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POROSITY OF HISTORIC MORTARS

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Abstract

This paper presents the results of the investigation of porosity of historic mortars by Rilem TC 167 Characterization of Historic Mortars with Respect to their Repair It is well known that porosity is one of the most crucial properties for the function and durability of a mortar. The character of this relation is insufficiently known. The aim with the work has been to summarise this knowledge and to elucidate the significance of porosity in the existing mortar for the choice of repair measures as well as the choice of repair mortar. The processes for the formation of primary porosity during mixing, applying and hardening of the mortars as well as the formation of secondary porosity in the hardened mortar is discussed. A brief introduction to methods used for assessment of the porosity is given. The influence of porosity on functional, durability and compatibility properties is discussed.

Key Words

Porosity, historic mortars, lime mortars

1 Introduction

The role of porosity of a historic mortar is important in terms of moisture transport, mechanical properties, durability and compatibility of the mortar to masonry as a whole. Methods to determine porosity are as diverse as its influence. Any characterization of historic mortar through porosity determination should not simply be done to obtain a value. Different techniques for determining porosity may give different values for the same sample. It is the interpretation of the value and its influence on the behaviour that is important.

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Porosity refers to the volume that is not composed of solids. Among existing classifications of pore sizes the most recognised is used by the IUPAC [1972] (Fig. 1) -- pores are classified as micropores, mesopores and macropores.

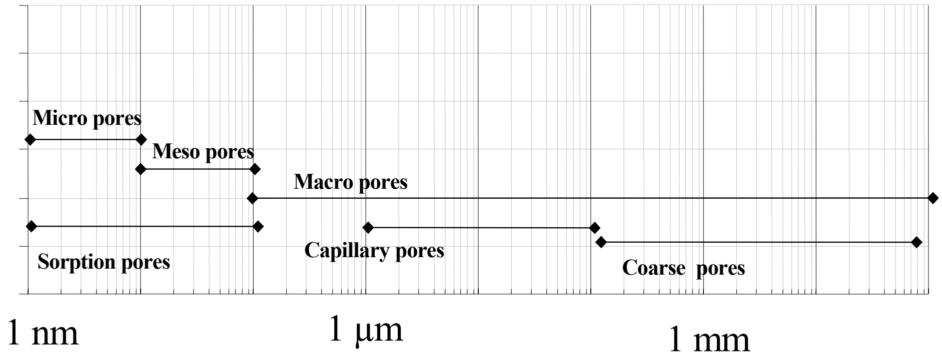


Figure 1. IUPAC pore size classification. The pore ranges of sorption, capillary and coarse pores are illustrated for reference.

An additional definition of pore size in mortars related to the moisture transfer properties is useful. Capillary pores range from approximately 0,1 to 100 micrometer and contribute to the capillary water transfer. Water present in finer pores is bound more tightly to the material and these pores provide a more limited moisture transfer contribution. Pores coarser than 100 micrometer contribute to the water permeability through gravity or wind driven water ingress.

The object of this paper is to provide a basic understanding of porosity in historic mortars with the focus on lime based mortars. This includes:

- how porosity is initially developed;
- how porosity changes over time;
- how porosity influences the properties and characteristics of a mortar.

2 Origin of Porosity in Mortars

2.1 Primary Porosity

Sorption-pores
Water is held to the surface of the sorption pores and no moisture is transported between these pores. Sorption pores occur as gel pores in the C-S-H gel (calcium-silicate-hydrate) of hydraulic mortars. Sorption pores are less abundant in lime mortars.

Capillary Pores
When the mortar is first mixed it is a dense slurry, with the aggregate and binder particles (ideally), uniformly distributed and surrounded by water. Primary porosity is developed through the movement of water due to either absorption into the surrounding masonry unit or evaporation to the air. These pores are highly interconnected and fluid transport through these pores is by capillary transport. The total porosity is further influenced by the carbonation of $Ca(OH)_2$ to $CaCO_3$ which represents a 10% volume increase. Accommodation of the volume change is taken up by a reduction of total porosity but no significant shift in pore size distribution (Papayianni and Stefanidou, 2001). Carbonation is a long-term process, and can take many years to complete. Hydrating hydraulic phases, if present also contribute to primary capillary porosity.
The water/binder ratio and the binder/sand ratio will also influence the capillary porosity. Capillary pores form at the contact of the binder to the sand the substrate. A higher water content and a lower sand content results in a higher relative total porosity

