The following are some of the compatibility requirements outlined by Válek *et al.* (2019).

Similar or higher drying rate to the substrate

Selecting the repair mortar that will allow adequate moisture evaporation is important. The selected mortar should have a similar or higher moisture evaporation rate than the substrate. However, careful consideration should be taken since fast evaporation in the presence of soluble salts might lead to salt crystallisation and damage the substrate. On the other hand, slow evaporation could result in frost action-related problems. The common issues related to drying rates that are too low or too high include delamination, disruption, and detachment of the repair and/or the original materials. The solution to this problem lies in the mix design (for example, the pore size distribution and the capillarity coefficient), which will affect the major properties related to mortar drying behaviour as follows: free, forced and capillary absorption, open porosity, drying rate and water vapour permeability.

Moisture ingress resistance - Reduced crack development and shrinkage chances

It is essential to prevent or at least reduce the shrinkage of the repair mortar. This depends mainly on the sand gradation properties as well as the binder-to-aggregate ratio that would provide good workability with no excessive water added for easier mixing. The selected sand should have a continuously graded particle size distribution to allow adequate water absorption. Additionally, the correct application and curing (to avoid rapid drying) procedures play a key role in how such mortar would behave; therefore, such factors should be considered as well.

Similar thermal and moisture expansion properties to the substrate

The thermal expansion coefficient of historic materials usually ranges between $3 – 10 \times 10^{-6}$ C^{-1}. A thermal coefficient of $\pm 2 \times 10^{-6}$ K^{-1} is recommended for repair mortars and substrate materials to prevent materials from exceeding their load-bearing capacity (Romero-Noguera *et al*., 2018). It is important to note that the stress induced by differential thermal expansion is a function of the E- modulus, the temperature gradient (e.g. sun-heated surface), the geometry of the repair, and the depth at which the repair mortar interfaces with the substrate. It is recommended to apply repair mortars with a lower E modulus than the substrate to protect the substrate.

2.7.4 Technical specifications

To ensure that the repair materials are functional, the selection of mortar components is based on both aesthetic criteria (type, color, and texture of the material to be reinstated) and mechanical

and physical properties (strength, elasticity, porosity, and coefficient of thermal dilatation). Arizzi (2012) outlined a wide range of technical requirements based on the functionality of the mortar. Renders, she claims, have distinct technical requirements than plasters and bonding mortars. Renders must meet the following technical requirements: moderate capillary water absorption, high water vapour transmission, surface hardness, low levels of released salts, resistance to soluble salts, and freeze-thaw cycles. Bonding mortars, on the other hand, require a high compressive strength as well as elasticity. Hughes *et al*. (2012) emphasised that these qualities might be modified by adjusting components and quantities to help meet the technical criteria.
Van Balen *et al*. (2005), Groot *et al.* (2007) and Romero-Noguera *et al*. (2018) outlined the following decisive technical properties for compatibility between new and old mortars:

- Compatible aesthetic features (colour, texture, flatness, brightness, surface finish).
- Composition similar to the original mortar (type of binder, type of aggregates, grain size distribution) to avoid significant differences in material behaviour that has adverse effects on repeatability.
- Proper pore size distribution.
- Lower strength (compressive, tensile and bond) than existing mortars and substrates.
- Low modulus of elasticity to that of masonry to allow deformability.
- Proper water movement properties (porosity, specific gravity, pore size distribution, water absorption by capillarity and vapour transport) – high permeability but not too high to avoid sub-florescence.
- Thermal dilation coefficient.
- Soluble salts or impurities (low to avoid efflorescence and crystallisation damage).
- Resistance to freeze-thaw cycles and other adverse environmental conditions.
- Workability (flexibility) that matches the application of the repair mortar.
- Curing conditions influence longevity, shrinkage and long-term deformation.

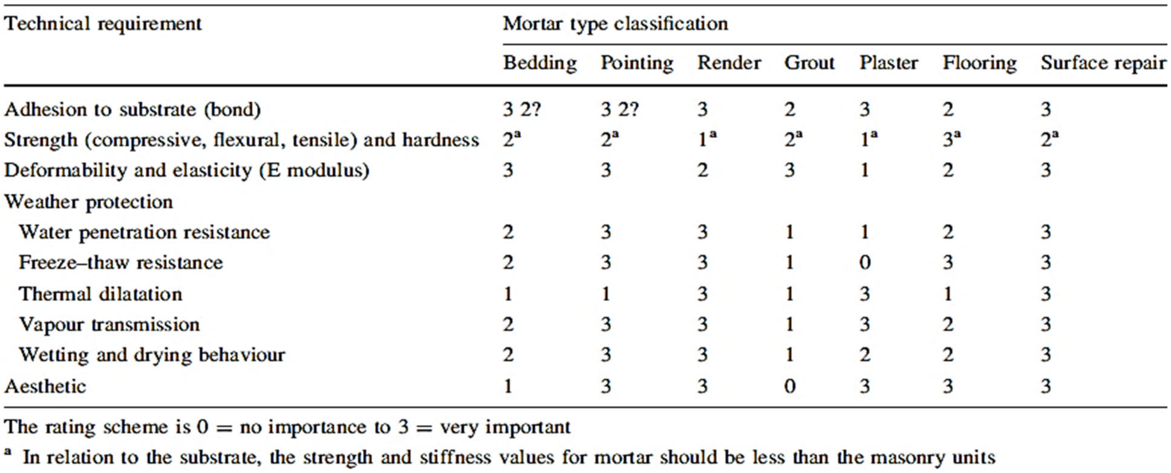
Aesthetics – Appearance and Colour

Replicating the original element's visual appearance is often considered the most important consideration for adopting replacement materials. Many other guidance publications and the preservation practitioners underline the importance of compatible aesthetics, with "matching appearance" receiving the highest overall importance score out of the criteria given (Weeks & Grimmer, 1995).

The repointing mortar's colour and texture (finish) should be matched to a reference mortar as required for historical authenticity and aesthetics. Maurenbrecher *et al*. (2008) and Ergenç *et al*. (2021) noted that the mortar components, mostly sand, contribute to the mortar's colour and texture. Sometimes modifications are added with the use of pigments. Such pigments should be inorganic oxides, and the amount added should not significantly alter the characteristics of the mortar (no more than 10% by weight of the dry binder). When selecting heritage restoration mortars, the color of the material must be taken into account. The colour change of the selected building material is often related to the spectral composition of the light source (Subbotin, 2019). Attempting to match the color of the replacement material to an original, on the other hand, is seen as a huge challenge, despite the fact that it is possible by retaining the chromaticity and adjusting the lightness to differentiate the original material from the new one. Theologitis *et al.* (2021) described the use of ochre and/or ceramic powder as additions to produce the desired coloration of plaster's physical qualities – porosity and permeability.

The physical properties are critical to substitute materials' compatibility and durability, as Hughes *et al*. (2012) ranked below in Table 2.1. The physical properties include aspects such as water flow through a material, the aptitude of the substitute material to trap moisture within the pore network and cause the decay of other materials, hygroscopic expansion, material expansion due to wetness and its degree, vapour permeability, thermal expansion and erosion resistance (Van Domelen, 2009). When developing restoration mortars, new mortars must be considered to eliminate differences in permeability and water transport qualities compared to the substrate materials (Válek *et al*., 2019).

Table 2.1 The key technical requirements rating versus mortar type classification (extracted from Hughes *et al*., 2012).



Technical requirement	Mortar type classification						
	Bedding	Pointing	Render	Grout	Plaster	Flooring	Surface repair
Adhesion to substrate (bond)	3 2?	3 2?	3	2	3	2	3
Strength (compressive, flexural, tensile) and hardness	2[a]	2[a]	1[a]	2[a]	1[a]	3[a]	2[a]
Deformability and elasticity (E modulus)	3	3	2	3	1	2	3
Weather protection							
Water penetration resistance	2	3	3	1	1	2	3
Freeze–thaw resistance	2	3	3	1	0	3	3
Thermal dilatation	1	1	3	1	3	1	3
Vapour transmission	2	3	3	1	3	2	3
Wetting and drying behaviour	2	3	3	1	2	2	3
Aesthetic	1	3	3	0	3	3	3

The rating scheme is 0 = no importance to 3 = very important

[a] In relation to the substrate, the strength and stiffness values for mortar should be less than the masonry units

Mechanical characteristics

The mechanical properties of a mortar (mortar durability, drying shrinkage, Young's modulus, and Poisson's ratio) are affected by the type of binding material, its amount, chemical composition, aggregate particle size distribution, particle surface, and the binding material-aggregateadmixture ratio (Jonaitis *et al*., 2019). The response of a material to tensile, compressive, and shear stresses are some of the most important mechanical parameters to consider during the design of restoration mortars. According to Young (2008), Van Domelen (2009), and Hughes *et al.* (2012), alternative materials should have equivalent or lower strength than the original material in most circumstances. Furthermore, mechanical properties such as tensile, compressive, and shear forces, flexibility (brittle or flexible), reactivity to potential impact, hardness, and creep are crucial when selecting restoration mortars (Van Domelen, 2009). The elastic modulus of mortar helps determine that of the masonry on which it is applied. However, the modulus of mortar layers is impossible to obtain thus a strength ratio is used to estimate the elastic modulus of masonry (Benedetti & Tarozzi, 2020).

2.7.5 Functional requirements

The design of a compatible repair mortar depends on the functional role the mortar performs within the structure, which should be considered during the design process, as summarised in Table 2.2 (Romero-Noguera *et al.,* 2018).

Table 2.2 Technical and functional requirements for the replacement rendering mortars (extracted from Romero-Noguera *et al*., 2018).

Technical requirements	**Functional requirements**
Low capillary water absorption to reduce the infiltrated water, in terms of both the capillary coefficient and the asymptotic value; however, too low capillary absorption may decrease the drying ability.	M
Proper water vapour permeability, high enough for infiltrated water to be able to evaporate, but not so high as to favour the occurrence of subflorescence.	M, PR
A proper pore size distribution, to allow enough space for salts to crystallise.	PR
Lower strength than masonry, but high enough to ensure adhesion to masonry and prevent cracking (caused by salt damage).	A, M, PR
A lower modulus of elasticity than masonry, to ensure enough deformity.	M
Dimensional compatibility is used to avoid stress induction to masonry or between the coats that compose the render, which may lead to cracking or loss of adhesion, respectively. Dimension compatibility requires a proper modulus of elasticity and a thermal expansion coefficient similar to the original mortars.	M, PR
Low soluble salts content.	M, PR
Mechanical properties (strength and stiffness) decreasing from inside to outside when render applied in two or more coats.	PR
Good aesthetic appearance that preserves the image of the building (colour, texture, flatness and brightness similar to original mortars).	A
Structure and components are similar to original mortars to avoid major changes in behaviour and respect the retreatability principle.	M, PR

Notes: M - to protect masonry; PR – to prevent the degradation of renders;A – to contribute to aesthetics.

The functional requirements influence the design parameters since different mortars with different functions have unique functional requirements. For example, renders have lower strength requirements than bonding mortars since the latter is required to receive and transfer the load to the masonry elements, i.e., stones and bricks.

The functional requirements are derived from:

- The role or function of the mortars in the masonry element. This refers to the rendering, plastering, pointing, jointing and flooring.
- The role of the masonry element in the building influences the entire masonry behaviour (Romero-Noguera *et al.*, 2018).

The primary role of mortar in heritage masonry is to conserve and sustain the masonry via the categories indicated in Section 2.2. Because the mortar categories serve diverse purposes in masonry, it is vital to distinguish between them while designing restoration mortars. This is related to the various properties. For example, jointing mortars require more compressive strength than rendering mortars. The two's functional requirements differ significantly, as do their property requirements.

2.7.6 Performance requirements

According to Apostolopoulou *et al.* (2017), the performance of repair mortars is critical. It is crucial to evaluate the behaviour of mortars under various settings and when exposed to specific conditions. Park (1988) elaborates that replacement materials can be utilised to replace poor performing materials and are acceptable at the time of application. However, their appearance and performance might deteriorate quickly; therefore, it is critical to assess the replacement material performance prior to application. This will aid in the decision-making process for selecting appropriate repair mortars. Some performance factors to consider throughout the design phase are summarised in Table 2.3.

Table 2.3 Criteria for technical performance of new pointing mortars (extracted from Fontaine *et al.*, 1999).

Performance characteristics	**Limits Explanatory remarks**	
Compressive strength of mortar	1 to 8 MPa	Compromise between too strong and dense mortar and too weak, cracking and allowing water to enter. Bedding mortar should be a minimum of 2 MPa.
Split tensile/compressive strength of mortar	≥ 10%	Low tensile strength results in the cracking of mortars. This could be considered as a material quality measure for brittle materials, which have a ratio between the compressive and tensile strength of around 10. The tensile strength rarely exceeds the bond strength for cement and lime mortars; therefore, no upper limit is defined.
Young modulus	1 to 8 GPa	Describes deformability of mortars under stress. Mortars are valued for the ability to adjust to a minor movement. Too stiff mortar can cause cracking to the adjacent material. However, this depends also on the elastic (Young's) moduli of all materials involved.
Flexural bond of masonry	≥ 0.3 MPa	Ideally, the interface (the bond) of mortars and stone should be as strong as the mortar.
Expansion (freeze/thaw test) of masonry	≤ 0.04%	Unidirectional freeze/thaw test where the damage is quantified by the change in the width of the mortar joint. Expansion between 0.04 to 0.4% is considered marginal.

2.8 Design and development challenges for heritage mortars

As summarised in Table 3.3, the production of compatible restoration mortars has been explored using the majority of natural hydraulic lime resources (Isebaert *et al.,* 2014 and Apostolopoulou *et al.,* 2019). This process is difficult because of the large difference in properties between old/ancient and modern materials, undefined methods, and a misunderstanding of the functional and performance criteria (Figure 2.7).

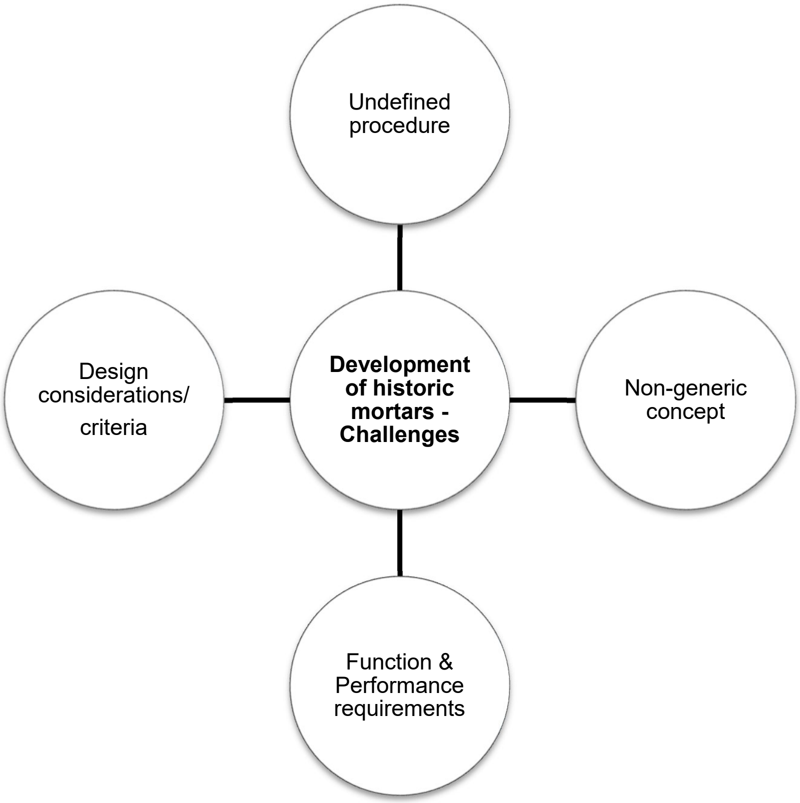


Figure 2.7 Historical mortar design and development challenges

2.8.1 Undefined development procedure

The selection of restoration mortars for historic constructions is unclear and poorly documented. Abdel-Mooty *et al.* (2009); Aggelakopoulou *et al*. (2019); Válek *et al*. (2019), and Lima *et al*. (2020) investigated multiple restoration mortars with various types of binder, such as cement, lime, natural hydraulic lime, and only clay (Table 3.3). The literature evaluated lacks details on adequate step-by-step design and development guides for future restoration practitioners. It is now impossible to design a repair mortar with certainty that the chosen mortar will perform sufficiently.

2.8.2 Concept that is not generic

Material design and production is not a generic concept. Materials from different times clearly have distinct characteristics (Loke, 2020), and so their performance will differ. Repair mortars from various eras are likely to differ in terms of properties, composition, and performance depending on their category and application region. It becomes difficult when restoring historic monuments because the expectation is to employ restorative mortars that are mostly based on the original material properties.

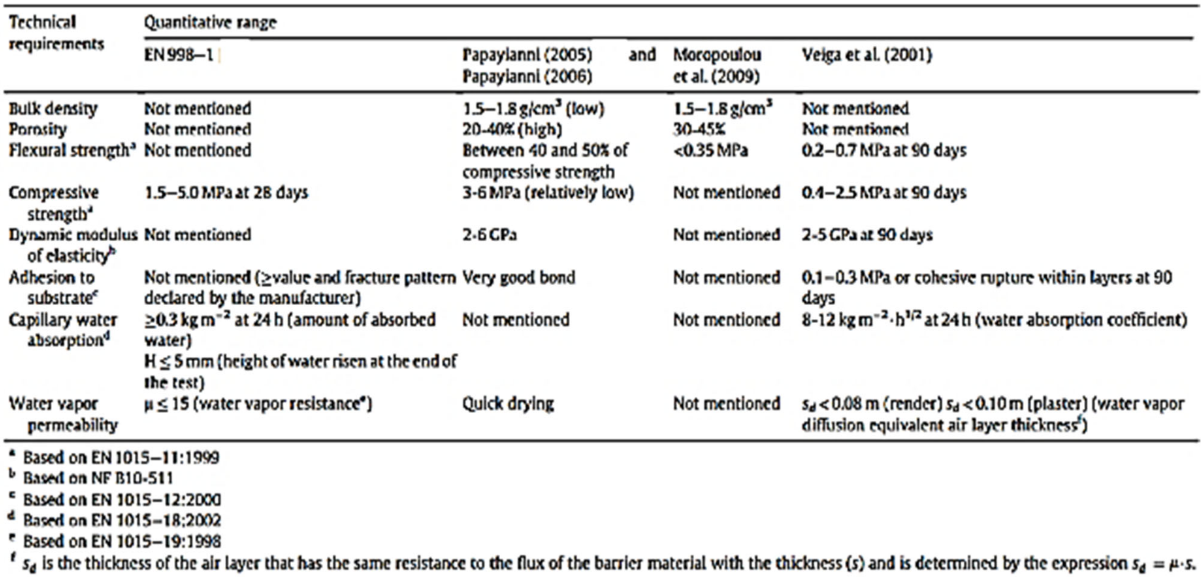
2.8.3 Design and development requirements

In masonries, mortars must be distinguished according to their roles, mostly for design and development purposes. The primary function of mortar in heritage masonries is to maintain and sustain the masonry from deterioration caused by either human or environmental factors. This aspect is related to the functional needs. The design and development requirements, such as the performance and functions of restoration mortars, are critical, according to Apostolopoulou *et al*. (2017). It is critical to evaluate the behavior of mortars under various settings and when exposed to specific environmental conditions. Thus, before application to historical constructions, material performance should be analysed.

2.8.4 Mortar design criteria and process

According to Groot *et al.* (2007), the design of new mortars has traditionally been centered on composition, but it is now based on characteristics. When designing heritage mortars, one needs to keep the following in mind: the characteristics of existing materials, such as binder type, binder-to-aggregate ratio, and climatic circumstances to which the mortar will be subjected, such as dry, low moisture and high moisture/humidity (Arizzi, 2012). It is a challenge when such information must be obtained since no guidance is available. Romero-Noguera *et al*. (2018) outlined the design selection criteria for repairing mortars, including optimum workability, three-day setting time or a tolerance of ten days, alkaline elements as low as 8 mg/kg, and flexural strength in the 0.4 – 2.5 MPa range. Additional replacement criteria are indicated in Table 2.4 as provided by different authors.

Table 2.4 General criteria for the selection of heritage repair mortars (extracted from Romero-Noguera *et al.,* 2018).



Technical requirements	Quantitative range			
	EN 998–1	Papayianni (2005) and Papayianni (2006)	Moropoulou et al. (2009)	Veiga et al. (2001)
Bulk density	Not mentioned	1.5–1.8 g/cm^3 (low)	1.5–1.8 g/cm^3	Not mentioned
Porosity	Not mentioned	20-40% (high)	30-45%	Not mentioned
Flexural strength[a]	Not mentioned	Between 40 and 50% of compressive strength	<0.35 MPa	0.2–0.7 MPa at 90 days
Compressive strength[a]	1.5–5.0 MPa at 28 days	3-6 MPa (relatively low)	Not mentioned	0.4–2.5 MPa at 90 days
Dynamic modulus of elasticity[b]	Not mentioned	2-6 GPa	Not mentioned	2-5 GPa at 90 days
Adhesion to substrate[c]	Not mentioned (≥value and fracture pattern declared by the manufacturer)	Very good bond	Not mentioned	0.1–0.3 MPa or cohesive rupture within layers at 90 days
Capillary water absorption[d]	≥0.3 kg m^{-2} at 24 h (amount of absorbed water) H ≤ 5 mm (height of water risen at the end of the test)	Not mentioned	Not mentioned	8-12 kg m^{-2}·h$^{1/2}$ at 24 h (water absorption coefficient)
Water vapor permeability	$\mu \leq 15$ (water vapor resistance[e])	Quick drying	Not mentioned	$s_d < 0.08$ m (render) $s_d < 0.10$ m (plaster) (water vapor diffusion equivalent air layer thickness[f])

[a] Based on EN 1015–11:1999
[b] Based on NF B10-511
[c] Based on EN 1015–12:2000
[d] Based on EN 1015–18:2002
[e] Based on EN 1015–19:1998
[f] s_d is the thickness of the air layer that has the same resistance to the flux of the barrier material with the thickness (s) and is determined by the expression $s_d = \mu \cdot s$.

In addition to the criteria above mentioned, the process of designing repair mortars is summarised by Arıoglu and Acun (2006) in Figure 2.8. The current study followed a similar process of designing and producing the restoration mortars, which involves four major activities and added the necessary modifications to suit the study area.

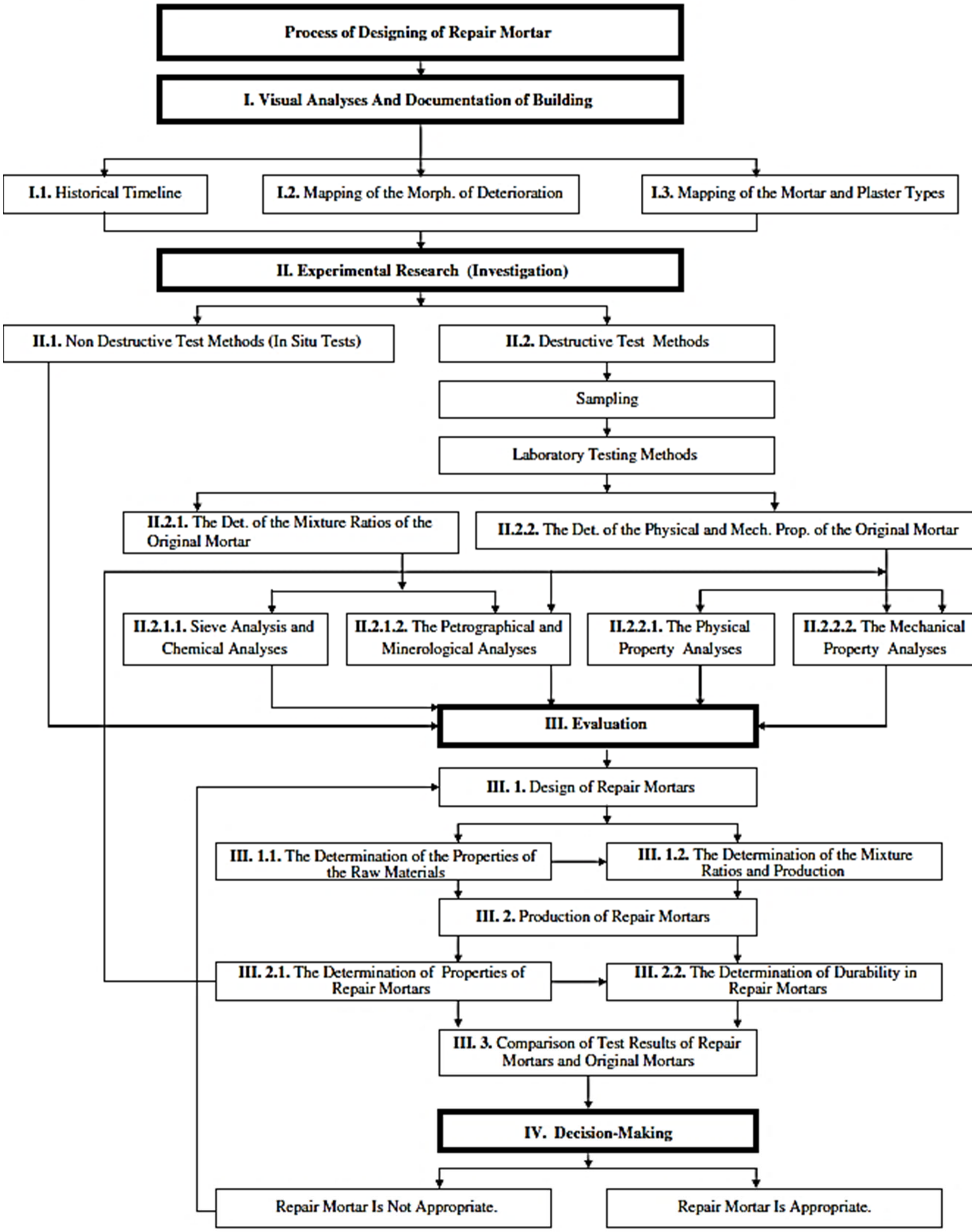


Figure 2.8 A flow diagram for the experimental design of repair mortars and plasters for historic buildings (extracted from Arıoglu & Acun, 2006)

2.9 Material testing and design Standards

The identification of heritage mortar properties has not been well practised in many parts of the African continent. This is mainly because of the lack of standards guiding this region's design and development of sustainable and compatible repair mortars. However, several international standards exist whose contribution to the design of heritage mortars is enormous. These standards address the complications associated with the design and application of repair mortars. Arizzi (2012) points to the Ten Books on Architecture by Vitruvius as the earliest known regulations pertaining to construction materials.

In addition to the long-existing Ten Books of Architecture, the modern construction industry relies on European (EN) and North American (ASTM) standards for the test methods to manufacture and control the quality of both ancient and modern mortars. The main difference between these standards and the RILEM recommendations, which focus mainly on historical materials, is that the former refers to industrial mortars in which a hydraulic binder is used by the state of the mortar (fresh or hardened). In contrast, the latter focuses mainly on lime-based mortars and their final function (structural, grouting, pointing, flooring, rendering, plastering etc.). The two binder types have different influences on the final mortar behaviour. Hence, the selection of applicable standards is critical.

2.9.1 European Standards (EN)

These standards provide descriptive procedures and limitations in terms of material sampling and physical, chemical, mineralogical and mechanical characterisation of historic mortars. There are deficiencies in some of these standards that the RILEM recommendations cover. The commonly used EN-standard includes the standard methodology for sampling from materials of cultural property - general rules, determination of water absorption by capillarity, determination of water vapour permeability, determination of drying properties, technical terms concerning mortars for masonry, renders and plasters as well as the characterisation of mortars used in cultural heritage structures.

2.9.2 American Standards for Testing Material (ASTM)

The ASTM standards are rarely used for cultural heritage materials because they focus on cement-based mortars. However, in cases where they are applied, modifications of the standard test procedures and sample sizes are always made. This is primarily because the sampling and testing of historical mortars is complex compared to modern materials. The restrictions and limitations on historical structures somehow result in non-standard sample sizes, which sometimes compromises the quality of results.

2.9.3 RILEM Recommendations

These standards provide test methods and specifications for evaluating lime-based repair materials for historic structures. Some RILEM recommendations stated by Alvarez et *al.* (2021) are listed in RILEM TC LHS- 277. These provide more specific and detailed guidance and support to restorers, architects and researchers pursuing cultural heritage restoration and conservation. Nonetheless, the scientific community and industries still focus mainly on EN and ASTM standards for assessing heritage repair mortar properties. This has resulted in several irregularities from applying these test standards to lime-based repair mortars, leaving a gap between industry and restoration. The problems emanate from differences in testing application between the lime-based and cement-based mortars in terms of equipment and methods used, sample sizes used and established curing conditions. Therefore, the results can be solely for comparative purposes and not for application in heritage restoration, thus making them irrelevant to the design of restoration mortars.

2.9.4 South African National Standards (SANS)

In South Africa, the commonly used standard methods for concrete and mortars are the SANS 2006 and the Cement and Concrete Institute Methods - 2012 (C & CI). The two guidelines, like the ASTM, focus on the analysis of cement-based concrete and very little on lime-based historical mortars. C & CI provides even further detailed guidance in terms of concrete mix designs towards specific target properties, as shown in Table 2.5. It highlights the details of different concrete applications while elaborating on each category's standard properties. But, no such guidance is provided for the lime mortars of historic buildings.

Table 2.5 Concrete mix design guidelines (extracted from Concrete manual 5-694.312, 2003). Legend: GI – Gradation index, GR - Gradation Range, vib- vibratory.

Kind of work	Method of placement	Type	Grade	Slump (mm)	GI	GR	Optional Gradations of aggregates	Mix no.	Remarks
Spec. 2411 Retaining walls									
Reinforced type walls	Manual + Vib	3	Y	75-100 (3-4)	1	3	35, 45, 50,	3Y43	
Gravity type walls	Manual + Vib	3	B	50-75 (2-3)	1	2	15-50 Incl.	3B32	
Concrete sub-foundation	Manual + Vib	1	A	75-75 (3-4)	1	3	35, 45, 50	1A43	
Spec. 2506 Manholes and catch basins									
Structures of Design A, C, E, F or G, drop inlet & surface block	Manual	3	B	75-100 (3-4)	1	2	15-50 Incl.	3B42	
All other	Manual	3	Y	75-100 (3-4)	1	3	35, 45, 50	3Y43	
Erosion control structures									
Culvert headwalls	Manual	3	A	75-100 (3-4)	1	2	15-50 Incl.	3A42	
Reinforced type dams	Manual	3	Y	75-100 (3-4)	1	3	35, 45, 50	3Y43	
Gravity type dams	Manual	3	B	50-75 (2-3)	1	2	15-50 Incl.	3B32	
Flumes, aprons, Spillways etc.	Manual	3	A	50-75 (2-3)	1	4	35-60 Incl.	3A34	
Spec. 2521 Sidewalks									
Plain	Manual	3	A	50-75 (2-3)	1	2	15-50 Incl.	3A32	
Exposed aggregate	Manual	3	A	50-75 (2-3)	1	6	50-70 Incl.	3A36	
Spec. 2531 Curb and gutter									
Slipform curb & gutter	Vibratory	3	A	25-50 (1-2)	1	2	15-50 Incl.	3A22	
Hand curb & gutter	Vibratory	3	A	50-75 (2-3)	1	2	15-50 Incl.	3A32	
Spec. 2533 Median barriers									
Cast-in-place barriers	Manual + Vib	3	Y	50-75 (2-3)	1	2	15-50 Incl.	3Y32	
Slipform barriers	Manual + Vib	3	Y	12-25 (1/2-1)	1	2	15-50 Incl.	3Y12	
Precast barriers	Manual + Vib	3	Y	50-75 (2-3)	1	2	15-50 Incl.	3Y32	
Prestressed concrete noise barriers									
Wall panels	Manual + Vib	3	W	50-75 (2-3)		6	50-70 Incl.	3W36	41 MPa required
Concrete posts	Manual + Vib	3	W	50-75 (2-3)		6	50-70 Incl.	3W36	

2.10 Development of restoration mortars

Several past restoration projects of heritage buildings have shown that designing and developing restoration mortars for pre-existing masonry is not a straightforward activity. It requires understanding from the researcher's perspective and close cooperation between the research and industry. Arizzi (2012) highlighted the lack of such a relationship between mass industrial production and smaller specialised needs for restoring historic structures. She mentions the existence of the concern as follows: "*Is it possible to produce a mortar to repair any type of historic building regardless of the wide variety of building materials and techniques originally employed in the construction? And will this mortar always fulfil the specific requirements of sustainability and compatibility*?" Researchers have used various mortars to practise compatible repairs of historic buildings, such as aerial and hydraulic lime-based ones (Isebaert *et al*., 2014; Apostolopoulou *et al*., 2019).

2.10.1 Mix design

A mix design refers to the selection of an appropriate ratio of materials such as lime, cement, water, stone, sand and additives, in some instances, used to produce fresh concrete or mortar of specified performance requirements such as strength, porosity and workability (Shi *et al*., 2015). Their study indicates that, for concrete purposes, the mix design is the first step in ensuring that the concrete performs as intended, both in the fresh and hardened state. The commonly used method for defining concrete mix design is the C & CI method, while the eyeball mix design, table of trial mixes, and nominal proportion methods are rarely used (Addis, 1998). In South Africa, the application of concrete and cementing materials needs to conform to the South African Bureau of Standards (SABS 5863: 2006) before application in construction works. On the other hand, this requirement leaves room for different interpretations where historical mortars are involved. In the South African context, there is not much guidance on these types of materials, so, it is unclear what methods and standards should be followed in relation to lime-based materials.

During the production of concrete and mortars, it is crucial to ensure good quality. This can be achieved through consistent mixing of constituents to avoid lumps, using an impermeable surface with no impurities that could affect the concrete properties and avoiding mixing the constituents in a place with external factors such as wind or rain as these would affect the intended final properties. It is also vital to accurately weigh the raw materials' mass in correspondence to the design to form a paste. This stage is crucial since any errors may result in properties completely different from the ones intended because of an existing relationship between water-to-cement ratio, workability, density, and compressive strength, among others (Xu *et al.,* 2012). A typical procedure for designing a concrete mix is depicted in Figure 2.9, with some deviations highlighted (in red) for heritage mortars. For compatibility purposes, evaluation of the chemical properties of the binder (see appendix A), which plays a major role in overall mortar behaviour is of great importance.

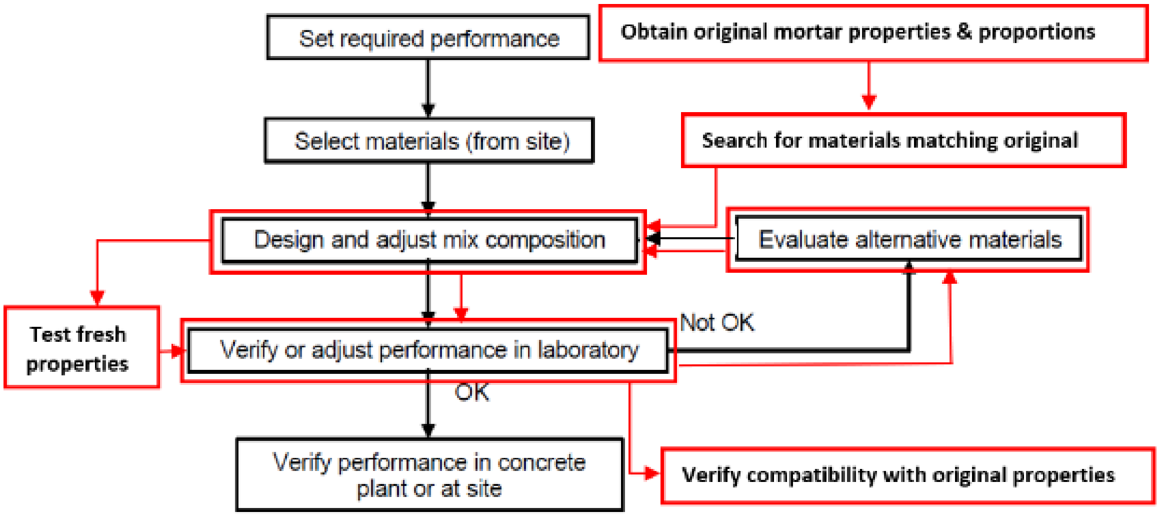


Figure 2.9 Concrete mix design procedure versus historic mortar design procedure (highlighted in red) showing the critical steps to be followed in the design process (modified from EFNARC, 2002)

2.10.2 Mixing ratio and procedure

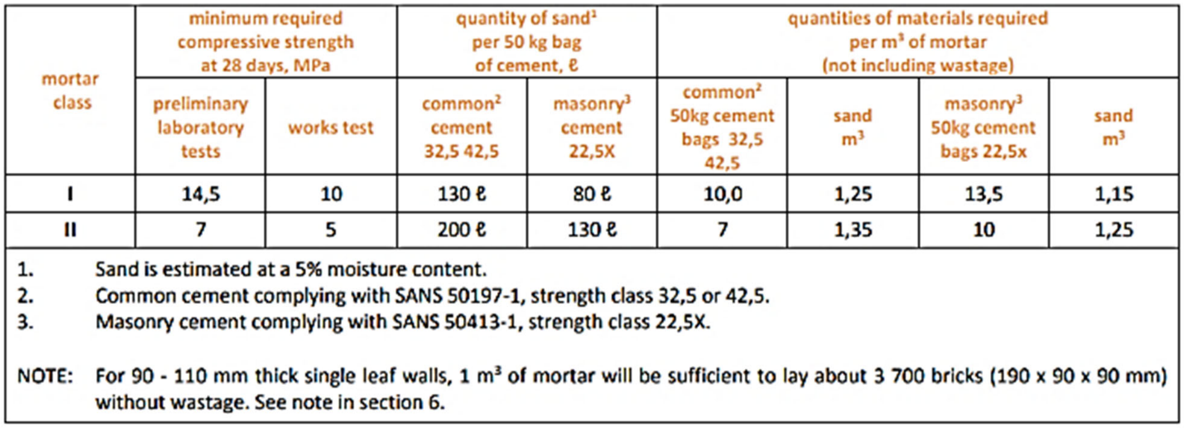
The aggregate to lime (binder) ratio selection is critical in establishing the final nature and qualities of the mortar. There is currently no standardised mixing ratio for lime mortars as there is for cement mortars, as indicated in Table 2.6. However, as per Moropoulou et al. (2002), the highly recommended lime-to-sand ratio is 1:3 by weight of materials for high-strength mortars. There appears to be a discrepancy among authors in terms of the mortar ratio. Cazalla *et al*. (2000) suggest a ratio of 1:4, which yields mortars with no cracks, while Lanas and Alvarez (2003), on the other hand, argue that the 1:1 lime/sand ratio offers the highest strength.

