DEPARTMENT OF CONSERVATION

The Correlation of Workability and Mechanical Strength in air lime binders with different aggregates and pozzolan'

a comparative study

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ABSTRACT

In this study are investigated two air lime binders common in conservation practice. The first one is a commercial air lime, emerging from dry slaking, which was left to age for 4 months. The second is a natural quicklime emerging from the burning in a traditional lime kiln from Mariestad, Sweden. Their workability, compression and flexural strength have been tested in correlation with the addition of rounded natural aggregates, angular (crushed) aggregates and pozzolan. The main aim of this study is to seek for the optimal interaction between the aggregates and these two binders with and without the addition of pozzolan. This study showed that the commercial material was more workable than the natural quicklime and it had inferior mechanical performance. With the addition of pozzolan the mechanical performance was improved for both binders. The natural quicklime from Mariestad, after slaking (hydration) was less workable compared to the commercial material, and the angular aggregates significantly worsened its workability. It became more workable and performed mechanically better with the addition of pozzolan, independent from the aggregates' shape.

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Contents

Foreword and Acknowledgements **9**

Glossary **10**

Introduction **11**

Chapter 1: Background **12**

1.1 Lime mortar; an overview 12

1.2 Pozzolana 14

1.3 Air lime as a binder; the lime cycle 16

1.4 Approaching binders as fluids 16

1.4.2 Aging of Air limes 17

1.5 Aggregates and their granular behaviour in a binder matter 18

Chapter 2: Properties of Mortars **22**

2.1. Fresh Properties: Workability 22

2.1.1 Aggregates influence on Workability 23

2.2 Hardened Properties: Mechanical properties 24

2.2.1 Water and Binder Content influence on mechanical properties 25

2.2.2 Grain size distribution and influence on mechanical behaviour 25

2.2.3 Additives and their relation to strength 25

2.2.4 Curing of Lime mortars 26

2.3 Types of mechanical testing 26

Chapter 3: Research Objectives **28**

Chapter 4: Experimental Procedure **30**

4.1 Experimental Design Overview 30

4.1.1 Materials 30

4.1.2 Experimental Design and Observational Requirements 30

4.2 XRF and XRD Analysis 32

4.2.1 Burnt lime from Mariestad, Sweden 33

4.2.4 Commercial lime hydrate (Murkalk 'Finja') 35

4.3 Selection of aggregate size distribution curve 35

4.4 Aging of commercial air lime 36

4.6 Slaking CaO from Mariestad 37

4.7 Mixtures and Fresh Properties 38

4.7.1 Flow table test 39

4.7.2 Electric Current Measurement while mixing for workability estimation 41

4.7.3 Electric Current and Flow Table test Results 43

4.7.4 Rheometer measurements 44
4.7.5 Observations on the Fresh Properties 45
4.8 Curing of specimens 46
4.8.1 Observations on Curing 47
4.9 Mechanical Strength 47
4.9.1 Results of Mechanical Tests 48
5. Conclusions 50
6. Limits of the study 52
References: 51
List of Figures 56
APPENDIX A 58
APPENDIX B 60
APPENDIX C 61

Foreword and Acknowledgements

During my little, academic mainly, experience in conservation I have come across a chasm between opinions and knowledge about building materials and mortars. At the beginning, my orientation was painting, icon and wall painting conservation, but soon conservation of historic building and building materials got my attention. The reason why I find conservation of architecture interesting is that it is an interdisciplinary field, that combines chemistry, engineering, and mineralogy. Here in the University of Gothenburg, with the opportunity of my postgraduate studies I decided to dig into the development of mixtures for conservation and restoration of historic structures and get an in-depth view on built heritage. With the opportunity of this thesis, I wanted to investigate factors that affect restoration render mixtures. I did my research at the Research Institutes of Sweden (RI.SE) at the department of Material design. I chose to do my research in a research centre because I realised that there is a dichotomy between theory and practice in my profession. However, I cannot also deny that before the industrialisation of binders the craft of masonry with the available local resources used to be more investigated by individual practitioners than it does today (Hansen et al. 2008). These days should be the conservators' duty to develop conservation mixtures and building materials. The study of lime-based mortars could also potentially contribute to the development of more sustainable contemporary building materials in the long run. The aim of this thesis is to set, and hopefully answer a small spectrum of research questions, that can be useful to a professional conservator and set a common ground between conservation practitioners and scientific conservation.

Several people have helped me complete my research and I ought to acknowledge:

I would like first to thank my supervisor Urs Mueller for trusting me to run my tests at the department of Material Design in Borås and giving me free hand to all the equipment and of course for advising me for the entire procedure. I would also like to thank Jonny Eriksson Lecturer at the Department of Conservation of University of Gothenburg who provided me with materials and advice. Patrick Rogers from the department of Environmental Analysis and Construction of RI.SE who provided me with information concerning the electric current test. I owe my gratitude also to all the people who work in the Material Design Department of RI.SE and a special thanks to Emilie L'Opital who helped me with my X-Ray diffraction test and her thorough explanations. To Gilles Plusquellec for his help with the X-Ray Fluorescence test, to Ilias Outras for his help supervising me during my laboratory work, to Ida Gabrielsson for her advice on aggregates and handling of equipment and to Alexander Oliva Rivera who helped me with my rheology tests.

Glossary

Additive: a material added to a mortar mix during mixing to modify certain properties. They can react chemically and mechanically with the materials of a mortar mix.

Aggregate: particles of natural or industrial origin, used as fillers; sand, shell, gravel, fragments of crushed stone etc. They vary in mineralogy, shape, particle size and porosity. They do not react chemically with the binder. Their selection depends on the purpose for which the aggregate is being used.

Air lime: a term used for non- hydraulic lime. Pure air lime only dries and carbonates in the atmosphere.

Binder: the material that binds together all the particles (aggregates an additives), it reacts chemically with the environment

Calcium hydrate or *hydrated lime:* $Ca(OH)_2$ the product after the slaking of quicklime

Fresh properties: the properties that a mortar mix has before the drying, setting or carbonation, the properties that possess when it is still a fluid.

Hardened properties: the properties that the mortar has after the hardening (full hydration and/or carbonation), the properties that has when it is in a solid state and has fully reacted. Depending on the mix these properties can be observed after one month or several months after the preparation of the mixture.

Hydraulic lime: a lime that sets and hardens primarily by the hydration reaction with water.

Portlandite: a hydration product occurring in lime and cements. It is a name for Calcium Hydroxide [$Ca(OH)_2$] or slaked lime used in mineralogy.

Pozzolan: a material rich in silica and sometimes alumina, it appears in form of particles of different sizes and in the presence of a chemical activators, like calcium hydroxide [$Ca(OH)_2$] it reacts chemically to produce a hydraulic set in a mortar. Natural Pozzolans or Pozzolana emerge from silicious volcanic ash.

Quicklime: lime mainly in oxide form (CaO). It is a reactive substance obtained by the burning of limestone in a lime kiln.

Slaked lime: calcium hydroxide $Ca(OH)_2$ is a hydrate of lime, and can be found in a powder or putty form, depending the amount of water used during slaking.

Slaking: the addition of water to quicklime (CaO) in order to be formed a hydrate such as $Ca(OH)_2$

Introduction

The aim of this thesis '*The Correlation of Workability and Mechanical Strength in air lime binders with different aggregates and pozzolan*' was to contribute to the understanding of the compatibility between binders and aggregates from a workability and strength viewpoint. This thesis's position on the topic is from a conservation perspective, so the materials used are binders that are used in conservation practice. Therefore, this thesis begins with a presentation of materials which are common within the conservation of built heritage. The overall scope of the study was the investigation of mortar mixes using engineering tools. These tools can be useful to a conservator in order to give a more complete view on the material properties. Here it should be mentioned that the use of this type of equipment is not possible for every conservation project, but the mechanical behaviour of a conservation- material should be more researched and discussed under mechanical aspects. This research started with literature review that considers fresh binders as fluid suspensions and aggregates as larger particles, which is one way to characterise these materials behaviour within a fresh mortar mix. The methods described were not used in the experimental part (chapter 4). In the experimental section the methods that were used test; consistency, viscosity, rheology and mortar application dealing with the mixtures as homogeneous units. The outcome of this study is on workability, resulted by the binder- aggregate interaction as well as the strength capacity of that interaction.

The binders used were lime hydrates of different origin (which can also be named as slaked limes or air- limes). The air lime binders that were compared were: a commercial lime hydrate and a burnt lime from Mariestad, which was slaked in the laboratory. The mixtures were also tested with the addition of a natural pozzolan from Greece, which gives the mortar hydraulic properties. The aggregates used, were natural rounded aggregates (natural sand) and crushed aggregates, produced by the crushing of stone. Crushed aggregates are not very common in conservation since they are not usually compatible with the historic mixtures, but they are studied in this thesis to be investigated and compared to the rounded ones.

Chapter 1: Background

1.1 Lime mortar; an overview

Lime mortar as a manufacturing material dates back to 12,000 BC in the Near East and as a construction material from 10,000 BC. The lime binder used at those times consisted of air lime, a material that dries and hardens in the atmosphere. Lime mortars at the time were obtained by the burning of limestone. Pure limestone ($CaCO_3$) when it is burnt at 800- 1000ºC, calcines to CaO (quicklime) under loss of CO_2 and is a fragile solid material. Quicklime reacts with water to a hydrate, which is called Portlandite ($Ca(OH)_2$). This is often referred to as hydrated lime, lime hydrate or slaked lime (Kang et al. 2019). The process of hydration is called slaking and releases a considerable amount of heat. Before the industrialised production of lime hydrate, the slaking of the lime created a smooth and malleable creamy putty (Hansen et al. 2008). Modern lime hydrate production is a strongly controlled process and uses a defined amount of water in liquid or steam form to produce a dry white powder. This process is called dry slaking and stoichiometric amount of water used to hydrate the quicklime (Hansen et al. 2008) and (Angelakopoulou et al. 2019).

Fig 1.1 Natural non-commercial air lime from Mariestad. CaO (left), middle while slaking and right $Ca(OH)_2$ putty after slaking (hydration).

Fig 1.2 Commercial air slaked lime $Ca(OH)_2$ in powder form, emerging from dry hydration.

In this thesis the lime binders that were used in order to be compared, using different aggregates are: A commercial dry slaked lime $Ca(OH)_2$ and a burned lime, which was slaked similarly to the historic practices and was used in a putty form. Lime hydrates exposed to the atmosphere react with the CO_2 to form calcium carbonate, this is the hardening reaction of lime mortars. A full carbonation of lime mortars can take from a few months to several decades, depending where the mortar was used (e.g. plaster or masonry mortar) (Kang et al. 2019). Until full carbonation the properties of the mortar change. When mortars include hydraulic or pozzolanic components a different reaction occurs; the hydration reaction, which contributes to the hardening of lime mortars. Hydraulic lime forms stable calcium silicate or aluminate hydrates by hydration and calcium carbonate by carbonation (Muheise-Araalia and Pavia, 2021). In hydraulic lime mortars both reactions contribute to the hardening process. Setting is the process where a mortar transforms from a plastic to a solid (more brittle) state. In cement mortars setting begins usually after 4 to 5 hours (ASTM C191). In the case of lime mortars, however, the setting process is less pronounced for pure lime- based binders, the setting process can best be translated to the drying and solidification of lime hydrate (Da Costa et al., 2018). With hardening, the mortar becomes more brittle but also increases its mechanical strength. For hydraulic materials these procedures can take place in humid environments and even underwater. Air lime, which was the material of interest in this thesis, sets by losing its water content, being transformed from a plastic material into a solid mortar of low strength. If the drying is too fast, the mortar may form cracks. In this study natural hydraulic lime was not used, but a hydraulic mortar was created by mixing with a natural pozzolan. Therefore, hydraulic binders will be defined in the following paragraph.

A lime mortar with hydraulic properties sets and hardens in part through a chemical reaction with water which is called hydration. The degree of hydraulicity increases with the amount of hydraulic components. Hydraulic components are usually reactive silicates or aluminates, mostly in form of calcium silicates or aluminates or in form of reactive silicates such as volcanic glass or calcinated clays (e.g. in brick dust) (Elsen et al. 2012) and (De Matos et al. 2020). In natural hydraulic limes the hydraulicity degree is not only related to the composition, but it is also affected by the burning temperature of the limestone during calcination. Hydraulicity classification was first developed by Louis Vicat in the 1830s. He classified hydraulic limes according to their setting time under water. The European Norm EN 459-1 defines three different types of hydraulic lime. These include the natural hydraulic lime (NHL), where hydraulic components were formed during calcination, e.g. clay containing limestone. Hydraulic lime (HL), where hydraulic components such as natural or industrial pozzolana or Portland cement is mixed into the lime and formulated lime (FL), which contains natural hydraulic lime and/or pozzolana/Portland cement but where the composition of the lime needs to be declared. NHL is produced by burning mostly clay containing limestone (so called marl) (Pánek et al., 2020), resulting in the formation of mostly portlandite, calcium silicates and calcium aluminates (Kang et al. 2019). Depending on the clay content in the marl source NHL can be as hydraulic (ANCADE, 2009). Consequently, NHL sets and hardens both via the hydration reaction during early curing and they gain further strength in the long run via carbonation (Kang et al. 2019). NHL is not universally available, and some of the producers of NHL in Europe are Germany (Otterbein and

Hessler-Kalkwerke), Portugal (Secil), UK (Singleton Birch and Roundtower), France (SOCLI-group, Lafarge and St. Astier), Italy (Tassullo) (Elsen et al. 2012). During the classical Greek and Roman periods natural hydraulic lime was relatively a product of coincidence rather than a designed technological achievement. However, hydraulic properties of lime binders were, in particular during the Roman period, achieved by the use of volcanic earth, first from Pozzuoli near Naples (hence the name pozzolan) and later from other European volcanic areas the Romans occupied (e.g. the Eifel region in Germany). A second pozzolan the Romans introduced were calcined clays in the form of brick dust and brick aggregate. Brick was the Roman staple of building materials and were produced in vast amounts, together with also large amounts of too low fired bricks, which showed an activation of the clays but not sufficient compressive strength. After the Roman Empire declined, the knowledge of using hydraulic lime in Europe vanished and was not rediscovered until the end of the middle ages (Mark and Hutchinson 1986).