(Schäfer and Hilsdorf , 1992, Hayden et al., 2001). In addition, to the initial water/binder ratio of the mortar the insitu water/binder ratio will vary with the absorption behaviour of the contacting masonry unit (Groot and Larbi; 1999, Brocken, 1998; Sugo et al., 1998). Given the same initial water to binder content, mortar in contact with a highly absorptive masonry unit will tend have a lower total porosity than mortar in contact with weakly absorptive masonry.

Coarse Pores.
Coarse pores in historic mortars are generally formed by:

- *Entrapped Air*
  Entrapped air pores are irregular in shape and distribution and can account for up to 8% total porosity. They form through the entrapment of air during the mixing process. The water content and application of the mortar can influence the total coarse porosity of the mortar. Mortars, which are very dry, tend to entrain air more than wetter mortars. The placement and mechanical treatment of mortars such as tooling will reduce the entrained air content near the surface by pressing the air out.
- *Shrinkage cracks*
  Cracking due to drying shrinkage is not uncommon. Shrinkage cracks increase with increasing water content of the mortar and the rate of evaporation of the water from the mortar. Coarse shrinkage cracks will contribute to water ingress and mechanical weakening (see fig 1).

2.2 Secondary Porosity

Secondary porosity results from the interaction of hardened mortar with the environment. Chemical reactions, as well as mechanical processes, can create secondary porosity.

Dissolution/Re-precipitation
The transfer of moisture in the pores of a mortar also includes the transfer of the soluble ions. Calcium is the most ubiquitous ion, coming from the calcium hydroxide and calcium carbonate of the mortar binder. Wetting and drying cycles will cause the transfer of fluid through the mortar. The calcium ions are transferred and may come in contact with fluids enriched in CO_2 as carbonic acid and calcite will precipitate. Ring-like textures of calcite are common along the interior of larger voids and at the exterior of mortar and are related to dissolution and re-precipitation. Narrow cracks may heal by calcite precipitation, known as autogenous healing. The influence of dissolution/re-precipitation on porosity is not completely predictable. Dissolution will cause an increase in porosity, but re-precipitation, probably at a different locality will reduce porosity. If the calcite precipitates on the mortar surface, the surface becomes more impermeable and there is a loss of mass to the mortar.

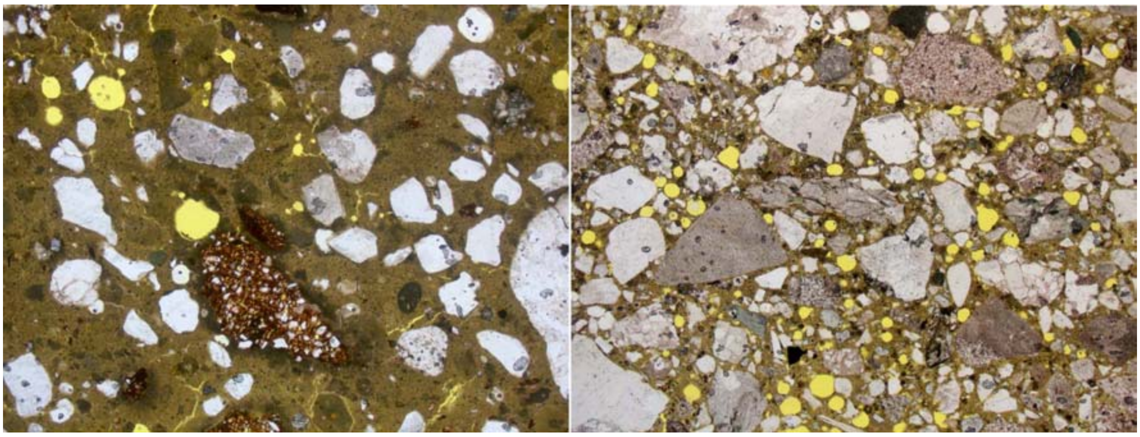


Figure 1. The image to the left shows a 14th century mortar from the brick masonry of the citadel Kärnan, southern Sweden. To the right is a modern lime cement rendering mortar with air entertainers. Note the low amount of aggregate and fine air voids in the historic mortar. Small shrinkage cracks can be seen in the medieval mortar. Aggregate are angular, air voids are circular and the paste is dark. The size of the images is 5*4 mm^2.