Table 2.6 The concrete versus lime mortar mixing ratios (by mass) IS 2250 (1981): Code of Practice for Preparation and Use of Masonry Mortars (As per government) Indian Standard, first revision, 1993.

Mortar application	Mix ratio (cement: sand) by weight	Recommended mix ratio (lime: sand) by weight
Ordinary masonry work	1:3 – 1:6	1:3
Reinforced brick work	1:2 – 1:3	-
Load bearing structures	1:3 – 1:4	-
Architectural work	1:6	-
External plaster	1:3 – 1:6	1:3
Internal plaster (rough sand)	1:5	-
Internal plaster (fine sand)	1:6	1:3
Flooring	1:4 – 1:8	-
Ponting	1:1 – 1:3	1:3

Some studies opted to mix the powder components and add water to repair mortars. In contrast, a study by Arizzi (2012) proposed mixing lime with water first before adding aggregates using the binder-to-aggregate ratio of 1:3 by weight. This mixing method is believed to help avoid lumps that usually form in lime mixes and helps achieve the desired mixing properties. As most of the literature suggests, two types exist; hand mixing and using a mixer with an average mixing time of 5 minutes. In addition, different types of binders require different mixing methods. For example, slaked lime is usually soaked in water for 24 hours before mixing. As stated by Cazalla *et al.* (2000) and Lanas and Alvarez (2003), this practice improves lime carbonation and reduces crack development. In South Africa, mortars' design and mixing proportions follow a guide summarised in Table 2.7. This design guide applies to cement mortars and not lime-based mortars. Hence, this research emphasises the need to address the existing gap in the design of heritage restoration mortar in terms of the design procedure.

Table 2.7 Mortar strength requirements and mix proportions (complying with SANS 2001-CM1:2012)



mortar class	minimum required compressive strength at 28 days, MPa		quantity of sand[1] per 50 kg bag of cement, ℓ		quantities of materials required per m^3 of mortar (not including wastage)			
	preliminary laboratory tests	works test	common[2] cement 32,5 42,5	masonry[3] cement 22,5X	common[2] 50kg cement bags 32,5 42,5	sand m^3	masonry[3] 50kg cement bags 22,5x	sand m^3
I	14,5	10	130 ℓ	80 ℓ	10,0	1,25	13,5	1,15
II	7	5	200 ℓ	130 ℓ	7	1,35	10	1,25

1. Sand is estimated at a 5% moisture content.
2. Common cement complying with SANS 50197-1, strength class 32,5 or 42,5.
3. Masonry cement complying with SANS 50413-1, strength class 22,5X.

NOTE: For 90 - 110 mm thick single leaf walls, 1 m^3 of mortar will be sufficient to lay about 3 700 bricks (190 x 90 x 90 mm) without wastage. See note in section 6.

Notes: Class I is for highly stressed masonry incorporating high-strength structural units that might be used in multi-storey load-bearing buildings or reinforced masonry.

Class II is for normal load bearing applications, parapets, railings, retaining structures, freestanding and garden walls, and other walls exposed to possible severe dampness.

2.10.3 Fresh mortar properties

Immediately after designing and producing restoration mortars, the fresh mortar properties need to be evaluated in terms of compatibility and performance, as summarised in Figure 2.10.

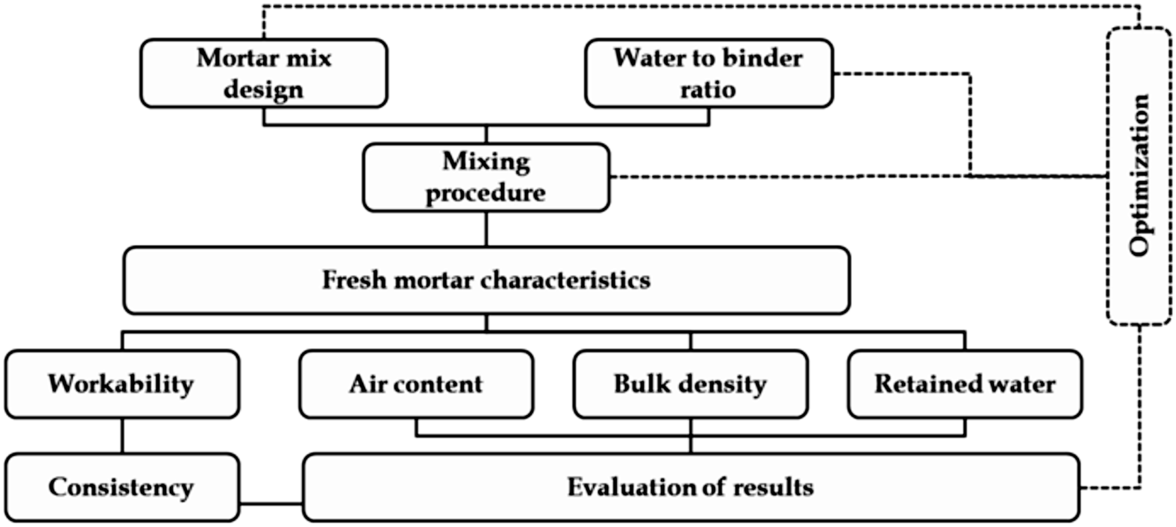


Figure 2.10 Methodology for assessing and optimising the properties of fresh mortar (extracted from Gherardi & Maravelaki, 2022)

Consistency

This property is determined using a flow table, in accordance with EN 1015-3 (2007) standard. According to Hanley and Pavía (2008), consistency is interlinked with the workability of the mortar. It increases in a non-linear manner as the water-to-binder ratio increases (Apostolopoulou *et al*., 2020).

Air content

This property is determined in accordance with EN 1015-7 (1999) standard. The air content entrapped in mortar during mixing is said to become part of the pore structure as the mortar sets and hardens. As elaborated in EN 459-1 (2015) standard, the allowable maximum air content for natural hydraulic lime mortars is 5%, with high percentages alleged to lower mortar strength and, thus, not recommended. However, Izaguirre *et al*. (2010) studied other researchers' perceptions of the requirement of air content below 5%, reporting the advantages of higher air content mortars in extremely cold climatic regions. The pore structure is said to allocate space for absorbed water to expand on freezing occasions (Izaguirre *et al*., 2010).

Bulk density

This is determined in accordance with EN 1015-6 (2007) standard to quantify the raw materials required for a restoration project. The test establishes the bulk density that the mortar will acquire after setting and hardening. In contrast to air content, the bulk density should be as high as possible.

Retained water

Retained water in historic mortars is determined through EN 1015-8 (2007) standard. This condition helps stabilise the mortar system after its application, during setting and hardening processes (Gherardi & Maravelaki, 2022).

2.10.4 Curing conditions and casting of repair mortars

The repair mortars for historic structures are mainly made of lime and not cured in water like cement. They are cured at regulated temperature and relative humidity, which determine the hardening pattern of the mortar. The drying conditions of mortars differ according to different binder types, and such conditions determine the mortar's characteristics. For instance, mortars produced from hydraulic binders yield optimum properties when early curing occurs under high humidity. In contrast, mortars composed of aerial binder require a lower humidity curing environment for the carbonation of the calcium hydroxide (Gherardi & Maravelaki, 2022).

2.10.5 Hardened mortar properties

After testing their properties in a paste form, the fresh mortars are cast into standard cube moulds of 40 × 40 × 40 mm and beam prisms of 40 × 40 × 160 mm for further assessment. The specimens are stored and allowed to be set and hardened in an environment with controlled temperature and humidity prescribed for achieving the desired properties. After curing the samples for the specified duration in monitored temperature and humidity, the cubes and beams are tested for their physical, chemical, mineralogical and mechanical properties using a summarised proposed methodology shown in Figure 2.11.

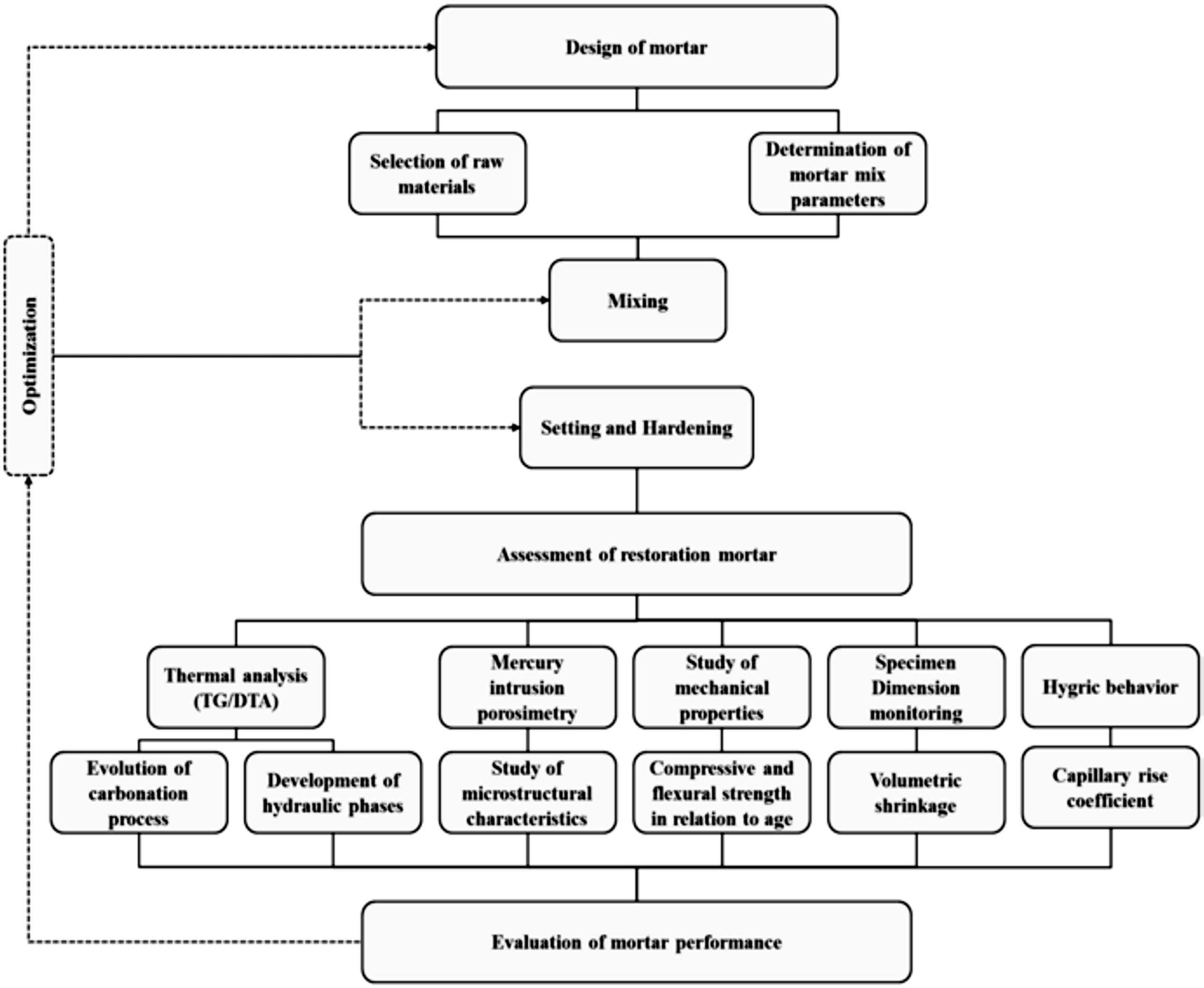


Figure 2.11 Methodology for evaluating and optimising hardened restoration mortar properties (extracted from Gherardi & Maravelaki, 2022)

Evolution of chemical reactions

The evolution of the chemical reactions within lime-based mortars is monitored throughout the mortar ageing up to carbonation completion. These reactions are commonly identified through thermogravimetric and differential thermal analysis (TGA/DTA) complemented by mineralogical analysis (Zhang *et al*., 2018). Any unwanted reactions that could affect the mortar performance are identified in this process.

Microstructural characteristics

The mortar's microstructural properties are important in achieving durability and compatibility. These properties further contribute to the mortar’s behaviour to substrate materials. For this analysis, mercury intrusion porosimetry (MIP) is a trusted technique for analysing the mortars' pore size distribution (Stefanidou, 2010; Anovitz, & Cole, 2015). Thomson *et al*. (2004) mentioned total immersion in water to identify the basic microstructural characteristics. They do indicate the shortcoming of this method as it does not provide details of the pore structure.

Shrinkage (related to volume)

Shrinkage refers to a difference in volume between fresh and hardened mortar expressed as a percentage. Mortars with low shrinkage are considered to be of high quality and are therefore recommended. Apostolopoulou *et al.* (2017) noted the relationship between mortar shrinkage and its deterioration rate and patterns that high shrinkage mortars tend to show microcracking, affecting their mechanical and hygric performance. In instances where low shrinkage mortars are applied in joints, minimised movements are experienced, leading to structural stresses (Gameiro *et al*., 2014).

Mechanical behaviour

The mechanical performance and compatibility of mortars are very important since they affect the behaviour of the entire structure (Apostolopoulou *et al*., 2018). Figueiredo *et al.* (2016) stated that compressive and flexural strengths are evaluated over time in accordance with EN1015-11 (2007) standard emphasising that the mechanical strength of heritage mortars should meet the specified strength requirements (Table 2.4), which increases over time. The differences in strength requirements for different mortar categories are also worth noting. Nonetheless, as far as this research is concerned, there is a limited indication of the specific strength requirements for different mortar categories.

Hygric behaviour

This refers to how water moves within the mortar, allowing breathability, which subsequently influences water and air movement within the masonry structure as a whole. Some hygric properties are capillary uptake, free and forced water absorption, and vapour permeability. These properties greatly influence mortar and masonry durability as water can transport soluble salts. Historic lime mortars are well recognised for their excellent hygric properties, which allow the structures to breathe while at the same time allowing adequate water ingress, as opposed to the application of modern cement-based mortars in historic structures, which trap the moisture and prevent air circulation within the structure (Isebaert *et al*., 2016).

2.10.6 Restoration materials

The industrial production of historic mortars involves mixing the dry components and adding water, according to the technical specifications and guidelines. The predominant historic materials are lime-based as studies indicate the use of cement in the construction industry only towards the end of the 19th century (Ngoma, 2009). According to Arizzi (2012), ready-to-use mortars are preferred over traditional ones. The former provides better component quality control and easy application with controlled or minimised workmanship errors. It is also vital to understand that no specific mortar exists for the various historic buildings because mortars are site-specific with different mix designs and components based on original material properties. Generally, all restoration raw materials should portray low quantities of soluble salts. The common materials used to produce restoration mortars include different types of binders and aggregates. Some of the commonly used binders include the following:

Aerial lime ($Ca(OH_2)$)

When simulations on historic lime mortars and research on pure lime mortar systems are done, aerial lime powder or putty is used (Gherardi & Maravelaki, 2022). In some cases where lime-pozzolan mortar is used, a mixture of aerial lime and pozzolanic additives in natural or synthetic form is often produced. This addition of pozzolanic additives enhances the mortar properties. According to Ngoma (2009), pozzolanic materials come in two forms, either natural - from volcanic materials or diatomaceous earth with dissolved calcium hydroxide ($Ca(OH)_2$) or artificial - a result of a reaction of dissolved calcium hydroxide ($Ca(OH)_2$) with fired clay or tile, shale, certain silicious rocks or fly-ash.

Calcitic and dolomitic hydrated limes

These types of lime mainly consist of calcium oxide and/or calcium hydroxide (Ngoma, 2009). Their microstructure significantly affects how they behave when in suspension, resulting in high workability. Arizzi (2012) discovered that the performance of dolomitic mortars during hardening exhibited greater shrinkage than calcitic lime mortars. Ngoma (2009) also warns against confusing hydrated and hydraulic limes. He further elaborates on the need to pay attention to the hardening behaviour, which is the main distinguishing factor between the two types of lime. According to Ashurst and Ashurst (1988), the hardening process of hydrated lime is a reaction between $Ca(OH)_2$ in the mix and atmospheric CO_2, while in hydraulic lime, water is essential for facilitating the chemical reactions with silicates and aluminates. During these chemical reactions in hydraulic mortars, some hydrated compounds are formed, thereby resulting in hydraulic mortars portraying hydrated lime mortar properties (Ashurst & Ashurst, 1988).

Quicklime (CaO)

This is the type of lime used primarily by researchers to simulate the hot lime technology of historical mortars (Margalha *et al.*, 2011). It is formed from carbon dioxide emitted while burning limestone, chalk, marble, shells or coral, producing hydrated lime (Ngoma, 2009).

Natural hydraulic lime (NHL)

Pinho Figueiredo *et al*. (2016) compare the use of NHL to that of pozzolanic additives. NHL is said to enhance mortar properties. This type of lime consists largely of calcium silicate, calcium aluminates and calcium hydroxide and is produced by burning argillaceous or marl limestone (Ngoma, 2009).

Gypsum and clay

These two materials could be used as independent binders or additives for choosing the lime binder. Gypsum is related to fast-setting mortars, while clay provides stability to mortars (Sophia *et al*., 2016 and Gomes *et al*., 2018).

Cement

It is also used as a binder or in combination with aerial lime. Nevertheless, Arizzi and Cultrone (2011) warn that research discourages the use of even a small amount of cement in lime mortars for heritage structures.

Table 2.8 summarises some of the design considerations, mixes and materials used by researchers across the world for historic mortars. Even though the researchers detailed such mortars, a concise procedure for designing and developing these mortars is still undefined and lacks clear detail.

Table 2.8 Historic mortar design methods and materials used in literature over the years.

Author	**Design and selection methodology and criteria for restoration mortars**
Abdel-Mooty *et al*. (2009)	They created lime-based mortars with white cement, gypsum, lignin sulfonate, and silica fumes to improve the mortar's performance. At 7, 28, and 56 days after mixing, these mortars were tested for compressive, tensile, and shear strengths. As a durability test, the weight loss of mortar cubes subjected to wetting and drying cycles was measured. In order to explore the composite structural behavior and bonding of stones and mortars, models of wall prisms were made using these mortars and tested under in-plane compressive stresses.
Aggelakopoulou *et al*. (2019)	They investigated the effect of calcium hydrated lime (lime putty or hydrated lime powder) on the chemical and physicomechanical properties of lime mortars. These were created with the same lime/aggregates mixing ratio and tested after 18 months of curing. The carbonation rate was determined using TGA/DSC, the pore structure was determined using MIP, and the hygric characteristics were determined using capillary rise and water immersion experiments. The mortars' shrinkage and apparent density were also tested, as well as their mechanical strength and static and dynamic modulus of elasticity, which were assessed using traditional mechanical testing and ultrasonic techniques. According to the findings, lime powder mortars have a higher carbonation rate and compressive strength than lime putty mortars. Lime powder mortars also exhibit a unimodal pore size distribution (while lime putty mortars present a bimodal one) and higher capillary rise coefficient and porosity accessible to water than lime putty mortars.
Apostolopoulou *et al*. (2018)	Prepared lime-metakaolin mortars with optimum compatibility and compliance with all set compatibility and the majority of performance criteria. These mortars are said to have early age strength and the highest mechanical strength values.
Arizzi (2012)	Designed rendering mortars using dry hydraulic lime. The study opted for mixing lime with water first before adding aggregates using the binder-to-aggregate ratio of 1:3 by weight. This was done to avoid lumps in the mix in order to achieve mixing proportion.
Freire *et al.* (2021)	They produced seven gypsum and lime-based plasters for interior walls with the addition of other components using commercially available materials such as limestone filler, water retaining agents and set retarders. Similar to Arizzi (2012), dry materials not lime putty were used.

Author	**Design and selection methodology and criteria for restoration mortars**
Hughes *et al*. (2012)	They recommend evaluating and quantifying the technical requirements such as adhesion, strength, elasticity, water and vapour transmittance, drying behavior, thermal dilatation, ability to deal with salt contamination and freeze-thaw cycling, and aesthetic features.
Válek *et al*. (2019)	They propose that the individual mortar components be chosen based on aesthetic factors (the type, color, and texture of the material to be reinstalled) as well as mechanical and physical attributes (such as strength, elasticity, porosity, and coefficient of thermal dilatation). As a result, these qualities must be examined ahead of time.
Kumar and Kumar (2022)	The authors studied lime-based mortars with the mix proportions of 1:3 and 1:1. In these mortars, they used lime-fly ash and lime-GGBS binders. The mortars had compressive and transverse strengths of 9.02 MPa and 2.46 MPa, respectively, less porosity, water absorption and rate of strength loss to alkali (NaOH) immersion with 31.23%, 18.52% and 9.42%, respectively. The two mortars displayed good thermal resistance, while the 1:1 mortar exhibited better salt crystallisation resistance.
Lima *et al*. (2020)	The article explains how to make three earth-based mortars using clayish earth rich in illite, kaolinite, and montmorillonite. Mortars were made with a 1:3 volumetric ratio of clayish earth and siliceous sand, and their characteristics were thoroughly characterised in both fresh and hardened states. Drying shrinkage and adherence are considered the most important properties to evaluate. Concerning the former, cracks were observed on montmorillonite mortar but are rare on illite mortar and absent on kaolinitic mortar.
Nĕzerka *et al*. (2014)	It was determined that the binder to aggregate volume ratio of 1:3 is the best for repairing mortars, achieving the greatest strength, and that mortars with greater ratios suffer from shrinkage cracking. Regarding the aggregate size, the sand of grain size up to 4 mm is the most favourable to use because this fraction produces mortars of the highest compressive strength (3.6 MPa), while the bigger grains cause a reduction in mortar strength.
Papayianni *et al.* (2000)	They suggested designing a new mortar according to its functional behaviour. This means that compatibility should be measured by properties characterising the functions of mortar in the structure. Characteristics were as follows: colour and surface structure strength, elasticity and deformability porosity and porosity properties coefficient of thermal dilation. The new mortar design was controlled mainly by composition (binder and filler proportions) combined with porosity and strength.
Papayianni *et al*. (2019)	Four different binding systems were used to prepare and test four different lime-based mortars: pure lime CL90 (according to EN459), Natural Hydraulic Lime NHL3.5 (according to EN459), Hydrated Lime + Volcanic Pozzolan 1:1 (CL90-Poz) and Hydrated Lime + Volcanic Pozzolan + White Cement 1:0.7:0.3 (CL90-Poz-Cem). There is no mention of durability testing to confirm the material's suitability.

Author	**Design and selection methodology and criteria for restoration mortars**
Pavía (2005)	RILEM (1980) standards were used to measure porosity, densities, compressive strength, capillary suction, and water absorption. The new mortars' compressive strength was determined using the BS 4551 (1980) standard, and their capillary suction was determined using the European standard EN 480-5 (1996). Petrographic microscopy and XRD were used for analysis. She emphasised the need for new replacement mortars to become durable and compatible with the original fabric but did not mention how to ensure this.
Pintea and Manea (2019)	Natural polymers, such as starch, cactus extract, animal clay, olive oil, jelly rice paste, and clay additions, were employed in the mortar plaster to improve mechanical qualities, increase water resistance, and improve texture. In this work, sticky rice was employed to consolidate lime mortar as well as the features of its microstructure. The rice is believed to have strong adhesive resistance, durability, and water resistance.
Porter *et al*. (2020)	The study proposes formulations of micro-grouts based on diethyl oxalate for stabilising painted lime plasters removed from limestone substrates.
Shivakumar et al. (2022)	For a study in India, the authors designed different mixes of lime, sand, and fermented organics with additions of bio-molecule components such as fatty acids, carbohydrates, polysaccharides, and proteins. Their mortar ratio was 1:3 by weight of materials based on European standards for restoring historic mortars.
Speziale *et al*. (2020)	Coatings with a 3D superhydrophobic structure based on inorganic ceramic oxides and photocatalytic nanoparticles have been suggested. TiO_2 - ZnO nano-heterostructures with mass compositions of 10-90 and 50-50 were used. The authors used superplasticizers to keep the nanoparticles from agglomeration, resulting in more effective mortar coatings.
Theologitis *et al.* (2021)	Natural hydraulic lime known for its similar physical characteristics and mechanical behaviour to the original historic binders was used with pozzolanic (Lafarge NHL-z 3.5) and metakaolin binders. The benefits of these types of binders include the absence of soluble salts, provision of hydraulic compounds and sustainability against environmental degradation. Metakaolin is an additive binder used to enhance the hydraulic behaviour of the mortar and its mechanical strength, but its use must be controlled for compatibility purposes. Binder-to-aggregate ratio of 0.5 was preferred for the purposes of strength properties, and water to binder ratio of 0.69 for attaining good workability. The grain size distribution was used to identify the suitable aggregates to be used in the mixes.

Author	Design and selection methodology and criteria for restoration mortars
Tsardaka and Stefanidou (2020)	They provide insight into the effect of various types of nanomaterials on the properties of air lime pastes, specifically nano-silica, nano-alumina, and nano-calcium oxide. The addition of nano-silica increases C-S-H formation, enhancing the compressive strength of the pastes and resulting in a denser structure. The primary function of nano-alumina is to serve as a filler, forming a continuous crystal net at the microstructural level. The combination of nano-alumina and nano-calcium oxide produced astonishingly favorable results, improving compressive strength, open porosity, water absorption outcomes, and microstructure. However, the aesthetics were not covered in this work, hence, it is unclear whether the mortar matches the original in terms of appearance.
Van Domelen (2009)	The proposed evaluation and selection method consists of ten steps: 1. Describe the original material's properties and performance (aesthetics, physical, chemical, mineralogical, and mechanical). 2. Determine the causes of failure in the original material and which qualities need to be modified with a new material to avoid repeat failure. Did the original material fail because of faults or specific material properties? Was it a result of poor design or detailing? Was it a result of bad installation? If the original material did not perform well, a replacement material could be used to improve performance. When considering alternative replacement materials, determining the original failure cause will disclose material attributes or performance characteristics that should be prioritized. 3. Develop a project-specific preservation philosophy and intervention target. 4. Create a "short-list" of possible replacement materials. 5. Assess the choices in terms of preservation philosophy, material qualities and performance, economic and environmental costs, and identify a suitable replacement material. 6. Keep a record of the review and selecting process. 7. Create design and installation standards and manage project planning. 8. Document and observe the installation procedure. 9. Complete a long-term follow-up assessment of in-situ performance. 10. Disseminate long-term material performance information for use by other preservation practitioners.

2.11 Mortar durability

Vukindu (2021) describes durability as “the ability of a structure to withstand the design environment over the design life without undue loss of serviceability or need for major repair.” Repair mortars need to withstand weathering from environmental conditions and biological decay factors (Freire *et al*., 2021). The major challenge associated with historic mortars and durability is meeting the current construction constraints. It becomes difficult to achieve the required strength since lime binders provide relatively weak mortars with a slow hardening rate. Even though sustainability only recently received much attention, historic mortars prove that the concept has long been practised. Sustainable durability is of paramount importance in historical mortar restoration. This can be achieved by using original raw materials which have proven their resistance to deterioration over time, and therefore, original material properties are key to achieving the durability of heritage mortars (Arizzi, 2012).

2.11.1 Admixtures and additives to improve durability

The use of admixtures to modify heritage mortar properties has been practised over the years. According to Arizzi (2012), materials such as blood, hair, straw, milk and eggs were commonly used. The additives such as retaining agents and hydraulic components such as pozzolans, fly ash, silica fumes, calcined clay, brick pebbles or lime dust help improve workability, waterproofing, mechanical resistance, durability, and shrinkage. Some of these materials were used even during the Roman times when volcanic material proceeding from the city of Pozzuoli in Naples was added to mortar constituents. Arizzi noted the non-existence of dosages prescribed for the use of these admixtures, thus creating a gap in a clearer understanding of their use in ancient mortars. Only such admixtures were used, and nothing on the quantities or the application method is mentioned. Super absorbent polymers (SAP) act as water-retaining agents and reduce shrinkage. However, the SAPs have shown some shortcomings in terms of workability, which, fortunately, could be resolved using plasticizers. In an attempt to protect the mortar against acid rain and sulphate attack, Arizzi (2012) stated the addition of barium hydroxide. Furthermore, for protection against sulphate attack, salt crystallisation processes and freeze-thaw cycles, the application of linseed oil (around 1%) is proposed.

2.12 Section summary

Ever since Portland cement was introduced to the construction industry and became one of the popular binders used in most modern constructions, industrial binder production has become somewhat reluctant to produce binders such as lime. Portland cement has been used in many remarkable construction projects with the highest success rate. Regrettably, the continuous use of Portland cement on historic structures creates adverse effects that may endanger the authenticity of these structures. The design and development of heritage restoration mortars becomes a topic of great significance as it would assist in reaching a deeper understanding of the procedure to be followed when designing new mortars with compositional characteristics and performances suitable for the original materials.

Thanks to recent advances in studies of suitable lime mortars, mostly in European countries, industries are swiftly encouraged to appreciate and pay attention to the radical differences between ancient and modern mortars. In this way, producing more suitable mortars becomes a promising task. It is acknowledged that there exists enough research around the design and production of mortars of both historic significance and modern ones. The studies have addressed the use of lime-based and cement-based mortars in heritage structures, with awareness made on maintaining the structure’s authenticity and achieving durability. However, the mix design and production procedure topic has yet to receive sufficient attention.

From the existing material standards, especially in South Africa, there is an insufficient guide for heritage mortar design and development. The existing construction standards focus mainly on cement-based materials and less on lime-base heritage materials. It becomes a challenge during the preliminary stage of heritage restoration in terms of describing a basic approach for the mix designs of the repair mortars and subsequently generating suitable mortars. It is simply a theoretical idea that could be debated on whether the aforementioned compositions are relevant and beneficial for the case studies. There is a need to create a precise procedure that will lead to the production of mortars that can be tested for durability.

2.13 Conclusion

The main purpose of this research was to apply high-end testing techniques to investigate the properties of heritage mortars from the two significant historic buildings in South Africa and propose a methodology to the existing South African National Standards for design procedure as well as production of suitable repair mortars. The reviewed literature indicates that repairing historic structures does not necessitate the use of readily available materials because it is nearly impossible to find equivalent traditional materials that were originally used during construction. This leads to the common option for the restoration teams of the present era, which involves using substitute materials. Nonetheless, this option requires a thorough examination of various raw materials, mix designs, and the application of multiple appropriate additives to match the mortar properties. This method of historical restoration is quite challenging and requires caution and careful evaluation of the original material properties. This is due to the potential consequences if the substitute components do not integrate properly with the original. It may result in a poor visual appearance as well as possible damage to the original surface and loss of authenticity.

The incompatibility between historic and substitute materials has been an issue for most restoration projects in most parts of the world. This is caused by the evolution of binder usage in the construction industry. Since nearly three centuries ago, when Portland cement was invented, its usage has grown enormously, and it is beginning to replace lime binder. Cement-based mortars and concrete have generally risen to a point where these materials are considered highly used in the construction industry. Regrettably, this popular construction material has failed to solve the decay problems on historic buildings by providing the key restoration goals; compatibility, suitability and longevity. Instead, it has resulted in visible inconsistencies and damage to the historic masonries.

Although much work pertaining to historic mortar characterisation and production of different repair mortars has been accomplished, there are still critical areas that still require investigation to improve the proper restoration process of these structures. Most researchers have carried out the characterisation of heritage mortars but have not provided a detailed process for the design and manufacture of the new mortars which are site-specific, as no single repair mortar is suitable for all heritage structures. Limited work has been undertaken to ensure these mortars' durability and applicability to achieve the desired outcomes. This research carried out durability and applicability tests associated with heritage mortars.

The most important conclusion that can be taken from the literature surveyed is that it is essential to convince the material production industry to follow scientific research to distinguish between historic and modern materials, especially with regard to their properties and behaviour in heritage restoration. Incorporating the straightforward design procedures, production guidelines, and performance testing of the heritage repair mortars into the South African National Standards would be a credible source for future restoration projects. This research work is believed to contribute to a broader understanding of the importance of involving the material manufacturing sectors and bridging the existing gap between the industrial production of historic restoration mortar components and research for proven compatible restoration materials in historically significant structures.

Chapter 3 Research design and methodology

3.1 Introduction

The majority of countries around the world have experienced the effects of wrongful selection and application of repair mortars to historic masonries. This is due to misunderstandings and insufficient data on the know-how of compatible historic material restoration. There is no doubt that the research in the area of original material characterisation before restoration works has advanced more satisfactorily in Europe, America, Asia, and Australia, but has yet to receive the necessary attention in Africa. Nonetheless, the design of different mortar categories and their development are regarded as areas of concern that lack the necessary due diligence.

The current research aims to address the present concern by the majority of historic authorities relating to an additional step after the successful characterisation of historical mortars, namely guidance in terms of design procedures and production of suitable repair mortars. Within the scope of this study, an initial historical and contextual literature review was undertaken to provide justification and background for executing this project. After that, the characterisation of original mortars followed. This is considered a prerequisite in historical mortar restoration. The testing philosophy, experimental variables, equipment and raw materials used for the production of the new mortars and the data obtained are presented in this chapter. Approaches in terms of standards and procedures recommended by Arıoglu and Acun (2006) and Gherardi and Maravelaki (2022) for a concise understanding of the materials selection criteria were considered and modified for application in this study.

The experimental work was split into two phases. The first phase involved investigating the characteristics of existing materials from two heritage structures in South Africa's Western Cape. Phase two was a further investigation of the various mix designs and the production of repair mortars for specific sections. Finally, the new materials underwent performance testing to verify their compatibility with the old materials.

3.2 Research design

The current study was a quantitative experimental procedure that involved quantifying heritage mortars' components and potential repair mortars' components. The properties of materials were assessed with reference to the RILEM Recommendations and EN standards. The initial stage involved a desk study whereby the literature related to heritage restoration material design and production was reviewed. Following that, characterising the mortars collected from the Castle of Good Hope and the two oldest buildings on Robben Island in the Western Cape Province, South

Africa. After studying the original material properties, we initiated the process of formulating the mix designs used to produce the repair mortars for these heritage structures.

A shortlist of potential replacement materials was drawn up, and the best-fit materials were eventually selected. The selected raw materials were tested for SANS-recommended properties, such as grading for the grainy materials. Thereafter, a methodology similar to the one proposed in the majority of the literature reviewed was followed to analyse the fresh and hardened properties of new mortars, even though some details were missing for different categories of mortars. The new mortars were assessed for their compatibility with the original, and the selection process was conducted based on the mortar properties and durability characteristics. A further analysis of these new mortars for their durability was evaluated and confirmed to pass the recommended criteria as stated in the literature. It has to be pointed out that this criterion is not a well-accepted and documented standard but is the view of several researchers.

3.3 Case studies

The Castle of Good Hope (built around 1600s), the maximum-security prison, and the pre-primary school on Robben Island (built from the 1700s to 1900s) were the selected case studies, both located in Cape Town, South Africa (see Figure 3.1). These structures hold significant historic status in South Africa, Southern Africa, and the world (Loke *et al.,* 2020). The structures are in different topographical locations: the Castle is inland even though very close to the coastline, while the latter is on an island in Table Bay in the Atlantic Ocean. For the Island buildings, the maximum-security prison is regarded as the most remarkable and historical precinct because of its use for detaining political prisoners, particularly Nelson Mandela.