Lime binders though, with hydraulic properties were produced by mixing lime with pozzolans (clays, volcanic ash, slag fragments, charcoal) (Elsen et al. 2012). Later other types of hydraulic lime were developed; either by burning impure limestone (rich in clays) or by adding during the burning process clays into the limestone (Elsen et al. 2012). As mentioned before, the hydraulic properties are depending on the amounts of calcium silicates and calcium aluminates in the mortar. A classification of hydraulicity was suggested by Boynton (1980) and classifies hydraulic limes according to their chemistry with a cementation index (CI), which lead to a classification in feebly, moderately and eminently hydraulic. In the European Norm EN 459-1 the natural hydraulic, hydraulic and formulated lime is categorized into strength classes: NHL/HL/FL2, NHL/HL/FL3.5 and NHL/HL/FL5, with 2, 3.5 and 5 MPa minimum strength after 28 days of curing, respectively. In order to enable the hydration of clinker phases or enable a pozzolanic reaction, hydraulic lime needs to be cured under humid conditions. That means after applying a lime plaster or render, the materials needs to be protected by wet burlap and plastic sheets. Drying out of the mortar too early stops any hydraulic or pozzolanic reaction, which may result in too low strength. Generally, there are no rules for the curing time of hydraulic mortars. Lime with natural hydraulic components, such as dicalcium silicate or calcium aluminate cure under humid conditions faster than lime with pozzolana, which react slower. However, the time needed for finishing any hydraulic or pozzolanic reaction depends on the amount of clinker phases or pozzolana and the environmental conditions but is generally faster than the carbonation reaction (Benmohamed et al. 2016).

1.2 Pozzolana

Pozzolana took their name from the city of Pozzuoli in Italy where many quality varieties of volcanic ash are found (Dodson, 1990). Natural pozzolana were used during the Roman period in mortars for masonry and plasters and as well as a key ingredient in Roman concrete (Mark and Hutchinson, 1986). There is evidence of Pozzolana use since the Aegean Middle Bronze Age (2000-1500 BC). The Romans though, developed the technology and systematised the use of Pozzolana. Volcanic ash is a natural pozzolan, that is a product of explosive volcanic activities, where magmatic components together with bedrock are finely dispersed in the air and afterwards deposited over large areas surrounding a volcano (Schneider et al. 2011). Pozzolana were used throughout the centuries mostly in lime binders but with the advent of Portland cement in the 19th century and beginnings of the 20th century there were also utilised in binder systems based on Portland cement. In the late 20th century cement companies used more and more industrial and natural pozzolana for lowering the cement clinker content to create the so- called blended cements whose production releases less CO_2 than pure Portland cement (Santana-Carrillo, et al. 2021) (Snellings et al. 2012). Some industrial pozzolana are: Fly ash (from combustion of coal) and silica fume (from smelting silica) (Khayat and Aïtcin, 1992) (Mounjouohou et al. 2019). Blast furnace slag from smelted or pig iron, it is also called latent hydraulic component (see below). Pozzolana are materials rich in silica and sometimes alumina, they appear in form of particles of different sizes and in the presence of a chemical activators e.g. $Ca(OH)_2$ and moisture can react chemically resulting to a stable calcium silicate hydrate (C-S-H) or calcium aluminate silicate hydrate (C-A-S-H) (Pardal et al. 2009) (L'Hôpital et al. 2016). When portlandite $Ca(OH)_2$ reacts with a pozzolan rich in silica, e.g. with SiO_2 and water the following reaction may happen:

$$3(CaO \cdot H_2O) + 2SiO_2 + nH_2O \rightarrow 3CaO \cdot 2SiO_2 \cdot nH_2O \text{ (C-S-H)}$$

(Justs et al. 2015)

Latent hydraulic materials, e.g. ground granulated blast furnace slag or some high calcium fly ashes, do not need an activator to start the hydraulic reaction (Suraneni et al. 2019). However latent hydraulic materials react for the most technical applications too slow and therefore they are in many cases used with an activator, e.g. Portland cement, gypsum, or alkali components.

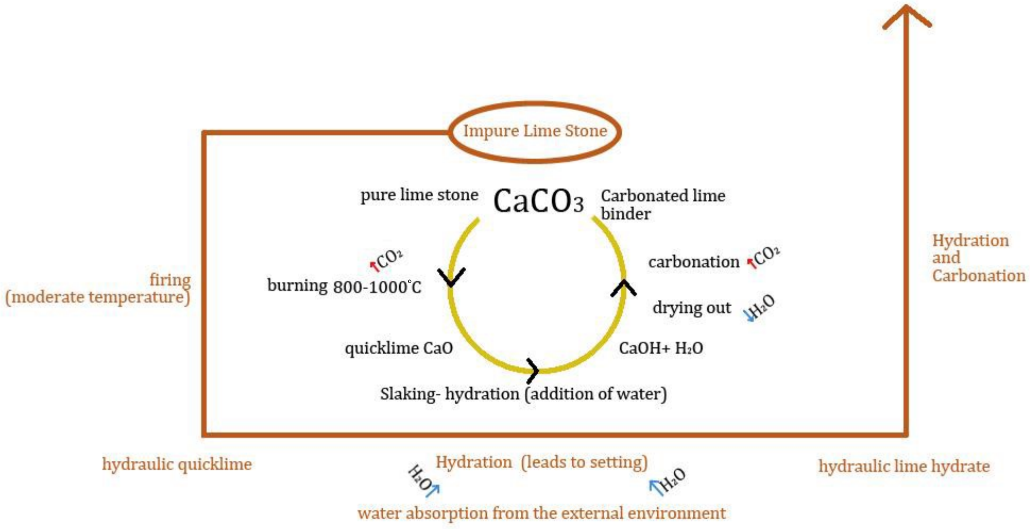

Fig 1.3 Illustration inspired from the book *'Building Limes in Conservation'* by Brocklebank Ian, 2012 p. 11

1.3 Air lime as a binder; the lime cycle

Binders are both adhesive and cohesive materials found in either solid or liquid form. When solid binders are mixed with a liquid (usually water) they turn into a malleable putty. Industrial mortars can be found in silos, in bags and can also be found wet ready to use in bags or containers. Air lime can be calcium lime or dolomitic lime. Calcium limes emerge from the calcination of pure limestone. Dolomitic limes come from the calcination of dolomitic limestone or dolomite, containing magnesium and will not be further discussed. After limestone is quarried it needs to be burned in order to turn into a reactive material. With burning, limestone loses its CO_2 and turns into CaO (quicklime), a substance that is highly reactive with water. The process of slaking refers to the reaction of calcium oxide to calcium hydroxide. Slaked lime is any air lime mainly in hydroxide form, obtained by the hydration (or slaking) of quicklime (Henry and Stewart 2011). The slaked lime, as mentioned, is lime hydrate. It can be in the form of a dry powder or a putty depending on the amount of water used for slaking (dry or wet slaking). Air lime is not able to set and harden underwater. The industrial calcium lime hydrate is classified in EN 459-1 according to its chemical composition starting from the most to the least reactive: CL 90-S, CL 80-S or CL 70-S. The most reactive air lime is the one with high concentration of free lime CaO (ANCADE, 2009). Binders mixed with aggregates form mortars. Mortars are defined as a mix of one or more inorganic or organic binders, aggregates, fillers, additives and/or admixtures (EMOdico[1]).

1.4 Approaching binders as fluids

Fluids can be liquids, gases, and plasmas. Solutions, colloids and suspensions also fall under the category of fluids, but the size of their particles is what differentiates them from each other (Tab. 1.1). A suspension is a heterogenous mixture of particles with diameters of above 0.5 µm that are distributed throughout a second phase (examples: paint, blood, hot chocolate, aerosol sprays). A colloid is also a heterogenous mixture, but the particles of a colloid are typically smaller than those of a suspension, generally in the range of 2-500nm (Ruiz-Agudo and Rodriguez-Navarro, 2010).

Types of fluids	**Particle size**	**Characteristics**
solution	-	Tthey do not separate out when calm/ do not scatter visible light
colloid	2- 500 nm	Scatter visible light, translucent or opaque/ non filterable/ do not separate standing
suspension	> 500 nm	Cloudy or opaque/ filterable/ separate out when standing

Tab. 1.1. Categories of fluids in relation to their particle size and natural properties.

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[1] EMO, European Mortar Industry Organisation, online dictionary, accessed October 2020, https://www.euromortar.com/emodico/en/show/conveyor-systems

From a mechanics perspective, fluids can be Newtonian or non-Newtonian. Newtonian fluids are those that generally have a low molecular weight and they experience shear stress that is linearly correlated to the strain rate. According to Bagnold (1954) the common case of high concentration of large solid spheres in a Newtonian fluid is a phenomenon occurring on the frontier between rheology and hydrodynamics. However, polymeric liquids, suspensions, pastes slurries and other composite materials show a yield stress and are not characterised as Newtonian but as Bingham fluids (Bird et al. 2002). In physics, a fluid is a substance that continually deforms (flows) under an applied shear stress, or external force (Boháč and Nečas 2016). Lime putties can be described as Bingham fluids; these types of fluids have a constant viscosity independent of shear rate but need to overcome a yield stress until they flow. Other non-Newtinian fluids contain macromolecules or finely ground solids and their viscosity can change to either a more liquid or solid form depending on the shear rate. When a fluid with increasing shear rate turns less viscous it is described as a shear thinning or thixotropic. On the contrary when it turns more viscous it is a shear thickening or anti-thixotropic.

1.4.2 Aging of Air limes

Aging of calcareous slaked lime stored under water has been recognised as a successful method that improves the quality of air lime (slaked lime) (Balksten, 2007). Studies' results vary when the portlandite crystal ($Ca(OH)_2$) development under storage in water is investigated. In some cases, Calcium Hydroxide aggregations turn into larger portlandite crystals, a fact which contradicts studies that have shown reduction of size over time. The creation of plate- like nano- portlandite crystals contributes to an increase of surface area. The particle shape also influences the properties of the lime putty. This structure has an improved capacity for water absorption, and this contributes to better plasticity (Rodriguez-Navarro 1998).

The reason why aging improves the quality of lime putties is still a matter of debate. The aging turns portlandite which has more prismatic crystals to sub-micrometre more plate like crystals and later to nanometre sized plate like crystals (Mascolo et al. 2010) (Ruiz and Navarro, 2010). This can enhance the lime putty with the desirable viscosity and plasticity or can lead to unwanted aggregation. In their study, Ruiz and Navarro (2010) conclude that the high burnt lime has the optimal rheological properties immediately after slaking. The smaller the particle size of the portlandite crystals the higher the surface area, which will enhance the carbonation of the lime putty. This applies to high burnt limes immediately after slaking where the plate- like crystals are mostly nano- aggregated already. The optimal qualities worsen quickly for high burnt lime, after slaking yet recover partially after longer periods of aging. On the contrary, in the case of soft burnt limes the rheology improves with long- term storage in water. The majority of portlandite crystals of soft burnt limes, are prismatic non-colloidal with fewer nano- aggregated colloidal plate- like crystals. In the case of vapour slaking (commercial materials) the aggregation is reversible and aging benefits the material.

1.5 Aggregates and their granular behaviour in a binder matter

Aggregates are granular mineral materials used for mortars and concrete. They derive in most cases from natural resources and do not contribute to the hardening reaction of a mortar (EMOdico[1]). For mortars often more or less rounded aggregates were used deriving from fluviatile or glacial deposits (Gutierrez, 2013). They are used to lower the amount of binder in a mortar but also to improve physical and mechanical properties of a mortar or concrete (e.g. shrinkage). Natural aggregates are quarried from their natural environment, often riverbanks, lacustrine or maritime deposits. The quality of the aggregates depends on their mechanical properties, their durability against corrosion, the morphology of their granules, and the amount of harmful components which can impair the durability of a mortar. The compatibility between binder and aggregates is also a topic that needs to be investigated in more detail. Naturally available sands contain fine grained materials in the form of silt ($2\mu m<r<63\mu m$), and clay components ($r<2\mu m$) (Assallay et al. 1998). Clay minerals such as smectite in the clay fractions can show pozzolanic behaviour within a lime binder, but their pozzolanic activity is usually insignificant (Moon et al. 2019). Fine particles though are associated with mortar strength but mainly because they act as fillers, increasing the density of the mixture (Moon et al. 2019). High packing density emerging from the presence of significant amounts of fine particles is not a necessary condition for mechanical strength. Especially in the case of air lime, higher amounts of fine aggregates can increase the water demand of the mortar mix during blending (Ince et al. 2015).

Granular flow is a topic that has been under research quite recently and is associated with workability due to aggregates presence in mortar mixtures. The main approaches to the topic are the discrete and the continuum descriptions (Ottino, 2000)[2]. An interesting work conducted by Hendrickx 2009, attempts to study and define workability of lime mortars, associating workability with the granular behaviour of aggregates. The solid particles in a mortar collide with each other repeatedly, and the momentum of one particle is transferred to the other. The momentum travels from a region of high velocity to a region of low velocity as similarly heat also travels from a region of high temperature to a region of lower temperature (energy flow) (Bird et al. 2002). This transfer of kinetic energy leads to rotational and translational motion which leads to flow patterns. Consequently, migration of suspended particles from a region of high shear rate to a region of low shear rate has been reported (Wallevik, 2003). Meaning that the aggregates do not comply with their shear thinning carrier's (binder) behaviour. Quantities that are relevant to the movement of granules in a fluid are: pressure, shear stress and strain, velocity, and viscosity.

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[2] Ottino, J.M (2000). [Review of the book *Sands, Powders, and Grains: An Introduction to the Physics of Granular Materials, by* J. Duran] *J. Fluid Mech*, *419*, 345-347. Retrieved from Cambridge University Press Journals Online.

Here are some definitions related to stress that should be defined:

- Stress (N/mm^2 or MPa): Is the restoring force of a body subjected to a deforming force, equal in magnitude, but opposite direction; tensile and compressive (longitudinal)
- Strain (m): Is the measure of how much an object is stressed or deformed
- Shear stress: The force is applied parallel to the cross section of the material
- Shear thinning: fluids become less viscous when shear stress is applied
- Shear thickening: fluids become more viscous when shear stress is applied

(Bair, 2015) and Brilliant.org (retrieved 22/02/2020)

Particles in a fluid matter can be investigated as the subject of granular mechanics. Due to this perspective, materials with both solid and fluid nature can be investigated not only as one rigid fluid, but as a suspension that is made of discrete entities. The aggregates in a mortar as particles are the subject of Dynamics; a branch of classical mechanics that deals with the accelerated motion of a body. Dynamics are both the kinematics and the kinetics of a particle. Kinematics refer to the motion of a rigid body with a specific shape and they furthermore study the geometric aspect of motion. Kinetics focus on the causes of motion and deal with the object of interest as a particle (volume- less mass) (Kim A. and Kim H.J, 2017). Particle dynamics in classical mechanics covers the range from the molecular level to larger particles such as the aggregates of a mortar.