Acid Leaching

The solubility of calcite in the mortar increases with decreasing pH. Acid leaching results in the dissolution of the mortar binder, and carbonate aggregates if present. It rarely leads to re-precipitation and therefore results in an increase of total porosity and the pore size is usually coarse. Acidic environments with pH values as low as 3 are not uncommon to historic masonry. It can come from humic acids of soils or lichens, or sulphates and nitrates from dry deposition on the masonry surface (Charola, 2000). While the latter type of acid leaching is generally associated with industrial environments, it is possible that localised dry deposition from a fire pit or fireplace can produce the same type of deterioration of the mortar.

Mechanical Actions.

Cracking due to mechanical actions occurs where resistance to different types of stresses is exceeded. The cracks may be coarse or capillary sized. Frost damage and salt crystallisation are special cases of mechanical action.

Secondary minerals.

Secondary mineral formation in mortar after hardening may greatly influence the pore structure of the mortar. Secondary mineral growth is the result of the dissolution of the primary paste, and the formation of a new mineral not including calcite. The new mineral formation usually results in cracking, and the cracks are typically coarse. Secondary minerals such as calcium or magnesium sulphate may form if soluble calcium or magnesium and sulphate are available. Ettringite or thaumasite may form if alumina and sulphate are available, resulting in pore or crack filling and possibly expansion of the mortar. If a damaged mortar contains any of these elements it does not automatically indicate the presence of the minerals and mineralogical studies are necessary.

3. Methods for Assessing Porosity: Potential and Limitations

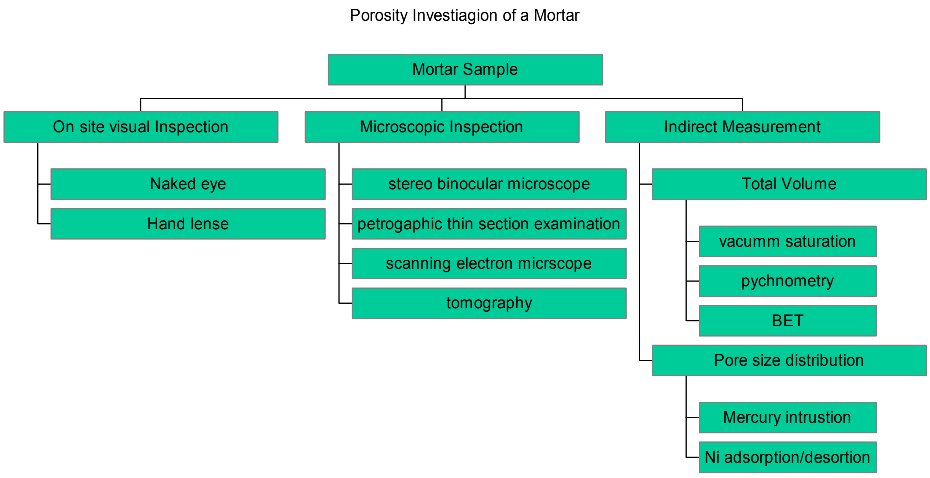
The three values, which are fundamental to describe porous materials, are:

- **Total Porosity (TP)** is defined as the fraction of the total volume that is occupied by pores. The porosity is further subdivided into open porosity and closed porosity. Open porosity consists of pores which are connected to one another and is measured by microscopy and intrusion techniques. Closed porosity consists of pores which are not connected to each other and is measured by microscopy techniques.
- **Pore Size Distribution (PSD)** is defined as the fraction of total pore volume in which the pores lie within a stated size range. It can be expressed either in integral or differential form. Pore size distribution values representing total porosity must be obtained by using different measurement techniques.
- **Specific Surface (SS)** is defined as the area or accessible surface area contained in unit mass of volume of solid and can, in principle, be calculated using total porosity and pore size distribution or from analytical techniques.