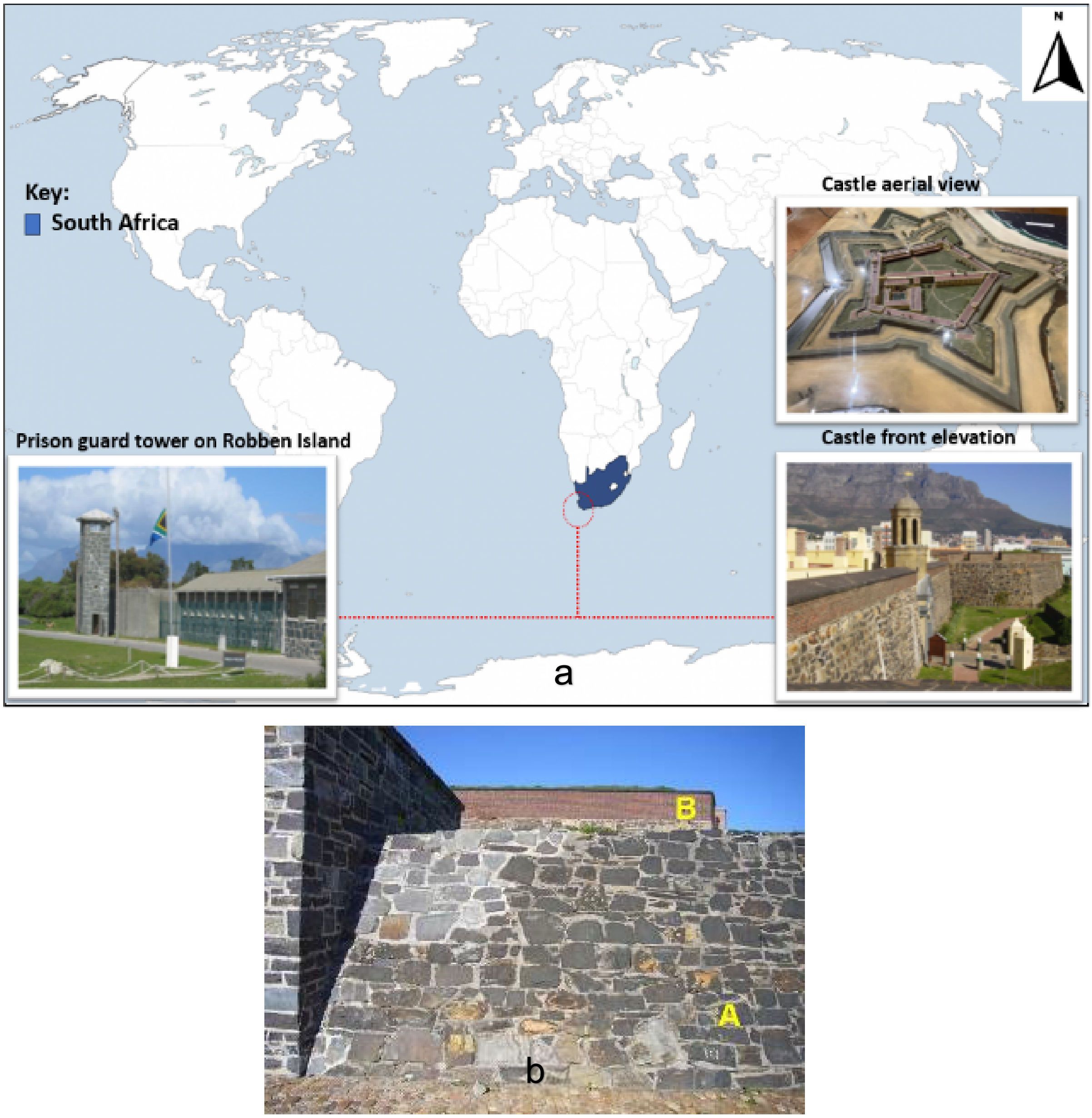


Figure 3.1 Geographical map of South Africa and aerial view of the Castle of Good Hope, an elevation (on the right) showing the castle's entrance. The prison guard tower on Robben Island constructed using blue slate is shown on the left. A) The Dutch wall section constructed using slate, and B) The British wall extension using reddish brickwork (modified from: https://www.castleofgoodhope.co.za/index.php)

3.3.1 Castle of Good Hope

The Castle of Good Hope is one of South Africa's most profoundly significant heritage site, encapsulating meaning for the nation itself. It is of historical value for certain European regions like the Netherlands and the United Kingdom. The castle's origins date back to 1666 when the Dutch East Indian Company (VOC) laid its foundational stone in the Cape (Zwarteveen, 2016).

The VOC, actively engaged in spice trade across Europe, Asia, and Africa, left an enduring mark on this structure. This colonial masterpiece, a testament to VOC's architectural prow-ess, has stood the test of time and is arguably one of the best-preserved remnants of VOC's global architectural endeavours. Much credit is owed to the relentless efforts of SAHRA in safeguarding such valuable structures.

The distinctive architectural design of the castle's pentagon (Figure 3.1), characterised by its five principal bastions, offers a captivating aerial vista from Cape Town's heart, situated on Strand Street. Its construction involved the excavation of 3.5-meter-deep foundations, followed by constructing 0.6-meter-wide, 10-meter-tall blue slate walls. In the 1830s, when the British took control of the castle from the Dutch, they extended the structure by raising the walls with reddish brickwork, as shown in Figure 3.1b. Further alterations brought about by the British included transforming the previously flat slate roof style to a pitched roofing style (source: https://www.castleofgoodhope.co.za/index.php). The materials used to construct this monument were sourced from Signal Hill and Robben Island (Cape Town), with decorative clinker bricks imported from the Netherlands. According to a report on Historical Buildings in South Africa (1979), some of the mortars used on the castle consisted of one part lime from shells (calcitic lime) mixed with one part clay. At the same time, other mortars were made from mixing a binder produced by burning shells in lime kilns at Robben Island, shells and sand (Historical Buildings in South Africa, 1979). Since 1922, the South African government has been responsible for overseeing the castle's maintenance, which presently serves as the headquarters for the Western Cape Military and remains a prime attraction, drawing scores of tourists daily.

The castle has undergone major deterioration in its masonry due to decayed mortars. Multiple site visits were conducted to assess the status of the Castle’s mortars in different locations. Without a doubt, it was evident that this structure requires restoration using compatible and durable mortars. This will foresee the long-term existence of masonry for centuries to come. The restoration work conducted on the Castle in the mid-to-late 1960s played a major role in ensuring this monument's existence (Gilbert, 1994). Regrettably, among the most recent repair works on the masonry of the Castle are a true reflection of restoration without a clear understanding of the materials being replaced versus the replacement materials. There is visual evidence of failure in the repairs, which negatively impacts the aesthetics of the Castle. In some of the walls, structural failure is also the case. This threatens the structure's existence, as there is no guarantee that the walls can withstand the applied loads for long without effective mortars.

3.3.2 Robben Island buildings

Robben Island is on Table Bay Island, which is accessible by a 30-minute ferry ride from the Nelson Mandela gateway at Waterfront in Cape Town. The Island is a UNESCO world heritage site encompassing both cultural and environmental heritage. It was used as a political prison to incarcerate the freedom fighters against the apartheid government, a military base and a leper colony. The buildings on the island date from the 1700s including additional structures constructed in the 19th and 20th centuries. The structures were built using blue slate and lime believed to have been quarried from the island's limestone quarry, which has thus far been closed for quarrying activities. Not much is documented regarding the mortar proportions for the buildings on this site.

From 1999 onwards, there were repairs to the Island's masonry (Conditional Assessment of Buildings on Robben Island, 2018 and Loke, 2020). Some major works included applying raised pointing on the stone walls of some building precincts on the island. The rendering of the oldest building (Pre-Primary school) using Portland cement-based rendering mortar is one other restoration mistake of the past. The majority of the repairs are starting to fail, and the evidence provided by Loke (2020) showed the use of materials different from the original materials to repair the decayed pointing on the masonry. Similar to the Castle, this threatens the Island's oldest building precincts.

3.4 Materials and methods

The primary goal of this research was to explain the design and production process of historical restoration mortars utilising locally available raw materials. The reverse engineering procedure was first carried out to provide an informed guide to the sourcing of the replica substitute materials. The study used an experimental approach with a series of aesthetic, physical, chemical, mineralogical and mechanical analyses, as shown in Figure 3.2. This methodology could be used as a guide for the design and development of heritage restoration mortars (in the South African context) by restoration practisioners and researchers. It was observed that the process of compatible restoration of historical masonries requires a multidisciplinary research exchange and collaboration between the Department of Mineralogy and Petrology at the University of Granada (UGR) in Spain and the Department of Civil Engineering and Geomatics at the Cape Peninsula University of Technology (CPUT) in Cape Town, South Africa were formulated. The initial experiments were conducted at the Centro de Instumentación Científica (CIC) of the UGR. The results from the second phase of experiments in this section were compared with the qualitative results in the initial phase, whereby original material properties were determined.

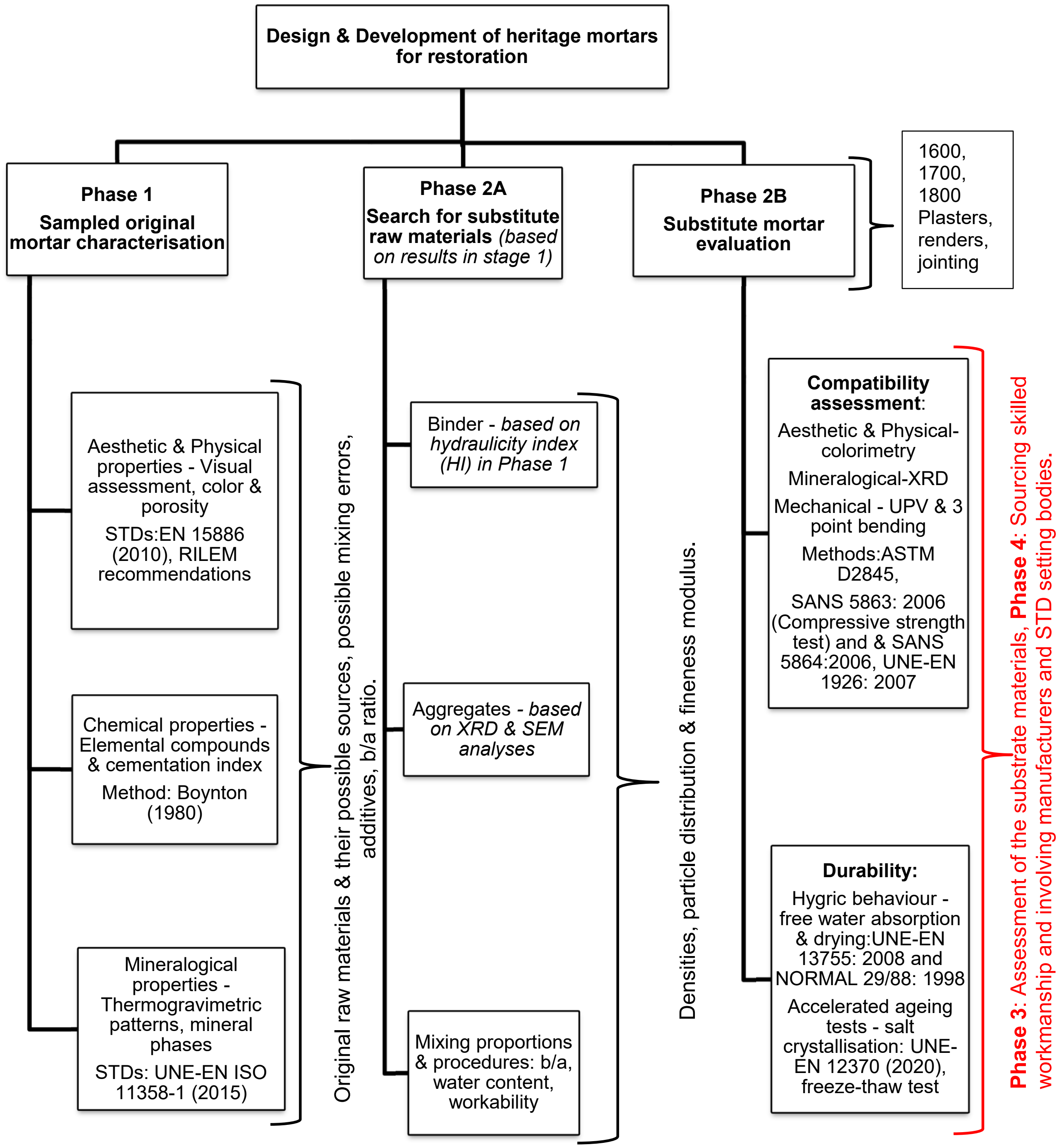


Figure 3.2 Derived methodology flow chart for designing and developing compatible and durable heritage repair mortars showing different phases: Phase 1-original material characterisation, Phase 2A-New mortar design and testing and Phase 2B-Compatibility and durability testing of the new mortars and finally Phases 3 and 4 encompassing the assessment of the mortar substrate materials and ensuring the use of skilled mason for restoration work

3.5 Phase 1: Original mortar analysis

A reverse engineering technique was used to propose a compatible restoration mortar. This involved collecting different types of original mortars from the Castle and Island building masonry and then carrying out an in-depth characterisation of the original samples. The results will be useful in producing the repair mortars based on reference properties to match the original. Information on binder type, aggregates, additives, and their respective proportions was obtained and used as a basis for searching for suitable replacement materials where necessary. The semi-quantitative results obtained in a Masters thesis by Loke (2020) were verified, and a full quantitative analysis was conducted in this doctoral thesis. The alternative methodology is presented in Table 3.1, indicating the systematic methods followed by Loke (2020) versus the present work.

Legend
Master Thesis: ▨
Doctoral Thesis: 🟩
A: The amount of time it takes to complete a test.

B: The amount of material needed for a test.

C: The quality of technical data required to develop repair mortars.

D: The method's ability to provide information on the mortar composition.

Table 3.1 Methodology selection – Master project *versus* detailed quantitative Doctoral project.

Properties	Property & Technique	A	B	C	D	Advantages	Disadvantages
Aesthetic	Cohesion (human hand)	<1 h	10 g	Adequate	Yes	Specimen may be used in further analysis, technically easy to perform	Results may differ according to personal views (subjective)
	Colour test (colorimetry)	<3 s	<1 g	Adequate	Yes, the colour for aggregates would serve as the guide for searching for new aggregates	Provides a quantitative measurement of the colour allowing to distinguish colours which can appear identical to the human eye	Cannot be used for colourless compounds
	Optical microscopy	<1 h	10 g	Adequate	Yes (limited)	Allows determination of grain size distribution, texture and mineralogy	At least 1 day for preparation of thin section hence, time-consuming
	Visual investigation (Colour chart)	<1 h	10 g	Adequate	Yes (limited)	Specimen may be used in further analysis, technically easy to perform	Results may differ according to personal views (subjective)
Physical	Frost resistance (freeze-thaw test)	30 days (30 cycles)	>100 g	Adequate	No	Technically easy to perform	Sample cannot be used in further analysis, time-consuming
	Moisture content (Gravimetric drying)	48 h	3 cm^3	Adequate	No	Specimen may be used in further analysis	Time-consuming
	Permeability (surface probe)	1 h	3 cm^3	Adequate	Yes (limited)	Specimen may be used in further analysis	Time-consuming
	Porosity (mercury intrusion porosimetry)	1 h	<1.5 g	Adequate	Yes	Rapid & accurate testing procedure, determines pore size distribution on small sample size, open porosity and specific surface area	Expensive lab equipment is needed, specimen cannot be reused

Properties	**Property & Technique**	**A**	**B**	**C**	**D**	**Advantages**	**Disadvantages**
	Porosity (water absorption by immersion)	Approx. 14 days	3-5 cm^3	Adequate	Yes (limited)	Technically easy to perform. Sample can be used in further analysis	Time-consuming
	Sieve analysis (by using HCl)	1-2 h	At least 100 g	Adequate	Yes (Aggregates)	Technically easy to perform	Large material quantity needed; sample is destroyed
	Sieve analysis (dry)	1 h	50-100 g	Adequate	Yes (Aggreg.)	Technically easy to perform, sample can be used in future	Separation of the aggregate, large material quantity needed which is usually impossible to obtain
	Thermal conductivity	7 days	>200 g	Adequate	No	Technically easy to perform	Very time-consuming
	Drying test	2 days	3-5 cm^3	Adequate	Yes (Approx.)	Technically easy to perform. Sample can be used in further analysis	Time-consuming
Chemical	Atomic absorption spectroscopy (AAS)	1-2 h	5-10 g	Adequate	Yes (elements)	High sensitivity, technically easy to perform	Expensive element, time-consuming when preparing the sample
	Hydrogen potential test	<1 h	5-10 g	Inadequate	No	Technically easy to perform	Incomplete analysis details
	Induced coupled plasma (ICP)	1-2 h	1-10 g	Adequate	Yes (elements)	High accuracy	Expensive lab equipment needed, time-consuming when preparing the sample
	Scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS)	<2 h	~ 2 g	Adequate	Yes (elements)	Less preparation time of the sample	Expensive lab equipment
	Titration of element oxides	1 h	5-10 g	Adequate	Yes (Binder)	No need for high end equipment, easy to perform	Some level of operator skill required, can be time-consuming
	X-ray fluorescence (XRF)	1 h	1-5 g	Adequate	Yes	High accuracy & easy sample preparation	Expensive lab equipment needed

Properties	Property & Technique	A	B	C	D	Advantages	Disadvantages
Mineralogical	Fourier transform infra-red spectrometry (FTIR)	<1 h	<2 mg	Adequate	No	Highly sensitive	Expensive lab equipment needed
	Gas chromatography	<1 min	<1 g	Adequate	No	High accuracy in few minutes	Expensive lab equipment needed
	Ion chromatography (IC)	24 h	0.1 g	Adequate	Yes (ions)	Accurate as well as reliable	Time-consuming
	Salt crystallisation test	15 days (15 cycles)	>100 g	Adequate	Yes (limited)	Technically easy to perform	Large quantity of sample required, sample cannot be used in further analysis
	Soluble salt analysis (titration)	<1 h	0.1 g	Adequate	Yes (major salts)	Technically easy to perform	Qualitative not quantitative analysis
	Thermogravimetric - Differential scanning calorimetry (TGA/DSC)	3 h	~ 50 mg	Adequate	Yes	Small consumption of material and highly sensitive	Expensive lab equipment needed
	Powder X-ray diffraction (PXRD)	<1 h	~ 1-2 g	Adequate	Yes	Considered least expensive and provides the necessary minerals in mortar samples. The sample can be reused.	It is not able to detect amorphous phases as well as crystalline phases in very low amounts.

3.5.1 Collection and processing of samples

The conservation plan for historic masonries through the design and development of mortars should be established prior to sampling, as suggested by Arizzi (2012). Thereafter, the relevant historic data in terms of construction and materials usage history, visual and photographic survey and restoration history needs to be collected and critically analysed. The following stage consists of drawing up a sampling and characterisation plan in alignment with the aim of the study. Laboratory availability, materials, and equipment are the main factors influencing the characterisation parameters. The original mortar samples were collected from the existing heritage structures using minor to non-destructive methods. Sampling was conducted in accordance with EN 16085 (2012) standards using a sample information table recommended by Ngoma (2009). As most of the experiments were carried out at UGR in Spain, an export license for the materials was granted by SAHRA, in the form of finely ground powders, as shown in Figure 3.3.

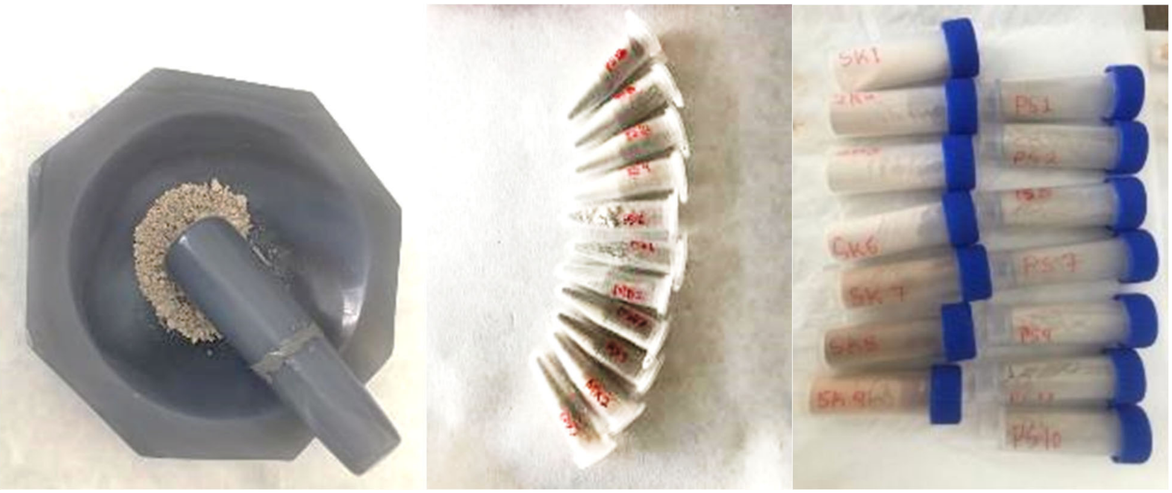


Figure 3.3 Mortar preparation by means of gentle crushing using an agate mortar and pestle for TGA/DSC, PXRD and XRF analyses

A summary of samples collected from the heritage buildings in the form of powders, thin sections and fragments is presented in Table 3.2. The samples were carefully selected to represent different mortar categories such as bonding/jointing, plaster, floor and render. The methodology in Phase 1 incorporated analysis of original in-situ and powder mortars, mechanical separation of binder from the aggregates using a 0.063 mm sieve and individual analysis of both components.

Table 3.2 Summary of original mortar samples and their properties based on visual observation.

Sample ID	Age	Category	Description	Original Mortar
SK1	1666	Plaster	Whitish mortar with black and reddish aggregates & finely crushed shells	Lime
SK2		Plaster	Whitish grey mortar with finer black-grey aggregates & finely crushed shells	Lime
SK3		Plaster	Whitish yellow mortar with black-grey aggregates & finely crushed shells	Lime
SK4		Floor	Whitish grey mortar with dense black-grey aggregates & finely crushed shells	Lime
SK5		Bedding /joints	Whitish mortar with black-grey aggregates	Lime
SK6		Bedding /joints	Cream white mortar with black-grey aggregates & medium crushed shells	Lime
SK7		Bedding /joints	Light yellowish soil mortar	Earth
SK8		Bedding /joints	Reddish brown soil mortar	Earth
SK9		Bedding /joints	Yellowish orange soil mortar	Earth
MX1	1700	Raised pointing	Grey mortar	Cement
MX2		Joints/bedding	Whitish mortar & finely crushed shells	Lime
MX3		Joints/bedding	Whitish to grey mortar & finely crushed shells	Lime
PS1	Before/around 1846	Rendering	Dark grey with 2nd layer of whitish-grey material attached	Cement
PS2		Rendering	Dark grey with whitish-grey layer attached to it	Cement
PS3		Rendering	Dark grey mortar	Cement
PS4		Plaster	Whitish grey with dense white & grey shell fragments	Lime
PS5		Rendering	Grey mortar with slate particles	Cement
PS6		Plaster on steps	Greyish with brown aggregates and traces of white finely crushed shells	Lime
PS7		Plaster	Grey mortar	Cement
PS8		Floor	Grey mortar with large stone(slate) aggregates	Cement
PS9		Plaster on steps	Whitish grey with traces of finely crushed shells	Lime
PS10		Rendering	Whitish grey with white medium-sized shells & slate aggregate (not dense)	Lime
PS11		Rendering	Whitish grey with white medium-sized shells (very dense)	Lime
PS12		Floor	Grey mortar with medium crushed slate aggregates and minor traces of white shells	Cement

3.5.2 Colorimetry analysis – Aesthetic analysis

There was an increase in curiosity for observing the colour difference around 1931. The Commission Internationale de l'Eclairage (CIE) used this to introduce the standard observer to colorimetry. This colour determination method reliably determines the tolerance of prints, colours, materials, inks, photographs, and the calibration of multimedia equipment. They, however, indicated that the following factors influence colour perception:

- Physical properties of the observed object, particularly its absorption characteristics,
- Spectral composition of the light source and characteristics of the environment through which it passes,
- Properties of the observer's visual system, including the state of their neural centres and transmission receptors, and
- Proximity to other objects, their properties, and experience gained from observation of similar objects.

A non-destructive test (NDT) was developed for mortar and aggregate samples independently to acquire a complete understanding of the colour of the mortar components as part of the priority properties for the production of new mortars as defined by Hauková *et al.* (2013). Lightness and chromatism of mortars are considered important features to examine during a historic mortar study. It is best to analyse the portion of the mortar that has had the least amount of exposure to the deterioration factors. This accurately represents the original color of the mortar in question. This system is a three-dimensional model that uses chromatic parameters related to the CIELAB space, where L* represents the lightness coordinate (0 black to 100 white), a* is the chromatic coordinate between red (+60) and green (-60), b* is the chromatic coordinate between yellow (+60) and blue (-60), C* is the chroma and represents the saturation of a colour (from 0 to 100), and hº is the hue angle (from 0º to 360º) as depicted in Figure 3.4.

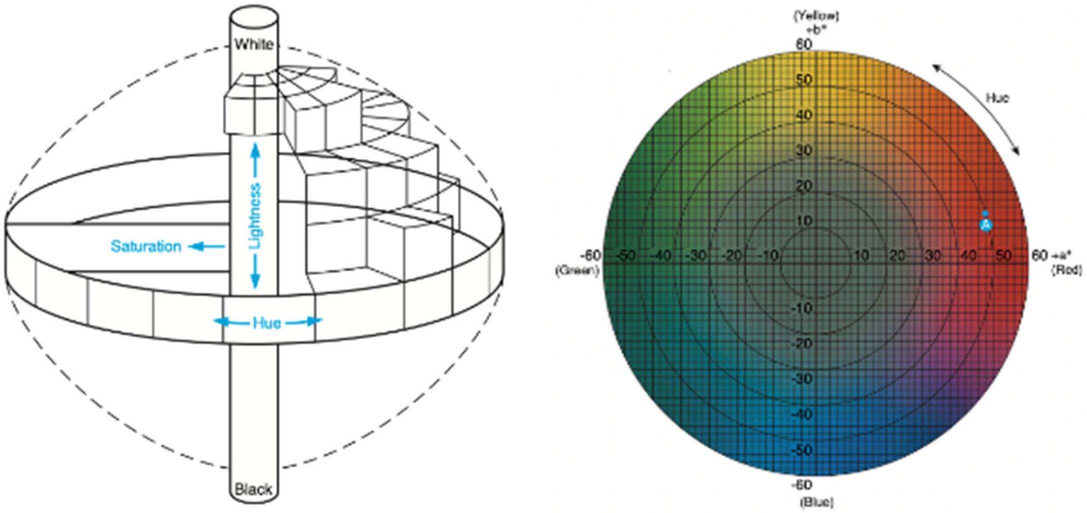


Figure 3.4 Colour measurement in three dimensions: hue, brightness, and chroma (saturation) (Konica Minolta, Precise colour communication)

The L*a*b* colour space is defined using the equations 3.1, 3.2 and 3.3 respectively:

$$L^* = 116\left(\frac{Y}{Yn}\right)^{1/3} - 16 \tag{3.1}$$

$$a^* = 500\left[\left(\frac{X}{Xn}\right)^{1/3} - \left(\frac{Y}{Yn}\right)^{1/3}\right] \tag{3.2}$$

$$b^* = 200\left[\left(\frac{Y}{Yn}\right)^{1/3} - \left(\frac{Z}{Zn}\right)^{1/3}\right] \tag{3.3}$$

Where:

X, Y, Z represent Tristimulus values XYZ for 2° Standard Observer or X_{10}, Y_{10}, Z_{10} for 10° Supplementary Standard Observer of the specimen.

Xn, Yn, Zn represent Tristimulus values XYZ for 2° Standard Observer or X_{10}, Y_{10}, Z_{10} for 10° Supplementary Standard Observer of a perfect reflecting diffuser.

It is, however, to be noticed that, if the X/Xn, Y/Yn or Z/Zn value is less than $(24/116)^3$, the changes indicated in equations 3.4, 3.5 and 3.6 apply.

$$\left(\frac{X}{Xn}\right)^{1/3} \text{is replaced by } \frac{841}{108}\left(\frac{X}{Xn}\right) + \frac{16}{116} \tag{3.4}$$

$$\left(\frac{Y}{Yn}\right)^{1/3} \text{is replaced by } \frac{841}{108}\left(\frac{Y}{Yn}\right) + \frac{16}{116} \tag{3.5}$$

$$\left(\frac{Z}{Zn}\right)^{1/3} \text{is replaced by } \frac{841}{108}\left(\frac{Z}{Zn}\right) + \frac{16}{116} \tag{3.6}$$

The degree of colour difference (ΔE^*) was conceived in 1948. With time, there were evolutions to this expression over the years (1950s and 1960s), and in 1966, R. S. Hunter expressed this difference using the currently well-accepted equation 3.7 (Mokrzycki & Tatol, 2011):

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \tag{3.7}$$

Where:

ΔL^*, Δa^*, Δb^* are the changes in L*, a* and b* values between the specimen colour and the reference.

To compare the new and old mortars, the following standard ranges of ΔE with regard to the observer were employed, as determined by Mokrzycki and Tatol (2011):

0 <ΔE <1: The observer is unaware of the difference.
1 <ΔE <2: An expert observer can identify the difference.
2 <ΔE <3.5: Even a novice observer can detect the difference.
3.5 <ΔE <5: There is a distinct color difference.
5 <ΔE: the observer detects two distinct colours.

This is a more accurate colour-measuring technique than the human eye, which could fail to distinguish between the finest lines of colour difference. The testing process followed the EN 15886 (2010) standard for surface color measurement. The colorimeter/spectrophotometer provides high accuracy and is reliable for identifying colour characteristics in objects and powders (Mokrzycki & Tatol, 2011). The process involved scanning the powder samples with the colorimeter and activating the reading through a single click that produced an instant reading using two flashes. This experiment takes about 2-3 seconds, making it one of the most time-efficient among numerous heritage mortar analysis techniques.

3.5.3 Mercury intrusion porosimeter (MIP) - Physical analysis

There are currently no European standards for conducting porosimetric studies on historic mortars, according to Hauková *et al*. (2013). However, several researchers, such as Apostolopoulou *et al.* (2017), have conducted microstructural analysis using the RILEM recommendations and methods such as mercury intrusion porosimetry (MIP). This technique uses low and high pressure to drive mercury into the pores of the sample and measure the amount of mercury necessary to fill them. The technique is known to offer high accuracy for the sample properties. The MIP was only performed on four samples (PS7, PS9, SK1, SK5) due to the limitations in collecting historical materials. One sample per mortar type of about 1 cm^3 was oven-dried at 60 ºC for 24 h before being analysed.

3.5.4 Environmental Scanning electron microscopy (ESEM) – microtexture

The ESEM is equipped with energy-dispersive X-ray spectroscopy (EDS) to generate high-resolution mineral maps through electronic processing of the wave nature. The ESEM analysis procedure involved cleaning approximately 2 mm^3 of mortar fragments from dirt and ensuring their dryness. The samples were carbon coated, mounted on an aluminium stub, and placed on sample holders in ESEM chamber for analysis. The accelerating voltage of 10 kV was used for six samples with different magnification ranges. After scanning the samples under a microscope, the image spectra were analysed for different elemental crystals (from selected points of a sample) by means of peaks using the Bruker Quantax EDS Esprit microanalysis software.

3.5.5 X-ray fluorescence (XRF) – Chemical analysis

The mortar quantitative chemical composition (major and minor element compounds) was determined by X-ray fluorescence (XRF). About 5 mg of finely crushed mortar samples was used during the analysis. However, due to limitations to heritage sample sizes, only 16 representative mortars were analysed by XRF.

3.5.6 Powder X-ray diffraction (PXRD) – Mineralogical analysis

This method is used to identify the crystalline phases of materials. The following analysis conditions were set for this analysis: 45 kV voltage, 40 mA current intensity, 3 to 70° 2θ investigated area, and 0.01 2θ/s goniometer speed. The analysis included nine samples from the Castle of Good Hope, twelve from the pre-primary school, and three from the maximum-security prison on Robben Island. Firstly, the materials were finely grounded using an agate mortar and pestle. Thereafter, the powder samples were sieved using a 0.063 mm sieve in order to separate a binder rich material from the aggregates, then both the bider and aggregate rich components were tested seperately. It is worth noting that the details of the original materials' geology are necessary for guidance in the PXRD data analysis. Different mineral phases were identified on the samples using X'Pert HighScore Plus 3.0 (Malvern Panalytical) software.

3.5.7 TGA and DSC – Mineralogical analysis

The carbonation degree, calcium hydroxide, calcium carbonate and carbon dioxide of the mortar binder fraction were determined by means of thermogravimetry and differential scanning calorimetry (TGA-DSC). Thermogravimetric analysis (TGA) is a technique commonly used to analyse cement hydration based on the decomposition temperatures of cement constituents and hydration products. The TGA-DSC complemented the powder X-ray diffraction (described in section 3.5.6), with the combination of the results from the two tests providing details on the type of binder, the aggregate and the additives. For TGA/DSC experiment, 50 mg samples per mortars were heated at 5°/min rate over a 25-950 °C interval. The data analysis followed the UNE-EN ISO 11358-1 (2015) standard. This type of analysis determines the thermal decompositions of brucite at $300<T<400$ ºC (Arizzi*,* 2012), portlandite at $400<T<600$ ºC and calcite at $600<T<800$º C (Shivakumar *et al.*, 2022). These intervals are used to estimate the percentage carbonation degree index (ICD) using Equation 3.8 (Arizzi *et al.*, 2012):

$$\mathrm{I_{CD}} = \frac{(\mathrm{P_0} + \mathrm{B_0}) - (\mathrm{P_x} + \mathrm{B_x})}{(\mathrm{P_0} + \mathrm{B_0})} \times 100 \tag{3.8}$$

Where:

P_0 and B_0 are the initial portlandite and brucite content at time zero as determined by TGA/DSC

P_x and B_x are the amounts of portlandite and brucite at a chosen time x as determined by TGA/DSC

3.6 Phase 2A: Design, development and testing of new mortars

To fulfil the second objective of this study which focuses on developing new compatible restoration mortars, the mix design procedure relied on the literature and results obtained from the properties of the original mortars. This is a different technique than the normal concrete design used globally and in South Africa, following the concrete and cement institute (C & CI) procedures as well as the SANS. However, as discussed in Chapter 2, the complexity associated with the design and development of heritage mortars remains a challenge, especially regarding guidelines; hence, individual literature is considered the main guiding tool. This could threaten the integrity of the methods followed since the choice for following a particular method over another is subjective. Due to limited documented procedures in the literature associated with the design of historical mortars, the design approach is mainly based on functional performance (Papayianni *et al.,* 2000) and on the composition of the original mortars (Groot *et al.,* 2010).

3.6.1 Selected raw materials

The choice of raw materials used in this study depended on the availability of materials similar to the original materials in the study area, the cost, and the compatibility with the original materials. Where the duplicate materials were unavailable for a number of factors such as discontinued use or production, alternatives yet very close to the original in terms of properties and benefits were opted for. The proposed restoration mortars were prepared at the Cape Peninsula University of Technology concrete laboratory using lime, sand, calcium carbonate material (chemically pure), seashells, gypsum skimmer and potable water.

Binder – Pressure hydrated lime

The original mortar characterisation revealed lime as the predominant binder used for mortars of both case study structures. It was also observed that the lime used in Robben Island was quarried from the island itself, while the lime used at the Castle was produced by burning the seashells from Robben Island (https://www.castleofgoodhope.co.za/index.php, 2023). Due to the absence of hydraulic lime around the study area, a type A2P (SABS 523) building and plastering dolomitic hydrated lime (air-hardening) produced by a nearby lime company was used in this study (See Figure A1, Appendix A). The lime was packaged in 25 kg bags. The South African Bureau of Standards (SABS) approved the binder and conformed to SANS 523. According to Ngoma (2009), hydrated lime-based mortars are more flexible than the Portland cement-based mortars which are strong and hard, thus, the former resist cracks better. Hydrated lime also increases mortar workability, consistency, and waterproofness.