In general, the granules that are distanced from each other are moving with greater freedom. However, when the effects of grain inertia dominate, turbulence takes place depending on consecutive instant collisions between layers of granules. In other words, the faster moving layer occupies the space of the slower moving layer (Bagnold, 1954). Bagnold also states that the effects of the fluid's viscosity can be dominant and influence the phenomenon differently. Bernoulli's principle relates pressure to two phenomena; if a fluid moves faster, the individual particles will spread out more, decreasing the pressure between them. The other idea is that if the fluid above decreases (reduction of apparent pressure), the particles experience less pressure thus are less compacted. The scale of observation of some phenomena and parameters which the material is considered as continuum, should be further defined. For example, the local density, according to Hendrickx, (2009), is a parameter which if it is not examined at an appropriate scale then the density and concentration of components can vary, but also the rate of deformation. Movement of particles is studied in fluid mechanics through stimulation methods in various scales such as conventional molecular dynamics (MD), Brownian Dynamics, Stokesian dynamics, Dissipative Hydrodynamics and the Discrete Element Method (DEM).

Mortar mixtures for different applications need to meet different workability requirements. For example, fresh mortars for plaster and render applications should be able to flow easily, but they should also rapidly behave as solids, once applied to a vertical wall (Fourmentin et al. 2015). Other mortars need to be flowable over a longer time period, e.g. when grouting a masonry infill. Mortars need to be balanced between the finer and larger particles as well as the amount of liquid in order to avoid separation and sedimentation. Lack of compatibility between aggregates and binders negatively affects the mechanical phenomena on the interface of the grain and the binder matter. Other early age phenomena related to compatibility and workability can also be related to durability properties (Hendrickx, 2009). Properties such as rheology and water transport in solid matter can affect workability, in addition the interface of the mortar mixture with other surfaces (Hendrickx, 2009). The interaction with other surfaces especially for render and plasters is also very much affected by the way of application. Aggregates such as sand grains, gravel or crushed stones are predominantly affected by gravity (Bergström, 1997), so when the shear rate is not high during application, that can lead to uneven distribution. Plasticity, water retention, soundness and sand carrying capacity and aggregate porosity are also properties that should be considered in order to define workability. Porous aggregates can lower workability (Matar et al. 2019), reduction in slump has been recorded by Tareq et al. (2020), after the use of porous aggregates. However porous aggregates can reduce the shrinkage and consequently enhance the mechanical properties (Jochem et al. 2020) after hardening.

Granular mechanics is the theoretical framework for aggregate behaviour and colloid[3] physics is the field that can explain lime particles behaviour (Hendrickx, 2009). Particles are discrete entities with individual density, size, shape, surface, roughness and porosity (Pavía and Toomey, 2008). Consequently, these characteristics can lead to different particle behaviours, for example, particle sedimentation, clogging, arching, bridging, rat-holing, and segregation (Blais, 2019). Cohesion is also a characteristic of liquid granular matter. When a binder- aggregate system is oversaturated cohesion can be lost (Mitarai and Nori 2006). According to Mitarai and Nori (2006) there are four stages associated with cohesiveness depending on the degree of moisture:

- the pendular state: the particles collide to each other with bridges of liquid at their interface point
- the funicular: some pores are filled with liquid but there are still areas left where there is trapped air
- the capillary: all the gaps between the particles are filled with water
- the slurry: all particles are soaked in liquid

Summarising: density, shape and size of aggregates, water retention and aggregate porosity are some of the macroscopic phenomena that affect workability relevant to the aggregates. After hardening, they influence the mechanical properties of the mixture as well. This study aims to focus on such macroscopic phenomena and seek for an optimal connection between different aggregates and binders.

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[3] Colloid: two phase systems of which one has at least one dimension between a nanometre and a micrometre

Chapter 2: Properties of Mortars

2.1. Fresh Properties: Workability

An empirical definition of workability could be the combination of factors which determine the degree of easiness in application of a mortar mixture, as determined by a mason (Hendrickx, 2009). The definition of workability in literature is commonly qualitative and sometimes confused as a function of workmanship (Narayan et al. 2016). According to the European Mortar Industry Organisation, workability is '*the sum of the application properties of a mortar which give its suitability (the ease with which a mortar may be used)*'. Workability, more in depth, depends on phenomena relevant to properties of the components of the mortar and the mixing ratios of those components (Hendrickx, 2009). Workability can be explained within the theoretical framework of rheology. Rheology is defined as the theory of deformation and the flow of matter in a liquid, gaseous or plastic state (Fig 2.1). The physical parameters that define rheology are stress, strain, rate of strain and time (see chapter 1.5) (Narayan et al. 2016).

Workability can be described with the following terms: plasticity, consistency, cohesion, adhesion, water retention, time of set, weight and penetrability (Hendrickx, 2009) and (Kampff, 1961).

Plasticity: Ability of solids to deform permanently under an applied force (EN 206-1).

Consistency: Describes the plasticity state of the mortar including its workability, pumpability and its applicability: e.g. stiff consistency or flowable consistency of a mortar (EN 206-1).

Cohesion: The property of mortars to keep all particles suspended, even after removal of the shear stress, thus, to resist segregation and 'bleeding' (Pacheco-Torgal et al. 2015).

Water retention: The ability of a fresh (hydraulic) mortar to retain its mixing water when exposed to substrate suction or evaporation (EMOdico[1])

Weight: overall the particles of a suspension are affected by gravity (Bagnold, 1954). Local density and concentration of particles can vary at different areas of the mixture (formation of clusters) and that can also influence the rate of deformation (Mitarai and Nori 2006).

Setting time: The time after which the mortar begins to harden. After this time the mortar is normally stable in the presence of water (EN 1015-4)

Overall rheology is the most distinguishable parameter found in the literature that mainly affects workability. Rheology is the branch of science that deals with the deformation of the materials' flow. For Newtonian fluids rheology is only affected by viscosity, which is a time-dependent property for a specific temperature, and it is not affected by strain. In the case of non-Newtonian fluids, the viscosity changes when the strain rate changes. A classic example is ketchup which reduces viscosity when shaken. Rheology is the study of materials with such nature. The flow of a lime or cement mortar can be described as a Bingham fluid. Bingham fluids are non-Newtonian fluids that have the tendency to behave as a fluid at a higher stress with a constant viscosity, but as a rigid body in lower stress (Roussel et al. 2006). For a mortar to behave as a fluid the applied stress should overcome its yield stress, this phenomenon was first explained by Bingham (1879- 1945) who was a professor at Lafayette College of Indiana.

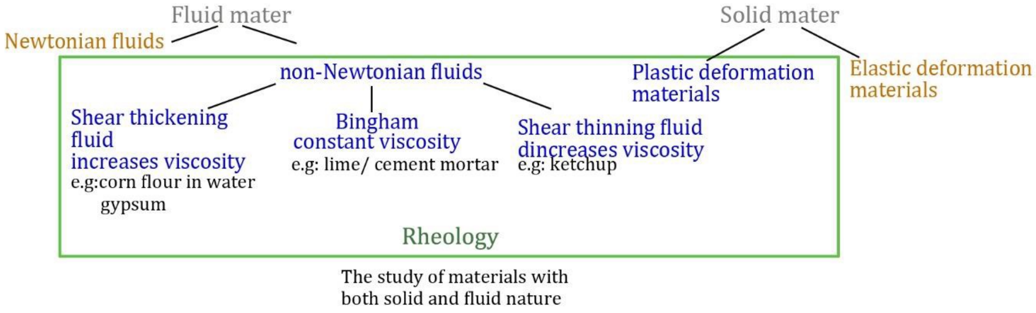

Fig 2.1. Materials that are characterised by rheology.

Mortars are non-Newtonian Bingham fluids, but they are also affected by chemical phenomena, like setting and hardening. So, from a fluid mechanics perspective; they are Bingham fluids, but from a chemical point of view the fluid properties change over time due to the setting and hardening of the binder. Within the time span, where a mortar is applicable (open time), it should not show significant changes of the workability properties. The rheology test on mortars should take place within the workable life of the mortar, usually 15 minutes after mixing (Mortar Industry Association, 2013).

2.1.1 Aggregates influence on Workability

The aggregate size distribution significantly affects the mechanical properties of a mortar and its workability. Historically it is suggested that a broad aggregate size distribution enhances both aforementioned properties. Aggregates influence the quality of a mortar blend by their size distribution, shape, density, porosity, water absorption capacity and their mechanical properties (Pavía and Toomey, 2008). The size distribution is usually described by the median size d_{50}. The median refers to the diameter where half of the sizes are smaller than this value and half are larger (HORIBA, Technical Note, 2009). Size, shape and porosity of aggregates are features that affect purely the workability (Pavía and Toomey, 2008) (Matar et al. 2019). Porous aggregates require a pre-wetting or excess water in order not to dry- out the mortar mix (Zhang et al. 2020). Furthermore, porous aggregates have in many cases lower mechanical strength compared to dense aggregates.

In non-Newtonian fluids the relation between viscous stress and strain rate can be extremely non- linear and viscous stress may even depend on other features of the flow besides strain rate (Wang et al. 2012). When the kinetic energy is high the collisions create flow patterns which are influenced by the collisional interactions, the lubrication force and gravity (Wang et al. 2014 and Wang et al. 2012). Inelastic collapse takes place at high particle number densities and the kinetic energy is significantly lower (Wang et al. 2012). There are three types of liquid- solid local flows: the dense phase that characterizes the clusters (high particle- density areas), the dilute phase outside the clusters, and the interaction phase that is in contact with the clusters and interacts with them (Wang et al. 2014).

In conclusion, the movement of aggregates in a binder is affected on a smaller scale by particle-particle, particle- fluid interactions and collisions that lead to migration of particles and rotational movement. On a larger scale, more dense areas (clusters) interact with less dense areas creating flow patterns. The aggregates are affected by gravity and they tend to move from areas of high shear rate to areas of lower shear rate, this behaviour can potentially be regulated by a wide range of aggregate sizes and optimal compatibility between aggregates and binders.

2.2 Hardened Properties: Mechanical properties

The mechanical strength of a mortar is part of the hardened properties of the composite material and an important performance indicator (CIVL University of Memphis 2019). The most significant components of a mortar mix are the qualities of the binder, its water/binder ratio, the aggregates and the ratio between the binder/aggregate. However, there are other parameters that influence the mechanical properties of a mix, such as water content, curing conditions and aggregate size distribution. The mechanical properties of mortars are crucial performance parameters for building masonry, in particular compressive strength, modulus of elasticity, poisson's ratio[4] as long as flexural, tensile and adhesive strength (Marques et al. 2020) (Gercek, 2007). Depending on the application (e.g. masonry mortar, pointing mortar, plaster) one or the other mechanical properties have a higher significance respectively. Mechanical properties of mortars define a large portion of the compatibility between old and new materials. Many compatibility related damages in historic masonry are related to the use of repair mortars with the wrong mechanical properties (e.g. a cement mortar with a too high modulus of elasticity in combination with building blocks of much lower stiffness). Mechanical tests measure the properties in a standardised way but are usually designed for cement mortars. Lime mortars show a different setting and hardening behaviour and the measuring times and curing procedures are less well described as for cement mortars. In Europe in many cases the test procedure is according to EN 1015-11 and performed on prisms of 160 x 40 x 40 mm^3. First the prisms undergo a flexural 3-point bending test and afterwards the half prisms are subjected to a compressive strength test. For the modulus of elasticity there are no standards, but tests procedures are usually performed on prisms or cylinders by measuring not only the load but also the strain during the test (Marques et al. 2020). Tests can be done by single loading or by applying load cycles to e.g. 1/3 of the maximum load. If the strain is measured not only parallel to the loading but also perpendicular, the Poisson's ratio can be determined (Mohamad, et al. 2018).

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[4] It is the elastic constant, known as Poisson's ratio. The measure of deformation of a material in directions perpendicular to the direction of loading.

2.2.1 Water and Binder Content influence on mechanical properties

There is a relation between mechanical properties and porosity. In porous materials mechanical strength depends on the porosity. The higher the porosity the lower the strength. In mortars, porosity is controlled by the water/ binder ratio. The higher the water binder ratio, the higher the number of capillary pores, the lower the strength (Lanas et al. 2003). Another factor is the curing conditions. High relative humidity (97±10%) contributes to hydration of lime-based binders compared to laboratory ambient conditions (RH 50±10%, 23±3°C). Garijo et al. 2019 conclude that the curing conditions in chambers enhance mechanical properties.

Lanas et al. 2003 noticed good results to the mechanical properties in laboratory mixtures with high binder content, however in unregulated conditions a flowable mortar could increase shrinkage, during drying. Eventually the strength properties can decrease (Kassimi and Khayat, 2020). On the other hand, compressive strength may not be the ultimate performance value for mortars; the modulus of elasticity also has a significant role. In particular in masonry mortars, lower elasticity moduli (E) are often preferable. High E-modulus can build up stress between mortar and stone during temperature and moisture fluctuations in the masonry and that can prove harmful for the masonry stones/units. This is often seen in repairs of masonry with cement mortars, where a too high E-modulus-mortar damages stones with lower E-modulus.

2.2.2 Grain size distribution and influence on mechanical behaviour

In the case of historic lime-based mortars the historic literature review of (Pavía and Toomey, 2008) concludes that it is generally accepted that the strongest mixtures are produced with the use of clean and sharp aggregates and a broad particle size distribution. The grain size distribution is a very important attribute in relation to aggregate characteristics (Garijo et al. 2019). Most of the literature agrees that a broad size distribution enhances the mechanical properties and workability. Angular aggregates exhibit better interlocking behaviour than round particles (Garijo et al. 2019). The mixtures that contain a wide size range of aggregates increase the bulk density and consequently the mechanical strength of the mortar. In addition, Pavía and Toomey (2008), suggest that flexural strength increases with the angularity of the aggregates' in conjunction with a wide particle size distribution. Studies have shown that a wide particle size distribution including larger coarse aggregates improves the durability of mortars compared to ones with similar binders and water content but smaller aggregates (Pavía and Toomey, 2008). A variety of small aggregates increases density. When density is increased the porosity decreases, however this does not necessarily lead to improved strength. Mixtures rich in small aggregates require more water. More water increases the water/ binder ratio and that negatively affects the bulk density, leading to shrinkage (Garijo et al.2019) (Pavía and Toomey, 2008).

2.2.3 Additives and their relation to strength

Additives like pozzolans or other hydraulic components, like brick dust, are substances that can increase strength. Thanks to their small particle size pozzolans can behave also as fillers because they tend to fill the voids of relatively larger grains (Ponikiewski et al. 2013).