Techniques for determining these values can be subdivided into direct methods, where the pore structure is evaluated by direct observation; and indirect methods where the presence or size of pores is inferred from measurements. It is not uncommon for two techniques to provide two different values for the same sample. An overview of techniques to measure porosity characteristics is given in the RILEM publication Characterisation of Old Mortars with Respect to their Repair (to be published in 2004).

Table 1 organizes the techniques described in the RILEM publication: top to bottom represents an increase in complexity of analysis and detail of information to be obtained. The most direct and the simplest tools include the naked eye, hand lens and stereo binocular microscope. These allow for the examination of macro pores and cracks. A petrographic or scanning electron microscope examination allows for more detailed investigation. Primary and secondary porosity can be viewed and values for total porosity, and pore size distribution, using image analysis is possible.

Table 1 Organization chart for porosity determination techniques.



The simplest indirect porosity measurement is total pore volume determination by vacuum saturation. Intrusive techniques using pychnometry and mercury intrusion

porosimetry (MIP), will also provide total porosity and pore size distribution, but equipment is required. Provisions for mercury in the work environment must also be considered. Specific surface measurements are best completed by BET methods (based of condensation of gases at low temperatures). Tomography, which includes nuclear magnetic resonance (NMR) is in its developmental stage, and is showing itself as an excellent research tool for moisture transport, but application to characterization is limited.

In any analysis and interpretation of porosity of mortar the following must be taken into consideration:

- There is no analytical method available, which will give the precise nature of porosity of a material.
- One analytical method may not give the same results as the other. For instance when a porosity property is measured by intrusive methods only those pores that are interconnected are measured. This provides a relationship of porosity to permeability. When measured by optical techniques, all pores, which can be observed, are measured. Some of these pores may not be interconnected to each other. It becomes apparent that depending on the nature of the measurement a different value might be obtained.
- Without a complete statistical sampling the analytical results are estimates. Other data such as chemical analysis or petrographic examination must be used to understand if the sample is representative of the investigated mortar.

4 The significance of porosity characterisation

The porosity of a repair or replacement mortar must be compatible with the surrounding masonry unit and the masonry as a whole in the following areas:

- Mechanical properties,
- Moisture transport properties
- Durability

4.1 Mechanical Properties

Compressive and Tensile Strength
Laboratory studies have investigated the mechanical properties of lime and hydraulic lime mortars (Schäfer & Hilsdorf 1992, Van Balen & Van Gemert 1994, Hayen *et al*, 2001, Papayianni *et al*, 2001). From these studies it was noted that lime mortars have higher total porosities than hydraulic lime mortars. Comparing hydrated and hydraulic lime mortars Schäfer & Hilsdorf (1992) concluded that the compressive strength is a function of the porosity *and* the type of binder (hydraulicity). Hayen *et al*, (2001), Papayianni *et al*. (2001) further show that progressive carbonation of lime mortars results in an increase of compressive strength and a decrease of porosity.

Mechanical Properties in Practice
Determining the mechanical properties of a historic mortar can be difficult because of the size of the sample, or the condition of the sample. Porosity analysis can be helpful to gain some comparative information on compressive strength, because of the inverse

relationship of a decrease in compressive strength with a gain in total porosity. There should be no expectation of absolute values.

Where a decrease in compressive strength might be suspected a non-damaged sample should be compared against a damaged sample. An increase of total porosity is an indication of decreased compressive strength. This would also influence the critical tensile strength. Pore Size Distribution would indicate if the gain in total porosity represents a coarsening of pores or an increase in the amount pores. In most damage cases the increase is due to an increase in pore size.