Aggregates – sand

Sand significantly affects masonry mortars' volume stability, durability and performance in the masonry unit. Therefore, the sand utilised in this investigation was chosen with care based on the role played by sand in mortar performance and the cost of acquiring materials on the Island and the Castle. Relatively coarse-grained aggregates, with an average fineness modulus of 1.64 were used in this study. Philippi building and plaster dune sand with the particle size distribution depicted in Figure 3.5 were opted for. The particle size distribution on three sand samples A, B and C (for ensuring accuracy) was determined in accordance with SANS 3001-AG1(2014) whereby a stack of sieves in accordance with their sizes was used to measure the material retained on each sieve. The grain size ranging between 0 and 4 mm is considered the most favourable to use because, as indicated by Shivakumar *et al*. (2022), this fraction produces mortars of the highest compressive strength (3.6 MPa), while the bigger grains reduce the mortar strength.

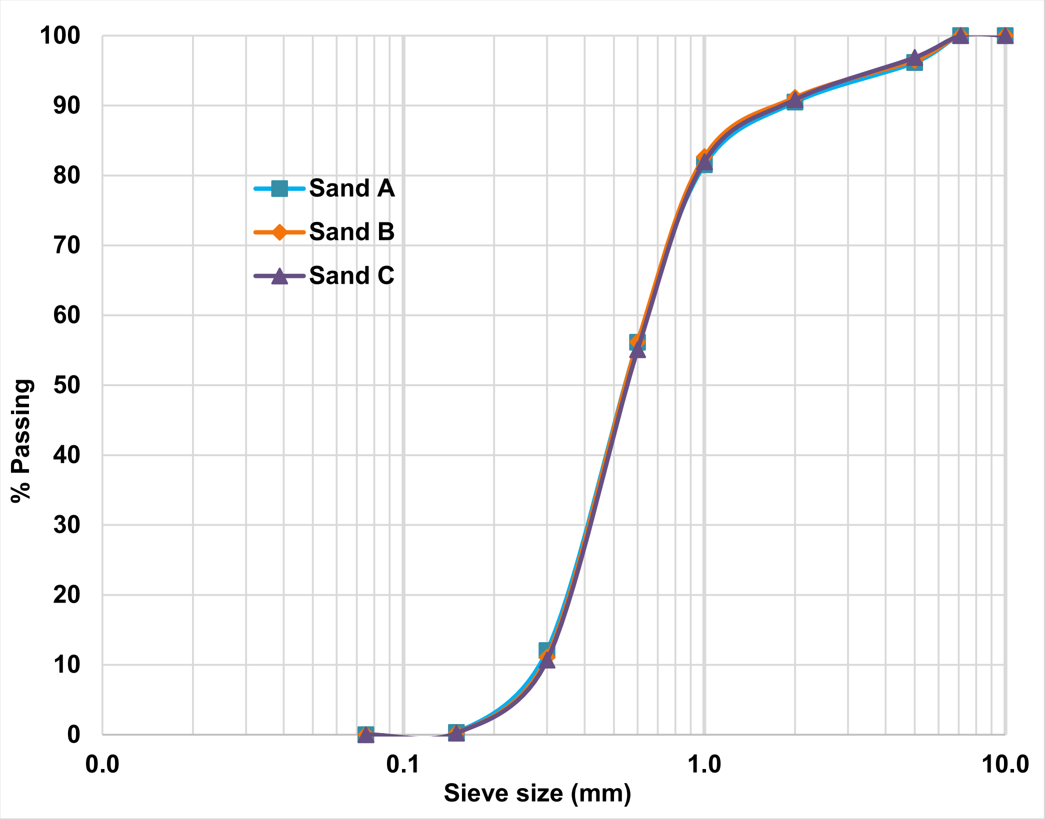


Figure 3.5 Gradation curves for 3 samples Sand A, Sand B and Sand C of Philippi building and plaster dune sand according to SANS 3001-AG1 (2014)

The cost is the main factor in restoring any historic or modern structure. This being the case, the cost of acquiring restoration materials should be cautiously evaluated and minimised as much as possible. With the main focus on cost control, an option to use the commercial Philippi sand was not much favoured or rather studied with great scepticism as opposed to applying the readily available sea sand, more accurately for buildings on Robben Island. The latter option would certainly reduce the restoration cost of purchasing and transporting the sand to the Island. Hence, such factors played a role in selecting the raw materials applied in this research. Also, to duplicate the original materials as identified during the first stage of this project, coarse sea sand (grading depicted in Figure 3.6) with fineness modulus (FM) of 3.84 obtained from Sea Point beach, on the Atlantic Ocean in Cape Town was opted for this study. Hence, this sand was assessed for its effectiveness in producing repair mortars on the island and Castle. The sand contained fine to coarse seashell fragments. The particle size distribution for three samples, S1, S2 and S3, was determined for accuracy.

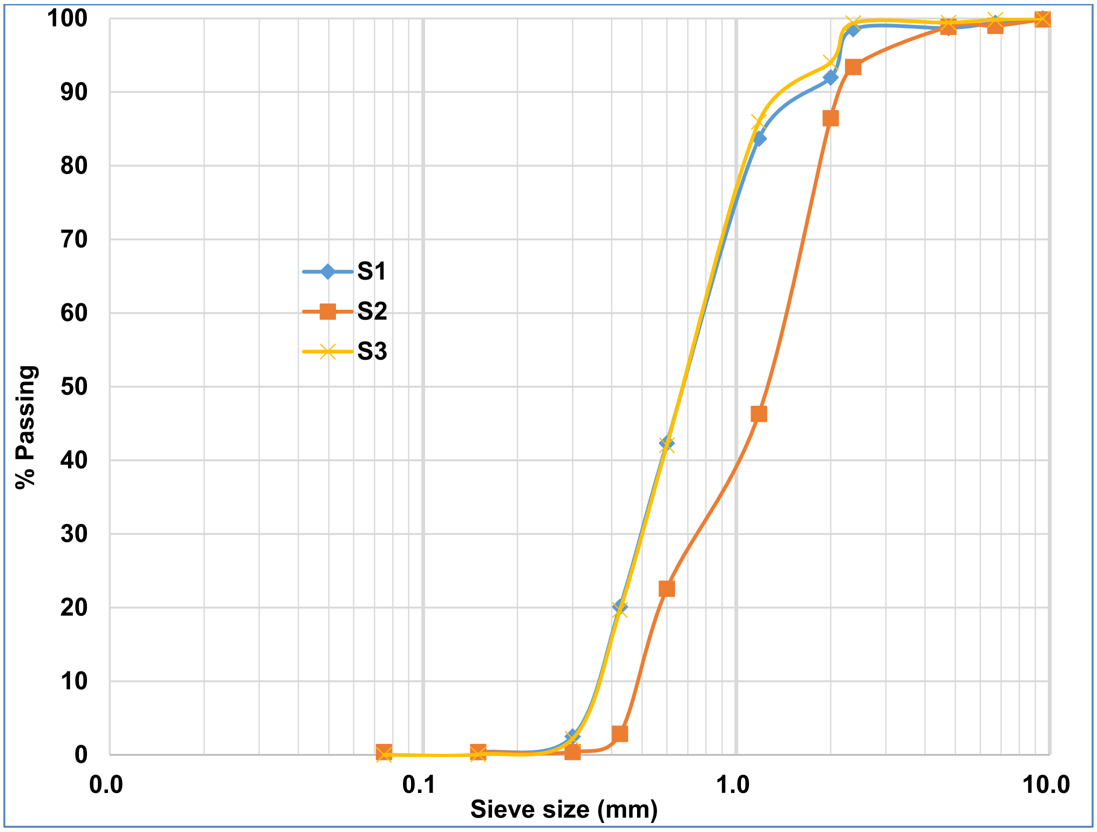


Figure 3.6 Gradation curves for sea sand with S1, S2 and S3 samples tested for accuracy according to SANS 3001-AG1 (2014)

The practice of using sea sand as aggregates in mortars was present since Roman times and is supported by Sun *et al*. (2020); He *et al*. (2022); Pineda *et al*., (2022). However, as noted by Mack (1998); Ngoma (2009), cations such as sodium and potassium and the excess chlorides are a major concern when using marine aggregates. These authors suggest that in the production of mortar, it is necessary to use aggregates that are free from impurities, especially salt, in order to avoid salt crystallisation in the masonry and corrorion of reinforcement in concrete structures. In this study, to minimize the impact of these elements present in sea sand, the aggregates were soaked in potable water (believed to be free from impurities) for 48 hours and thoroughly washed prior to mixing. But, for comparison purposes between the original and washed sea sand in terms of durability, the two were used and assessed for their durability in heritage restoration.

Additives – Calcium carbonate, seashells and gypsum

Shell fragments were present in the original mortars. This was correlated with the sand originating from the sea. Ngoma (2009) also associates the presence of shells in historic mortars to the lime production, which, in the case of Robben Island, such possibility was discarded due to the lime originating from the lime quarry on the island. Calcium carbonate with FM of 1.48 (see Figure 3.7) was added where Philippi sand (commercial product), free of shell fragments, was used. This was used to analyse the chemical impact of shells or calcium carbonate (replaing sea shells) in mortar properties. Since some original mortars contained traces of gypsum identified by PXRD, a gypsum skimmer purchased from a local supplier was also incorporated into the mix for some mortars.

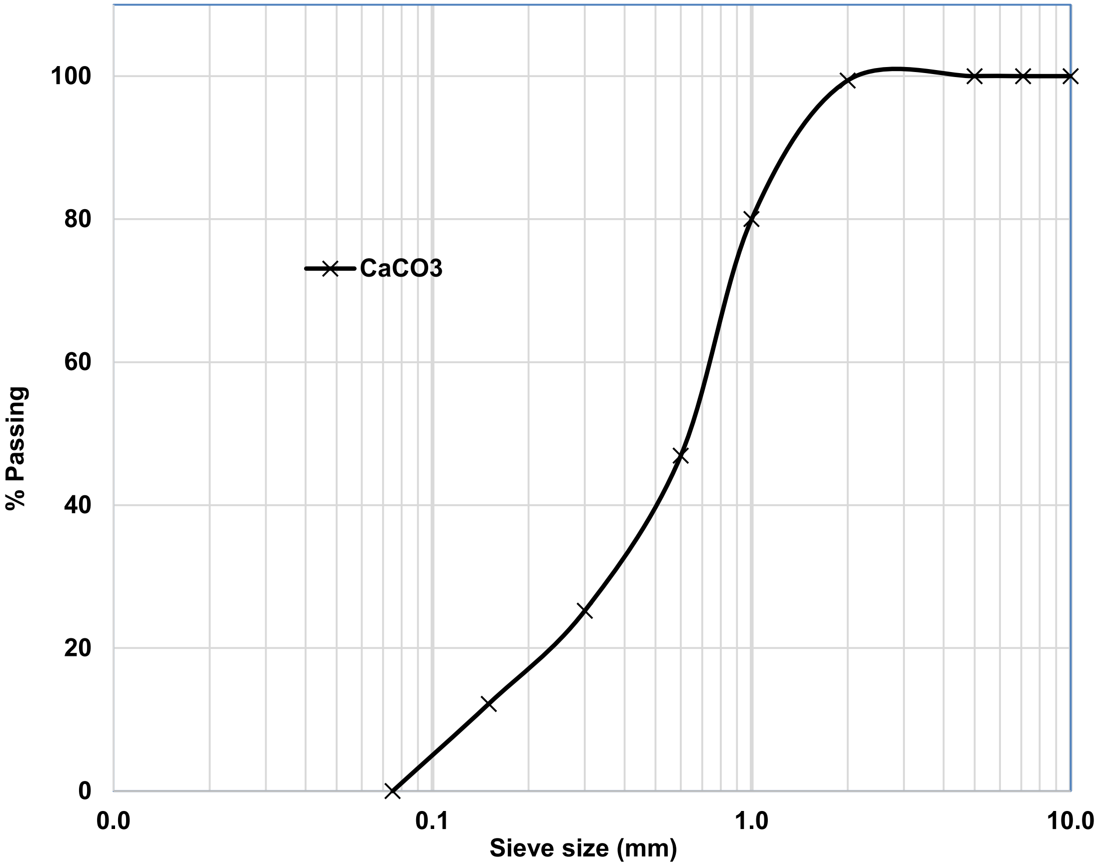


Figure 3.7 Particle size distribution for commercially sourced calcium carbonate compound

Clean water

This study used potable water from the tap to mix the solid components to form a paste. The 1:3 mortars required an average 0.73 water to binder ratio while the 1:1 mortar required a higher ratio of 0.95, to achieve optimal consistency on mortars.

3.6.2 Aggregate densities

The bulk densities of both Philippi and sea sand were determined through SANS 3001-GR6, Part 6 standard, as shown in Figure 3.8. The method involved filling the bulk density container with 3 layers of sand, giving each layer 25 blows. The mass difference between the container with sand and an empty container is then recorded. The compacted bulk density for Philippi sand was 1750 kg/m^3, while sea sand had a density of 1610 kg/m^3 and hydrated lime had a bulk density of 0.55 g/cm^3 (see Appendix A).

Figure 3.8 Determining the densities of aggregates using SANS 3001-GR6, Part 6

3.6.3 Mixing procedure

The mixing and production processes are important steps in mortars used for restoration purposes (Arizzi, 2012). For this study, after weighing the required material quantities, the ingredients were hand-mixed in a stainless-steel mixing bowl and an automatic mortar mixer, as indicated in Figure 3.9. The study opted for pre-mixing the dry components before adding water and kneading/mixing to form a paste. The most cited ratio for lime to sand 1:3 by weight, was used for 90% of the mixes in this study with different aggregate and additive quantities. The amount of added water to 1200 g of dry mortar components ranged between 220 – 320 ml in order to obtain the desired workability.

Figure 3.9 Raw materials (from left to right): hydrated lime, sea sand, seashells and calcium carbonate and mixing procedure for new mortars using both machine mixing and hand mixing

3.6.4 Mixing proportions

The study opted for a mixing ratio lime to sand of 1:3 by weight based on the results obtained for the existing mortar analysis and the existing literature. However, for mortar mix number 9, the ratio was 1:1 based on PXRD results (presented in Table 4.1 and Table 4.3) of the corresponding original mortar (SK6). The following mortars were then prepared whereby optimum mortars for the two case studies were selected from the list:

Mix 1 (M1) - 1:3 hydrated lime, Philippi commercial sand

Mix 2 (M2) - 1:3 hydrated lime, washed Sea point sea sand, 5% (of overall mix) finely (0.5-5 mm) crushed seashells

Mix 3 (M3) - 1:3 hydrated lime, Philippi commercial sand, 10% (of overall mix) commercial calcium carbonate

Mix 4 (M4) - 1:3 hydrated lime, Sea point sea sand, 5% (of overall mix) finely (0.5-5 mm) crushed seashells

Mix 6 (M6) - 1:3 hydrated lime, Sea point sea sand, 5% (of overall mix) medium (5-15 mm) crushed seashells

Mix 7 (M7) - 1:3 hydrated lime, Sea point sea sand, 5% (of overall mix) finely (0.5-5 mm) crushed seashells, 3% gypsum

Mix 8 (M8) - 1:3 hydrated lime, Sea point sea sand, 30% (of overall mix) finely (0.5-5 mm) crushed seashells

Mix 9 (M9) - 1:1 hydrated lime, Sea point sea sand, 5% (of overall mix) medium (5-15 mm) crushed seashells

The above mortars were designed to correspond with the original mortars listed in Table 3.2. It is to be noted that no mortars were produced for the earth and cement mortars.

Table 3.3 Summary of original mortars and proposed mortars derived from the characterisation of original materials using optimised basic characterisation and the advanced test.

Original Sample ID	Category	Original Mortar type	New proposed mix
SK1	Plaster	Lime	M7
SK2	Plaster	Lime	M7
SK3	Plaster	Lime	M7
SK4	Floor	Lime	M2/4
SK5	Bedding /joints	Lime	M6
SK6	Bedding /joints	Lime	M9
SK7	Bedding /joints	Earth	-
SK8	Bedding /joints	Earth	-
SK9	Bedding /joints	Earth	-
MX1	Raised pointing	Cement	-
MX2	Joints/bedding	Lime	M7
MX3	Joints/bedding	Lime	M7
PS1	Rendering	Cement	-
PS2	Rendering	Cement	-
PS3	Rendering	Cement	-
PS4	Plaster	Lime	M2
PS5	Rendering	Cement	-
PS6	Plaster on steps	Lime	M2
PS7	Plaster	Cement	-
PS8	Floor	Cement	-
PS9	Plaster on steps	Lime	M2/4
PS10	Rendering	Lime	M6
PS11	Rendering	Lime	M8
PS12	Floor	Cement	-

3.6.5 Mortar workability

The essential properties of fresh mortar were determined at the concrete laboratory of the CPUT under controlled temperature and humidity content to achieve optimum results. The workability of a mortar was determined by a flow table test specified in ASTM, C230/C230M-14 (2014), as shown in Figure 3.10. This test involves placing a cone mould with top and bottom diameters of

70 and 100 mm on the middle of the circular plate and filling it up with freshly mixed mortar. The mould was gently removed, and the mortar was given 25 drops. The drop impact led to mortar flowing on the circular plate. The flowability/workability of the mortars was then measured as a diameter of the flow.

Figure 3.10 Fresh mortar workability testing using a flow table test

3.6.6 Mortar casting and curing

After mixing and testing fresh mortar properties, cubes of 40 mm edge and rectangular prisms of 40 × 40 × 160 mm were cast in steel and custom-made moulds (see Figure 3.11). Before casting the mortars into the moulds, the inside surfaces were lightly greased with oil to aid easier following demoulding. The fresh mortar paste was cast to half-fill the mould times with a metal rod. Then, the mould was filled to capacity, tamped 15 more tampers, and levelled using a steel rule for a smooth, even surface. The samples were labelled, indicating the casting date and mix proportion. To obtain accurate results, 3 cubes and beams per test were prepared.

Figure 3.11 Mortar cubes preparation, casting, demoulding and curing procedure

For curing the cubes and beams, the curing conditions indicated by Cazalla *et al.* (2000) were adapted for the mortars that were produced. The samples were stored in a room with relative humidity of 60 ± 5% and temperature of 20 ± 5 °C, to allow evaporation of excess water, thus drying before removing them from the moulds after 7 days of curing. Strength tests were conducted at 28, 60 and 90 days, while hygric and durability tests were carried out after 60 ± 5 days of atmospheric curing.

3.7 Phase 2A: Compactness and mechanical properties

The compactness and strength of mortars are of great importance since they are interlinked with the mechanical performance of the entire building (Apostolopoulou *et al*., 2018). The compactness of new mortars was determined by means of ultrasound and mechanical tests (compressive and flexural strengths).

3.7.1 Ultrasonic pulse velocity (UPV)

Non-destructive/indirect testing technique, namely ultrasound pulse velocity (UPV) was used to measure the sample strength. The direct transmission method (Fusade & Viles, 2019) was used following the ASTM D2845 (2005) standard. Equation 3.9 was used to calculate the total anisotropy ($\Delta \mathrm{M}$) (Cultrone, 2022):

$$\Delta \mathrm{M} = \frac{\mathrm{V_{max}} - \mathrm{V_{min}}}{\mathrm{V_{max}}} \times 100 \tag{3.9}$$

Where:

V_{max} is the maximum velocity of the three tests measured irrespective of the measurement direction,

V_{min} is the minimum velocity of the three tests measured, irrespective of the measurement direction.

3.7.2 Flexural and compressive strength

In addition to non-destructive testing, the direct/destructive testing methodology was used for compression and flexural strength of the new mortars following modified SANS Method 5863: 2006 (Compressive strength test) and SANS Method 5864:2006 (Flexural strength). Instead of the standard sizes of 150 mm edge cubes and 100 × 100 × 600 mm prisms, analysis was carried out on 40 mm edge cubes and 40 × 40 × 160 mm prisms as suggested for heritage mortar analysis by Cristofaro *et al.* (2022). The centre-point loading test was used to determine the flexural strength, which was later calculated using Equation 3.10 (Válek & Veiga, 2005):

$$f = \frac{1.5Fl}{bd^2} \quad (3.10)$$

Where:
f: the flexural strength by centre loading test in MPa
F: the maximum failure load in N
l: the beam span (distance between the axes of the supporting rollers) in mm
b: the beam width in mm
d: the beam height measured in mm

The casting and curing conditions were as described in Section 3.6.6, with 6 cubes and 3 prisms per test per curing time. The mortar strength variation over time was assessed after 28, 60, and 90 days of atmospheric curing.

3.8 Phase 2B: Compatibility and durability testing of new mortars

To ensure the compatibility and ultimate performance of a heritage restoration mortar, a thorough characteristics investigation of all mortar properties, from aesthetics to chemical composition, must first be carried out. A mortar is then selected based on its capacity to meet both compatibility and performance requirements. This section covers the compatibility requirements and durability assessment carried out on the new mortars.

3.8.1 Compatibility

In addition to the fresh and hardened mortar properties described above, compatibility and comparison tests such as colorimetry and X-ray diffraction were conducted using the same procedure used for the original mortar samples. It should be noted that for the produced mortars, the PXRD analysis was carried out on two parts of each sample (the inner and outer layer that is more exposed to carbon dioxide) to assess their carbonation degree.

3.8.2 Hygric properties/pore system

Water plays a significant role in the deterioration of construction materials; thus, evaluating the absorption and drying kinetics of the mortars is fundamental for a better understanding of their durability (Beck *et al.,* 2003). Three cubes (40 mm sides) per new mortar mix were cured for 60 ± 5 days after fully hardening to avoid premature crumble during the test cycles. The cubes were tested to determine free and forced water absorption (A_b and A_f, respectively) and drying following RILEM Recommendations (1980), UNE-EN 13755 (2008) and NORMAL 29/88 (1998) standards. The procedure for this test involved oven-drying three cubes per mortar type at 100 °C, 24 hours before the immersion of the sample in deionized water (Figure 3.12A). A balance with an accuracy of 0.01 g (Figure 3.12B) was used to monitor the mass change after each cycle of water immersion, carefully wiping the samples to remove surface water until a constant mass was obtained over a 24-hour interval. The saturated mass under a vacuum was determined, as demonstrated in Figure 3.12C. The hydrostatic mass of the samples was also determined by hanging the specimen with a wire hook from the balance's plate into a container of water (hydrostatic weighing). After that, the samples were dried under controlled room temperature.

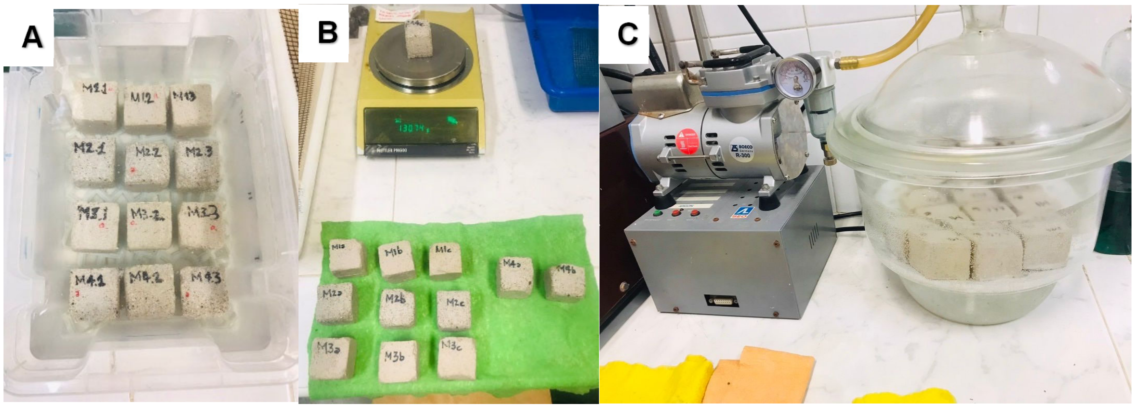


Figure 3.12 Free water absorption (A) drying (B) and mortar cubes forced (under vacuum) water absorption procedure (C)

The free water absorption (A_b), forced water absorption (A_f), pore interconnection degree (A_x), drying index (D_i), saturation coefficient (S), open porosity (P_o), and the apparent (ρ_a) and real densities (ρ_r) were determined using the Equations 3.11 to 3.16 after measuring the changes in the mass of mortar samples over a specific time owing to water penetration into the samples.

$$A_b = \frac{M_L - M_0}{M_0} \text{ x } 100 \qquad (3.11)$$

$$Di = \frac{\int_{t_0}^{t_f} f(M_t)dt}{M_s \, t_f} \qquad (3.12)$$

$$\rho_r = \frac{M_o}{M_o - M_s} \text{ x } 100 \qquad (3.13)$$

$$\rho_a = \frac{M_o}{M_H - M_s} \text{ x } 100 \qquad (3.14)$$

$$P_o = \frac{M_s - M_o}{M_s - M_H} \text{ x } 100 \qquad (3.15)$$

$$S = \frac{M_{24h} - M_o}{M_s - M_o} \text{ x } 100 \qquad (3.166)$$

Where:

M_L is the mass of the sample saturated with water at atmospheric pressure measured in grams

M_o is the initial dry mass of the sample in grams

M_H: hydrostatic mass in grams

M_t represents a decreasing water weight content as a function of time

M_s is the saturated mass in grams

M_{24h} is the sample mass after 24 hr in water

t_f is the end time of the drying test

t_o is the initial time of the drying test

ρ_a: is the apparent density in g/cm^3

ρ_r: is the real density in g/cm^3

P_o: is the open porosity

3.8.3 Durability: Ageing tests

To assess the damage after filling the mortar pores and fissures with soluble salts and exposing them to extreme weather conditions, a set of 3 cubic samples with 40 mm edges (tested after 60 days of atmospheric curing) per mortar type were exposed to 15 cycles of the salt crystallisation test using a 14% w/w solution of decahydrate sodium sulfate ($Na_2SO_4 \times 10H_2O$) and following the UNE-EN 12370 (2020) standard. For the freeze-thaw test, mortars were exposed to 30 cycles of freezing conditions in a freezer following UNE-EN 12371 (2011) standard. Fragment loss, formation of cracks and weight variation were monitored and recorded during the experiments.

Salt crystallisation resistance

The test involved drying the samples at 105±5 °C in an oven until a constant mass was reached (Figure 3.13A). A total of 15 cycles with one cycle involving immersion in a solution (Figure 3.13B) for 2 hours at 20 °C, drying the sample in an oven at 100 °C for 20 hours, cooling period at 20 °C for 2 hours and weighing the samples to complete a cycle. The samples were then thoroughly washed at the end of the test after the 15th cycle to remove all the remaining salts from the pores and fissures and to determine the weight lost by the simples, as depicted in Figure 3.13C.

Figure 3.13 Samples during salt crystallisation cycles demonstrating A) Drying of samples at 100°C B) Immersion in deionised water and C) Change in weight and shape over the 15 cycles

Freeze-thaw test

According to Křivánková *et al*. (2019), lime mortars are susceptible to freeze-thaw cycles because lime has a large porosity and little mechanical resistance. As a result, the capacity of historic mortars to withstand frost action is critical to their endurance, particularly in frigid regions. Even though Cape Town rarely experiences extremely low temperatures, freeze-thaw cycles were determined, considering the fact that the temperature behaviours continue to change over time due to the impacts of global warming.

The material was subjected to freezing and thawing tests to determine its resistance to crack development and weight variations. The procedure for the test involves drying the samples in the oven for 48 hours at 60 °C, then placing the samples in water for 48 hours, as shown in Figure 3.14A. The saturated sample weight was then recorded before freezing the samples at -15 °C in a freezer for 8 hours to complete a cycle, as illustrated in Figure 3.14B. A total of 30 cycles of immersion in water for 16 hours and freezing for 8 hours were completed for the mortars, resulting in changes in sample mass, as seen in Figure 3.14C.

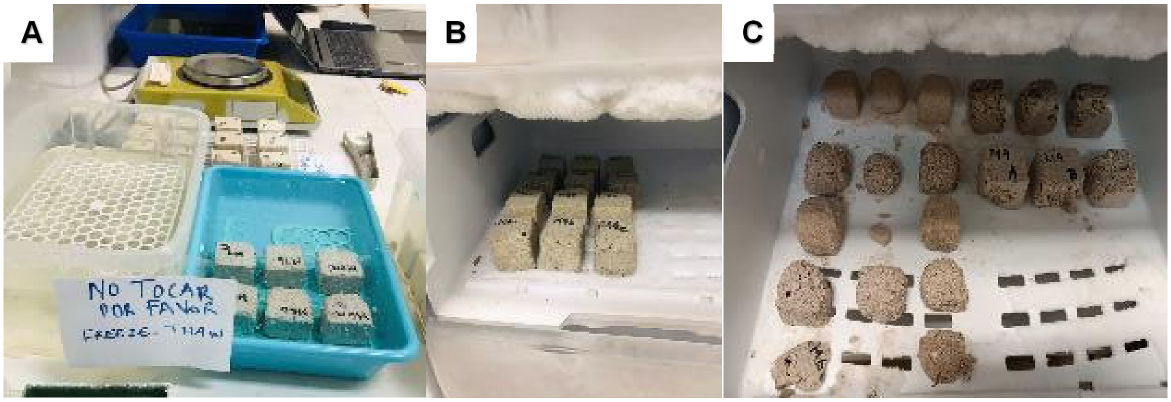


Figure 3.14 Freeze-thaw test for new mortars showing A) mortars immersed in water B) mortars subjected to freezing conditions C) mortars experiencing changes in mass over freeze-thawing process

3.9 Instrumentation and equipment used

To achieve the aim and objectives of this research, an experimental procedure was used to test the samples at the Cape Peninsula University of Technology laboratories and the Centre for Scientific Instrumentation at the University of Granada using the iequipment mentioned in this section. All the equipment and instruments met the calibration requirements for accuracy of the results.

Aesthetic properties

A portable spectrophotometer, which adheres to a CIE-developed system, was utilised to measure colour, as shown in Figure 3.15. A portable Konica Minolta CM-700d apparatus was used, and a CIE illuminant D65 that simulates daylight with a colour temperature of 6504 K was selected in the 400 – 700 nm wavelength range with the SpectraMagic NX Colour Data Software used for recording the measurements for original mortars and the newly produced mortars.

Figure 3.15 Spectrophotometer used to measure the colour of the mortar samples as well as the aggregates after mechanical separation

Physical properties

Figure 3.16 shows a Micrometrics AutoPore V 9600 porosimeter used to determine the pore size distribution, open porosity (%), pore surface area (m^2/g), and sample densities (bulk and skeleton) in (g/cm^3).

Figure 3.16 AutoPore V Series mercury porosimeter used for determining the mortar porosity

Microtexture

An environmental scanning electron microscope (ESEM) QEMSCAN 650F with the ability to provide high-resolution electronic images was used in this study for the microtextural analysis of mortars (see Figure 3.17).

Figure 3.17 The ESEM microscope used for identifying elements present in samples under magnification

Chemical properties

The Claisse LeNeo fusion instrument was used to prepare glass disks for XRF; after that, a PANalytical X-ray spectrometer Zetium Polymer Edition, as shown in Figure 3.18, was used to carry out the analysis.

Figure 3.18 The XRF Claisse LeNeo fusion and PANalytical Zetium compact spectrometer

Mineralogical properties

A Malvern Panalytical X'Pert PRO diffractometer (Figure 3.19) with an automatic loader was utilised for the current project,

Figure 3.19 X-ray diffractometer Malvern Panalytical X'Pert PRO

In addition to PXRD, weight loss due to heating a sample up to 950 °C was investigated using a Mettler-Toledo TGA/DSC-1 instrument, as indicated in Figure 3.20.

Figure 3.20 The TGA/DSC 1 HT/851 DIC-2021-2 instrumentation for analysing the thermal weight loss of binder content in mortars

Compactness

An ultrasonic pulse velocity tester (Controls 58-E4800) with 54 Hz frequency transducers was used on 3 cubic samples of 40 mm edge per mortar type to evaluate their compactness, as displayed in Figure 3.21.

Figure 3.21 The UPV tester used to test mortar compactness using transducers with 54 kHz frequency on three perpendicular sides of 40 mm^3 cube mortars

Mechanical properties

A digital compression universal testing (Foote test press auto) machine (See Figure 3.22A and 3.22B) at 50 kN/min loading speed was used to observe the compression failure for the mortars. The tests followed SANS Method 5863 (2006) with modifications aligning to UNE-EN 1926 (2007) standard. The flexural loading resistance was tested using a loading rate of 0.25 kN/sec loading rate on a concrete beam press machine as depicted in Figures 3.22C and 3.22D.

D

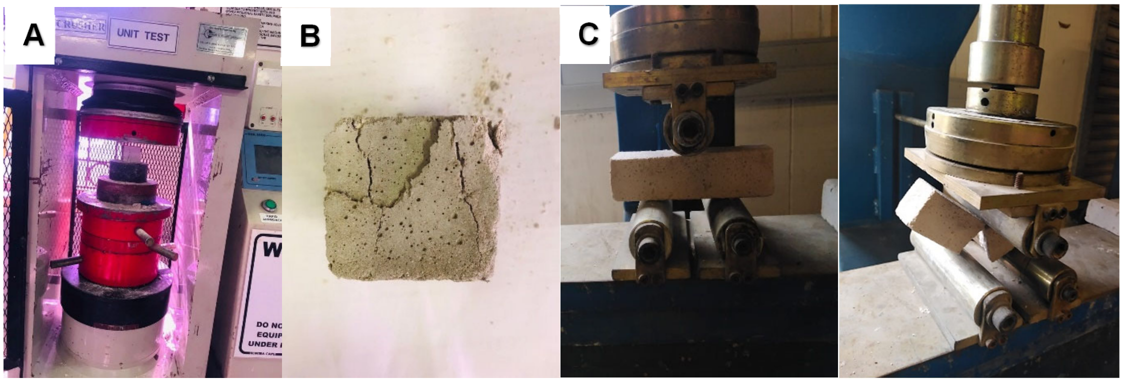


Figure 3.22 A) Mortar cube testing equipment B) Cube failure pattern C) Prism centre point loading and D) Prism flexural failure

3.10 Experimental limitations

There are some limitations to be pointed out from the analyses carried out in this research. Despite being a widely used technique for measuring mortar porosity, MIP analysis on historic mortar samples with weak cohesiveness can break the sample or cause micro-cracking during the analysis due to mercury injection into the pore network at rising pressures. This can lead to misleading porosity readings in the sample. Additionally, because of its destructive nature, the samples used in the MIP analysis cannot be reused for other analyses. MIP is also environmentally harmful due to mercury being used in this test; hence, it faces discontinuity (Arizzi & Cultrone, 2021).

In terms of mineralogical analysis, PXRD cannot differentiate between carbonatic minerals in aggregate and binder. For this reason, PXRD was conducted on mortar components (binder rich material and aggregates) after mechanical separation using a 0.063 mm sieve as suggested by Middendorf *et al.* (2005). Arizzi and Cultrone (2021) indicated the capability of PXRD to identify amorphous phases only present in excess (usually 3–5% of total sample weight). The authors suggested dissolution approaches for enriching calcium aluminates, calcium silicates, and acid-insoluble. According to Arizzi and Cultrone (2021), the Rietveld method helps reduce overlapping hydraulic phases such as C_2S and C_3S, clearly displaying the mineral phases (Arizzi & Cultrone, 2021).

The major limitations associated with a study of historic mortars' pore structure (hygric tests) are that they require large sample sizes (minimum 40 mm^3) and the timely duration of the test. As a result, hygric tests were impossible to carry out for original mortars due to sample limitations on historical structures; hence, the MIP technique was used instead. This technique also requires cohesive specimens over brittle ones to survive long exposure to water. Therefore, earth mortars could be challenging to analyse. The salt crystallisation and freeze-thaw tests similarly require relatively large quantities (4 cm^3) of samples and are time-consuming.

3.11 Summary

The use of contradictory materials for restoration of heritage structures is a common practice that requires immediate action. Without addressing this, the disparity in materials and the need for repeated repairs shall continue to prevail. The current research concentrated on producing suitable materials for the Castle of Good Hope and buildings on Robben Island using the RILEM, European, Spanish, and South African National Standards where applicable. The old and new materials were characterised using the techniques published in the literature that provide detailed and useful information for the production process of new materials. During this study, it was observed that designing mortars to be used in compatible repair of heritage structures requires systematic guidance for restoration practitioners, especially in developing countries where studies relating to the restoration of heritage buildings are very limited.

A clear understanding of the necessary steps and procedures to be undertaken and the possible enforcement of certain requirements before any restoration project is of paramount importance. Sufficient efforts in this regard would benefit the visual appearance of heritage structures and yield a positive restoration lifespan and, thus, economic sustainability. This chapter outlined procedures for characterising original mortars for their aesthetic, physical, mineralogical and chemical properties. It has also provided the procedures for designing new heritage mortars with details on relevant local and international standards to follow. To safely suggest a certain mortar mix over the other, all produced mortars were subjected to extreme environmental conditions such as freeze-thaw and salt crystallisation to assess their tolerance to such conditions.