2.2.4 Curing of Lime mortars

Lime mortars require a long time to carbonate and therefore acquire their maximum mechanical capacity. A strong increase in strength of mortar mixtures after 365 curing days as compared to 28 curing days had been found (Lanas et al. 2003). Even hydraulic mixtures it is possible to require more than a month to harden, mobility of Ca^{+2} ions have been reported during the secondary formation of portlandite during the hydration reaction (Amenta et al. 2020). The adequate ratio of $CaCO_3$ to $Ca\ (OH)_2$ (portlandite) had not been reached even after 365 days of curing, in some cases. When the portlandite carbonates completely it is succeeded a porosity decrease and therefore a strength increase can be observed. It has been shown that compressive strength doubles itself from 28 to 365 days for lime binders (Lanas et al. 2003). The literature agrees that the EN 1015-11 European standard for mortar testing is applicable for mortars with hydraulic binders than pure lime ones (Drougkas et al. 2016). Drougkas et al. 2016 suggest that the increase in mechanical strength of air lime and hydraulic lime mortars is significant long after 28 days compared to Portland cement samples. Lucia Grijo et al. 2019 started the mechanical tests on NHL after 56 days of hardening. This is due to the fact that carbonation and hydration of pozzolana takes more time than just 28 days (Villca et al. 2021).

2.3 Types of mechanical testing

There are many types of mechanical testing but the most common and the ones that were applied in this thesis are the compressive strength and the flexural strength tests according to EN 1015-11.

According to EN 1015-11 compressive strength is performed on cubes. For this, prismatic specimens (40×40×160 mm) are subjected to flexural strength tests. The two remaining halves of the prisms are used for compressive strength tests with 40 x 40 mm^2 square stamps. Cube samples are optimal for absolute values of compressive strength. If strain should be determined, prismatic or cylindric specimens of 2:1 length: diameter ratio is a better option. Strain is measured by strain gauges parallel to the loading, this test results to stress-strain curves. From those the E-modulus can be determined. For a more accurate determination of the modulus of elasticity a cyclic loading is preferable. Compressive strength values measured on prisms and cylinders with higher than 1:1 length: diameter ratio show lower compressive strength than cube samples. Here should be noted that strength of mortar specimens is affected by the moisture content and to a lower degree, the temperature. It is paramount to store samples in a climate room until mass constancy prior to the test, in order to ensure comparable equilibrium moisture contents in the specimen.

Tensile strength is not standardised for mortars. Instead it is tested indirectly by measuring the flexural or splitting strength. Splitting can be measured with the use of two circular dowels perpendicular to the direction of casting and the flexure can be measured with the three or four-point bending tests (Fig 2.2). However, there are methods that can estimate tensile strength. 10N/s could be too high if very low strength air lime mortars are tested under flexural strength (Drougkas et al. 2016).

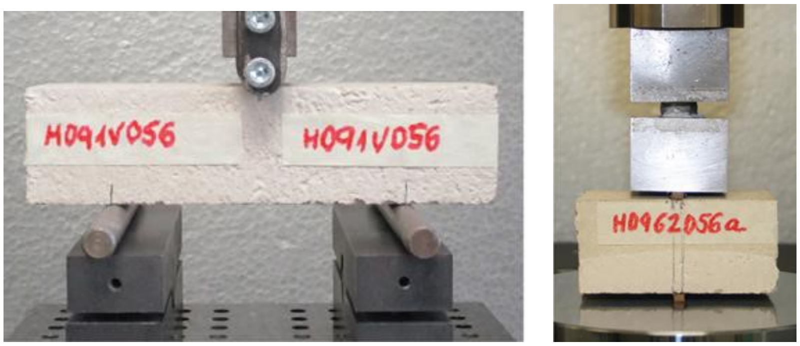

Fig 2.2. Three-point flexure test (left) and splitting test (right) (archive of Garijo et al. 2019).

For mortars the type of loading during a test is relevant. If just standard tests are performed a load- controlled mode is preferred. That means during the test, the cylinders of the testing machine increase the load gradually with a constant loading rate. Most tests require that the time of failure is between 50- 100 s. This means that the loading rate in N/s has to be adjusted accordingly. If stress- strain relations should be investigated e.g. for modulus of elasticity, a strain- controlled loading is preferable (Gong, 1995). In this case a strain gauge at the specimen, controls the loading and loading rates are adjusted in mm/s.

Chapter 3: Research Objectives

Fresh and hardened properties of lime mortars are not studied to the same depth in the scientific literature as cement mortars. Tests to study the fresh properties of cement mortars are not always suitable for the study of lime mortars. The flow table test is a very common method that tests the consistency of a mixture, but even in the European Standard EN 1015-3 the consistency is a measure of fluidity and/or wetness and the results give an estimation of plasticity when the fresh mortar is subjected to a vertical force. These results are not necessarily associated with the behaviour of a mixture when used by a craftsperson (EN 1015-3). Consistency is dependent on the material's yield stress and density which are both affected by chemical composition, particle specific gravity and particle size (Clayton et al. 2003). Some mortars are flowable and deform mostly by gravity and others are plastic and need additional force to deform. A mortar can be flowable when it has high binder content and less aggregates, this however can lead to shrinkage during drying. Some factors that affect shrinkage are: the w/b, elastic modulus and the curing environment. In addition, the lime mortars have higher air content, therefore the target values for cement are not ideal for workable lime mortars (Clayton et al. 2003). Shrinkage affects negatively the mechanical performance of a mortar (see chapters 2.2.1, 2.2.2). These mortar mixtures of this study were all flowable.

The aim of this research though was not to establish new methods or tools for the workability measurement, the reason the above are mentioned is because they constitute the framework of this research. Understanding the concept of fresh mortar properties from a scientific point of view leads to more critical evaluation on the outcomes resulting from the standard practices. This study targeted the research of fresh lime binder properties associated with consistency- workability and their interaction with different aggregates. The second aim was the comparison of the fresh properties with the hardened properties of the selected lime binders. The binders that were compared were commercial aged lime hydrate paste and fresh- slaked lime putty emerging from burning in traditional kiln. Previous studies aiming the research of hydrated lime paste and slaked lime putty, have been conducted by Rodriguez-Navarro et al. (1998), Ruiz-Agudo et al. (2010) and Balksten (2007).

In this research were studied the fresh and hardened properties of lime mortars in association with aggregate shape and pozzolanic activity. The fresh properties that were studied are the consistency and viscosity of the fresh mixtures. In this study, these parameters were considered as responsible for the workability of the mixtures. The present work did not research all workability related parameters like consistency, viscosity, fluidity and deformability separately. The fresh and hardened mortar properties of this experimental research were regarded as a system of factors: the aggregate shape, the pozzolanic activity and binder interaction with the two. The interactions of the materials mentioned were researched as a homogenous system. The hardened properties were studied similarly and conclusions that attempt to associate fresh properties with the hardened ones were presented.

More particularly this research aimed to the following goals:

- The effect that has the aggregate shape on fresh and hardened lime mortar performance indicators.
    - Analysis of the two binders' consistency (commercial aged hydrated lime pastes and fresh slaked lime putty) (see chapter 4.7)
    - The influence degree that the different aggregates have on the two binders tested respectively.
- The effect of pozzolan on consistency/workability and strength for the two binders.
- Additionally, which of the two binders is more influenced by the presence of pozzolan.
- The tracking of electric current of the mixer, during the mixing of each mixture as a valid non-standard practice to estimate workability.
- The effect of pozzolanic activity during the curing of the specimens.
- Association of fresh properties with strength.
- Association of pozzolanic quality with strength.

The parameters investigated were the workability and strength in relation to different binders and aggregates' shape. The main goal of this study is to be shown and discussed the main factors that influence workability and strength and can be controlled by a conservator. The mechanical qualities and chemical consistency of building materials, it is crucial to be investigated because different materials require different handling to show their optimal performance.

Chapter 4: Experimental Procedure

4.1 Experimental Design Overview

This experimental study took place in the facilities of the Cement Lab of the unit Material Design at RI.SE Research Institutes of Sweden, in Borås. The research focused on the investigation of performance parameters between two different binders and two different types of aggregates, with and without the addition of a natural pozzolan.

4.1.1 Materials

The binders used were Finja Murkalk Lime Hydrated (aged for 4 months) and non-commercial lime, hydrated from CaO burnt in a traditional lime kiln at Mariestad (the hydration took place in the lab 2 days prior to the trial tests). The pozzolan used was a volcanic ash from Greece which is used mostly in concrete as a natural pozzolan under the trade name Micrasil' (produced by IMERYS Minerals). The main hypothesis is if the shape of aggregates influences the performance of mortars (workability and strength) with the aforementioned binders (see chapter 3). The binders differ in hydraulicity degree, hydration kinetics, chemical consistency, and microstructure. These variables effect the binder's fresh and hardened properties, so it is expected that these two different lime binders interact differently with the aggregates and the pozzolan. The two types of aggregates used, were: Sand with well-rounded aggregates and 0/2.8 mm in size, composed from different sources and crushed, very angular aggregates with 0/2.8 mm in size. The aggregates were tested with the binders mentioned and with and without pozzolan for each binder.

4.1.2 Experimental Design and Observational Requirements

The entire process took 6 months to be completed. It started the 16th of June of 2020 and was completed the 4th of January 2021 (Tab. 4.1). Prior to the tests took place the Aging of the commercial $Ca(OH)_2$. Followed the obtainment of adequate amount of aggregate sizes for the tests and the slaking (hydration) of the CaO of Mariestad. After that, trial tests took place before the actual procedure to be minimised possible errors concerning the water content of the mixtures. For the workability estimation, the flow table test and the electric current tracking method occurred the same days the mixtures were prepared for casting. The viscometer test took place 2 weeks after casting for the same ratios of materials. After the workability tests had taken place, the curing of the specimens followed and finally the mechanical tests.

Time Frame	
Aging of Commercial slaked lime (Släckt Murkalk E FINJA)	16/06/2020- 06/11/2020 (4.5 months)
Aggregate size distribution determination and collection of aggregates	July- August 2020
Trial Tests for the estimation of water content	September- October 2020
Slaking of Mariestad's CaO	30/10/2020
Preparation and Casting of samples	November 2020
Curing	Nov 2020-Jan 2021(50-57 days)
Mechanical tests	04/01/2021

Tab. 4.1. Experimental procedures within a six months period.

Tests and equipment used		
Procedure	Equipment and Models	Laboratory
Binders composition	Benchtop Powder X-Ray Diffraction (XRD) Instrument. 2D Micro-XRF M4 Tornado, Bruker	Chemistry Lab
Aggregate size distribution	Sieves with different mesh sizes from 0.063mm to 2.8 mm	Concrete Lab
Aging	Mortar mixer of the company VMI, model: PH6105V and plastic buckets (emsafe 14.4l, Lagan Plast)	Concrete Lab
Slaking (Hydration)	Plastic bucket and a hoe	Concrete Lab
Preparation of mixtures	Mortar mixer: Toni Technik model 6214.10/EN	Cement Lab
Slump test (consistency)	Hand- driven Flow Table (254 mm) by Humboldt MFG	Cement Lab
Electric current Test	Clamp Ammeter 400A, BILTEMA	Cement Lab
Casting	Steel molds 40×40×160 mm	Cement Lab
Curing	Chamber 93% RH 19ºC and 50% 20ºC	Cement Lab, Curing NTP
Rheology	Viscometer (Viscomat XL, Schleibinger)	Cement Lab
Mechanical Tests	Compression Testing: Model 2580-301, Instron. Flexural Strength testing: Toni Tecknik manual.	Concrete Lab

Tab. 4.2 The experimental procedures and the piece of equipment used respectively.

4.2 XRF and XRD Analysis

As a first step the starting materials were characterized according to their chemical and phase composition. This step was significant because burnt lime obtained from Mariestad was calcined from a highly impure limestone with inclusions of non-calcite minerals. These clusters were orange-brown and there were also some dark brown-charcoal black coloured clusters that showed a significant hardness. This material was burnt in a traditional lime kiln; therefore, the temperature distribution within the kiln was not homogenous, which explained the variation in shades. In total three samples were analysed in detail: Two from the Mariestad burnt lime (CaO): one from a dark area (charcoal black) and one from a lighter area (creamy white). The third sample was a commercial industrial lime hydrate (Finja murkalk). Energy dispersive X-ray fluorescence analysis (XRF) was used for analysing the chemical compositions of the limes. The instrument used was a Micro-XRF M4 Tornado by Bruker. The phase composition was determined by an X-ray powder diffractometer (XRD) by Rigaku with a copper tube and a silicon drift fast detector. The result diffractograms underwent a semi-automatic search for phases stored in the ICDD database. The phase content was estimated from the height of the peaks associated to a phase.

Fig. 4.1: The samples ready to be tested in the XRF and XRD. The samples were ground into a fine powder.

Oxides	**Mariestad dark areas (ML D)**	**Mariestad light areas (ML L)**	**Industrial lime hydrate (LH)**
SiO_2	19.4	5.0	1.2
TiO_2	0.96	not detectable	0.04
Al_2O_3	6.60	1.39	0.56
Fe_2O_3	5.77	1.17	0.48
MnO	0.40	0.60	0.03
CaO	62.80	91.0	95.9
MgO	0.86	0.62	1.19
Na_2O	3.07	0.12	not detectable
K_2O	not detectable	not detectable	0.15
SO_3	Not analysed	Not analysed	Not analysed
CO_2	Not analysed	Not analysed	Not analysed
H_2O	Not analysed	Not analysed	Not analysed

Tab. 4.3: Chemical composition of the Mariastad lime (MS D, MS L) and the industrial lime hydrate (ILH). The values are weight-%).

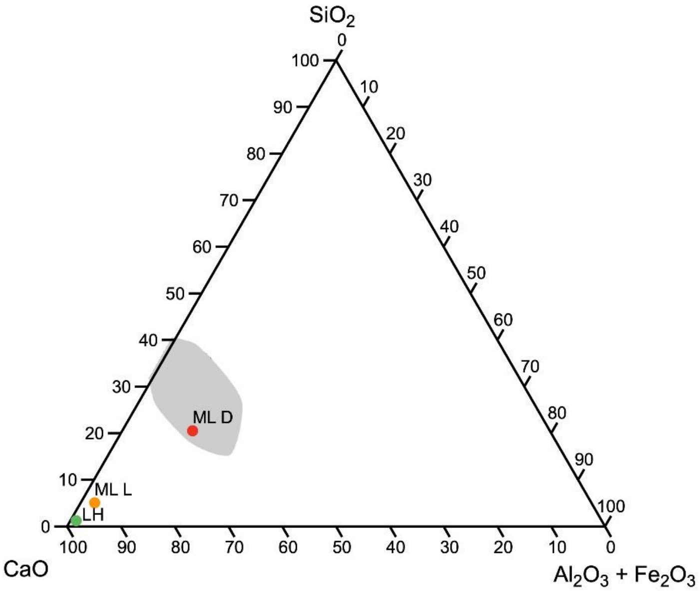

Fig. 4.2: Ternary diagram with the chemical composition of the Lime from Mariestad and the industrial lime hydrate. Given is also the approximate chemical composition of modern Portland cements as a comparison (grey shaded field).

Phase	Formula	**Mariestad dark areas (MS D)**	**Mariestad light areas (MS L)**	**Commercial lime hydrate (ILH)**
Calcium Oxide	CaO	+++	+++	-
Portlandite	$Ca(OH)_2$	+	±	+++
Larnite	Ca_2SiO_4	±	±	-
Gehlenite	$Ca_2Al(AlSiO_7)$	+	-	-
Calcite	$CaCO_3$	-	±	-
Magnetite	Fe_3O_4	±	-	-
Periclase	MgO	-	-	±
Quartz	SiO_2	±	-	-
Augite	$(Ca,Na)(Mg,Fe,Al)(Si,Al)_2O_6$	+	-	-

Tab. 4.4: Phase composition of the different limes from XRD results. The estimated amounts were derived from the peak height of the individual phases of the XRD patterns (see appendix). Relative amounts: +++ = abundant; ++ = intermediate; + = low; ± = traces; - = not found (see appendix A).

4.2.1 Burnt lime from Mariestad, Sweden

One sample has been extracted from a dark area, which represent clusters of impurities (Fig. 4.3) and one from the lighter areas, which represented the bulk of the material (Fig.4.4). These clusters of dark coloured impurities occur frequently. The sample also showed magnetic properties. The chemical composition of both is shown in Table 4.3 and Figure 4.2, the phase composition in Table 4.4.

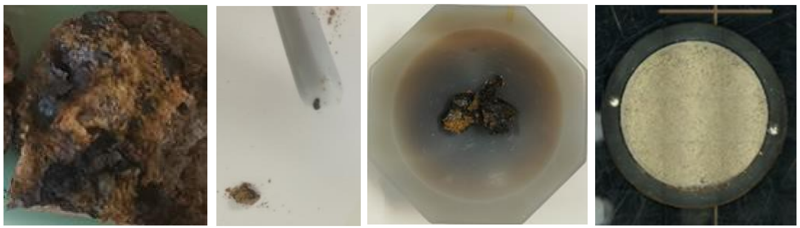

Fig. 4.3: Lime from Mariestad – Dark area with magnetic properties (2nd image from left).

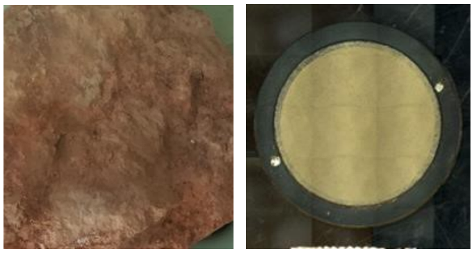

Fig. 4.4: Lime from Mariestad – Lighter areas.

The dark impurities contained calcium oxide as main phase (Tab. 4.4) with small amounts of portlandite and augite, as well as traces of larnite, gehlenite and magnetite. Portlandite was formed from hydration of CaO with humidity from the air. The original limestone of Mariestad is a sedimentary rock, rich in heavy minerals derived from neighbouring metamorphic and igneous rocks. Sweden is part of the Fennoscandian Shield, an area of old crystalline and metamorphic rocks. This is also demonstrated from the presence of pyroxenes in the form of augite and oxides in the form of magnetite, which resisted weathering and were sedimented when calcium carbonate was precipitated by microorganisms from the water. The ferromagnetic properties derive from magnetite. The high silicon and aluminium/iron oxide content derived partially from augite and quartz but possibly also from clay impurities, which would have been calcined together with the calcium carbonate and rendered amorphous and therefore undetectable by XRD. This is also visible in the higher amounts of silicon dioxide in Figure 4.2. However, the results have to be interpreted with care.

The light areas consist predominantly of calcium oxide with only traces of larnite, portlandite and calcite. The lime binder from Mariestad as shown by its composition, can be characterised as feebly hydraulic with hydraulic inclusions.

4.2.4 Commercial lime hydrate (Murkalk 'Finja')

The results indicate that the material is pure Portlandite $Ca(OH)_2$ with traces of Periclase (MgO). Periclase was still in the oxide form because it takes a long time for MgO to hydrate and turn into $Mg(OH)_2$ (Tab. 4.4).

Fig. 4.5: Commercial lime hydrated in powder form the company Finja (Murkalk 'Finja').

4.3 Selection of aggregate size distribution curve

For the performing the experimental procedures an aggregate sieve curve was defined for both, the angular and rounded aggregates. By using only one sieve curve it was possible to study the impact of the grain shape of the aggregate on fresh and hardened mortar properties. The aggregates used have a 0.06/2.8 mm sieve curve with a minimum particle size of 0.063 mm. Historical mortars or renders/plasters show a large variety of aggregate size distributions, usually within the size range of 0/4 mm, which is in line with the chosen particle size. The NORMSAND with a 0/2 mm grain size was not used, because it has a greater amount of very fine particles than the presented curve and that could result to aggregation of the finer particles to the bottom of the moulds due to the fact that the binders that are used have a long setting period. The sieve sizes that were used in mm, were: 0.063, 0.125, 0.250, 0.50, 1.00, 1.60, 2.00 and 2.80 (Tab.4.5). It was used a combination of natural (round) sands 0.063/2.80 their commercial names are: Råda sand 1-2 mm, Brogård 0.8- 1.2 and Brogård sand 55KT. The crushed aggregates were obtained after sieving particles sizes 0.063-2.80 mm from available material in the lab 0-4 mm.

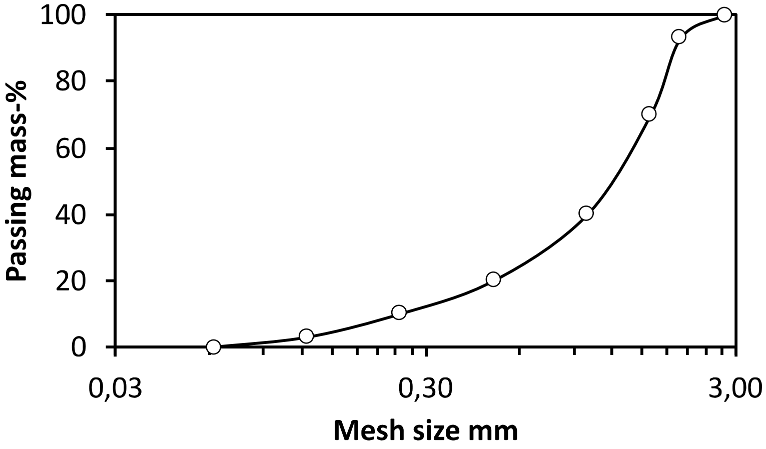

Fig. 4.6: Aggregate size distribution curve used.

Characterisation	Mesh size (mm)	% w/w	Total Passing %
Fine-medium coarse	2.80	0	100
Fine-medium coarse	2.00	7	93.00
Fine-medium coarse	1.6	23	70.00
Fine-medium coarse	1	30	40.00
Fine	0.5	20	20.00
Fine	0.25	10	10.00
Fine	0.125	7	3.00
Fine	0.063	3	0.00
Pan	0	0	0.00

Tab. 4.5 Aggregate size distribution

4.4 Aging of commercial air lime

The commercial lime hydrate was used for aging. In the aging process the lime hydrate was stored under water for an extended time. Usually, the lime improves its workability properties compared to a lime hydrate, which is mixed directly from the bag. The mixer used for the procedure was a large-scale mortar mixer of the company VMI, model: PH6105V.

During the aging, the lime putty enhances its plasticity. These qualities can consequently lead to improved mechanical properties (Rodriguez-Navarro 1998). The mechanical and workability results were compared to the Mariestad specimens (which was not aged). The main differences of these two binders were their burning temperature, reactivity, and purity.

The following describes the preparation for aging the industrial lime hydrate:

- A w/b (water/binder) ratio of 1.5 was used. The value was arbitrary and based on the water demand of the hydrate powder to create a smooth putty.
- 5 kg of lime hydrate were placed into a mixing bowl and 7.5 kg of water were added during the mixing of the putty. The mixing time was around 15 minutes (Fig. 4.7a, b). During this time the mixer was stopped 2 two times and putty, which was sticking to the walls of the mixing bowl was scraped into the bulk of the putty.
- After the mixing the bowl was emptied into a bucket. After that, a 10 cm water layer was placed carefully above the lime putty without disturbing the putty itself (Fig. 4.7c, d).
- After that, the bucket was closed with a lid and the putty was stored for 4 months.

The putty was stored for 4 months, but after three months some of the material was used for trial tests.

After the storage time of four months, the water content of the putty was determined again. In order to estimate the free water content of the aged lime, it needs to dry out at 105ºC and calculate the mass difference before and after drying. For this, three samples were taken from the bottom middle and top of the putty in the bucket. The putty samples were transferred into empty ceramic crucibles. The empty crucibles were weighed prior to the procedure. Crucibles and putty samples were then dried until mass balance in a drying oven at 105 °C. Afterwards the crucibles and dried putty samples were weighed again and from the different readings the water content was determined. There were taken 3 samples from the binder putty and were then dried out in 3 different ceramic containers. So, they had given 3 different water percentage values (see appendix B). The mean value of the three results determined the water binder ratio.

a b c d

Fig. 4.7: Preparation steps for aging lime putty.

The water content analysed in the aged putty was equal to the initial w/b ratio of 1.50 when the water was added to the dry binder. Previous studies indicated a settlement of the $Ca(OH)_2$ particles during aging, thus reducing the w/b ratio of the putty. Other effects of aging are broadening of the size distribution of the portlandite crystals and change of crystal morphology (Mascolo et al. 2010). However, change in w/b was not observed in this case. This could be due to a too short aging time or a because of the use of industrial lime hydrate. Most of the studies on lime aging was performed on slaked quicklime.

4.6 Slaking CaO from Mariestad

As mentioned before (see chapter 1.1) limestone loses during burning at 900- 1000 ºC CO_2 and turns from $CaCO_3$ into CaO, which is a solid highly reactive substance. In most cases it is used in mortars in hydrated form as portlandite $Ca(OH)_2$. The hydration is done with liquid water and the result is a malleable putty (Swedish Standard SS 134003). For this study, ca. 5 kg of CaO were hydrated with 6 kg of water with a w/b = 1.2 and a total amount of lime putty of 11 kg. Prior to the slaking, a trial slaking of a smaller amount of material took place with a w/b = 0.9. But when the trial mixtures were made workability was inferior and the addition of water was necessary to be achieved the desirable workability. Therefore, the amount of water was increased during the slaking of the lime. The slaking was done in the following steps:

- Quick lime aggregates were crushed to sizes below 20 mm.
- The water was added gradually by spraying it into a plastic trough while the material was constantly worked with the hoe, dissipating any excess heat. Quick

lime is very reactive and safety measures were taken in order to avoid overheating and splashing of lime putty[5].

- After slaking, the trough with the lime putty was sealed with plastic foil and left for two days to cool down.

Fig. 4.8: a. Part of the necessary equipment. b. Slaking of the quick lime in the trough. c. Covering the trough and cooling the putty.

4.7 Mixtures and Fresh Properties

Eight mixtures were prepared and tested on the flow table and for each of those mixes, 3 specimens were prepared testing mechanical properties. The curing time was from 50 to 57 days. The series ML refers to 4 mixtures of fresh slaked lime[6] from Mariestad with and without a pozzolan (P). And the series ALH refers to the aged commercial lime hydrate, the commercial 'Finja' murkalk with and without pozzolan as well (Tab. 4.7). The used aggregate types consisted of round and crushed ones described before with the sieve curve shown in Figure 4.6 (chapter 4.3).

Binders and Additives origin
Lime Binders
Quick lime from Mariestad calcined in an experimental field kiln and slaked two days before used for sample mortars
Commercial lime hydrate, aged for 4 months (Finja murkalk)
Pozzolan
Volcanic ash from Greece, consisting predominantly of glass, trade name 'Micrasil' by IMERYS

Tab. 4.6: Binder components.

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[5] A dust mask, protective goggles/clothing and chemically resistant gloves are very important during this procedure, since it is a highly exothermic reaction where, if no precautions are taken, the heat generated can boil up the water and highly alkaline putty can splash to nearby persons, causing burns or eye injuries.

[6] the mixtures were made 2 days after the slaking

Name	Description
ML-R	Mariestad's fresh slaked lime with round aggregates
ML-C	Mariestad's fresh slaked lime with crushed, angular aggregates
MLP-R	Mariestad's fresh slaked lime with 20% w/w pozzolan and round aggregates
MLP-C	Mariestad's fresh slaked lime with 20% w/w pozzolan and crushed, angular aggregates
ALH-R	Aged commercial lime hydrated with round aggregates
ALH-C	Aged commercial lime hydrated with crushed, angular aggregates
ALHP-R	Aged commercial lime hydrated with 20% w/w Pozzolan and round aggregates
ALHP-C	Aged commercial lime hydrated with 20% w/w Pozzolan and crushed, angular aggregates

Tab. 4.7: Abbreviations for the mortar series.

For the mortar mixes a binder: aggregate ratio (b:a) of 1:5.5 by mass was used. This is a common ratio amongst practitioners which usually results in adequate workability of the mortars. The binder part referred to the dry binder, not the putty. In the case of slaked quick lime from Mariestad the amount of CaO was used for the calculation and in the case of the commercial lime hydrate the amount of powder was used. For the calculation of the w/b ratio the total water content was accounted for. This included both, the amount used during slaking/aging and the added water content during mixing the mortar series. The w/b ratio of the slaked lime was 1.2 and the w/b of the aged lime hydrate 1.5. The mortars were mixed by adding the lime putty and aggregate together in a standard mixer bowl. The mix was then blended in a standard mortar mixer for ca. 2-3 minutes and then more water was added, which was followed by ca. 15 minutes more mixing.

4.7.1 Flow table test

Flow table tests were performed with a mortar flow table and a Hägermann cone. Tests were performed according to EN 1015-3 directly after mixing. For an adequate workability a diameter between 170 mm and 190 mm was the desirable. However, as it is shown in the Table 4.8 some values were over that limit, but the mixtures demonstrated the desirable workability as plaster during application on a test wall (Fig 4.9c).

Figure 4.9 shows set-up for the flow table test and the final stage of the test, where two diameters perpendicular to each other were measured and the average value of the two represented the final result. Figure 4.9c shows two trial mortars after their application to a test wall. The test mortars were flicked onto a brick wall with a triangular trowel. The mortars showed good adhesiveness towards the brick material. In order to prevent premature drying-out of the mortars, the wall was covered with a plastic foil.