Chapter 4 Results: Original and developed mortar characteristics

This chapter presents the characteristics of the original mortars and the proposed mortars for repairing the oldest masonry structures in Cape Town. The compatibility and durability comparison results for the existing mortars versus the recommended restoration materials are presented. The significant properties such as the aesthetic, physical, chemical and mineralogical were carefully selected and performed using colorimetry, MIP, ESEM, XRF, TGA/DSC and PXRD analyses. Additional fresh and hardened mortar properties such as workability, hygric, shrinkage, compressive and flexural strength and ageing tests were conducted in alignment with the European Standards, RILEM recommendations and the ASTM where applicable. The results represent only certain selected sections on the case studies for the academic research with limited scope. For the practical restoration works on these buildings, a detailed analysis will require a larger number of samples. This will give a wider representation of most of the masonry and, thus, detailed information to aid proper restoration.

4.1 Chemical properties

4.1.1 X-ray fluorescence (XRF)

The XRF results for mortars from both the Castle of Good Hope and the oldest buildings on Robben Island are shown in Table 4.2 with an indication of the loss of ignition (LOI), cementation (CI) and hydraulicity indices (HI). The HI and CI were calculated using Equations (4.1) and (4.2), derived from Boynton (1980) to interpret the results in terms of the binder type identification guide provided by Brosnan (2014) and are summarised in Table 4.1. The XRF analysis shows that samples SK1, SK3 and SK5 have the highest percentages of CaO (Table 4.2), suggesting that the binder used could be lime, confirming the visual assessment in Table 3.2.

$$\mathrm{CI} = \frac{(2.8 \text{ x } \%\mathrm{SiO_2} + 1.1 \text{ x } \%\mathrm{Al_2O_3} + 0.7 \text{ x } \%\mathrm{Fe_2O_3})}{(\%\mathrm{CaO} + 1.4 \text{ x } \%\mathrm{MgO})} \tag{4.1}$$

$$\mathrm{HI} = \frac{\mathrm{SiO_2} + \mathrm{Al_2O_3}}{\mathrm{CaO}} \tag{4.2}$$

Table 4.1 Cementation index (CI) classification guideline (extracted from Brosnan, 2014).

Binder description	CI	Active clay in the limestone
Pure or non-hydraulic (aerial) lime	CI<0.15	Very little clay
Sub-hydraulic lime	0.15 to 0.3	Very little clay
Slightly hydraulic limes	0.3 to 0.5	Around 8%
Moderately hydraulic limes	0.5 to 0.7	Around 15%
Eminently hydraulic limes	0.7 to 1.1	Around 25%
Natural cement	1.7	Up to 45%

Based on the CI values, SK1 and SK3 were prepared with a sub-hydraulic lime (0.15≤ CI ≤0.3) and very little clay content, while SK5 was prepared with a low hydraulic lime content (0.3≤ CI ≤0.5) and an active clay content of about 8%. The other three samples from the Castle, SK7, SK8 and SK9, were classified as earth mortars richer in silica and alumina, resulting in CI falling out of the range (beyond the maximum CI of 1.7 provided by Brosnan, 2014). These results prove the absence of hydraulic lime for producing these mortars. They further suggest a possible addition of cement (presence of CaO in lower quantities and higher alumina content), thus verifying that these samples were earth mortars with active clay content above 45%. The high hardening rate displayed by earth mortars SK7, SK8 and SK9, proven by high hydraulicity indices (HI), indicates the possible presence of a cement-based binder in these samples. Most mortars from the pre-primary school building on Robben Island (PS1, PS2, PS7 and PS11) revealed the presence of natural cement (1.1≤ CI ≤1.7) with PS9 and P10 having the CI range outside the criterio provided by Brosnan (2014), resulting to unclassified binder type. A hydraulic lime-based mortar (MX3) was used in the maximum prison building.

It is interesting to note the high CI value in SK2 even though this sample was visually classified as a lime-based mortar. Considering the other chemical compounds, SK8 and PS11 contained the largest concentration of Zr, which could suggest the use of dune sand deposits on the west coast (north of Cape Town) in which zircon crystals are present (Moumakwa, 2007 & Harlow, 2017). MnO and P_2O_5 appear in very low amounts (below 0.05 and 0.2%, respectively) in all the samples. As shown in Table 4.2, the higher LOI content clearly corresponds to the samples with higher CaO amounts due to the release of CO_2 during the calcination of samples.

Table 4.2 X-ray fluorescence results of major element oxides (in wt.%) except Zr (in ppm) used for computing the hydraulicity (HI) and cementation (CI) indices. LOI stands for loss on ignition and - for not detected.

Sample ID	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**MnO**	**MgO**	**CaO**	**Na_2O**	**K_2O**	**P_2O_5**	**Zr**	**LOI**	**TOTAL**	**HI**	**CI**	**Binder description, according to Brosnan (2014)**
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(ppm)	(%)	(%)			
SK1	2.94	0.47	0.19	-	0.27	53.32	0.63	0.17	0.14	65.4	41.58	99.76	0.06	0.17	Sub-hydraulic lime
SK2	25.30	5.89	2.09	0.05	6.08	27.60	2.21	1.58	0.26	280.7	27.9	99.27	1.13	2.18	Unclassified
SK3	2.61	0.42	0.18	-	0.18	53.03	0.69	0.17	0.13	59.4	42.31	99.77	0.06	0.15	Sub-hydraulic lime
SK5	6.85	1.44	0.58	0.01	0.20	48.87	0.55	0.53	0.10	73.6	40.59	99.82	0.17	0.43	Slightly hydraulic lime
SK7	47.27	12.89	3.96	0.02	0.68	11.93	2.85	1.66	0.17	499.0	16.98	99.09	5.04	11.59	Unclassified
SK8	60.47	14.67	7.70	0.04	0.84	1.51	1.69	1.75	0.11	878.4	10.2	99.76	49.76	71.05	Unclassified
SK9	60.12	17.47	5.60	0.02	0.72	3.30	0.40	2.05	0.14	357.8	8.84	99.55	23.51	44.45	Unclassified
MX1	26.90	3.94	2.42	0.03	2.22	37.93	0.40	0.51	1.01	119.7	23.54	99.13	0.81	1.98	Unclassified
MX3	15.97	2.13	1.35	0.02	9.23	32.03	2.35	0.61	0.93	77.6	34.47	99.17	0.57	1.07	Eminently hydraulic lime
PS1	19.83	3.32	1.94	0.03	1.04	40.07	0.42	0.44	0.33	56.6	31.38	99.27	0.58	1.46	Natural cement
PS2	21.65	3.49	2.12	0.03	1.09	39.22	0.53	0.47	0.55	60.7	29.83	99.21	0.64	1.62	Natural cement
PS5	30.84	3.23	1.92	0.03	1.26	33.98	0.68	0.45	0.56	94.3	26.17	99.34	1.00	2.55	Unclassified
PS7	20.87	3.77	2.02	0.03	0.97	39.55	0.44	0.56	0.46	62.5	30.49	99.41	0.62	1.56	Natural cement
PS9	25.35	3.57	2.59	0.04	1.00	35.86	0.38	0.35	0.35	67.3	29.48	99.24	0.81	2.06	Unclassified
PS10	23.64	3.77	2.05	0.03	1.01	36.86	0.63	0.53	1.49	92.8	29.09	99.34	0.74	1.88	Unclassified
PS11	19.82	3.49	2.13	0.04	0.96	39.11	0.43	0.46	0.79	113.8	32.10	99.57	0.60	1.50	Natural cement

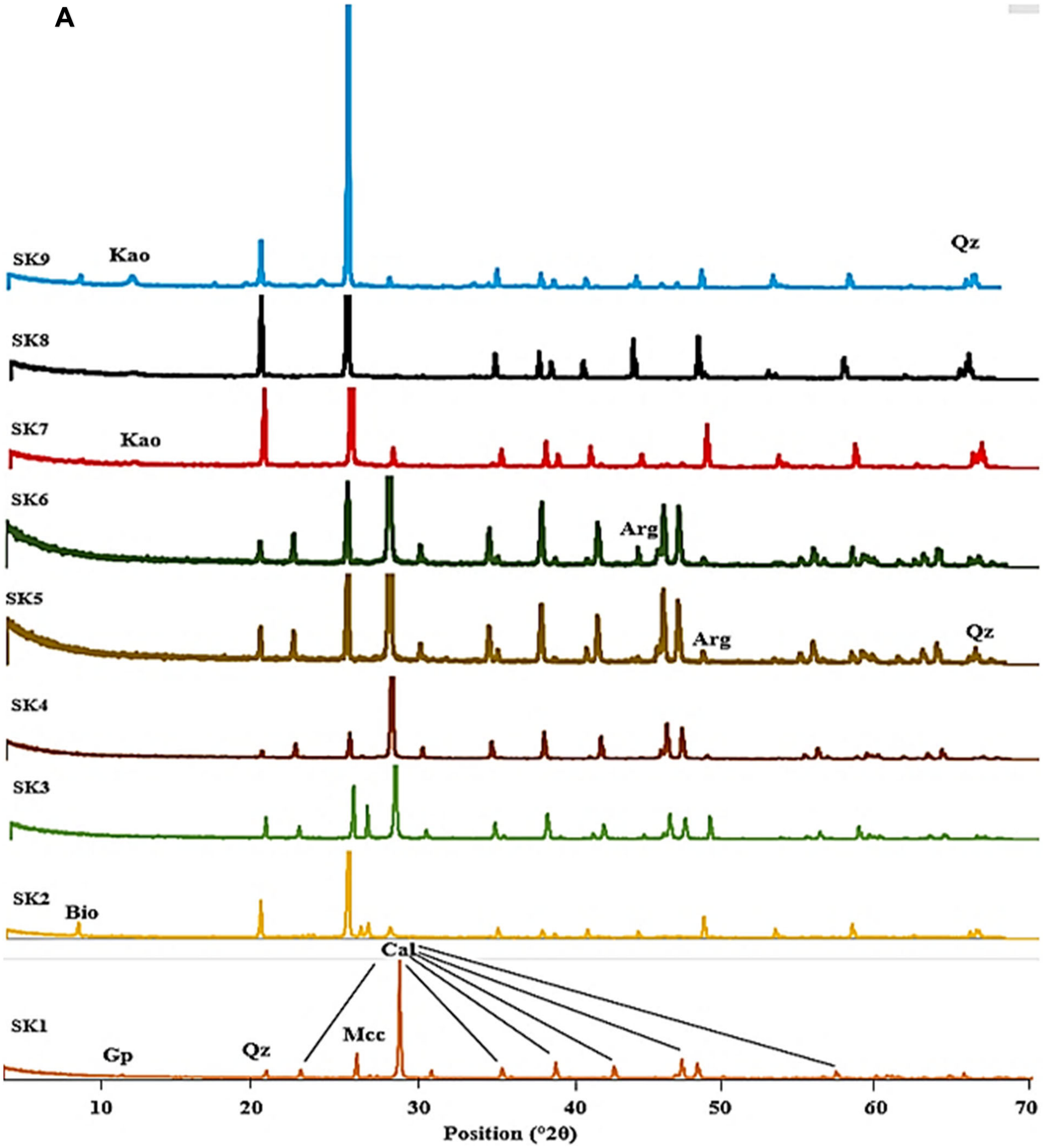
4.2 Mineralogical composition

4.2.1 Powder X-ray diffraction (PXRD)

The mineralogy of the samples depends on the geological history of the raw materials used in preparing such mortar. In this case, the mineralogy of the mortars is strongly influenced by the Geology of Cape Town, where most of the building materials are believed to come from. According to the geological history of the city provided by Compton (2004), the three main rock formations are the Stellenbosch batholiths, which were intruded into the Malmesbury group approximately 630 million years ago, the Kuilsriver-Helderberg, Cape Granite, which includes the massive Peninsula, and the late Precambrian Malmesbury group (sedimentary and metamorphic rock). About 450 million years ago, the Table Mountain group sandstones were deposited on the Malmesbury series basement and eroded granite surface. This provides an understanding of the minerals as observed by PXRD analysis.

The PXRD patterns of the samples from the Castle (SK1-SK6), Robben Island Maximum Security Prison building (MX1-MX3), and RI Pre-Primary School building precinct (PS1-12) showed the presence of calcite, aragonite, gypsum, quartz and feldspars (i.e., microcline and traces of orthoclase). Biotite was identified in one sample (SK2) (Figures 4.1A and 4.2 and Table 4.3). Calcite is common in all samples (the typical mineral in lime mortars) except in the three earth mortars (SK7, SK8 and SK9) and SK2, where this carbonate is scarce or in traces and quartz prevails.

Sample SK2 is rich in quartz but very poor in calcite (Figure 4.1B), and this composition does not match lime mortar. It is possible that in this case, white cement was used for this mortar elaboration and, therefore, depending on the first use of cement in the construction industry, the mortar in question was not an original mortar but an intervention for which there was no evidence of application date. Due to quartz crystals' high reflectance power and very low cementitious phases, it is very difficult to identify these last by PXRD. The earth mortars (SK7, SK8 and SK9) are rich in quartz and show low amounts of kaolinite. SK5 and SK6 contain considerable amounts of aragonite, which is clearly linked to the mineralogy of shells used in mortars' aggregate. Some traces of gypsum are detected in SK1, SK2 and SK3. This sulphate was probably added to aid in the fast setting of the mortars. Small peaks of hematite and microcline were other phases identified in these samples. Biotite was also detected in two of the three earth mortars (SK7 and SK8).


A
SK9
Kao
Qz
SK8
SK7
Kao
SK6
Arg
SK5
Arg
Qz
SK4
SK3
SK2
Bio
Cal
SK1
Gp
Qz
Mcc
10
20
30
40
50
60
70
Position (°2θ)

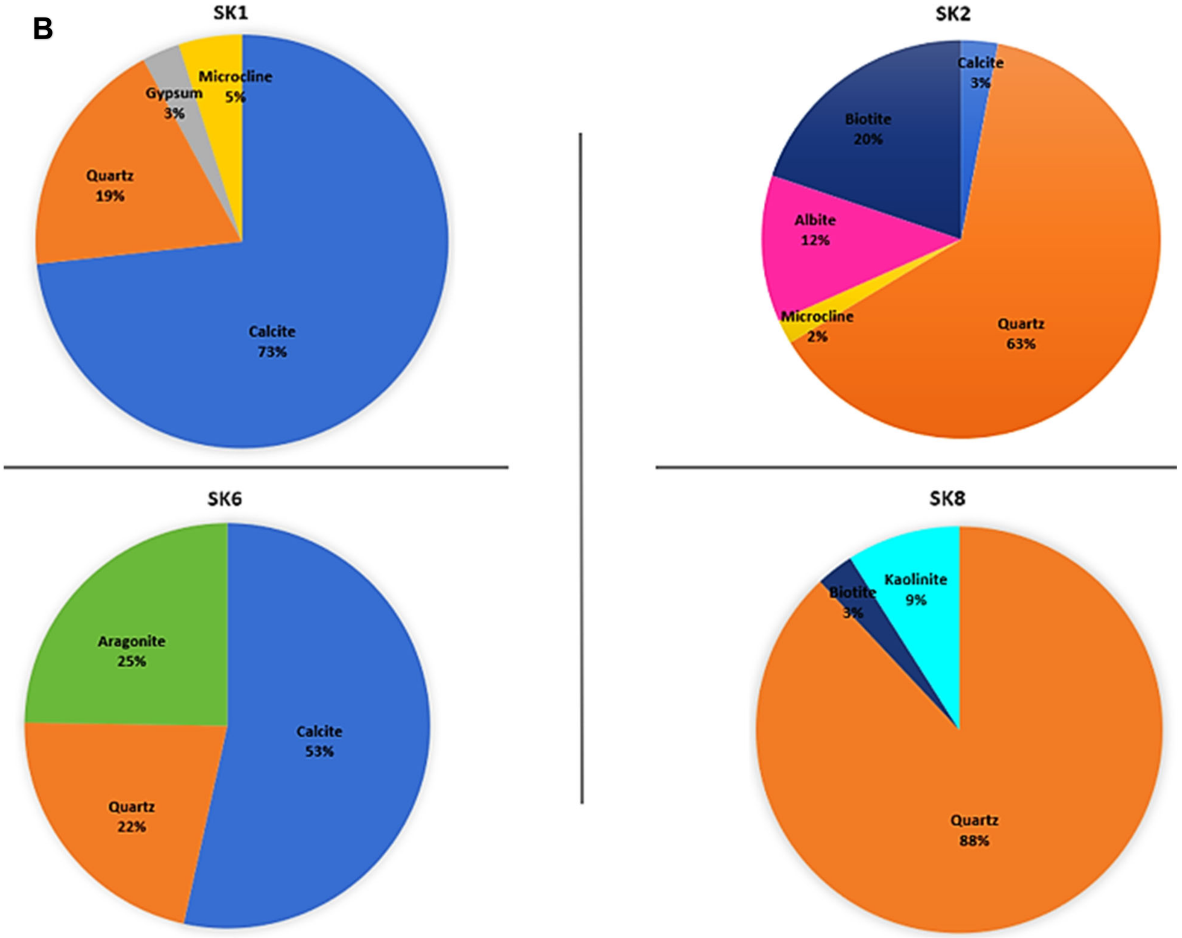


Figure 4.1 A) Powder X-ray diffraction (PXRD) patterns of the original mortars from the Castle of Good Hope. Legend: Arg - Aragonite, Bio - Biotite, Cal - Calcite, Gp - Gypsum, Kao – Kaolinite, Mcc - Microcline, Qz – Quartz B) PXRD semi-quantitative results showing the different mineral phases present in some original mortars from the Castle of Good Hope

According to Cape Town' geology (Compton, 2004), it is no surprise that the original mortar samples from these monuments are rich in phases related to outcropping rocks in the three main rock formations of the area, the late Precambrian Malmesbury group (sedimentary and metamorphic rock), the Cape granite comprising the huge Peninsula, Kuilsriver-Helderberg, and Stellenbosch batholiths (Loke *et al*., 2023).

Ettringite was identified in sample PS12, as displayed in Figure 4.2. Ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12}·26H_2O$) is typically formed due to the chemical reaction between the sulphates and aluminates usually present in the hydration products of cementitious materials, particularly in Portland cement. Its crystallisation involves a high mass increment, contributing to cracking and deterioration in mortar under certain environmental conditions. But mostly, this takes place during the first stages of hydration, before the hardening of the cement slurry. The presence of this phase in PS12 suggests using Portland cement for this mortar, either as an additional binder to lime or the sole binder. This implies that this particular mortar is cement-based, as determined by the visual assessment in Table 3.2. Even though samples MX1, PS1, PS2, PS3, PS5, PS7, PS8 and PS12 were visually assessed as cement mortars, their PXRD analysis did not display any ettringite phases.

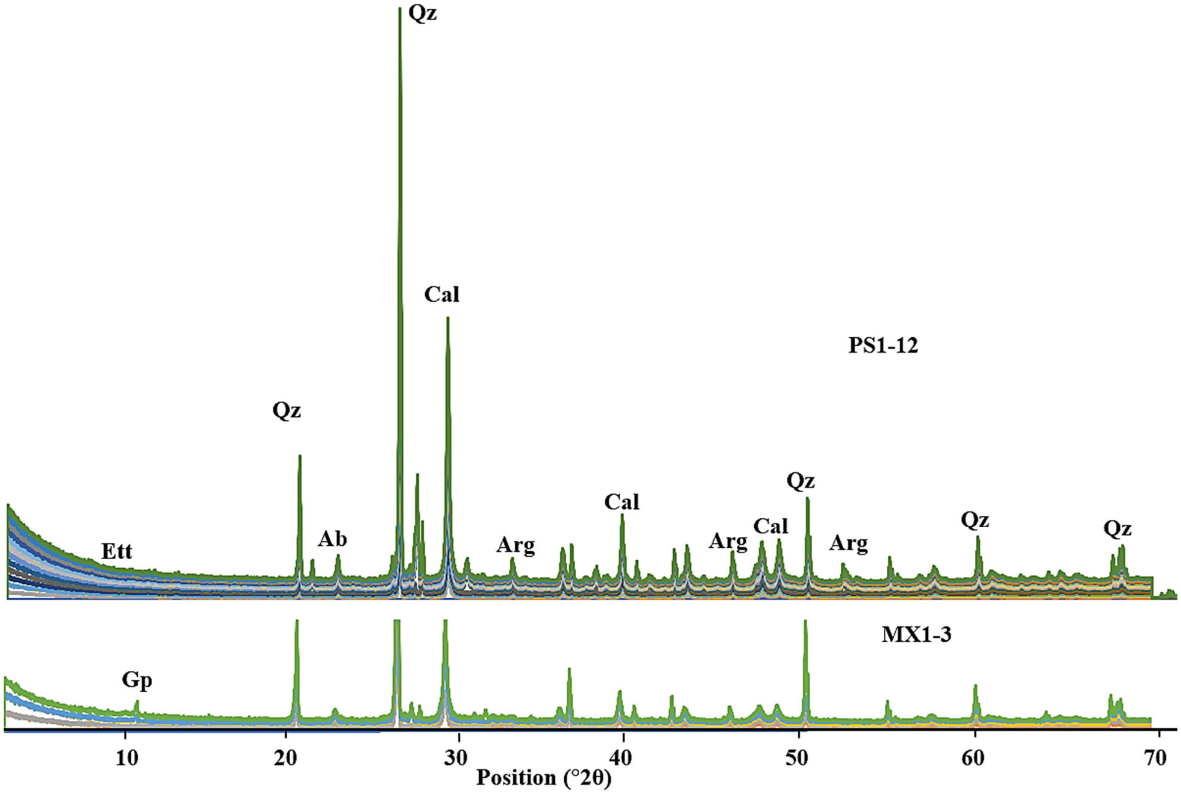


Figure 4.2 PXRD patterns of the original mortars from Robben Island buildings: Maximum security prison (MX1-3) and Pre-primary school (PS1-12). Legend: Ab – Albite, Arg - Aragonite, Bio - Biotite, Cal - Calcite, Ett – Ettringite, Gp - Gypsum, Kao – Kaolinite, Mcc - Microcline, Qz – Quartz

Figure 4.3 shows the PXRD patterns for the binder content (<0.063 mm), displaying ettringite phases for the cement mortars, confirming the results of the visual assessment in Table 3.2.

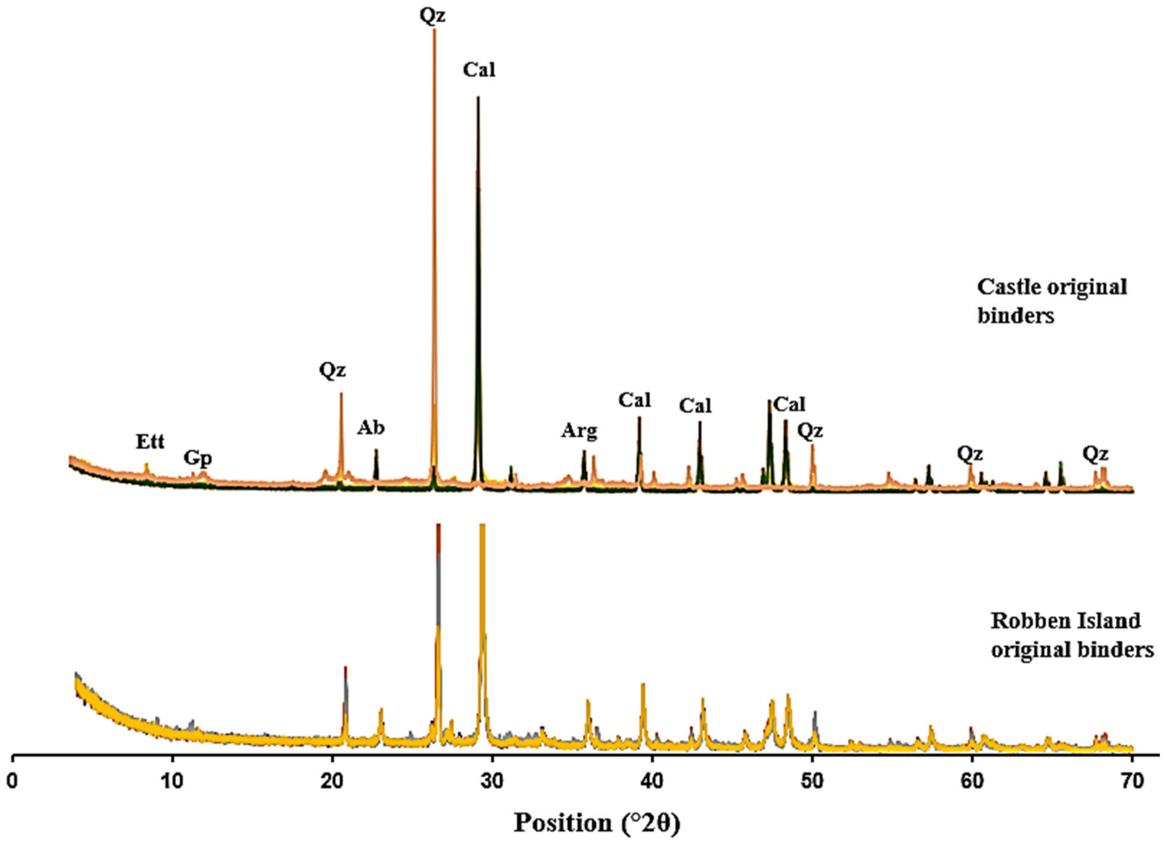


Figure 4.3 PXRD patterns of binder rich content samples from the Castle of Good Hope and Robben Island buildings. Legend: Ab – Albite, Arg – Aragonite, Cal - Calcite, Gp - Gypsum, Qz – Quartz

The new mortars proposed for the repair of respective sections on the Castle and Island buildings displayed minerals, as shown in Figure 4.4. The PXRD patterns for all 8 mortars were similar with common phases such as calcite, aragonite and portlandite since a similar binder type was used and only two types of sand were selected for the production of the samples. The carbonation degree did not have a noticeable effect on the mineral crystals of the samples, as it was observed that the inner part of the samples (less carbonated) had a mineral composition similar to that of the more carbonated outer area.

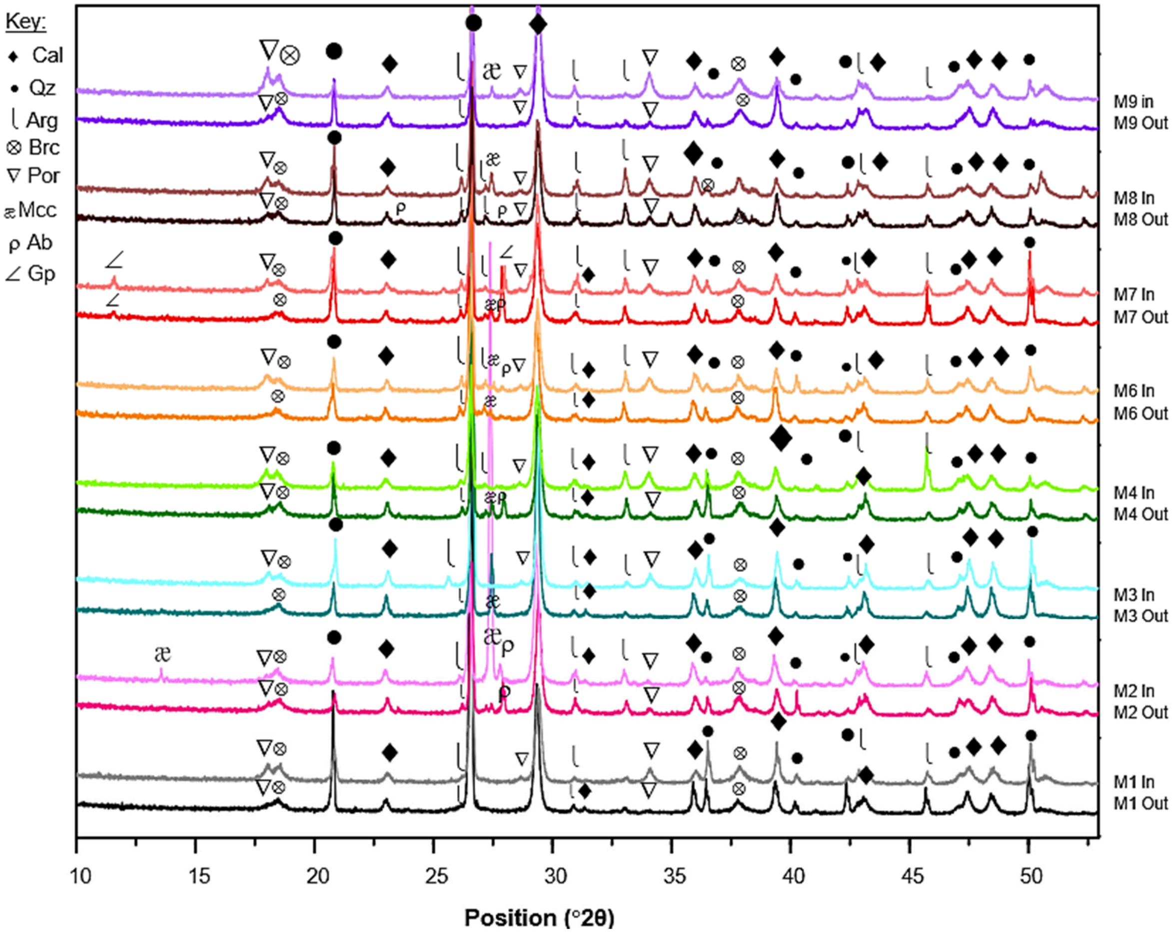


Figure 4.4 PXRD patterns of the new repair mortars. Legend: Ab – Albite, Arg – Aragonite, Brc - Brucite, Cal - Calcite, Gp - Gypsum, Mcc - Microcline, Por – Portlandite, Qz – Quartz

Qualitative mineralogical composition of the Castle, Island buildings and new mortars is provided in Table 4.3. The mineral phases were identified in the original mortars and on binder rich content obtained after sieving the mortar with a 0.063 mm sieve, revealing their amount as abundant, scarce, trace and absent. The repair mortars M1-M9 showed some traces of brucite and portlandite phases compared with the original mortars.

Table 4.3 Qualitative mineralogical composition by PXRD of the mortars showing: very abundant *; abundant **; scarce*; mineral in traces tr; absence of a mineral. Legend: Ab – Albite, Arg – Aragonite, Brc - Brucite, Cal - Calcite, Ett – Ettringite, Gp - Gypsum, Kao – Kaolinite, Mcc - Microcline, Por – Portlandite, Qz – Quartz.**

Sample ID	PXRD mineral phases									
	Qz	Cal	Bio	Mcc	Gp	Arg	Kao	Brc	Por	Ab	Ett
SK1	*	***	-	*	*	tr	-	-	-	-	-
SK2	***	*	**	tr	tr	tr	-	-	-	-	-
SK3	*	***	-	*	tr	tr	-	-	-	-	-
SK4	**	***	-	tr	tr	tr	-	-	-	-	-
SK5	**	***	-	tr	tr	*	-	-	-	-	-
SK6	**	***	-	-	tr	**	-	-	-	-	-
SK7	***	tr	tr	-	-	-	*	-	-	-	-
SK8	***	tr	tr	-	-	-	tr	-	-	-	-
SK9	***	tr	tr	-	-	-	*	-	-	tr	-
MX1	***	**	-	-	-	-	-	-	-	-	tr
MX2	***	**	-	*	tr	-	-	-	-	-	-
MX3	***	**	-	-	tr	-	-	-	-	*	-
PS1	**	***	-	-	-	*	-	-	-	*	tr
PS2	**	***	-	-	-	*	-	-	-	*	tr
PS3	**	***	-	-	-	*	-	-	-	*	tr
PS4	**	***	-	**	-	*	-	-	-	-	-
PS5	**	***	-	-	-	-	-	-	-	-	tr
PS6	***	**	-	-	-	*	-	-	-	-	-
PS7	**	***	-	*	-	*	-	-	-	-	-
PS8	***	**	-	-	-	*	-	-	-	-	tr
PS9	***	**	-	-	-	*	-	-	-	-	-
PS10	***	**	-	-	-	*	-	-	-	-	-
PS11	***	**	-	tr	-	*	-	-	-	-	-
PS12	***	**	-	-	-	-	-	-	-	-	tr
M1	***	**	-	-	-	*	-	tr	tr	-	-
M2	***	**	-	tr	-	*	-	-	tr	*	-
M3	***	**	-	-	-	*	-	tr	tr	-	-
M4	***	**	-	tr	-	*	-	-	tr	*	-
M6	***	**	-	-	-	*	-	-	tr	-	-
M7	***	**	-	*	*	*	-	tr	tr	*	-
M8	***	**	-	*	-	*	-	tr	tr	tr	-
M9	***	**	-	*	-	*	-	tr	tr	tr	-

4.2.2 Thermogravimetry analysis - Differential scanning calorimetry (TGA -DSC)

The thermograms for all the samples composing of lime from both the Castle and Island buildings had the same shape typical for lime mortars, as seen in Figure 4.5, with significant weight reduction after 700 °C. In more detail, the blue curve indicated as Batch 1 summarizes the behaviour of samples SK1, SK3, SK4, SK5, SK6, PS4, PS6, PS9, PS10 and PS11, whose trends are very similar. Batch 2 (in orange) represents sample SK2; Batch 3 consists of SK7 and SK9 samples and has a similar trend as representative SK7, shown by the grey curve; Batch 4 (in yellow) is composed again of one sample, SK8. Batch 1 is characterised by only one main inflexion that starts around 700 ºC and ends at 880 ºC due to the calcite decomposition (Arizzi & Cultrone, 2021).

Batch 2 differs significantly from Batch 1 even though SK2 was visually classified as lime mortar and has a similar thermogram to the earth mortars grouped in Batch 3. Batch 2 shows a first weight loss from 100 to 200 ºC due to the dehydration of gypsum detected by PXRD. Two more steps can be identified between 300 and 500 ºC and between 500 and 700 ºC due to the dehydroxylation of biotite and perhaps the presence of some portlandite, which suggests a fully carbonated mortar (Földvári, 2011). The main loss in Batch 2, between 700 and 800 ºC, is linked to calcite decomposition. Regarding Batch 3, there is no inflexion at 100-200 ºC as no gypsum was added to the earth mortars but a slow, steady descent up to 700 ºC due to phyllosilicate dehydroxylation. Again, between 700 and 800 ºC, the main loss is due to the presence of calcite in these mortars and its decomposition. Finally, Batch 4 is characterised by a small descent and is the mortar with the lowest weight loss (around 12%). According to the mineralogy detected by PXRD, the weight loss is attributable to the dehydroxylation of phyllosilicates in the mortar (see Table 4.2).

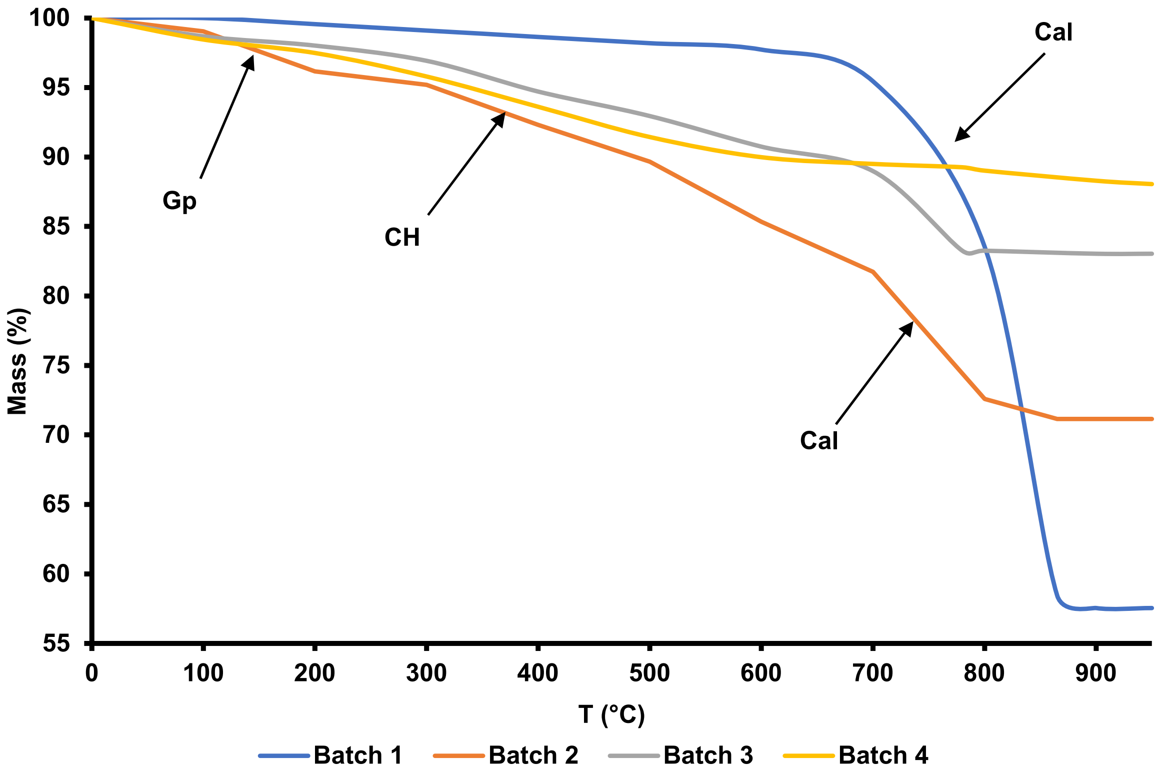


Figure 4.5 TGA curves for original Castle mortars show the weight loss over increasing heating temperature over a period of time. Legend: Batch 1 mortars represented by SK1 showed a similar pattern while Batch 2 represents sample SK2, Batch 3 represented by SK7 with a similar pattern, and Batch 4 represents SK8. Legend: Calcite: Cal, gypsum: Gp, portlandite: CH

For cement mortar samples PS1, PS2, PS3, PS5, PS7, PS8 and PS12, the TGA curves were similar to those of the mortars, as depicted in Figure 4.4 (Batch 1). A somewhat different material decomposition pattern is shown in Figure 4.6. In this cement-based sample (MX1), the continuous weight loss associated with the release of water from hydrated cement phases, such as calcium silicate hydrates (C-S-H), the dehydration of ettringite and perhaps of portlandite (even if PXRD did not detect this phase), and the decomposition of calcite can be seen in Figure 4.6. The detailed TGA-DSC results are attached in Appendix C.