Fig. 4.9: a. and b. Set-up for the flow table test. c. The mortar applied to a brick test wall.

Name	w/b	b/a	Agg (g)	Pozzolan (g)	Lime Binder dry (g	Water slaking/aging (g)	Water added (g)	Lime Putty (g)	Final w:b	flow table
ML-R	1.2	1/5.5	1350	0	243	291.6	110	534.6	1.65	216mm
ML-C	1.2	1/5.5	1350	0	243	291.6	110	534.6	1.65	177mm
MLP-R	0.96	1/5.5	1350	48.6	194.4	233.28	90	427.7	1.33	178mm
MLP-C	0.96	1/5.5	1350	48.6	194.4	233.28	125	427.7	1.47	175mm
ALH-R	1.53	1/5.5	1350	0	243	371.8	30	614.8	1.65	210mm
ALH-C	1.53	1/5.5	1350	0	243	371.8	30	614.8	1.65	204mm
ALHP-R	1.22	1/5.5	1350	48.6	194.4	297.4	40	491.8	1.38	206mm
ALHP-C	1.22	1/5.5	1350	48.6	194.4	297.4	55	491.8	1.45	201mm

Tab. 4.8 aggregated data of mixture ratios and flow table test results

The b:a ratio as well as the content of the pozzolan was constant for all mixtures. However, the water content varied in the pozzolanic mixtures since the water content is closely related to the consistency of the mortar, but of course other factors such as aggregate shape/size and type of the binder influences the water demand to make a workable mortar. Different mixtures with equal water content will give very different consistencies. During the trial tests w/b ratios between 1.4-1.6 were deemed to be appropriate. However, when the trials were tested on a brick wall only mortars with a w/b = 1.6 showed good performance. The aggregated data of the slump test in relation to the water content and aggregate shape, are presented in Table 4.9. In pure lime mixtures, where the water content was more predictable, it was decided to keep the w/b at 1.65. Indeed, that worked well for the ML-R and the ALH-R and ALH-C. The ML-C gave lower consistency value, but still within the desirable spectrum. The pozzolanic mixtures were judged to have lower water content since pozzolans enhance the fluidity of the mixtures (see chapter 1.2). The mixtures MLP-R and ALHP-R have similar water content values, but they give quite different consistency results. The same happens in the case of MLP-C and ALHP-C.

Binder Type	w/b	Flow Table Result (mm)	Shape of aggregates
Lime Binder			
ML-R	1.65	216	round
ALH-R	1.65	210	round
ML-C	1.65	177	angular
ALH-C	1.65	204	angular
Lime Binder and Pozzolan			
MLP-R	1.33	178	round
ALHP-R	1.38	206	round
MLP-C	1.47	175	angular
ALHP-C	1.45	201	angular

Tab. 4.9: The w/b ratios for the different mortar mixes.

4.7.2 Electric Current Measurement while mixing for workability estimation

The electric power that the mixer consumes during blending of a mortar can be used as indication for its consistency. However, this method is not a standard practice, but it can give indicative results, though interpretation must be done carefully. One of the simplest set-ups for this method is the use of a current clamp (Fig. 4.10).

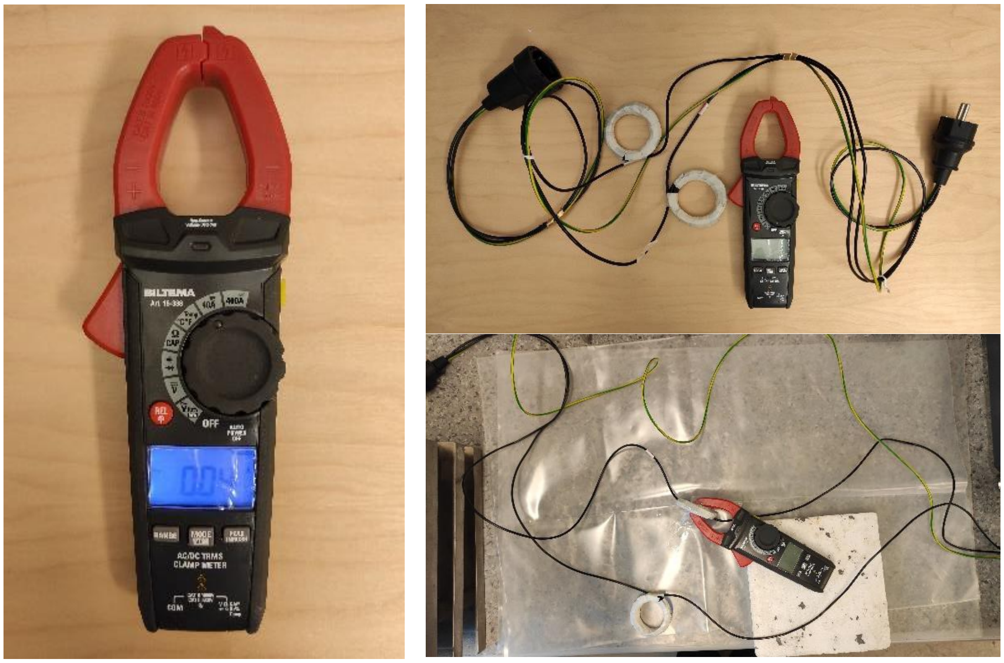


Fig. 4.10: Current clamp and experimental setup.

The use of an uninterruptible power supply (UPS) is optional, but preferable, because it would minimise the error (Fig 4.11). However, the UPS setup was not available for this project.

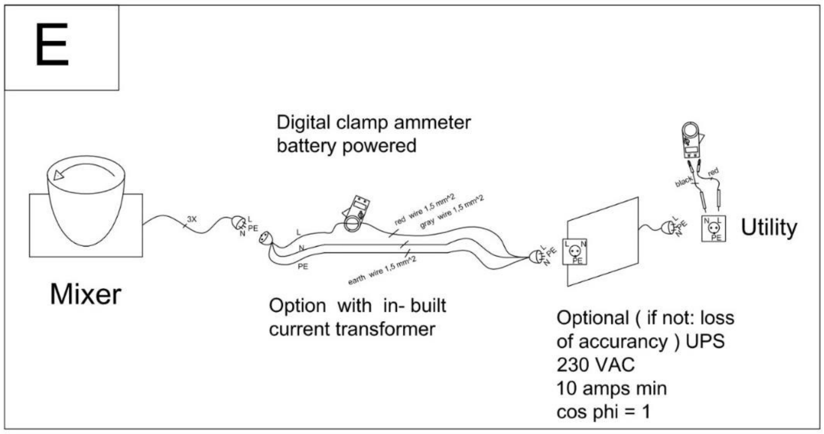
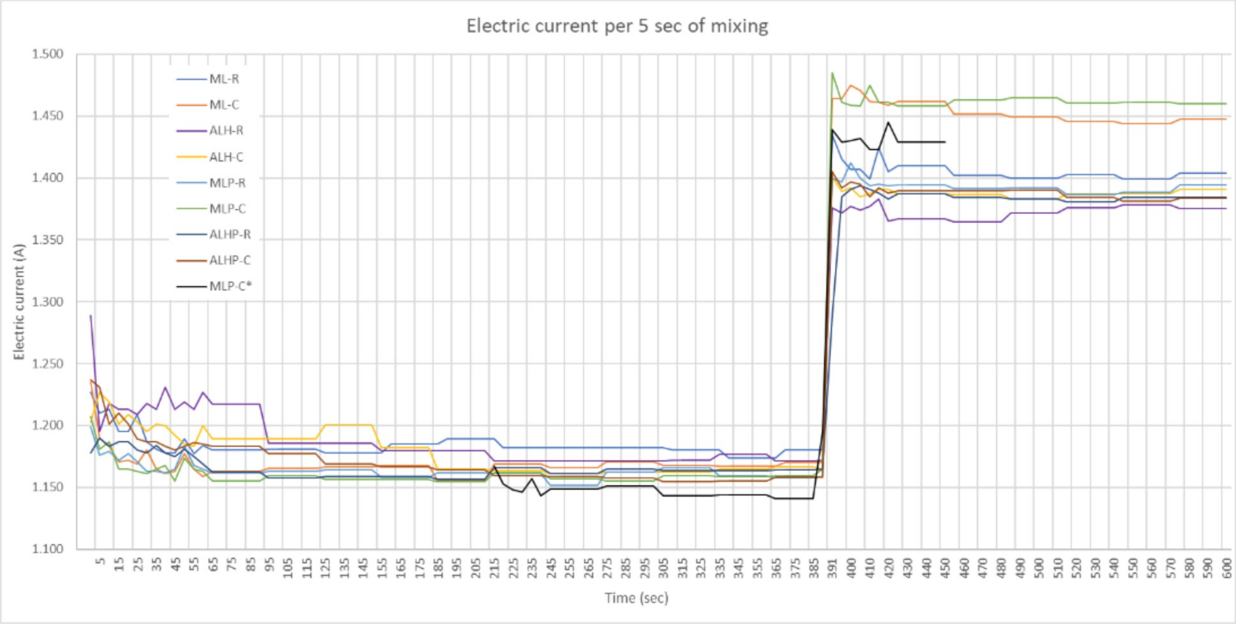


Fig. 4.11: The wiring diagram of the set-up.

The wiring connection consisted of three separated wires. The live wire, the neutral wire and the earth wire, connected to a female and male plug, which worked as a mediator between the mixer and the supply. The clamp ammeter is attached to the live wire while the mixer is in use. For greater accuracy the live wire was twisted creating a tenfold ring (Fig. 4.10). Therefore, the clamp gave ten times the value of the current and that resulted in a better readability of the result on the LCD screen. The actual results, however, needed to be divided by 10. A timer was used, and the procedure was video- recorded for every mixture for a duration of 10 minutes. For the first 6.5 minutes the values were recorded for low speed 65 revolutions/min and the last 3.5 minutes were recorded on high speed 120 revolutions/min. The values were registered every 5 seconds but after all the data had been listed, a mean value of every 6 values (30 seconds) was calculated in order to have a more readable diagram. In the Diagram (4.7.2) are presented the results of this method for every mixture.

The assumption behind measuring the electrical power demand for the motor of the mortar blender is that the consistency of the mortar would require more or less power to mix the mortar based on its consistency. The mortar blender had only two speeds and measurements were made at those two speed levels. More speeds of the mortar blender would have allowed a more accurate registration of the rheological behaviour of the mortar series, which would have been analogue to a rheometer.



Diag. 4.7.2: The electrical current for each sample vs time in seconds. For the first 6.5 minutes the mixer's speed was 60 rpm and for the last 3.5 minutes the speed was 120 rpm. Therefore, in the 391 second there is a sudden shift between the 1.350- 1.450 A. The final results were obtained from the high-speed area.

4.7.3 Electric Current and Flow Table test Results

The measurement of the electric current gave no significant differences in the current readings at 60 rpm. However, this changed when the speed was doubled to 120 rpm. The results were listed from high to low (Tab. 4.10). In Table 4.10 they were also compared to the flow table results and the w/b. The currents at 120 rpm show that two mixes were considerably less consistent, and both consisted of crushed aggregate, ML-C and MLP-C*[7], The results show that all ML series have the highest values. The mixtures that both contain crushed aggregates are first on the list and the ML-R and MLP-R come second. The mixture ML-C has the highest value. The MLP-R shows similar values with the ALHP-C, ALH-C and ALHP-R, in other words 3 out of the 4 pozzolanic mixtures. The ALH-R has the lowest values but does not differ significantly from the last 4 on the list. In the low speed the ALH series shows greater Amp values during the first 4 minutes of mixing, especially the non-pozzolanic mixtures, but after the transition to high speed they are the most workable mixtures. At this point it should

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[7] The mixture MLP-C has been tested twice because after the 10 minutes of mixing it did not give the desirable result in the slump test, so 20 ml of water was added and the measurement was repeated (for less time), an asterisk was added to the name of the measurement (represented by the black line in the graph). The measurement MLP-C (green line) should not be taken under consideration its w/b was 1.39. Its presence in the results though proves that due to low water content this sample had the highest Amp value.

be noted that the 2 mixtures series took place on different days, so there could have been a small error due to the provider's current.

High Amp Value	Mixture Name	Flow Table Test (mm)	w/b
↓	ML-C	177	1.65
	MLP-C*	175	1.47
	ML-R	216	1.65
	MLP-R	178	1.33
	ALHP-C	201	1.45
Low Amp Value	ALH-C (almost matching Amp values)	204	1.65
	ALHP-R	206	1.38
	ALH-R	210	1.65

Tab. 4.10: The results were listed from high to low, the mixtures in the blue box had very similar Amp values.

4.7.4 Rheometer measurements

Since the mixes with aged industrial lime hydrate showed a more plastic consistency, it was attempted to measure rheological properties. The mixtures tested were the ALHP-C and the ALHP-R (see Table 4.7, chapter 4.7). However, during the measurements, it was evident that the mortars' stiffness with the w/b values used for the flow table tests was hard on the border what the rheometer (Viskomat XL by Schleibinger GmbH) could detect. The measurement was performed similar to measurements for cement mortars and concrete. The instrument works with a rotating bowl, where the mortar was placed. A fixed fishbone shaped paddle, where the extension rod was connected to a force gauge, measured the torque the mortar was exerting on the paddle. The measurements started from higher (30 rpm) to lower shear rates in defined steps to bring the plastic mortar into an equilibrium. The shear rates and corresponding torque (shear stress) values are registered by the instrument and plotted afterward to a torque vs. share rate diagram.

The results of the two mixes subjected to the test are shown in Figure 4.12. The results demonstrated that both tested mortar mixes indeed behaved like a Bingham fluid. The results in Figure 4.12 indicated a large influence of the shape of aggregates towards the yield stress, with 118 Nmm for rounded and 197 Nmm for angular, crushed aggregates. However, the influence on the viscosity, which was represented by the slope in the graph of Figure 4.13, was almost the same. Please note that the values for the slopes in Figure 4.12 are proportional to the viscosity of the mortars but not their viscosities themselves. This is because the shear stress was expressed in torque (Nmm) and not in an actual stress (N/mm^2) reading. For the latter, the rheometer would need to be calibrated, which was not done in this case.

The explanation that the yield stress is considerably higher with angular aggregate shapes is due to the interlocking of the grains once they shear movement stops. An angular shape of the grains is increasing slightly viscosity during the shear movement but as long as it is movement in the mortar, the effects are less. Higher yield stresses also indicated the mortars with crushed aggregates. They are more suitable for plaster or render applications, since once the mortar is thrown to a vertical wall, it will stay that way and not flow downwards.

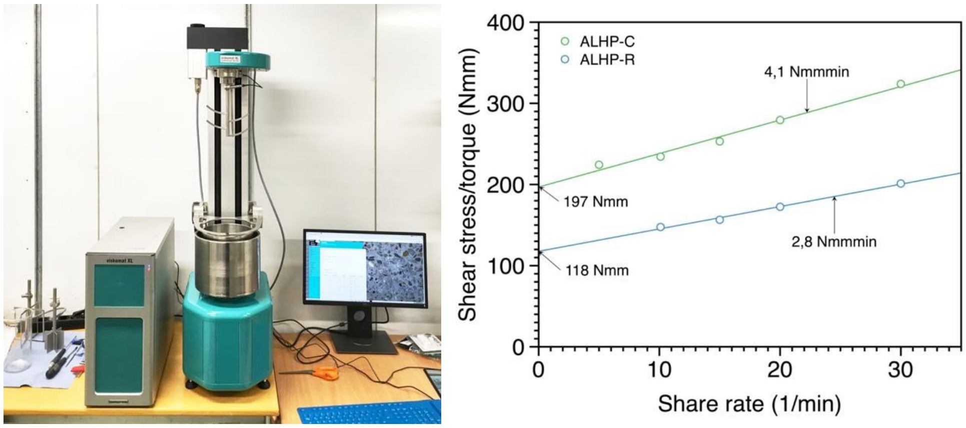

Fig. 4.12: The rheometer Viskomat XL. The torque vs. shear rate for specimens ALHP-C and ALHP-R.

4.7.5 Observations on the Fresh Properties

- In most cases the results of the flow table tests agree with the results of Ampere values (Tab. 4.11). Only the ML-R flow table results showed a large difference to the electric current test (in Tab. 4.11 indicated with red colour), probably due to an error. Additionally, based on the current tests the ML series was less workable than the ALH series as well as the rest 3 mixtures of ML series.
- Overall, the rounded aggregates show better workability than the crushed aggregates with the two methods (flow table, ammeter clamp).
- The ALH- series was more flowable compared to the ML-series.
- The non- pozzolanic mixtures (see Table 4.11, marked bold) had the same water content and the ALH were more workable. That led to the conclusion that aged commercial lime hydrate putty has a higher workability in comparison to the Mariestad's fresh slaked lime.
- MLP-R and MLP-C were more workable than the ML-C, but in the case of ALH series the ALHP-R and ALHP-C (pozzolanic mixtures), were less workable than ALH-R and ALH-C respectively. So pozzolan proved more effective in ML series even though the water content was similar to the ALHP mixtures. However, within the ALH-series the flow table results were very closely together.

- The tracking of the electric current proved an easy trustworthy method for a rough estimation of workability. Improvements could be made by adding periods of different speeds, analogue to a rheometer measurement.
- The rheology measurements showed a big influence on the yield stress of the mortars between angular, crushed and round aggregate grains but to a much lesser degree on the viscosity.

	Flow Table result (mm)	Flow Table results classification	Amp value results classification	
HIGH Flow Table Value	216	**ML-R**		LOW Amp Value
	210	**ALH-R**	**ALH-R**	
	206	ALHP-R	ALHP-R	
↓	204	**ALH-C**	**ALH-C**	↓
	201	ALHP-C	ALHP-C	
	178	MLP-R	MLP-R	
LOW Flow Table Value	177	**ML-C**	**ML-R**	HIGH Amp Value
	175	MLP-C	MLP-C	
			ML-C	

Tab. 4.11: Comparison of flow table tests with the power input during mixing of the mortars.

4.8 Curing of specimens

After the mixtures were prepared and the flow table test had been completed, the mortar was put back to the mixer for 15 seconds. The test specimens were casted in two layers and were vibrated to ensure an even distribution in the moulds. They were left to set in the 40×40×160 mm standard forms (Fig. 4.13). The mortars were left in the forms up to 5 to 6 days under high relative humidity. This was because they were too fragile for demoulding. After demoulding the specimens were stored in an airtight container over water for 2-3 days more in order to hydrate (Fig 4.13). The carbonation was done in a chamber under stable conditions 20 °C and 50 % RH. Table 4.12 shows the timeline of the curing procedure.

Fig. 4.13: The mortar in the forms and the ones with pozzolana stored over water.

Name	03.11.2020	06.11.2020	09.11.2020	11.11.2020	13.11.2020	16.11.2020	Days in chamber	Days in NTP over water	Days in Total left to Carbonate
ML-R	cast- 19.8°C 93.8 % RH	chamber 19.8°C 93.8 % RH	over water in container	room 20°C 50% RH			6	2	55
ML-C	cast- 19.8°C 93.8 % RH	chamber 19.8°C 93.8 % RH	over water in container	room 20°C 50% RH			6	2	55
MLP-R	cast- 19.8°C 93.8 % RH	NTP over water in container	room 20°C 50% RH				3	3	57
MLP-C	cast- 19.8°C 93.8 % RH	NTP over water in container	room 20°C 50% RH				3	3	57
ALH-R	-	cast- 19.8°C 93.8 % RH	chamber 19.8°C 93.8 % RH	chamber 19.8°C 93.8 % RH	over water in container	room 20°C 50% RH	8	3	50
ALH-C	-	cast- 19.8°C 93.8 % RH	chamber 19.8°C 93.8 % RH	chamber 19.8°C 93.8 % RH	over water in container	room 20°C 50% RH	8	3	50
ALHP-R	-	cast- 19.8°C 93.8 % RH	chamber 19.8°C 93.8 % RH	NTP over water in container	room 20°C 50% RH		6	2	53
ALHP-C	-	cast- 19.8°C 93.8 % RH	chamber 19.8°C 93.8 % RH	NTP over water in container	room 20°C 50% RH		6	2	53

Table 4.12 Timeline of Curing

4.8.1 Observations on Curing

The ML series set faster in the humid conditions compared to the ALH series which required more time to set and eventually be ready to be demoulded. This is probably because Mariestad’s lime is impure therefore feebly hydraulic. Similarly, the pozzolanic mixtures set faster in both series.

4.9 Mechanical Strength

There were tested 24 specimens of 40×40 160 mm for flexural strength and 47 halves emerged from the initial 24 for compressive strength testing. The first test was the flexural strength test. This test measures the flexural strength, and the set up was a manual 3-point testing machine according to the EN1015-11. The results are shown in the appendix C. Some of the specimens were very sensitive and broke before the flexural test occurred, these measurements are marked with the dash symbol.

Fig. 4.14: Experimental set-up for flexural and compressive strength tests.

After the measurement in flexural strength, the two remaining halves were tested under compression. The minimum loading rate recommended from the EN standard is 50 N/s but the ALH series proved sensitive to that loading rate. So, a loading rate of 10 N/s was used for the procedure and the load surface was 40×40 mm (Fig. 4.14).

4.9.1 Results of Mechanical Tests

All the results are listed in the form of a table in the appendix C. The flexural strength was not relevant. The loads were too low to identify any significant patterns and some specimens broke before the testing was completed, in Figure 4.17 the results of the flexural strength are presented. The compressive strengths are shown in Figure 4.16. The MLP-R and MLP-C specimens show the best performance under compression, with no significant difference to each other. ML-C and ML-R exhibited slightly lower values. Follow the ALHP-C, then the ALHP-R and the worst performance is the ALH-C with similar values as the ALH-R specimens.

There is a significant difference in the compressive strength between the type of binder, but no effect in type of aggregate shape. For both, slaked quick lime and aged commercial lime hydrate, the addition of the pozzolan increases the compressive strength markedly. In general, the compressive strength of the slaked quick lime mortars is in the expected range, even though according to the standard definition in EN 459-1 for hydraulic lime a value of ca. 1.5 MPa would not qualify for the lowest HL class of the standard. This is because hydraulic components in commercial HL limes are often added in form of cement, which reacts quicker. Natural pozzolana need longer time for hydration.

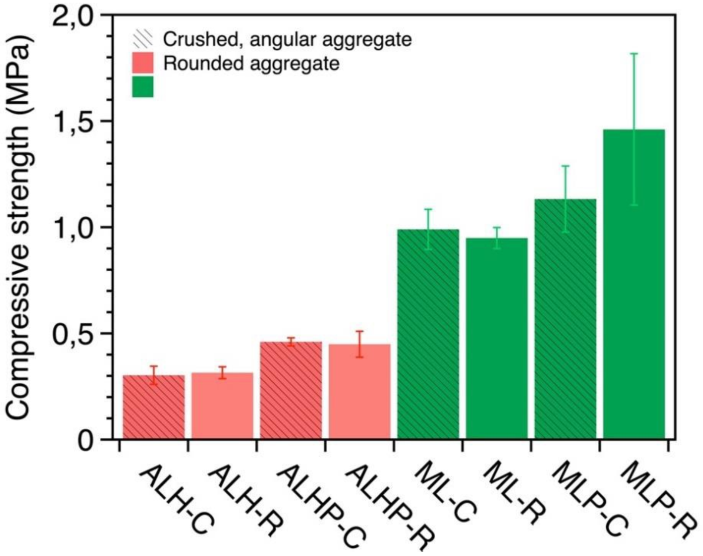

Fig. 4.16: Results from compressive strength measurements (mean values from 5-6 specimens). The error bars represent the standard deviations.

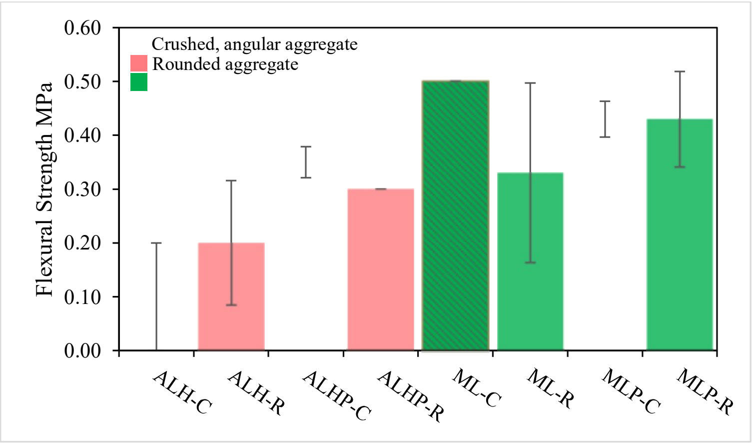

Fig. 4.17: Results from flexural strength measurements (mean values from 1-3 specimens). The error bars represent the standard deviations.

5. Conclusions

This experimental study showed that the round aggregates result in higher workability in both the Mariestad's fresh slaked lime (ML) and the Commercial lime Hydrated of 'Finja' (ALH). Pozzolan did not significantly favour rheology especially in the case of ALH due to lower water content, but workability in ALH series was sufficient in every mixture. In the ML series workability was enhanced after the use of pozzolan even though the water content was lower. However, this is inadequate for a safe conclusion and more testing should be done. Overall rheology is primarily affected by the aggregate shape (yield stress) and water content. A secondary role has the presence of pozzolan, however water content in relation to pozzolan should be further researched for these binders (Table 6.1).

Under compression the specimens with pozzolan addition yielded higher compressive strength but the aggregate shape did not contribute significantly to compressive strength. In non-pozzolanic specimens the aggregate shape seemed not to affect the compression strength of the mortars in a conclusive way. To sum up, compression was more affected by pozzolanic activity or hydraulicity in general and less affected by the aggregate shape. Aggregate shape had a primary role in tensile strength. The flexural strength test showed that crushed aggregates enhanced the flexural behaviour of the specimens and pozzolanicity had a secondary role (Table in appendix C).

The binder type influenced most the test results. The ML binder proved to give better strength properties, but it was the least workable. The ALH on the other hand had significantly inferior strength properties, but better workability. The binder type, in this comparative study, seemed to affect the most all three parameters tested (rheology, compression, flexure) but the cause of this behaviour was not investigated. Binder quality can be affected also by composition, hydraulicity, slaking method, microstructure and air content. The mixtures as mentioned in chapter 3 were investigated as homogenous unities, therefore the aforementioned binder parameters were not researched in this study.

Comparative Assessment		
Rheology	**Compression**	**Flexure**
ALH, ALHP	MLP, ML	ML, MLP
Round aggregates	Pozzolan content, hydraulicity degree.	Crushed aggregates
Pozzolan content	Aggregates shape does not affect significantly	Pozzolan content

Tab. 6.1 A synoptic presentation of the results from the most influential factor to the least for each parameter tested.

All things considered aging and pozzolan use is necessary in the commercial lime hydrate for conservation purposes. Especially when access to Natural Hydraulic Limes (NHL) is not possible. The workability of this binder is adequate but affected significantly by the aggregate shape and the pozzolan content. Air content for this binder is recommended to be further investigated. The traditionally slaked lime from Mariestad showed overall better performance. This is possibly due to its composition and its feebly hydraulic qualities. Due to low hydraulicity this material is not recommended to be aged; it performs better when it is still fresh slaked. With regulation of the water content it can be both workable and durable. It performed very well with round aggregates under compression and gave relatively good flexural strength values. This binder is less workable and higher water content is recommended for its better performance. Higher water content though can affect its mechanical performance, so it is recommended to be used with natural round aggregates. However, such materials are not extensively available, have not been under quality control and their performance in historic structures cannot be in every case successful. Their study though prior conservation projects can lead possibly to compatible mixtures with the historic ones. The electric current measurement during mixing proved to be a reliable, easy non-standard method for the estimation of workability.

6. Limits of the study

The water content was not regulated according to the flow table test results, the idea was to be either stable or similar for all mixtures. The water content in the pozzolanic Mariestad's mixtures should have been more deliberately regulated in relation to pozzolan. However, the pozzolanic mixtures had the best mechanical performance. More wall applications should have been made, due to inadequacy in binder most trials and wall applications took place with non-aged air lime as a binder. More wall applications would have helped to estimate the water content in more realistic circumstances, and they would have led to more complete conclusions related to workability. They are recommended to future studies in the scope of conservation practice. Air content of the binders is also something that affected this experimental study, but it was not considered as a parameter and it is recommended to be further investigated. Although air content could favour workability, it could increase porosity, causing fragility. Curing period could have also been longer, the standards recommend 28 days but according to the literature this has not proven enough for lime binders, and especially air lime. Studies have shown that the most significant changes in strength are between 29 to 49 days (Drougas et al.2016), and full carbonation can take more than a year. Therefore, all specimens were left to cure more than 50 days. For more complete conclusions, a greater number of samples could have been used.