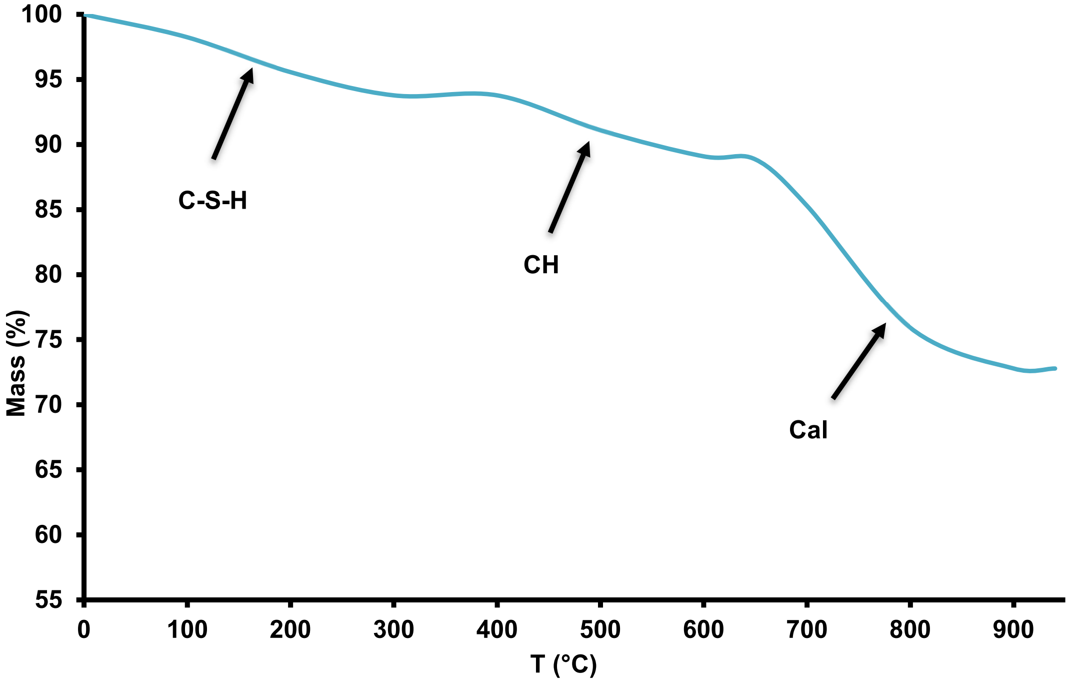


Figure 4.6 TGA curve for cement mortar PS1 showing the weight loss over increasing temperature over a period of time. Legend: Calcite: Cal, calcium silicate hydrates: C-S-H, portlandite: CH

4.3 Textural properties

4.3.1 Environmental scanning electron microscopy (ESEM) and Energy Dispersive X-ray Spectroscopy (EDS)

A better understanding of the mortar sample's composition was confirmed by assessing the high-resolution ESEM micrographs and EDS spectra for specific features that stood out during the analysis (Figure 4.7). The ESEM observations of SK1 revealed the presence of clustered flower-like morphologies (Oral & Ercan, 2018) with aggregated particles of approximately 5 µm in size in a porous matrix (Figure 4.6A). These crystals' EDS spectrum and scalenohedral morphology suggest they are calcite. Calcite is also observed with different morphology (i.e., tabular) and denser particle distribution (Figure 4.7B). Quartz grains scattered throughout the matrix have also been identified (Figure 4.7B). The presence of K, Al, and Si elements in SK5 suggests the presence of feldspar crystals (Figure 4.7C), possibly microcline, according to PXRD analysis. The presence of Ca in the same EDS spectrum is related to the lime binder, while Na and Cl are due to the sea spray and the precipitation of halite on the surface of any coastal buildings. A cubic crystal of halite is visible in the left margin of Figure 4.7B. Feldspars are usually prismatic and have marked cleavage (see black arrow, Figure 4.6D). Sporadic spherical contaminant particles have also been detected (Figure 4.7E). They are about 2 µm and are rich in Si (see EDS analysis). Sometimes, organic fibres (perhaps fungal hyphae or other types of roots) can be seen in the matrix of the original mortars (Figure 4.7F).

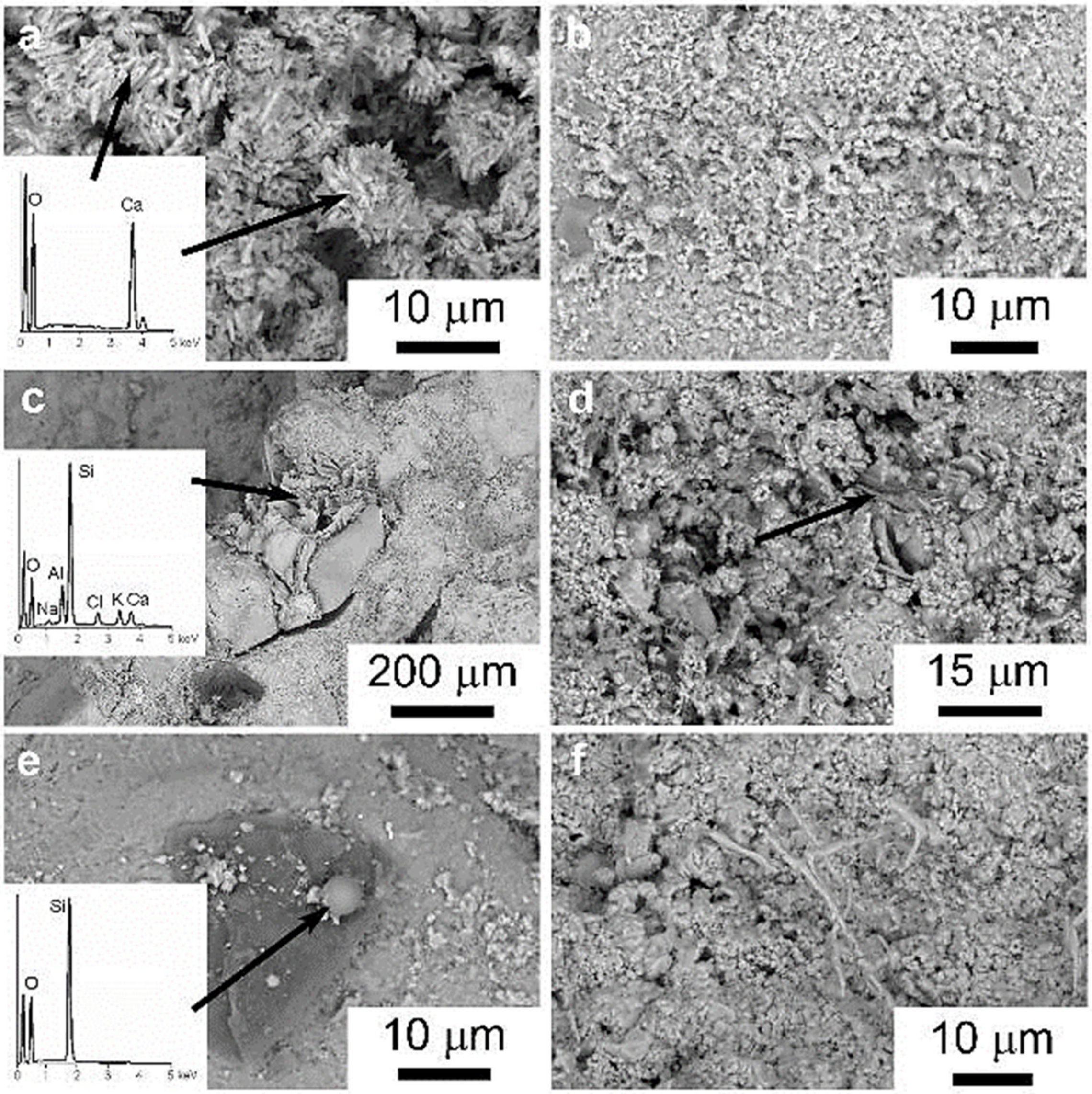


Figure 4.7 ESEM micrographs and EDS spectra for Castle samples SK1 (a-b) and SK 5 (c-f)

The ESEM observations for PS4 revealed similar sporadic spherical contaminant particles (see Figure 4.8A). Some diatom coral cells were also visible, with a possible presence of algae (Figure 4.8B). This suggests the mortar's exposure to constant water, which is considered harmful to masonry elements. Although PS9 PXRD analysis showed no ettringite peaks, ESEM observations highlighted some fibrous and platy morphology of ettringite particles as proven by the EDS spectrum, which indicates the presence of Al, Ca, S and O elements (Figure 4.8C). This suggests that this sample contained a cement component, which can be confirmed by the greyish colour that was observed visually (Table 3.2). A very porous structure was seen in the PS11 mortar (Figure 4.8D-E). The ESEM - EDS results for the 5 mortar samples are attached in Annex D.

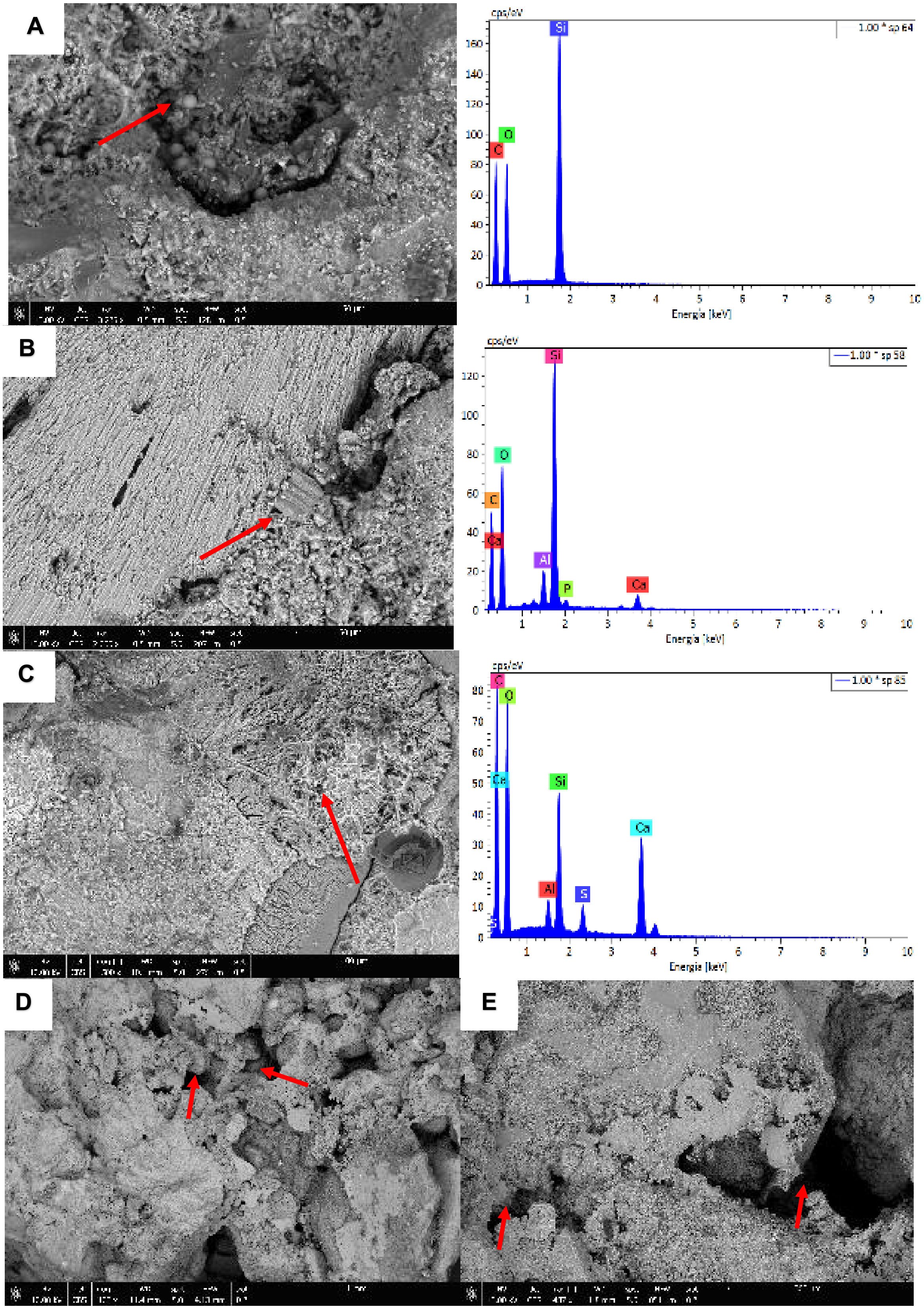


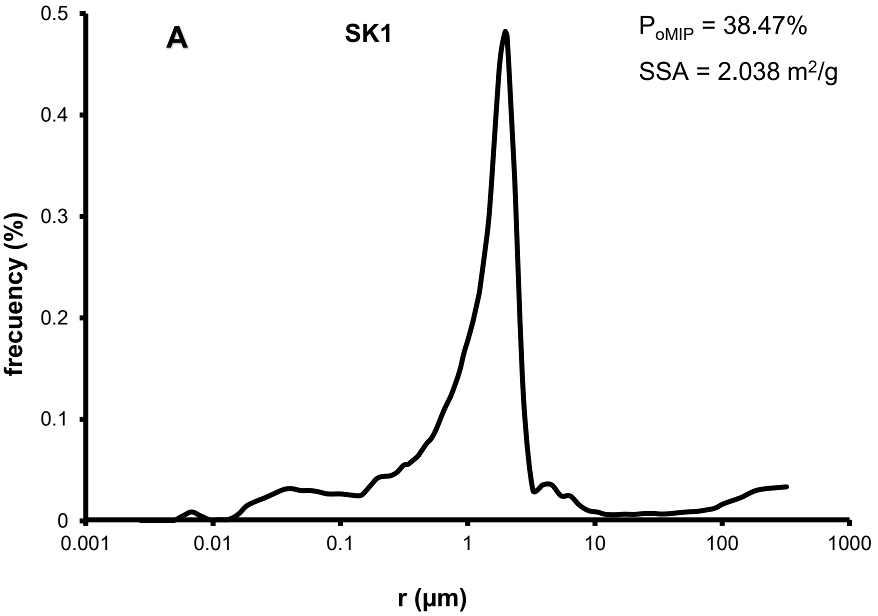
Figure 4.8 ESEM micrographs and EDS spectra for Robben Island's Pre-primary school building samples PS4 (A-B), PS9 (C) and PS11 (D-E)

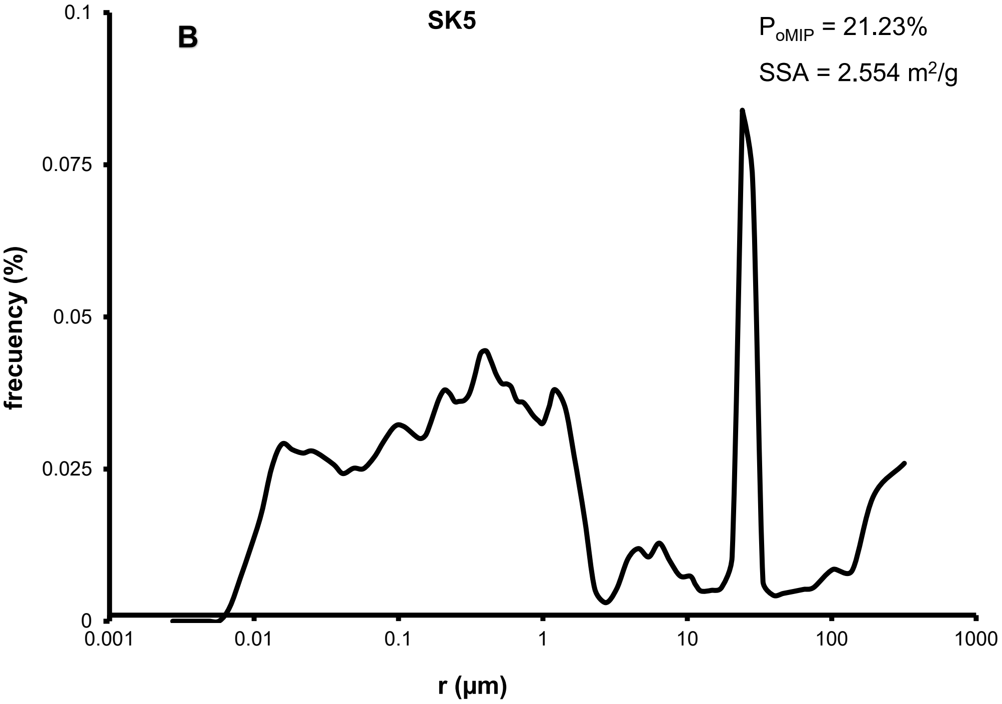
4.4 Physical properties

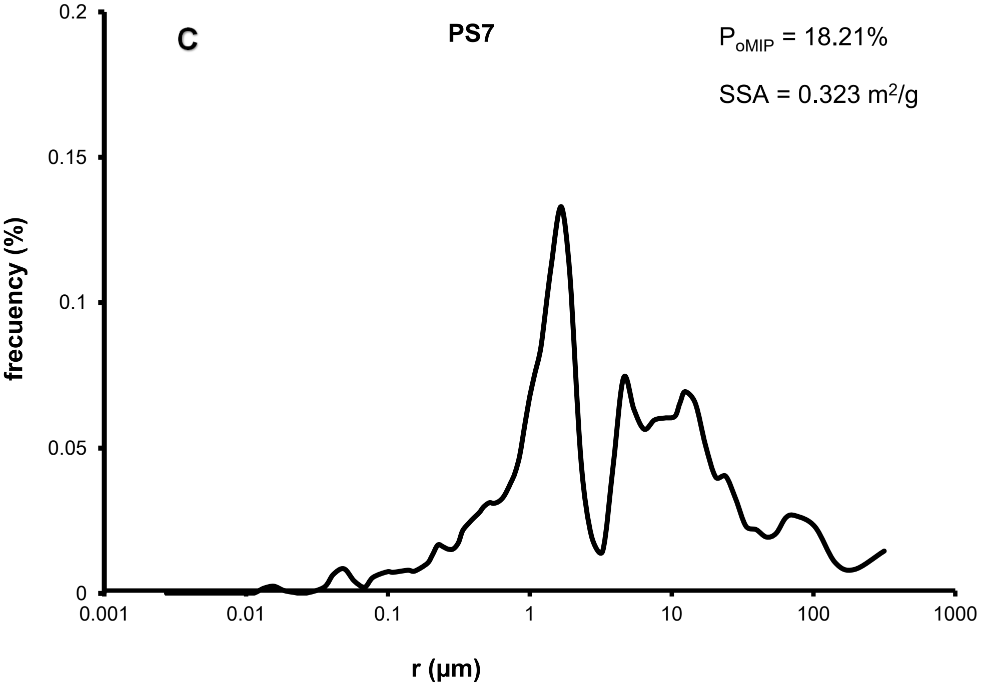
4.4.1 Mercury intrusion porosimetry (MIP)

The investigation of the porous system of the mortars shows that the lime mortars have a porosity ranging between 18 and 38%, SK1 being the most porous sample (Figures 4.9A - D). The cement-based mortar PS7 (Figure 4.9C) had the least porosity. This was expected since the literature clearly highlights the differences in porosity properties between the two mortars. Adequately porous mortars allow the masonry to breathe. However, one must be careful when using mortars that are too porous, as these would allow excessive water penetration and, thus, freeze-thaw possibilities (Loke *et al*. 2020).

As depicted in Figure 4.9A, the plaster mortar SK1 used in the Castle is more porous than the jointing (SK5) mortar (Figure 4.9B) from the same structure and sampling location, with 38% and 21% open porosity (P_{oMIP}), respectively. The pore size distribution curve of SK1 is virtually unimodal, with the main peak at roughly 2 μm pore radius. Very small second-family pores can be seen on the left of the main peak between 0.02 and 0.15 μm. SK5 presents a much less pronounced and polymodal curve with a peak at 24 μm pore radius and another sector of pores between 0.007 and 2 μm. The higher amount of smaller pores in SK5 compared to SK1 determines a slightly higher specific surface area (SSA) in the former. On the other hand, the porosimetric analysis for both PS7 and PS9 (Figures 4.9C-D) shows a polymodal pore size distribution with PS7 having a main peak at around 2 μm pore radius and another sector of pores between 5 and 100 μm. PS9, on the other hand, portrays a main peak at around 8 μm with minor peaks towards the left side.


A
SK1
P_{oMIP} = 38.47%
SSA = 2.038 m^2/g
frecuency (%)
0.5
0.4
0.3
0.2
0.1
0
0.001
0.01
0.1
1
10
100
1000
r (µm)


B
SK5
P_{oMIP} = 21.23%
SSA = 2.554 m^2/g
frecuency (%)
0.1
0.075
0.05
0.025
0
0.001
0.01
0.1
1
10
100
1000
r (µm)



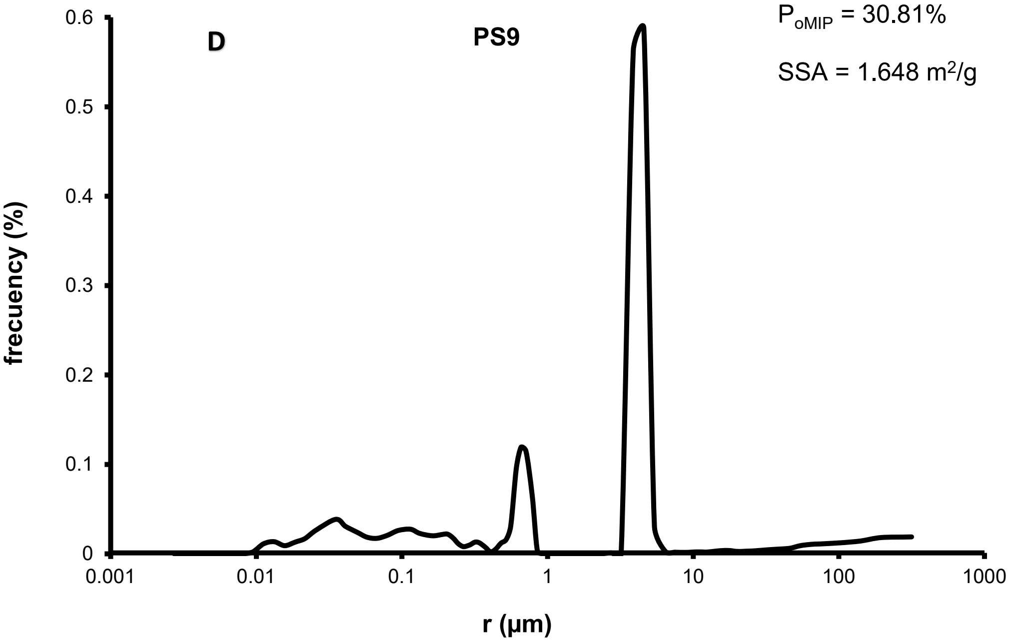


Figure 4.9 Pore size distribution curves of mortars from the Castle of Good Hope and pre-primary school on Robben Island determined by mercury intrusion porosimetry. Frequency (in %) versus pore radius (in µm). Open porosity (P_{oMIP}) and specific surface area (SSA) values are indicated in each diagram.

4.4.2 Hygric behaviour

For heritage repair mortars, a general expectation is to select a new mortar that will allow adequate moisture absorption and evaporation and with a similar or higher moisture evaporation rate than the substrate (Van Domelen, 2009). In any case, careful consideration should be taken since fast evaporation in the presence of soluble salts might lead to the development of sub-efflorescences and damage the surrounding materials. In contrast, slow evaporation could result in frost action-related problems. The hygric parameters listed in Table 4.4 and Figure 4.10 display a somewhat similar free water absorption trend (A_b, Table 4.4), with M9 having the highest $A_{b,}$ followed by M7. These two mortars (M7 and M9) portrayed the highest saturation coefficient (S) and higher porosity (P_o) than the other mortars. The control samples M1 and M3 also had high porosity.

Sample M3 displayed the lowest pore interconnection denoted by the highest A_x value. At the same time, M9 demonstrated the highest interconnection between pores (lowest A_x in Table 4.4 and lowest curve slope in sector b in Figure 4.10). The explanation for this difference in A_x lies in the use of the higher amount of binder in M9 (it is the only sample with a 1:1 binder-to-aggregate ratio), which may have favoured the development of a significant number of retraction fissures, hence improving the circulation of water in the pore network. A_x is inversely related to the saturation coefficient (S) (Table 4.4). This is logical since samples with poor interconnection among pores (high A_x values) saturate poorly (low S values). The lowest free (A_b) and forced (A_f) water absorptions in M6 suggest that this mortar has low retraction fissures, absorbing the least amount of water and attaining the lowest P_o and S values.

Regarding the drying of samples, control samples M1 and M3 have a similar drying pattern, as seen in segment (c) of Figure 4.10. M6 was the quickest to dry (highest D_i), unlike M9, which took longer. The values of real density (ρ_r, Table 4.4) are quite similar as they depend on the mineralogy of mortars. On the other hand, apparent density (ρ_a) is linked to the entire volume of samples (i.e., also the empty spaces). More porous samples generally have lower ρ_a values or higher differences between ρ_a and ρ_r. In the light of hygric tests, M6 emerged as the preferred repair mortar option as it had lower water absorption properties with a porosity (23%) similar to that of the original sample SK5; hence, it is less prone to water attack, resulting in higher durability expectations (Cultrone *et al.,* 2004). This will be evaluated later by salt crystallisation and freeze–thaw tests. M4 could be a second option if M6 does not meet the durability requirements. Regarding water flow in the pore system, M7 and M9 could be selected to replace SK1 mortars based on their high porosity values.

Table 4.4 Hygric behaviour of control mixes (M1 and M3) and the proposed repair mortars (M4, M6, M7 and M9). Legend: A_b - free water absorption (wt%); A_f - forced water absorption (wt%); A_x - degree of pore interconnection (wt%); D_i -drying index; S - saturation coefficient (%); P_o - open porosity (%); ρ_a - apparent density (g/cm^3); ρ_r - real density (g/cm^3). Standard deviations are shown in brackets.

Hygric property	M1	M2	M3	M4	M6	M7	M8	M9
A_b	12.86 (0.092)	11.47 (0.204)	12.41 (0.376)	11.20 (0.251)	9.72 (0.369)	13.82 (0.319)	9.45 (0.371)	15.79 (0.147)
A_f	15.45 (0.100)	14.73 (0.227)	15.58 (0.412)	13.06 (1.007)	11.64 (0.336)	15.69 (0.325)	11.64 (0.205)	16.63 (0.210)
A_x	16.77 (0.218)	22.14 (0.179)	20.37 (0.487)	13.95 (5.398)	16.52 (0.833)	11.90 (0.211)	18.81 (2.200)	5.06 (0.402)
S	70.33 (0.072)	65.43 (0.096)	66.04 (0.854)	73.67 (5.632)	68.63 (1.200)	78.11 (0.047)	71.83 (2.646)	87.69 (0.441)
D_i	0.932 (0.001)	0.933 (0.001)	0.930 (0.001)	0.934 (0.001)	0.939 (0.001)	0.930 (0.0003)	0.940 (0)	0.931 (0.001)
P_o	28.30 (0.146)	27.29 (0.297)	28.39 (0.694)	24.97 (1.551)	22.55 (0.622)	28.43 (0.607)	23.01 (0.347)	29.69 (0.266)
ρ_a	1.83 (0.003)	1.85 (0.010)	1.82 (0.004)	1.91 (0.028)	1.94 (0.004)	1.81 (0.005)	1.98 (0.005)	1.79 (0.009)
ρ_r	2.55 (0.003)	2.55 (0.008)	2.54 (0.020)	2.55 (0.015)	2.50 (0.017)	2.53 (0.024)	2.57 (0.006)	2.54 (0.010)

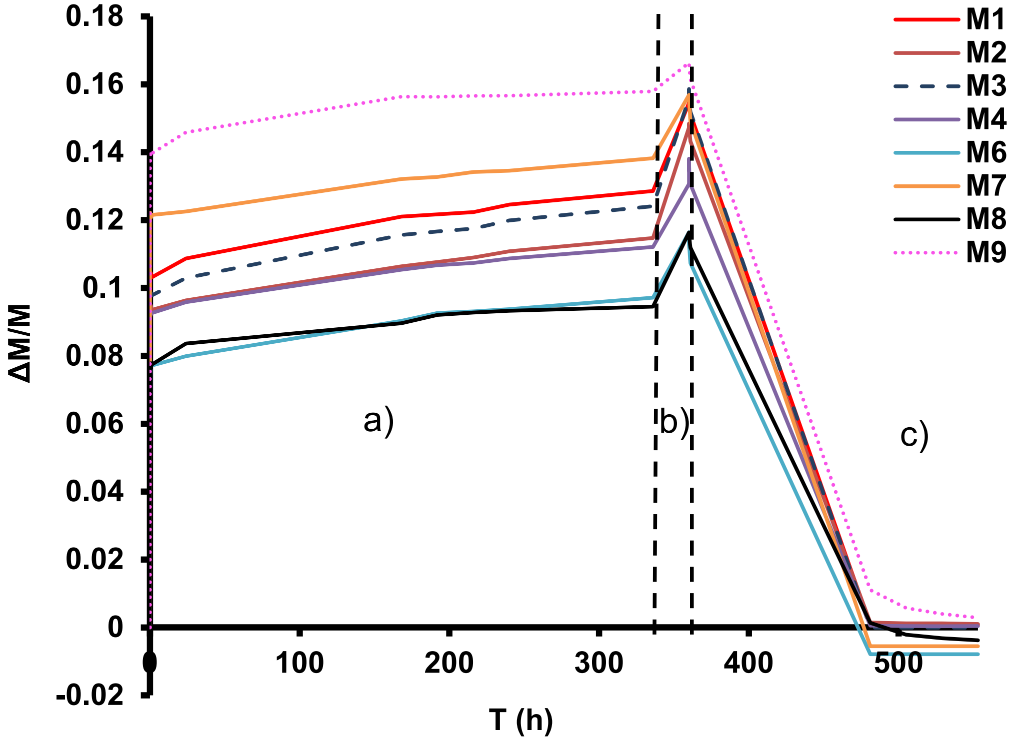


Figure 4.10 Hygric behaviour of new repair mortars a) Water absorption at atmospheric pressure, b) water absorption under vacuum and c) samples' drying curves. The graph depicts a variation in weight (ΔM/M) versus time (in hours)

4.5 Aesthetic properties

4.5.1 Colour

The binder matrix was primarily whitish in color, according to the visual assessment. Figures 4.11 and 4.12 show the mortar lightness (L*) ranging between 52 and 82 for both structures' original mortars, respectively, with the error bars indicating the variation in the measurements. The values around 50 (SK8) tend to be grey. This is a preliminary selection guide on using lime as a binder for all the mortar samples. This prediction was further confirmed through chemical and mineralogical analysis.

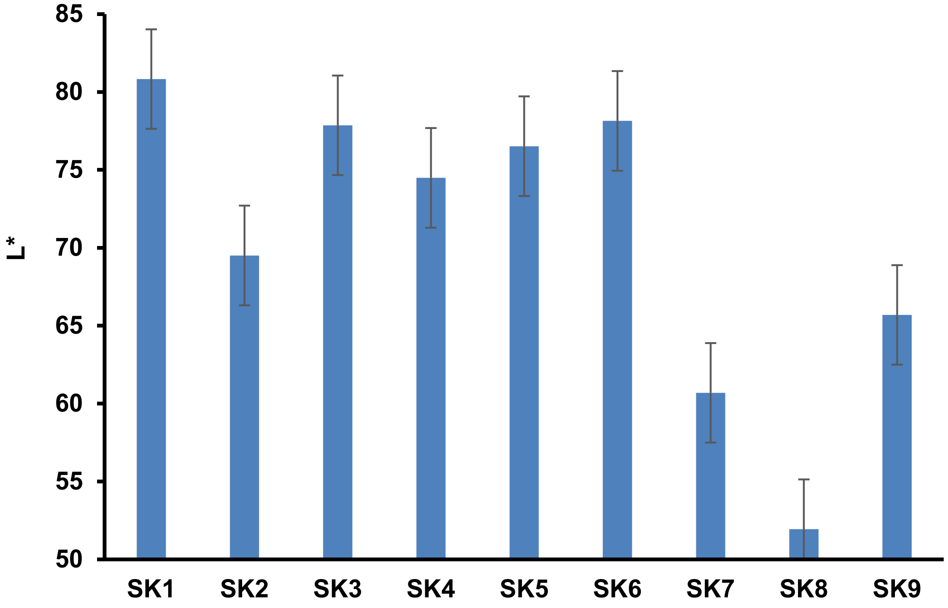


Figure 4.11 Lightness (L*) for mortars from Block B- kitchen (1666) on the Castle of Good Hope

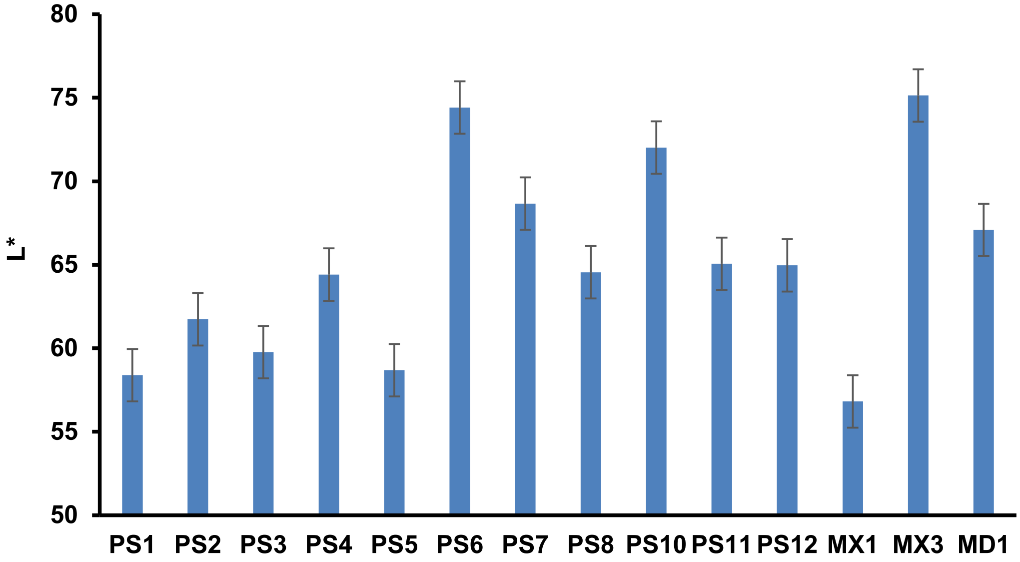


Figure 4.12 Lightness (L*) coordinates for the mortars from the pre-primary school and maximum-security prison buildings on Robben Island

The colorimetry analysis was also conducted on aggregates after mechanically separating them from the binder using a 0.063 mm sieve. The decision to separate the aggregates from the binder was based on the literature elaborating on the influence of the properties of the aggregate on general mortar properties. Hence, the colour of the aggregates plays an important role in the overall colour of the mortar. The aggregates used in the Castle indicated the lightness above 60, with just 10% of the binders indicating L* below 60, as depicted in Figures 4.13 and 4.14 for the two structures. All the L* values of the aggregates could be considered whitish.