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List of Figures

Figure 1.1: Natural non-commercial air lime from Mariestad. CaO (left), middle while slaking and right Ca(OH)2 putty after slaking (hydration), source: Natalia I. Dedousi, 2020
Figure 1.2: Commercial air slaked lime Ca(OH)2 in powder form, emerging from dry hydration, source: Natalia I. Dedousi, 2020
Figure 1.3: Illustration inspired from the book *'Building Limes in Conservation'* by Brocklebank Ian, 2012 p. 11, source: Natalia I. Dedousi, 2020
Figure 2.1: Materials that are characterised by rheology, source: Natalia I. Dedousi, 2020
Figure 2.2: Three-point flexure test (left) and splitting test (right), source: Garijo et al., 2019
Figure 4.1: The samples ready to be tested in the XRF and XRD. The samples were ground into a fine powder, source: Natalia I. Dedousi, 2020
Figure 4.2: Ternary diagram with the chemical composition of the Lime from Mariestad and the industrial lime hydrate. Given is also the approximate chemical composition of modern Portland cements as a comparison (grey shaded field), source: Urs Mueller, 2021
Figure 4.3: Lime from Mariestad – Dark area with magnetic properties (2nd image from left), source: Natalia I. Dedousi, 2020
Figure 4.4: Lime from Mariestad – Lighter areas, source: Natalia I. Dedousi, 2020
Figure 4.5: Commercial lime hydrated in powder form the company Finja (Murkalk 'Finja'), source: Natalia I. Dedousi, 2020
Figure 4.6: Aggregate size distribution curve used, source: Natalia I. Dedousi, 2020
Figure 4.7: Preparation steps for aging lime putty (a,b,c,d), source: Natalia I. Dedousi, 2020
Figure 4.8: a. Part of the necessary equipment. b. Slaking of the quick lime in the trough. c. Covering the trough and cooling the putty, source: Natalia I. Dedousi, 2020
Figure 4.9: a. and b. Set-up for the flow table test. c. The mortar applied to a brick test wall (a,b,c,d), source: Natalia I. Dedousi, 2020
Figure 4.10: Current clamp and experimental setup, source: Natalia I. Dedousi, 2020
Figure 4.11: The wiring diagram of the set-up, source: Natalia I. Dedousi, 2020
Figure 4.12: The rheometer Viskomat XL. The torque vs. shear rate for specimens ALHP-C and ALHP-R, source: Urs Mueller, 2020
Figure 4.13: The mortar in the forms and the ones with pozzolana stored over water, source: Natalia I. Dedousi, 2020
Figure 4.14: Experimental set-up for flexural and compressive strength tests, source: Natalia I. Dedousi, 2020
Figure 4.16: Results from compressive strength measurements (mean values from 5-6 specimens). The error bars represent the standard deviations, source: Natalia I. Dedousi, 2020
Figure 4.17: Results from flexural strength measurements (mean values from 1-3 specimens). The error bars represent the standard deviations, source: Natalia I. Dedousi, 2020

APPENDIX A

XRD- SPECTRA

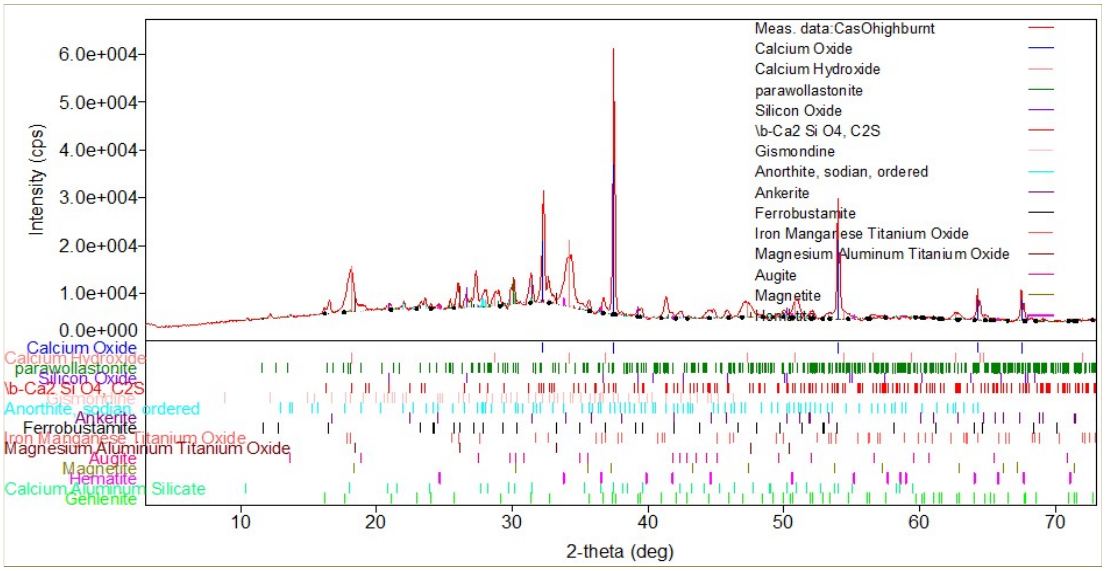

Fig. A1 Mariestad's dark area CaO

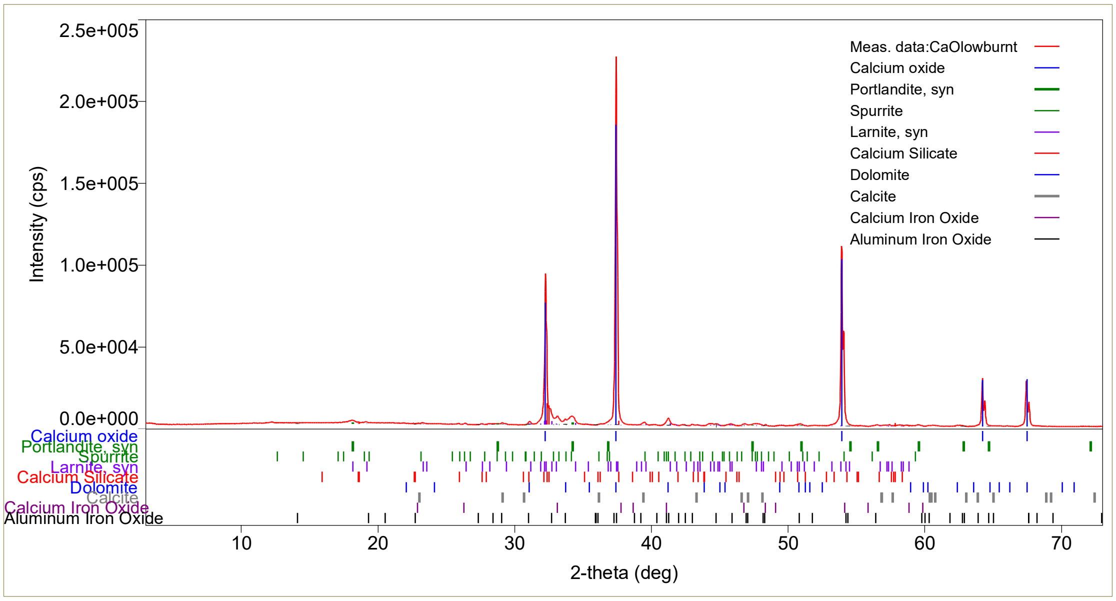

Fig. A2 Marestad's light area CaO

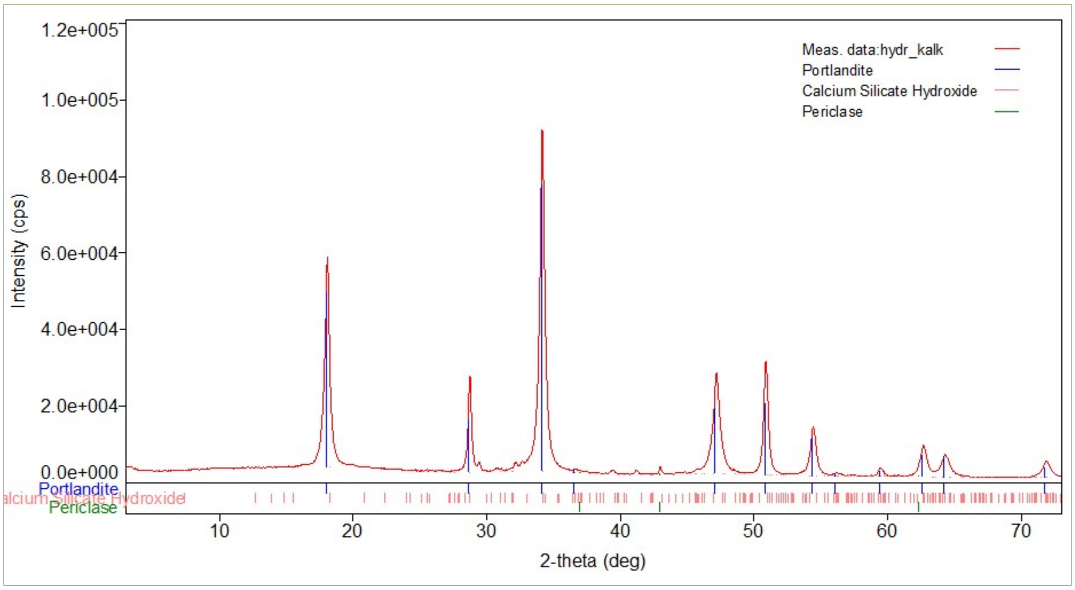

Fig. A3 Comercial CaOH 'Murkalk Finja'

APPENDIX B

Calculation of free water content in in Aged commercial hydrated lime 'FINJA' after 3 months of storage.

This binder has W/B ratio 1.5, this is how initially was made the day that it was prepared and left to age. In order to be estimated the free water content of the aged lime, it needs to dry out at 105°C and calculate the mass difference before and after drying. There were taken 3 samples from the binder putty and were then dried out in 3 different ceramic containers. So, they had given 3 different water percentage values. The mean value of the three results determined the water binder ratio (Tab B1 and B2).

	date	Container 1	Container 2	Container 3
Container mass (g)	07/09	76.4982	76.1909	79.0444
Container + wet mass binder (g)	07/09	104.4670	111.9262	120.7958
Container+ dry mass (g) 1	08/09	87.4950	90.2565	95.6151
Container+ dry mass (g) 2	10/09	87.5374	90.2860	95.6819

Tab. B1

	Container 1	Container 2	Container 3
Net wet mass (g)	27.9688	35.7353	41.7514
Net dry mass (g) 1	10.9968	14.0656	16.5707
Net dry mass (g) 2	11.0392	14.0951	16.6375
Net dry mass difference (g)	0.0424 (-0.38%)	0.0295 (-0.20%)	0.0668 (-0.40%)

Tab. B2

$W_{conternt\ 1}$= 60.60%

$W_{content\ 2}$ = 60.59%

$W_{content\ 3}$= 60.23%

$W_{cont.mean}$= 60.47% => W/B= 1.5297

In conclusion the water content detected is equal with the initial 1.5 W:B ratio when the water was added to the dry binder.

APPENDIX C

In the Table C the compressive strength results are listed from high to low. The specimens indicated all very low flexural strength; some specimens did not give values. The flexural strength results are listed in the per group of samples.

Specimen Name	Compressive stress at Maximum Max load [kN]	Compressive stress at Maximum Load [MPa]	Specimen Name	Flexural stress at Max load [MPa]
MLP-R 6/6	3.07	1.92073	ALH-R 1/5	0
MLP-R 5/6	3.00	1.87202	ALH-R 2/5	
MLP-R 4/6	2.20	1.37733	ALH-R 3/5	0.4
MLP-C 1/6	2.13	1.32816	ALH-R 4/5	
MLP-R 2/6	2.10	1.31196	ALH-R 5/5	0.2
MLP-C 2/6	2.05	1.28104	ALH-C 1/6	0
MLP-R 3/6	1.99	1.24209	ALH-C 2/6	
MLP-C 5/6	1.83	1.14444	ALH-C 3/6	0
ML-C 3/6	1.83	1.14089	ALH-C 4/6	
MLP-C 6/6	1.77	1.10782	ALH-C 5/6	0.3
ML-C 4/6	1.71	1.06796	ALH-C 6/6	
MLP-R 1/6	1.66	1.0388	ALHP-R 1/6	0.3
MLP-C 3/6	1.63	1.01852	ALHP-R 2/6	
ML-R 5/6	1.61	1.00701	ALHP-R 3/6	0.3
ML-R 4/6	1.60	1.00092	ALHP-R 4/6	
ML-C 5/6	1.55	0.97053	ALHP-R 5/6	0.3
ML-R 6/6	1.55	0.97001	ALHP-R 6/6	
ML-C 2/6	1.50	0.93914	ALHP-C 1/6	0.35
MLP-C 4/6	1.47	0.91589	ALHP-C 2/6	
ML-R 2/6	1.46	0.91496	ALHP-C 3/6	0.3
ML-C 1/6	1.46	0.91285	ALHP-C 4/6	
ML-C 6/6	1.45	0.90793	ALHP-C 5/6	0.4
ML-R 1/6	1.45	0.90625	ALHP-C 6/6	
ML-R 3/6	1.43	0.89609	ML-R 1/6	0
ALHP-R 2/6	0.82	0.51387	ML-R 2/6	
ALHP-C 6/6	0.80	0.49701	ML-R 3/6	0.5
ALHP-R 1/6	0.79	0.49491	ML-R 4/6	
ALHP-R 5/6	0.76	0.47281	ML-R 5/6	0.5
ALHP-R 6/6	0.75	0.46608	ML-R 6/6	
ALHP-C 5/6	0.73	0.45935	ML-C 1/6	0.5
ALHP-C 3/6	0.73	0.45836	ML-C 2/6	
ALHP-C 1/6	0.72	0.45128	ML-C 3/6	0.5
ALHP-C 4/6	0.72	0.4499	ML-C 4/6	
ALHP-C 2/6	0.71	0.44467	ML-C 5/6	0.5
ALHP-R 4/6	0.60	0.37572	ML-C 6/6	
ALHP-R 3/6	0.59	0.37087	MLP-R 1/6	0.4
ALH-C 6/6	0.59	0.37043	MLP-R 2/6	
ALH-R 4/5	0.58	0.36005	MLP-R 3/6	0.3
ALH-C 5/6	0.52	0.32804	MLP-R 4/6	
ALH-R 3/5	0.51	0.31655	MLP-R 5/6	0.6
ALH-R 5/5	0.50	0.31143	MLP-R 6/6	
ALH-C 4/6	0.49	0.30518	MLP-C 1/6	0.5
ALH-R 2/5	0.48	0.29934	MLP-C 2/6	
ALH-C 3/6	0.46	0.28886	MLP-C 3/6	0.4
ALH-R 1/5	0.46	0.28639	MLP-C 4/6	
ALH-C 1/6	0.44	0.27596	MLP-C 5/6	0.4
ALH-C 2/6	0.40	0.24898	MLP-C 6/6	

Tab. C