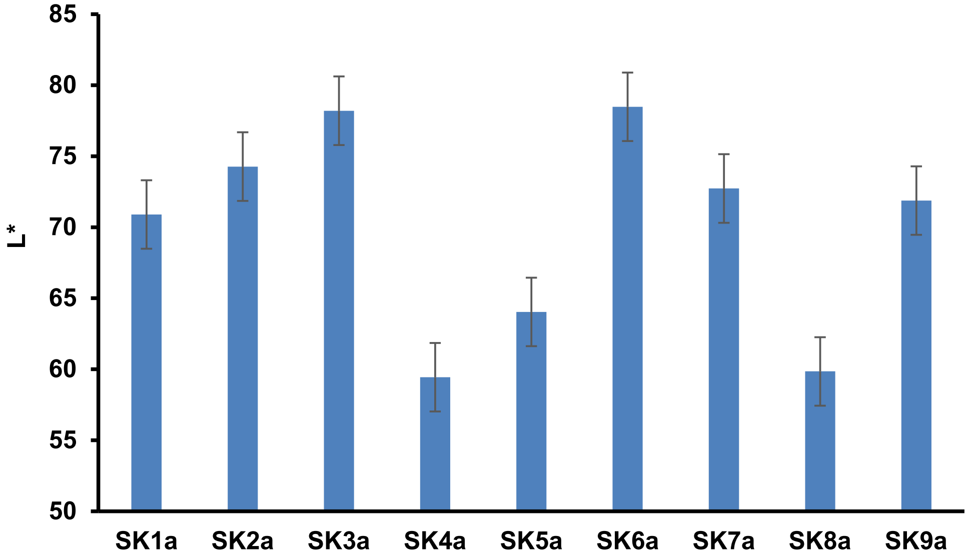


Figure 4.13 Lightness (L*) coordinates for binders from Block B- kitchen (1666) on the Castle of Good Hope

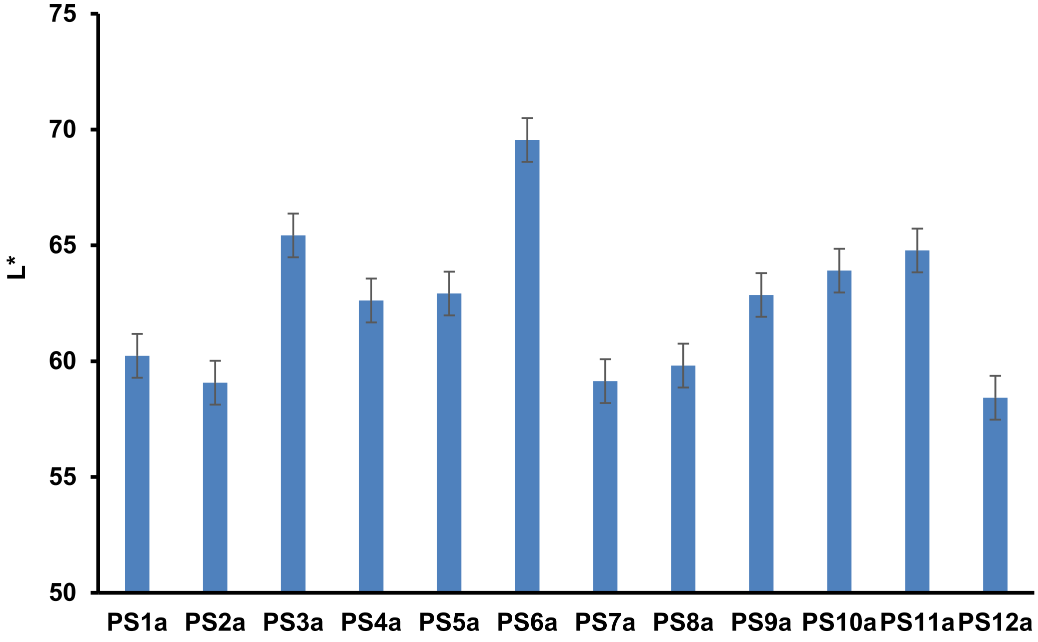


Figure 4.14 Lightness (L*) coordinates for binders from the Pre-primary school and Maximum-security prison buildings on Robben Island

In addition to the lightness, the chromatic coordinates a* and b* for both the monuments were recorded for the mortars after grinding and the binder's rich material portion after mechanical separation (Tables 4.5 and 4.6, respectively). As demonstrated in Tables 4.5 and 4.6, chromatic axes (a* and b*) indicated mortars with a trend toward the grey area as per the CIELAB space. Samples SK7, SK8 and SK9 (earth mortars) reach the highest values and, probably, the most marked colours among the studied samples. Sample SK4 displayed the lowest saturation (C*) values due to the lowest a* and b* values. These values are a valuable tool for restoration interventions as they are compared to the newly proposed mortars. A comparison with the allowable tolerances is made to come to a final decision regarding the substitute material application. All hue angles (hº) tend towards yellow colours.

Table 4.5 Chromatic parameters of historic mortar samples from Castle of Good Hope: a* and b*: chromatic coordinates, C*: chroma, hº: hue angle.

Sample ID	**Chromatic parameters of mortar samples**				**Chromatic parameters of aggregates**			
	a*	**b***	**C***	**hº**	**a***	**b***	**C***	**hº**
SK1	3.63	13.45	13.93	74.91	3.69	11.97	12.53	72.88
SK2	3.33	11.11	11.59	73.36	1.96	6.35	6.64	72.82
SK3	2.75	11.02	11.36	75.99	3.16	11.81	12.22	74.99
SK4	1.62	5.63	5.86	73.93	5.1	9.95	11.18	62.83
SK5	3.49	10.81	11.36	72.11	4.67	11.97	12.85	68.69
SK6	2.66	11.64	11.94	77.17	2.29	11.18	11.41	78.43
SK7	9.19	27.08	28.59	71.24	4.06	18.42	18.86	77.58
SK8	8.78	22.94	24.56	69.03	8.88	25.99	27.47	71.12
SK9	10.46	25.83	27.86	67.94	5.51	20.66	21.38	75.06

Table 4.6 Chromatic parameters of historic mortar samples from buildings on Robben Island: a* and b*: chromatic coordinates, C*: chroma, hº: hue angle.

Sample	Chromatic parameters of mortar samples				Chromatic parameters of aggregates			
ID	a*	b*	C*	hº	a*	b*	C*	hº
PS1	1.79	8.86	9.045	78.55	3.55	14.45	14.88	76.17
PS2	1.28	8.3	8.4	81.19	3.93	14.35	14.88	74.66
PS3	1.76	9.59	9.75	79.6	3.33	14.04	14.42	76.65
PS4	1.72	8.4	8.58	78.44	1.89	10.01	10.18	79.31
PS5	1.61	8.27	8.42	78.97	3.54	11.81	12.33	73.32
PS6	1.93	10.85	11.01	79.92	3.1	12.16	12.55	75.7
PS7	1.77	10.81	10.96	80.69	3.83	11.46	12.08	71.47
PS8	1.59	7.86	7.99	78.47	3.43	12.69	13.15	74.87
PS9	-				5.45	13.41	14.47	67.86
PS10	1.81	10.68	10.83	80.4	4.99	13.97	14.83	70.34
PS11	1.79	9.47	9.63	79.32	2.8	12.03	12.36	76.91
PS12	1.88	10.13	10.3	79.47	1.32	7.33	7.45	79.79
MX1	2.23	9.05	9.32	76.13	-			
MX3	3.4	12.1	12.57	74.28	-			

After carrying out the colourimetric measurements on the original and new mortars, the results were compared for the compatibility of the proposed new mortars with the original ones. The colour difference ($\Delta\mathrm{E}$, Table 4.7), was calculated for mortars, using Equation 3.7. All the new mortar samples have ΔE values greater than 5, which implies that a standard observer could notice the difference in colour between these samples and the original samples (Mokrzycki & Tatol, 2011).

The new repair mortars showed an almost similar lightness except for M6 and M9, which are lighter (higher L*), but less saturated (lower C*). This is because sample M6 has the lowest chromatic parameter a*, while M9 has the lowest b* (Table 4.7). The hue angle of samples M4 and M6 stand out from the others (hº, Table 4.8). Even if all samples fall in the grey area of the Munsell Soil Color Chart, the original samples, except SK4, show higher b* values. This is why when the original samples are compared with the new ones, ΔE is always greater than 5, the limit above which people can easily distinguish two colours as different (Mokrzycki & Tatol, 2011). M2, M6, M7 and M9 are the repair mortars with the highest ΔE values compared to the original ones PS4, PS10, SK2 and SK6. These differences were, at least in part, expected considering that the original mortars have survived weathering conditions for over 350 years, hence, colour change.

Table 4.7 Chromatic CIELAB colour space parameters: lightness (L*), chromatic parameters (a* and b*), chroma (C*), and hue angle (hº) for the proposed repair mortars for the Castle of Good Hope and buildings on Robben Island. ΔE is the colour difference between original mortars and new repair ones.

Sample ID	Sample Detail	L*	a*	b*	C*	hº	ΔE
M1	Repair control sample 1	83.84	1.53	6.74	6.91	77.10	-
M3	Repair control sample 2	83.92	1.63	7.03	7.22	76.95	-
M2	PS4 replacement	82.98	1.24	6.46	6.58	79.09	18.26
M2	PS6 replacement	82.98	1.24	6.46	6.58	79.09	9.37
M4	SK4 replacement	83.63	1.21	6.76	6.86	79.73	8.86
M6	SK5 replacement	85.06	1.13	6.24	6.35	79.65	9.66
M6	PS10 replacement	85.06	1.13	6.24	6.35	79.65	13.40
M7	SK1 replacement	84.19	1.34	5.62	5.77	76.52	8.69
M7	SK2 replacement	84.19	1.34	5.62	5.77	76.52	15.41
M7	SK3 replacement	84.19	1.34	5.62	5.77	76.52	8.17
M7	MX3 replacement	84.19	1.34	5.62	5.77	76.52	11.01
M8	PS11 replacement	82.42	1.19	5.97	6.08	78.76	17.25
M9	SK6 replacement	86.22	1.41	5.28	5.47	75.02	10.03

4.6 Compactness and strength

4.6.1 Ultrasound pulse velocity (UPV)

The mortars produced from commercial sand (M1 and M3) are more compact than the mortars produced from sea sand (Table 4.8). It is also interesting to observe that the lowest ultrasound velocity was measured in a gypsum-containing mortar (M7). This observation disputes the assumption that gypsum was only added to aid quick setting. The added gypsum influences the waves' velocity of the mortar (V_p, Table 6). If this was not the case, V_p of M4 and M7 were expected to be within the same range since the two mortars have the same components except for adding gypsum in M7. The control samples without seashell fragments are less anisotropic (ΔM) compared to the shell-reinforced mortars. These organic fragments should play a huge role in increasing the compactness of the mortar. In this respect, Shivakumar *et al*. (2022) observed that the proteins present in the shells help to bind the raw materials by augmenting the internal cohesion between the crystalline particles. Hence, higher strength and interlocked connections between particles result in rigid mortar properties.

Table 4.8 Ultrasound values of new repair mortars. V_{Pa}, V_{Pb} and V_{Pc} represent ultrasonic wave velocities in m/s along the three orthogonal mortar cube directions. ΔM is the total anisotropy (%).

Mortar ID	Wave velocity (m/s)				ΔM (%)
	V_{Pa}	V_{Pb}	V_{Pc}	V_P Aver.	
M1	1841	1848	1786	1825	4.41
M2	1648	1605	1592	1615	4.29
M3	1634	1690	1676	1667	5.31
M4	1461	1481	1544	1495	6.44
M6	1458	1580	1486	1508	8.69
M7	1189	1164	1106	1153	7.58
M8	1516	1480	1369	1455	10.04
M9	1535	1602	1573	1570	6.00

4.6.2 Compressive strength

The seashell fragments (and their proteins) had no impact on either the velocity, as shown in Table 4.9 or strength, as depicted in Figure 4.15, whilst the curing period plays the main role in the strength development of these mortars. The control (M1 and M3) and gypsum-containing mortars (M7) in their early stage of curing/carbonation (after 28 days) achieved significantly high (generally 50% more) compressive strengths when compared to more carbonated samples (after 60 and 90 days). On the other hand, the other samples (M4 and M6) seemed to gain compressive strength over a prolonged hydration period of 90 days. Sample M9 did not follow any trends between the two described groups, showing high mortar strength at 60 curing days. This out-of-trend strength development could be due to a different mixing ratio, considering that M9 is the only mortar with a 1:1 mix ratio. Logically, the particle sizes are smaller in this mortar, with higher calcite formation through the carbonation process, contributing to increased strength. In this respect, Shivakumar *et al.* (2022) suggested that the grain size between 0 and 4 mm provides the highest compressive strength, while the bigger grains reduce the mortar strength. A decrease in mortar compressive strength in sample M7 could be influenced by adding gypsum to this mortar.

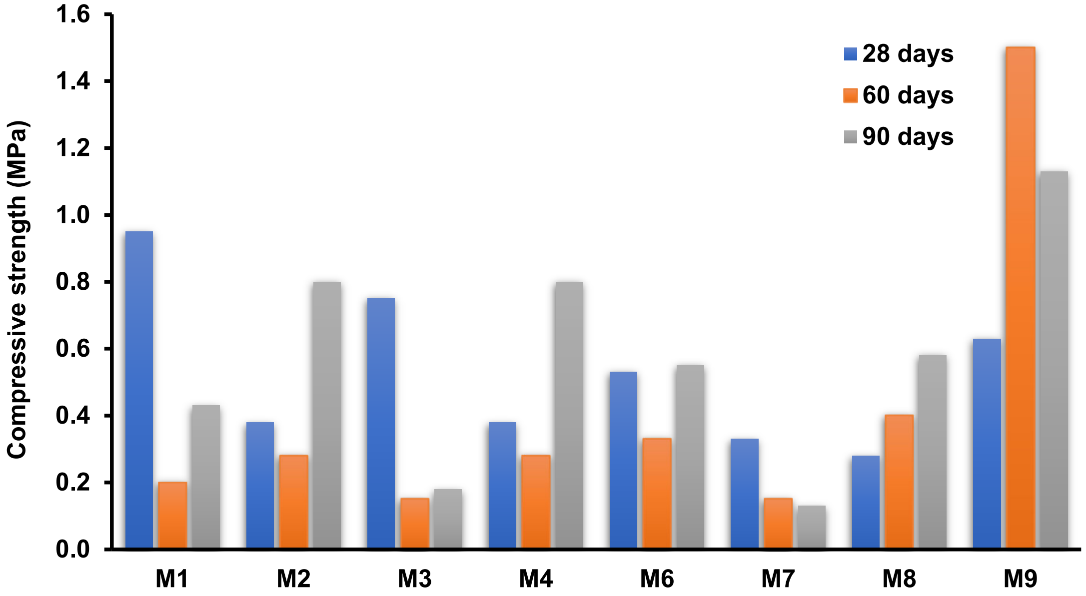


Figure 4.15 Compressive strength (in MPa) on new mortar cube samples after 28, 60 and 90 days curing under controlled humidity and temperature

4.6.3 Flexural strength

The flexural strength developed similarly; the 90-day mortars showed highest strength, followed by the 28-day mortars across all mixes, with 60-day mortars having the least stength. Although the flexural strength behaviour was similar for all 1:3 mixes, a different trend was observed for M9 (1:1) mortars, as depicted in Figure 4.16. With an average of 0.58 MPa at 28 days for 1:3 mixes, these mortars fall within the 40-50% of the compressive strength maximum strength recommended by Fontaine *et al*. (1999) for jointing mortars. However this was not the case for 90-day mortars having an average of 1.21 MPa which is higher than the 0.7 MPa recommended by Veiga *et al.* (2001).

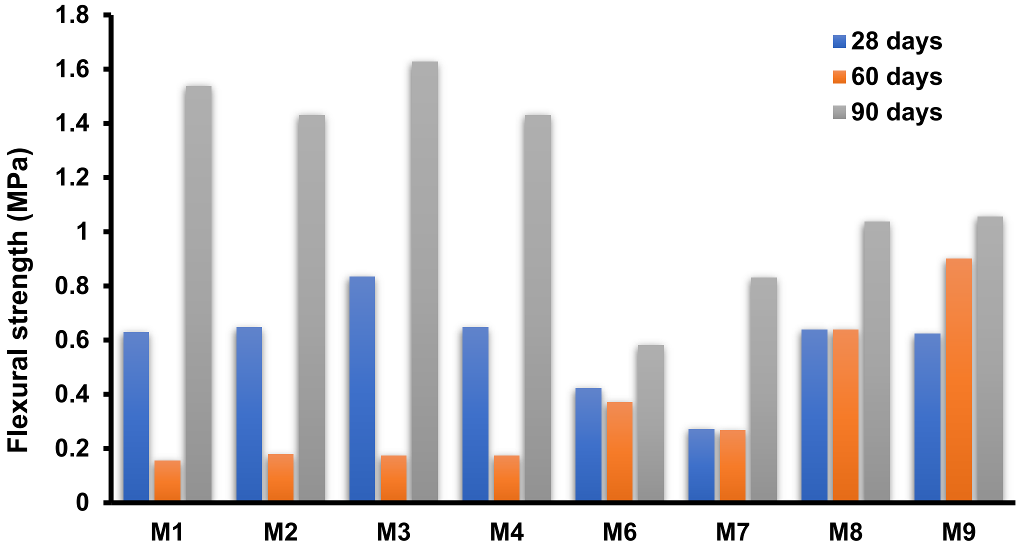


Figure 4.16 Flexural strength (MPa) on new mortar beams samples after 28, 60 and 90 days of curing under controlled humidity and temperature

4.7 Durability - Ageing tests

4.7.1 Freeze-thaw resistance/modulus of rupture

When water turns from liquid to ice, a 9% increase in specific volume causes pressure to build up in the pore walls of the mortar, which can lead to breaking and cracking (Ergenç *et al*., 2021). In particular, if mortars have developed poor mechanical strength, a high saturation coefficient, and a high porosity with smaller pore sizes, they are vulnerable to ice action. Compared with Portland cement mortars and hydraulic lime, lime and lime with pozzolan mortars are far less resistant to freeze-thaw cycles.

The deterioration patterns due to alternating freezing and thawing of the eight new repair mortars for Castle of Good Hope are demonstrated in Figure 4.17. During the decay test, samples M1 to M9 showed a small weight gain in the first and second cycles. This was due to the water filling the samples' pores and fissures. On the other hand, sample M7 had already started to lose weight from the second cycle. Visually, the deterioration pattern of mortars happens so that the edges crumble first, with weight loss occurring towards the centre of the sample. For samples M6, M7 and M9, the decay test could not be completed, with M9 performing the worst and not managing to pass the 9th cycle. In the hygric test, M9 was the most porous material (P_o, Table 4.5), influencing its resistance to freeze-thaw cycles (Křivánková *et al*., 2019). Its open porosity was, however, not so different from the other samples. The degree of pore interconnection differentiates it from the other samples (A_x, Table 4.5). This sample has a much lower A_x value (better pore interconnection) than the others. This will have favoured an easier water migration into this mortar's capillaries, causing its early breakage during the water-ice phase transition. The gypsum-infused mortar (M7) was the second least resistant to the freeze-thaw test, as the samples broke at the 12th cycle, while M6 lasted until the 19th cycle. The other types of mortar endured 30 cycles with an average 50% mass reduction at the end of the test.

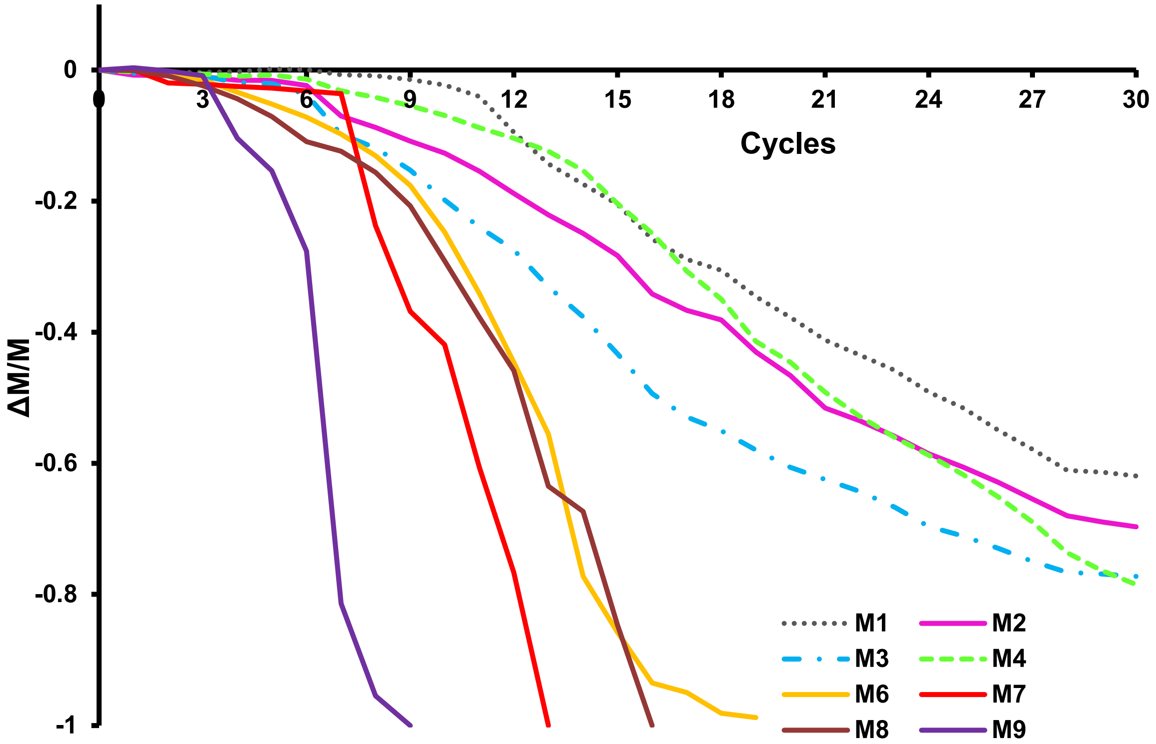


Figure 4.17 Freeze-thaw test of new repair mortars. Weight variation (ΔM/M) versus number of cycles

4.7.2 Salt crystallisation

The resistance of mortars to fifteen salt crystallisation cycles was almost similar for all the samples, irrespective of their composition, except for M7, which was the mortar that lost more weight (Figure 4.18). From the beginning to the 3rd - 4th cycle, all the samples gained small amounts of weight due to the crystallisation of sodium sulphate in their pore system. Later on, at the 7th cycle, M7 experienced a weight loss of around 70% instead of roughly 30%, as for the other mortars (Figure 4.18). Right after this cycle, the samples started to break off, displaying signs of crumbling, with the cubes changing shape as they shed off layers from the surface to the inside. At the end of the test, there was on average 50% mass loss for all the mortars except for M7, which lost 95% of its mass. Benavente *et al*. (2006) suggested that the materials' ability to withstand salt attack is related to its compressive strength and P-wave velocity. The fact that M7 is the mortar with the lowest compressive strength at both 60 and 90 days and ultrasound velocity can explain its poor resistance to degradation due to the crystallisation of salts in the empty spaces of the sample. Control samples (M1 and M3) performed best in this test.

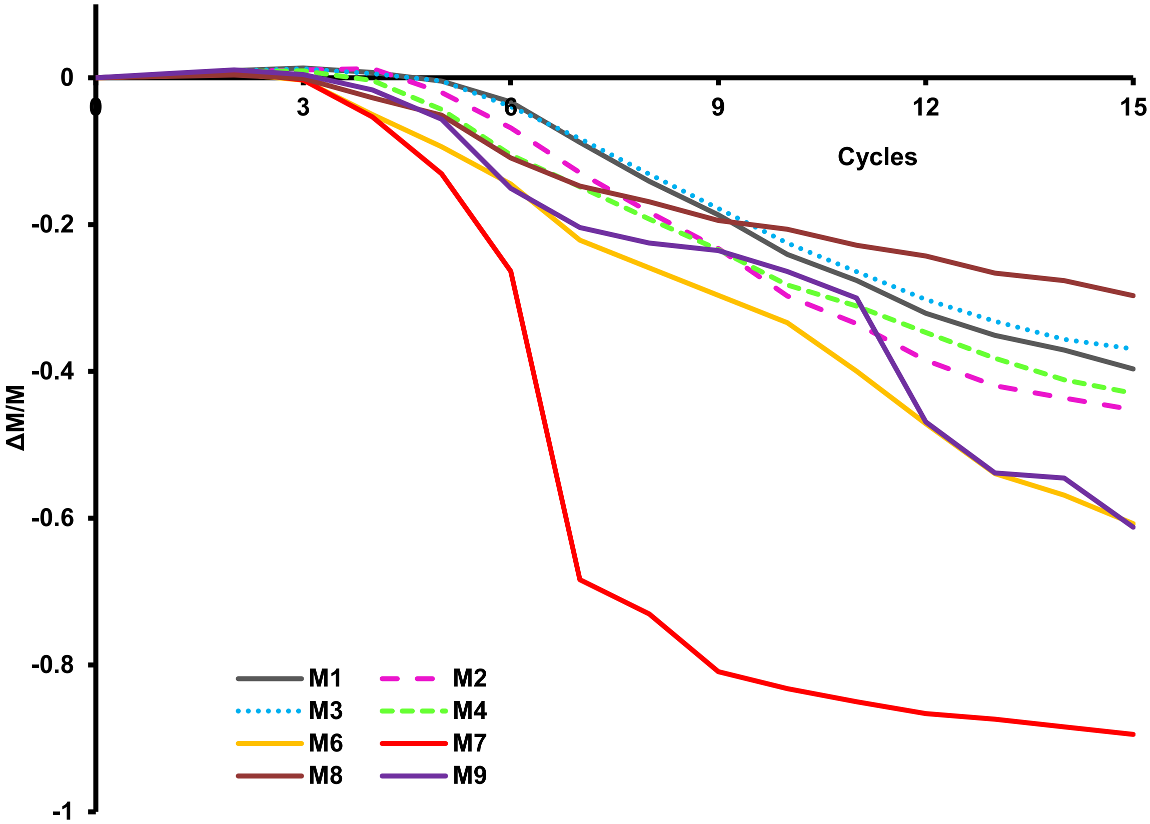


Figure 4.18 Salt crystallisation test of new repair mortars. Weight variation (ΔM/M) versus number of cycles

4.8 Conclusion

Using incompatible materials in the restoration of heritage structures is a widespread issue, leading to adverse consequences that threaten the authenticity of these historical treasures. Such misguided attempts at restoration often fall short of addressing decay problems in historic buildings and fail to achieve essential restoration objectives, which include compatibility, suitability, and longevity. This prevailing trend of using mismatched materials perpetuates the need for recurrent repairs. To rectify this problem, it is imperative to investigate the design and development of heritage restoration mortars, as this endeavour promises a better comprehension of the requisite procedures for designing new mortars with compositional attributes and performance characteristics that seamlessly align with the original materials.

The primary goal of this research was to examine the design techniques for heritage restoration mortars and develop appropriate materials in compliance with established standards, including RILEM, European and South African National Standards where applicable. By characterising the original sub-hydraulic lime-based and earth mortars, invaluable insights were gained to inform the production process of new materials. The findings highlighted the importance of a systematic framework for restoration practitioners, particularly in developing countries lacking sufficient resources for heritage building restoration studies. The new repair mortar M4 demonstrated a chemical and mineralogical composition congruent with most of the original mortars, displaying good resistance against freeze-thaw and salt crystallisation ageing tests. On the other hand, M6, M7 and M9, quickly crumbled during those tests, even though M9 had the highest compressive strength. A key observation was the interaction between durability and mortars' physical and mechanical behaviours. The high porosity and water absorption in M7 and M9 mortars were correlated with the increased vulnerability to deterioration induced by salt crystallisation and freeze-thaw cycles.

The aesthetic aspect generally deserves attention, especially the colour, to mitigate any inconsistencies during the restoration work. The high colour change values underscore the need to address this aspect, potentially through modifications involving colour-enhancing pigments that bear no effects on the mortar properties besides the colour. This requires research in this area to match the colour of mortar and predict future colour changes due to inevitable ageing and exposure to environmental conditions.

Chapter 5 Discussion

The main cause of most challenges faced in heritage restoration is the compatibility between original and repaired mortars. The limited guidance in clearly outlined procedures for the design and production of the repair mortars that will serve the desired purpose has, for many years, been an area of concern requiring research. Because of the lack of clearly documented information in this regard, uninformed restoration projects are executed, thus causing negative economic impacts due to early failures of such repairs. This chapter compares the properties of the Castle of Good Hope and Robben Island building precincts' designed mortar' with the documented and recommended performance and technical criteria for heritage mortar. The assessment of whether the suggested mortars are suitable for use in heritage restoration is provided in this chapter, with recommendations for improvement of such mortars. It further creates a systematic connection between the findings from the experimental and theoretical concepts obtained throughout the study and the research questions outlined in Chapter 1.

5.1 Introduction

Even though the design of restoration mortars is critical in any conservation project, more so for historic structures, because of the complexity of these mortars, this topic still attracts limited interest from the scientific and industrial communities. From the literature reviewed, it is clear that this process lacks guidelines and documentation, leading to subjective design and production procedures. Despite the challenges facing the compatible restoration of historic masonries, its importance cannot be overemphasised nor denied. For this reason, the sustainable conservation of historic mortars is an issue that is gaining much-needed momentum in society, abroad, but not much in Africa. Hence, the motivation for the present study.

5.2 General selection criteria for heritage repair mortars

During the selection of new compatible restoration mortars of the architectural heritage, it is important to take into account the important design and production aspects, namely: i) visual analysis of the heritage building in question, ii) experimental analysis of the original mortar samples for their physical, mineralogical and chemical properties, iii) characterisation of the potential raw materials that match the original properties and determination of their appropriate ratios, and iv) assessment of the new mortar durability. This general procedure provides valuable information that will ensure long-term existence of historical monuments through distinguished restoration interventions.

A preliminary visual assessment of the original materials preceding a detailed experimental characterisation by means of colorimetry, PXRD, XRF, MIP, TGA-DSC and ESEM analyses are key in achieving the best-fit repair mortars for historical constructions. Even though there is no suitable-for-all material characterisation standard applicable for this task, the European Standards (EN), American Society for Testing and Materials (ASTM) and primarily, the RILEM Recommendations are a step in the right direction in addressing the lack of standards in heritage mortar analysis as indicated in Section 2.9 of this thesis. It was discovered during this research that, for the South African context, the existing design standards do not specifically cater to the design of historical mortars but solely focus on modern materials, particularly cement-based. This calls for the need to incorporate the guidelines related to heritage mortars and subsequent heritage materials in the South African National Standards.

The characterisation of the original mortar samples representing different categories, such as plaster, render, bonding and floor mortars from the three eras, 17th to 19th century, led to the selection of hydrated lime repair mortars made of aggregates collected from the West Coast (Cape Town). The raw material selection was based on the results of the original mortar analysis. Despite this, due to the ageing of these original mortar samples, possible deviations in terms of composition from the initial application hundreds of years ago are to be noted. This describes the capability of the new materials to meet the recommended heritage material criteria outlined in Table 2.4 for heritage restoration purposes, providing recommendations should they not meet the recommendations. Table 5.1 summarises the heritage mortars’ design criteria specified in literature versus the key parameters obtained in the current study.

Table 5.1 Summary of the criteria for selecting heritage repair mortars modified from Veiga *et al.* (2001); Papayianni (2006); Moropoulou *et al.* (2009) versus the results obtained in the current study.

	Design criterion range in the literature				
Property	**EN 998-1 (2003)**	**Papayianni (2006)**	**Moropoulou *et al.* (2009)**	**Veiga *et al.* (2001)**	**Current study**
Workability	Optimum.	Details not provided	Details not provided	Details not provided	Optimum except high shell content mortar M8 & high binder (1:1) content M9 with lower workability.
Setting time	3-10 days. No curing conditions were provided.	No details	No details	No details	5-7 days under relative humidity of 60 ±5% & room temperature of 20 ±5 °C.
Compressive strength	1.5-5 MPa at 28 days	3.6 MPa	No details	0.4-2.5 MPa at 90 days	Aver. 0.5 MPa at 28 days & 0.4 MPa at 90 days for 1:3 mixes. 0.6 MPa at 28 days & 1.1 MPa at 90 days for 1:1 mix.
Flexural strength	No details	40-50% of Compressive strength	<0.35 MPa (no curing period given)	0.2-0.7 MPa at 90 days	Aver. 0.58 MPa at 28 days & 1.21 MPa at 90 days for 1:3 mixes. 0.62 MPa at 28 days & 1.05 MPa at 90 days for 1:1 mix.
Porosity	No details	20-40%	30-40%	No details	Average 27%

5.2.1 Mortar functional-based design

There is a strong relationship between the technical and functional requirements of the mortar, as indicated by Romero-Noguera *et al.* (2018). When designing the repair mortars for historical structures, the functional role of the final product within a structural component should be considered (Papayianni *et al.,* 2000). Romero-Noguera *et al.* (2018) summarised the key functional requirements in Table 2.2. Despite that, the authors provided limited quantitative details on these requirements. For instance, it is indicated that the rendering mortars ought to have proper pore size distribution that is high enough for water infiltration but not too high since it would promote sub-florescence. Providing descriptions without numerical ranges allows for subjective limits on pore size distribution. An additional requirement is the less soluble salt content for rendering mortars, which could lead to subjective decisions based on the designer’s discretion.

In the current study, it was challenging to assess the ability of new mortars to meet the functional requirements and thus propose the recommendations since no exact ranges could be found in the reviewed literature. Even so, Hughes *et al*. (2012) made a significant contribution by providing the key functional requirements for each mortar class, indicating the degree of importance for each technical requirement, with the aesthetics being the most important among all mortar classes except for the jointing mortars.

5.2.2 Evaluation of the existing criteria

Based on the comparison between the current research and the literature regarding acceptable design ranges and functional requirements, a conclusion can be made concerning the selection of repair mortars. The literature does not provide full details of some of the ranges given, such as the curing conditions for setting time and sample age for compressive and flexural although such conditions play a major role in the setting speed and strength development of mortars. Details on the mortar category would also provide a concise and specific criterion compared to the overall ranges, which may not be applicable to all the different mortar applications. As an example, the porosity of the sacrificial mortar (render) would be lower than that of the plaster or the pointing mortars, and so would the strength of bonding mortars, which are load bearing as opposed to the strength of the non-load-bearing plaster. The conclusion of such details on the criteria ranges will lead to a more precise choice better suited for the application.

It is unknown whether the requirements stated in Table 5.1 as indicated in the literature, are based on renders, platers, pointing or bonding mortars. Nonetheless, the new proposed mortars for the Castle of Good Hope and buildings on Robben Island are within these ranges and, therefore, are suitable for use in heritage restoration with the assumption that the difference in mortar properties among the different mortar categories is catered for in the ranges provided.

5.3 Interpretation of results: key technical performance requirements

The critical performance requirements for compatible repair mortars are outlined in Section 2.7.4. In this section, the aesthetic features using colorimeter, and mortar composition matching the original are assessed for suitability in the restoration of the case studies. The composition of the mortar dictates its overall behaviour and, subsequently, that of the masonry structure. The aggregates' type, particle distribution and density, the binder type and use of additives greatly influence the mortar behaviour. Hence, their selection should be done with great caution. Arizzi (2012) indicated that raw materials with similar properties to the original materials are key to durability and compatibility in heritage restoration works. Understanding the original material properties is important to understand better the expected mortar behaviour resulting from the suitable matching raw materials. It is also necessary to assess the mineralogical and chemical composition of the repair mortars in relation to the original, as the presence of undesirable chemicals in the mortar not only endangers the mortar component or masonry, but the entire structure. Based on the technical requirements in Section 2.7.4 and Table 2.3 provided by Van Balen *et al*. (2005), Groot *et al.* (2007), Romero-Noguera *et al*. (2018) and Fontaine *et al*. (1999), compatibility assessment of the new mortar is also provided in this section.

5.3.1 Similar composition to the original mortar

Even though it was unclear as to which original mortar properties were matched in their study, Aggelakopoulou *et al.* (2019), as well as the current study, assessed the suitability of hydrated lime (lime putty/hydrated lime powder) or used in heritage mortar restoration. The 1:3 ratio by weight was used in this study, aligning with most of the authors listed in Table 2.7. Most original mortars were hydraulic lime-based, with the possible undocumented repairs based on natural cement. However, since hydraulic lime could not be sourced within the study area, hydrated lime, having the chemical composition (Appendix A) closest to the original mortars was selected as a substitute binder type instead. Regarding the aggregates and additives, sand sourced from the Atlantic Ocean, along the West Coast, similar to the original aggregates of sedimentary origin, were used with seashells as additives. In addition to this type of aggregates being compatible with the original in terms of properties, their selection was based on the need to minimize restoration cost (purcharing aggregates) on raw materials especially for the large scale restoration projects. Considering that the both the Castle and buildings on the Island are located on a coastal area where sea sand could be a readily available raw material, the cost effectiveness for using sea sand could be beneficial.

5.3.2 Pore size distribution

Pore size and particle size distribution in mortars are directly related to water movement within a sample. The porosity and pore size distribution are essential for pointing, rendering and surface repairs and reasonably important for plasters, as concluded by Hughes *et al.* 2012 (See Table 2.1). For restoration mortars, Romero-Noguera *et al*. (2018) recommend a higher, yet not too high, porosity, specific gravity, pore size distribution, water absorption by capillarity and vapour transport to mitigate sub-florescence. The high porosity recordings between 18% and 38% of new mortars are a perfect replacement since they would allow the masonry to “breathe”, thus not trapping excess moisture.

5.3.3 Compatible aesthetic features

The aesthetic parameters, namely colour, texture, flatness, lightness, and surface finish, are among the most important properties to achieve during mortar replacement among different mortar classes, yet they are the most challenging. These features affect the entire structure as they present the monument itself. In the current study, as was expected, the new mortars portrayed a different colour to the original materials. This indicates the need to modify the mortar colour before application.

5.3.4 Split tensile/compressive strength

As part of the technical performance requirements for new pointing mortars outlined by Fontaine *et al*. (1999), pointing mortars should have a compressive strength between 1 - 8 MPa, as indicated in Table 2.3. Unlike the design criterion provided by EN 998-1 (2003), Veiga *et al*. (2001), Papayianni (2006) and Moropoulou *et al.* (2009), for this property, Fontaine *et al*. (1999) clearly state that this range is applicable for jointing mortars and not for any other mortar type. In this regard, out of the eight designed repair mortars in this study, for the repair of jointing mortars SK5 to SK9 from the Castle of Good Hope and MX2 and MX3 from the Maximum security prison building on Robben Island, the most suitable repair mortar is M9 in terms of its compressive strength of 1.13 MPa at 90 days, thus, meeting the range provided by Fontaine *et al*. (1999) for jointing repair mortars. Fontaine *et al*. (1999) elaborated that low tensile strength causes mortars to crack as they cannot resist vending. However, mortars that are too stiff are equally undesirable as they can affect the substrate materials and cause cracking. This performance requirement, which has average importance across mortar classes as mentioned in Table 2.1 by Hughes *et al.* (2012), does not only apply to the mortars but extends to the substrate (interface of mortar and stone/brick should be ≥ 0.3 MPa) materials in masonry, being either stones or bricks.

5.3.5 Durability and longevity

In order to ensure the durability and longevity of heritage repair mortars over extended periods, it is essential to investigate their resistance to freeze-thaw and salt crystallisation cycles. Four of the eight mortars proved resistance to freezing temperatures, while the other four (M6-M9) failed the extreme conditions, with weight loss of up to 50% of the total sample size being the failure sign. In terms of resistance to salt attack, all seven mortars displayed similar failure behaviour except M7 (sample with added gypsum), which displayed the highest weight loss. This ultimately discourages using this mortar to restore these structures located in a coastal area and, hence, susceptible to salt attack from sea spray.

5.4 Practical applications and significance

An essential tool in the restoration of historic masonry is the idea of creating appropriate repair mortars. The current research focused on using extensive property characteristics to design and develop suitable mortars to preserve these distinctive structures' heritage structural integrity and original aesthetics. Its goal was to direct the creation of restoration materials that would not damage the original surface in conjunction with long-term performance evaluations. The new materials in this research have proven their effectiveness in use on historical structures, with a need to modify the colour using pigments before applying the mortars on heritage masonries. This was proven by their compatibility with the original in terms of physical, mineralogical and chemical properties as well as their durability when exposed to ageing tests.

This research could serve the South African Heritage Resources Agency, the Robben Island Museum, the Castle of Good Hope management and the heritage construction community based on key aspects to investigate during a restoration project. The research provides comprehensive guidelines, relevant standards and procedures for carrying out the primary analyses for durable repair mortars and possible incorporation into the South African National Standards for regulating the application of lime-based mortars in a modern era. Even so, restoration professionals can improve the design and development of heritage mortars for practical restoration projects through a large-scale material sample that would allow material interpretation on a large project scale. This was not the case with this project since there are regulations and limitations to monitor and restrict the sample sizes on heritage structures.

5.5 Conclusion

The main objective of this research was to assess the material design standards, to design and produce suitable repair mortars for historic structures in the Western Cape using these standards, and to evaluate the durability of new mortars under accelerated weathering conditions. A greater understanding of the key technical performance requirements of heritage mortars based on functional parameters has been obtained in this work. The work highlighted some of the gaps in the criteria used to select the mortars, which, in most cases, lack precision in addressing the technical requirement ranges for different mortar classes. The new mortars were critically assessed for their appropriateness to remedy the defects occurring on heritage masonries that, for many years, have been addressed erroneously due to limitations in available construction standards guiding them. The design procedure showed the necessity to address the new mortar's vulnerability to water-induced degradation using hygric tests, which play a major role in how the entire masonry structure resists water movement. It was proven that a binder-to-aggregate ratio of 1:3, as suggested in literature by Moropoulou *et al.* (2002); Arizzi (2012); Nězerka *et al*. (2014); kumar and Kumar (2022) and Shivakumar *et al.* (2022) is the best in terms of hygric behavior and meeting the general performance standards. Simultaneously, the assessment of the mortar's durability showed that the mortars could survive salt contamination better than freeze-thaw conditions. Based on the typical average temperatures in Cape Town, the resistance to freezing could not be considered an essential aspect in discarding these mortars.

Chapter 6 Conclusions and recommendations

The design and development of heritage mortars for restoration is a complex task requiring expertise from different fields of archaeology, science, engineering, geology, historians, and mainly the material production industry and standard-setting bodies. Over the years, there has been a gap in the roles involved in the restoration of historic mortars. Some stakeholders are often left out of the practical restoration process, particularly researchers, the materials industry, and standard-setting bodies. This has led to a large amount of research addressing the pressing issues regarding heritage mortar restoration, but lacking implementation due to limited applicable standards put in place during the restoration work, mostly in developing countries, particularly in South Africa. There is a need to emphasise the engineering approach in the design and development of heritage mortar by taking input from various stakeholders. This research has proven the crucial role of engineers in ensuring that new materials are close to original ones in terms of their aesthetic, physical, chemical and mineralogical properties. It has introduced the involvement of engineers in heritage conservation, which was a missing concept in the past.

This gap in limited guidelines that address the specific needs of heritage structures has led to the loss of resources due to incompatible renovation projects that do not last long enough. This chapter presents general conclusions drawn from the reviewed literature and detailed experimental work leading to the development of mortars declared suitable for restoration. Some recommendations for supplementary research in heritage mortar restoration are also outlined in this chapter.

6.1 Specific conclusions

The specific conclusions drawn in this study after conducting the experimental investigations to address the set objectives are outlined in this section. For the 1600, 1700, and 1800s mortars extracted from the Castle and buildings on Robben Island, respectively, their chemical, physical, and mineralogical properties showed whitish hydraulic lime mortars (original) with the addition of seashell fragments. Meanwhile, the repair mortars were greyish, proving the use of Portland cement. Some of the original mortars were earth, as proven by the mineralogical analysis in which kaolinite was present. These mortars were constructed using the feldspar aggregates collected from the Cape Town coast many years ago.

As the reviewed literature suggested, a 1:3 mixing ratio by weight was suitable for developing compatible and durable ready-to-use mortars for heritage restoration. This ratio helped to achieve optimal workability, allowing the mortars to dry at a recommended speed of 5-7 days, as indicated in EN 998-1 (2003). The 1:3 ratio, as opposed to the 1:1 mixing ratio, yielded compatible mortars (duplicating the original) as suggested by Schueremans *et al*. (2011). Additionally, they could resist salt crystallisation and freeze-thaw cycles, indicating their long-term durability against the ageing tests chosen in this study. It is to be pointed out that further study in assessing the mortar durability when applied to surrounding heritage materials such as bricks and stones is yet to be researched. Such a study will assist in evaluating the harmonious integration of new mortars with the substrate materials. Finally, by virtue of using raw materials with similar properties to the original, an extended lifespan of heritage masonries is guaranteed based on achieved compatibility.

6.2 General conclusions

The practice of utilising incompatible materials in the restoration of heritage structures is a widespread issue, leading to adverse consequences that threaten the authenticity of these historical treasures. Such misguided attempts at restoration often fall short of addressing decay problems in historic buildings and fail to achieve essential restoration objectives, which include compatibility, suitability, and longevity. This prevailing trend towards the use of mismatched materials perpetuates the need for recurrent repairs. In order to solve this problem, it is imperative to study the design and development of new mortars for heritage restoration, with compositions and performance that perfectly match the original materials.

The primary objective of the current study was to scrutinise the design procedures for heritage restoration mortars and develop appropriate materials in compliance with established standards, including RILEM, European and South African National Standards where applicable. By characterising the original sub-hydraulic lime-based and earth mortars, invaluable insights were gained to inform the production process of new materials. The findings highlighted the importance of a systematic framework for restoration practitioners, particularly in developing countries where there are insufficient resources for restoration of heritage buildings.

The new repair mortar M4 demonstrated a chemical and mineralogical composition congruent with the original mortar, showing good resistance against freeze-thaw and salt crystallisation ageing tests. Conversely, mortars M6, M7 and M9 quickly crumbled during the ageing tests, even though M9 had the highest compressive strength. An overarching observation was the interplay between durability and the physical and mechanical behaviour of the mortars. The high porosity and water absorption in M7 and M9 mortars were correlated with the increased vulnerability to deterioration induced by salts and freeze-thaw cycles.

The aesthetic aspect generally deserves attention, especially the color, to reduce possible inconsistencies during restoration work. The high values of color difference (ΔE) between the original and new mortsrs underline the need to address this aspect, possibly through color modifications using pigments, if other physical properties are not modified

The aesthetic aspect, especially the colour, deserves attention to mitigate any inconsistencies during the restoration work. The high colour change values emphasise the need to address this aspect, potentially through modifications involving colour-enhancing pigments that bear no effects on the mortar properties besides the colour. For optimal performance within masonry structures, a comprehensive investigation into the interaction between the new mortars and surrounding materials, such as bricks or stone, is necessary to recommend the responsible application of these mortars. The conclusion reinforces that compatibility alone is insufficient if skilled artisans do not meticulously apply the restoration mortar. For this reason, a careful approach to the application of restoration mortars is essential and requires the expertise of skilled workers to safeguard the integrity of these valuable architectural structures. Further research into heritage compatible application techniques for restoration mortars deserves to be explored.

6.3 Novelty and recommendations for South African National Standards: Design and production of heritage mortars

The originality of this study is found in the formulation of guidelines and the provision of standardised solutions for the design and development methods of new compatible restoration mortars of heritage structures, which includes four main aspects, namely:

i) Visual analysis of the heritage building in question
ii) Analysis of the original mortar samples for their physical, mineralogical and chemical properties
iii) Characterisation of the potential raw materials that fit the original and determination of their appropriate ratios
iv) Assessment of the new mortar compatibility (with the original) and their durability based on ageing tests.

The research provides insights into the appropriate design and development of different heritage mortar categories: jointing, plaster, render and floor mortars that are compatible and durable for economic sustainability, whereby repeated repairs resulting from premature repair failures are avoided. A concise methodology encompassing various design standards from European (EN), RILEM Recommendations, North American standards (ASTM) and South African National Standards (SANS), among others, were identified to be suitable for reaching a desired repair mortar for heritage buildings, which is not as straightforward as the design of cement-based mortars.

The results from the analysis of the original material provided a clear search map for repair materials whose compatibility and durability were subsequently evaluated. The guidelines provided in this research can be useful to guide the restoration/conservation teams when executing projects on structures of historic significance in terms of the materials analysis for compatible repairs. Through the conclusions, the research highlights the often-neglected aspect of deploying skilled workmanship and appropriate application techniques for heritage mortars. This study believes that compatible repair mortars would not serve a purpose if applied incorrectly by unskilled masons. It further bridges a gap between the construction materials industry and the design of historic mortars as it highlights, in detail, a route to obtaining the compositional details on heritage binders and aggregates. Thus advising the material manufacturers on the way to supply materials for use in heritage conservation.

6.4 Recommendations for future research

This study evidently showed that it is critical to design and develop sustainable intervention approaches and new mortars to restore heritage masonries with the contribution of various stakeholders, especially the industries. It has also established the need to amend and edit the existing construction standards, as indicated in Section 6.3 of this thesis, to address the specific needs for heritage constructions. The study further highlighted that, even if one designs and produces a compatible and durable mortar for restoration, other factors play a role in the mortar behaviour and its appropriateness, such as: workmanship and interaction with the surrounding materials i.e. bricks or stone. Therefore, research should not be limited to the design of compatible heritage mortars. Still, analysing such mortars in a masonry element requires attention as far as research is concerned. Although the work presented in this thesis gives an extensive and comprehensive study of the design and development of heritage repair mortars, many areas for future research can be identified and suggested to improve the confidence level and reliability of the current results and suggestions.

When combined with poor workmanship, a compatible mortar fails to address the underlying problem of inconsistency and early deterioration in heritage structures. Therefore, a cautious approach is essential when applying restoration mortars, requiring the expertise of highly skilled workers to safeguard the integrity of these precious architectural relics. Further research into the application techniques for compatible heritage restoration mortars warrants exploration with the collaborative efforts and mutual understanding of all relevant stakeholders, not only the historians or heritage practitioners but also the engineers, contractors, conservators, architects, geologists, scientists, archaeologists, heritage authorities, material industries and standards establishing bodies. In terms of heritage material characterisation, the following areas need investigation:

- To investigate the causes of deterioration and processes on heritage materials in order to determine the cause of failure and thus propose improved mitigation strategies based on informed decisions.
- To assess the modern, sustainable, and eco-friendly construction materials for their use in restoration of heritage buildings and potential additives to enhance their properties for compatibility and durability purposes.
- To study the impact of climate change on the degradation of heritage material, especially in big cities with high greenhouse gas emissions, and subsequently, to propose adaptive and resilient restoration materials against climate-induced degradation.
- Introduction of artificial intelligence through innovative techniques for non-destructive testing and monitoring of heritage materials as well as simulative predictions of the heritage repair mortars' lifespan and their ability to withstand deterioration factors.
- Digital documentation, i.e. 3D scanning and modelling of heritage materials, is required for future restoration interventions.
- A development standard for heritage mortar testing and its development may be available in the near future.
- Finally, new materials should be researched to protect heritage materials from deterioration rather than to restore them.

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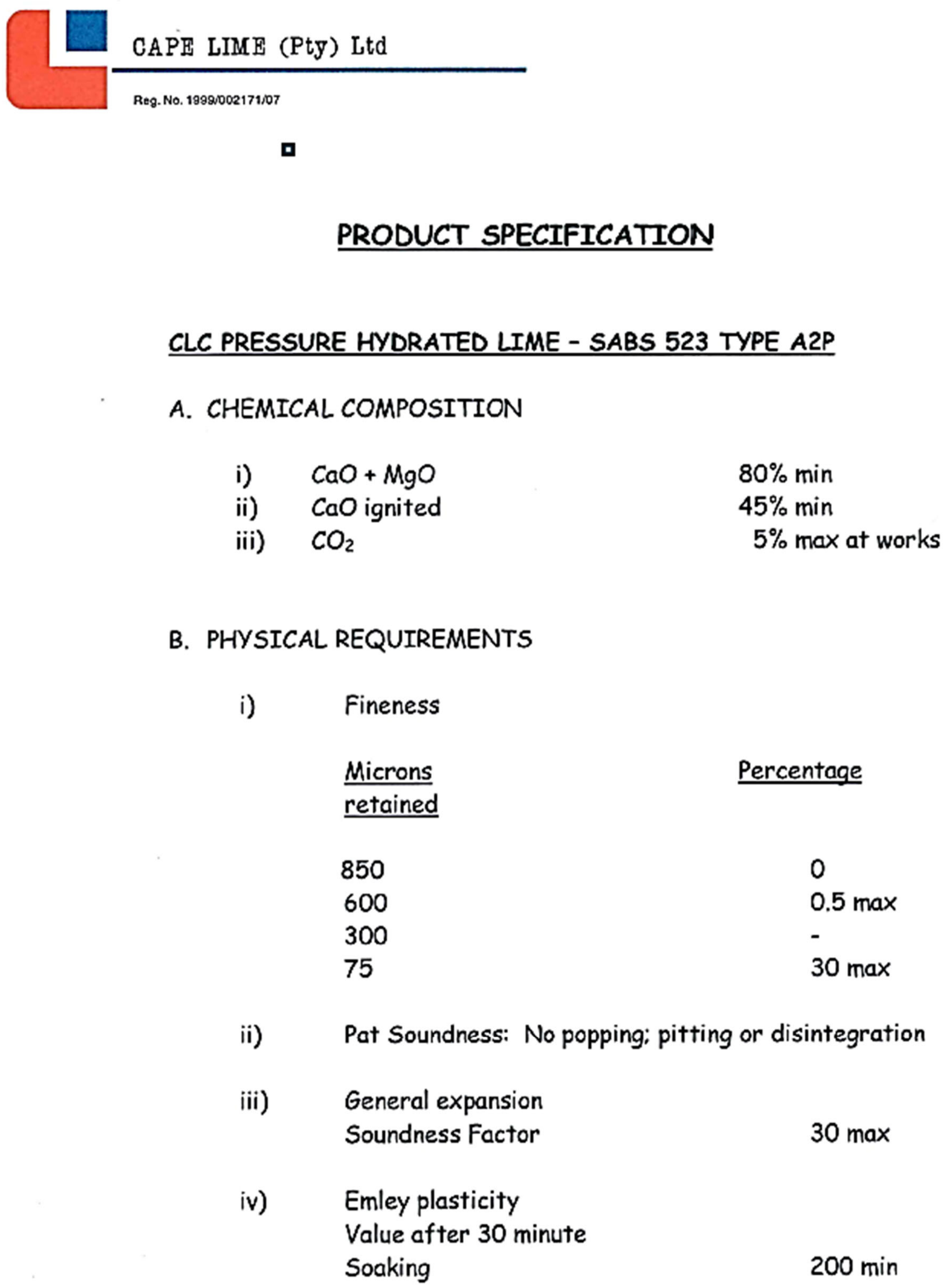
Appendices

Appendix A. Pressure hydrated lime specification

Table A.1 Cape Lime - Hydrated lime specification



CAPE LIME (Pty) Ltd

Reg. No. 1999/002171/07

PRODUCT SPECIFICATION

CLC PRESSURE HYDRATED LIME – SABS 523 TYPE A2P

A. CHEMICAL COMPOSITION

i)	CaO + MgO	80% min
ii)	CaO ignited	45% min
iii)	CO_2	5% max at works

B. PHYSICAL REQUIREMENTS

i) Fineness

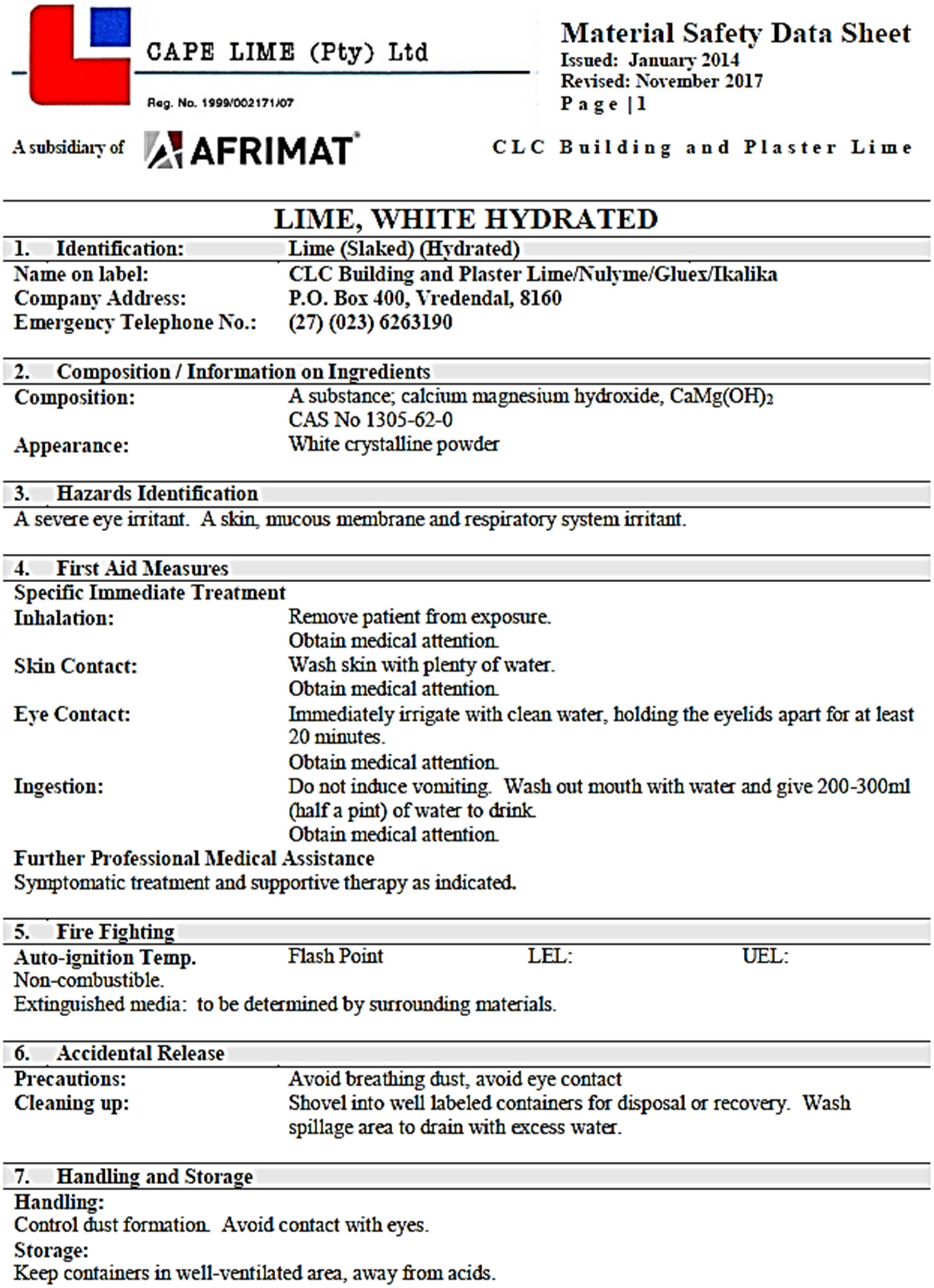
Microns retained	Percentage
850	0
600	0.5 max
300	-
75	30 max

ii) Pat Soundness: No popping; pitting or disintegration

iii) General expansion Soundness Factor 30 max

iv) Emley plasticity Value after 30 minute Soaking 200 min

Table A.2 Cape Lime - Hydrated lime material safety data sheet (MSDS)



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CLC Building and Plaster Lime

LIME, WHITE HYDRATED

1. Identification: Lime (Slaked) (Hydrated)

Name on label: **CLC Building and Plaster Lime/Nulyme/Gluex/Ikalika**
Company Address: **P.O. Box 400, Vredendal, 8160**
Emergency Telephone No.: **(27) (023) 6263190**

2. Composition / Information on Ingredients

Composition: A substance; calcium magnesium hydroxide, $CaMg(OH)_2$
CAS No 1305-62-0
Appearance: White crystalline powder

3. Hazards Identification

A severe eye irritant. A skin, mucous membrane and respiratory system irritant.

4. First Aid Measures

Specific Immediate Treatment

Inhalation: Remove patient from exposure.
Obtain medical attention.
Skin Contact: Wash skin with plenty of water.
Obtain medical attention.
Eye Contact: Immediately irrigate with clean water, holding the eyelids apart for at least 20 minutes.
Obtain medical attention.
Ingestion: Do not induce vomiting. Wash out mouth with water and give 200-300ml (half a pint) of water to drink.
Obtain medical attention.

Further Professional Medical Assistance

Symptomatic treatment and supportive therapy as indicated.

5. Fire Fighting

Auto-ignition Temp. Flash Point LEL: UEL:

Non-combustible.

Extinguished media: to be determined by surrounding materials.

6. Accidental Release

Precautions: Avoid breathing dust, avoid eye contact
Cleaning up: Shovel into well labeled containers for disposal or recovery. Wash spillage area to drain with excess water.

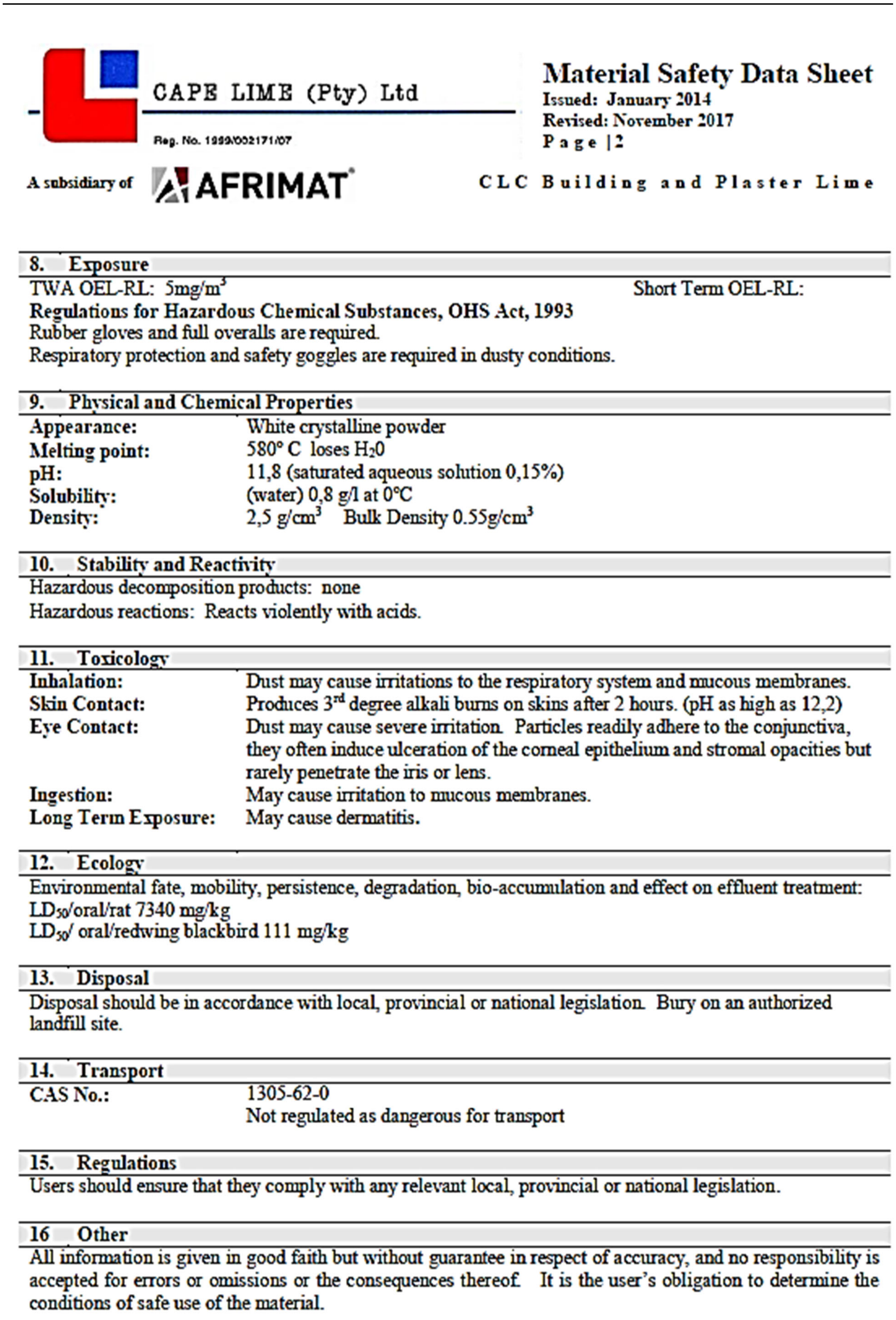
7. Handling and Storage

Handling:

Control dust formation. Avoid contact with eyes.

Storage:

Keep containers in well-ventilated area, away from acids.



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CLC Building and Plaster Lime

8. Exposure

TWA OEL-RL: 5mg/m^3 Short Term OEL-RL:

Regulations for Hazardous Chemical Substances, OHS Act, 1993

Rubber gloves and full overalls are required.

Respiratory protection and safety goggles are required in dusty conditions.

9. Physical and Chemical Properties

Appearance:	White crystalline powder
Melting point:	580° C loses H$_2$0
pH:	11,8 (saturated aqueous solution 0,15%)
Solubility:	(water) 0,8 g/l at 0°C
Density:	2,5 g/cm^3 Bulk Density 0.55g/cm^3

10. Stability and Reactivity

Hazardous decomposition products: none

Hazardous reactions: Reacts violently with acids.

11. Toxicology

Inhalation:	Dust may cause irritations to the respiratory system and mucous membranes.
Skin Contact:	Produces 3rd degree alkali burns on skins after 2 hours. (pH as high as 12,2)
Eye Contact:	Dust may cause severe irritation. Particles readily adhere to the conjunctiva, they often induce ulceration of the corneal epithelium and stromal opacities but rarely penetrate the iris or lens.
Ingestion:	May cause irritation to mucous membranes.
Long Term Exposure:	May cause dermatitis.

12. Ecology

Environmental fate, mobility, persistence, degradation, bio-accumulation and effect on effluent treatment:

LD$_{50}$/oral/rat 7340 mg/kg

LD$_{50}$/ oral/redwing blackbird 111 mg/kg

13. Disposal

Disposal should be in accordance with local, provincial or national legislation. Bury on an authorized landfill site.

14. Transport

CAS No.:	1305-62-0 Not regulated as dangerous for transport

15. Regulations

Users should ensure that they comply with any relevant local, provincial or national legislation.

16 Other

All information is given in good faith but without guarantee in respect of accuracy, and no responsibility is accepted for errors or omissions or the consequences thereof. It is the user's obligation to determine the conditions of safe use of the material.

Appendix B. TGA/DSC results

TGA-DSC Analysis.pdf

Appendix C. ESEM-EDS full results

SK1 report.pdf SK5 report.pdf SK6 report.pdf PS4 report.pdf PS9 report.pdf PS11 report.pdf

Appendix D. Compressive strength calculations

Table D.1 Compressive strength calculations for the new mortars

Mix	Area of cube (mm^2)	Failure load (kN)			Compressive strength (MPa)			Average compressive strength (MPa)		
		28 days	60 days	90 days	28 days	60 days	90 days	28 days	60 days	90 days
M1	1600	2.6	0.4	1.2	1.63	0.25	0.75			
	1600	1.4	0.2	0.4	0.88	0.13	0.25			
	1600	1.0	0.6	0.4	0.63	0.38	0.25			
	1600	1.2	0.2	0.8	0.75	0.13	0.50			
	1600	1.4	0.2	0.6	0.88	0.13	0.38	**0.95**	**0.20**	**0.43**
M2	1600	0.4	0.4	2.2	0.25	0.25	1.38			
	1600	0.4	0.4	1.2	0.25	0.25	0.75			
	1600	0.4	0.6	1.0	0.25	0.38	0.63			
	1600	0.8	0.4	1.0	0.50	0.25	0.63			
	1600	1.0	0.4	1.0	0.63	0.25	0.63	**0.38**	**0.28**	**0.80**
M3	1600	1.6	0.2	0.2	1.00	0.13	0.13			
	1600	0.6	0.4	0.4	0.38	0.25	0.25			
	1600	2.0	0.2	0.2	1.25	0.13	0.13			
	1600	0.8	0.2	0.2	0.50	0.13	0.13			
	1600	1.0	0.2	0.4	0.63	0.13	0.25	**0.75**	**0.15**	**0.18**
M4	1600	0.4	0.6	2.2	0.25	0.38	1.38			
	1600	0.2	0.2	1.4	0.13	0.13	0.88			
	1600	0.4	0.6	1.0	0.25	0.38	0.63			
	1600	1.0	0.4	1.0	0.63	0.25	0.63			
	1600	1.0	0.4	0.8	0.63	0.25	0.50	**0.38**	**0.28**	**0.80**
M6	1600	1.0	0.4	0.6	0.63	0.25	0.38			
	1600	0.6	0.6	1.0	0.38	0.38	0.63			
	1600	1.4	0.6	1.0	0.88	0.38	0.63			
	1600	0.6	0.4	0.8	0.38	0.25	0.50			
	1600	0.6	0.6	1.0	0.38	0.38	0.63	**0.53**	**0.33**	**0.55**
M7	1600	0.4	0.2	0.2	0.25	0.13	0.13			
	1600	0.6	0.2	0.2	0.38	0.13	0.13			
	1600	0.4	0.2	0.2	0.25	0.13	0.13			
	1600	0.6	0.2	0.2	0.38	0.13	0.13			
	1600	0.6	0.4	0.2	0.38	0.25	0.13	**0.33**	**0.15**	**0.13**
M8	1600	0.4	0.6	0.6	0.25	0.38	0.38			
	1600	0.6	0.8	1.2	0.38	0.50	0.75			
	1600	0.2	0.4	0.8	0.13	0.25	0.50			
	1600	0.6	0.8	1.0	0.38	0.50	0.63			
	1600	0.4	0.6	1.0	0.25	0.38	0.63	**0.28**	**0.40**	**0.58**
M9	1600	1.6	2.4	2.0	1.00	1.50	1.25			
	1600	0.8	2.2	1.6	0.50	1.38	1.00			
	1600	1.0	3.4	1.6	0.63	2.13	1.00			

Mix	**Area of cube (mm^2)**	**Failure load (kN)**			**Compressive strength (MPa)**			**Average compressive strength (MPa)**		
		28 days	**60 days**	**90 days**	**28 days**	**60 days**	**90 days**	**28 days**	**60 days**	**90 days**
	1600	0.6	2.0	1.6	0.38	1.25	1.00			
	1600	1.0	2.0	2.2	0.63	1.25	1.38	**0.63**	**1.50**	**1.13**

Appendix E. Flexural strength calculations

Table E.1 Flexural strength calculations for the new mortars

Mix	Failure load (N)					Beam width (mm)			Beam depth (mm)		
	28 days	60 days	90 days			28 days	60 days	90 days	28 days	60 days	90 days
M1	540	120	1320	40	40	0.63	0.14	1.55			
	540	140	1300	40	40	0.63	0.16	1.52			
	540	140	1320	40	40	0.63	0.16	1.55			
	540	120	1300	40	40	0.63	0.14	1.52			
	540	140	1320	40	40	0.63	0.16	1.55	**0.63**	**0.15**	**1.54**
M2	560	180	1220	40	40	0.66	0.21	1.43			
	560	140	1260	40	40	0.66	0.16	1.48			
	540	160	1080	40	40	0.63	0.19	1.27			
	560	140	1280	40	40	0.66	0.16	1.50			
	540	140	1260	40	40	0.63	0.16	1.48	**0.65**	**0.18**	**1.43**
M3	660	160	1400	40	40	0.77	0.19	1.64			
	740	140	1380	40	40	0.87	0.16	1.62			
	720	140	1380	40	40	0.84	0.16	1.62			
	740	140	1400	40	40	0.87	0.16	1.64			
	700	160	1380	40	40	0.82	0.19	1.62	**0.83**	**0.17**	**1.63**
M4	560	180	1220	40	40	0.66	0.21	1.43			
	560	140	1260	40	40	0.66	0.16	1.48			
	540	140	1080	40	40	0.63	0.16	1.27			
	560	140	1280	40	40	0.66	0.16	1.50			
	540	140	1260	40	40	0.63	0.16	1.48	**0.65**	**0.17**	**1.43**
M6	380	320	480	40	40	0.45	0.38	0.56			
	340	320	480	40	40	0.40	0.38	0.56			
	340	320	500	40	40	0.40	0.38	0.59			
	360	300	480	40	40	0.42	0.35	0.56			
	380	320	540	40	40	0.45	0.38	0.63	**0.42**	**0.37**	**0.58**
M7	200	340	720	40	40	0.23	0.40	0.84			
	240	320	720	40	40	0.28	0.38	0.84			
	240	160	680	40	40	0.28	0.19	0.80			
	240	160	700	40	40	0.28	0.19	0.82			
	240	160	720	40	40	0.28	0.19	0.84	**0.27**	**0.27**	**0.83**
M8	560	540	900	40	40	0.66	0.63	1.05			
	520	560	880	40	40	0.61	0.66	1.03			
	520	540	880	40	40	0.61	0.63	1.03			
	560	540	880	40	40	0.66	0.63	1.03			
	560	540	880	40	40	0.66	0.63	1.03	**0.64**	**0.64**	**1.04**
M9	580	720	880	40	40	0.68	0.84	1.03			
	520	780	980	40	40	0.61	0.91	1.15			
	520	780	880	40	40	0.61	0.91	1.03			
	520	780	880	40	40	0.61	0.91	1.03			
	520	780	880	40	40	0.61	0.91	1.03	**0.62**	**0.90**	**1.05**