4.2 Influence of Porosity on Moisture Transfer Properties

Theoretical Aspects

Different driving potentials govern the transfer process of moisture. Moisture transfer through vapour diffusion is governed by a difference in vapour pressure, while the liquid moisture transfer is governed by a pressure difference on the liquid. In coarse pores or cracks external pressure such as wind pressure and gravity loads affect moisture transfer. Due to the high moisture capacity of cracks and macro-pores, however, they can act as a reservoir drastically increasing the available water to be moved into the mortar. Temperature gradients also influence transport. In a material with a temperature gradient, water may be transported through vapour diffusion in one part and then condense and be transported through capillary suction in other parts. Condensation of vapour to liquid will occur at the dew point, and this occurs often within a masonry wall. An increase in salinity of the pore fluid will increase the relative humidity of pores and increase the dew point temperature.

In masonry, moisture exchange also occurs between the mortar and different masonry materials it bonds to. A simple rule of thumb is the direction of capillary transport will occur from coarse porous material to fine porous material. This is generally considered to be a fundamental premise to the concept of compatibility of mortar to masonry. The preferred direction of moisture transport is from the masonry unit to the mortar. This is so the moisture load is in the mortar which makes it sacrificial to the masonry unit. Brocken (1998) points out that the nature of the interfacial zone is important for the transport of moisture across this boundary and it is not the same as the body of the mortar. He explains that for a lime-cement mortar the lack of coarse aggregate and process of carbonation in the interfacial zone may well create essentially a moisture barrier. No thorough work has been dealt with how this relates to lime mortars and it needs to be investigated.

Moisture transport in practice

Characterizing moisture transport properties must be done in a context of knowing where the mortar is from and its function. The location of the mortar allows the investigator to consider its importance to moisture transport. Mortar in contact with the surface, masonry units or deep in the interior to the structure will have different moisture loads and therefore have different roles for moisture transport. Surface mortar, which would include exterior renders or the first 2 cm from the exterior surface of a joint can be either the wick for moisture or the reservoir. Moisture can be driven into the wall by rain, or drawn in by capillary transport. Near surface cracks or voids filled with water can act as ponds, supply a source of water for capillary transport into the interior of the mortar. IRA tests of the removed mortar can provide a good indication of the nature of the pore structure at the surface. Large cracks or voids cannot draw water into them, and the IRA values will be relatively lower than a mortar that has numerous capillary pores at the surface.

The porosity of mortar in immediate contact with the masonry unit is particularly important where the possibility exists where there is concern in changing the direction for moisture transport. Recall that moisture will move from coarse pores to smaller pores by capillary action. Petrographic examination to determine the composition of the interfacial zone and PSD are the best tools to investigate this. Knowing the PSD and IRA of the contacting masonry unit is also necessary. A relatively non-porous stone will not have the impact on moisture transport across the joint as a very porous stone.

Mortar in the interior of the masonry structure should be investigated to determine the nature of the primary mortar. Typically this mortar will not have secondary overprints associated with exterior and contact mortar. The total porosity and pore size distribution will be an indication of bearing capacity of the mortar and the nature of the materials. High total porosity (> 20%) with a small amount of pores <1 µm most likely indicates a lime-sand mortar. Higher values still of both total porosity and coarsening of the pores would indicate that the mortar is probably altered and friable. Low values of both total porosity and more fine pores might indicate the presence of a hydraulic lime phase. This would be confirmed using other analytical techniques.

4.3 Durability.

Durability is generally related to the resistance of a material in its environment. For masonry and mortar the greatest influence on durability is the presence of pore fluid and the composition of the pore fluid.

Salt Transportation and Salt Damage.
Soluble salts in mortar or masonry units can present a serious durability risk to a mortar Salts may come from the masonry materials themselves, or supplied from exterior sources Damage occurs at or just below the surface of the mortar, where there is a change in the degree of saturation of the salt laden solution (Charola, 2000, Lewin, 1982). Crystallisation and hydration may cause damage through expansion.

The influence of salts on composition of pozzolan or hydraulic mortars may also be significant. A pore solution saturated in chloride will alter calcium-aluminum-hydrate phases to calcium chloroaliminates, resulting in an expansion. A sulphate rich solution may react with calcium from the pore solution of the mortar to form gypsum. Gypsum deposits occur on the surface, often creating a coating, which may in turn reduce the surface evaporation of moisture and enhance sub-florescence.

Freeze-thaw durability.
The porosity and the related moisture properties have a major influence on the freeze thaw durability of a mortar. The degree of saturation, the total pore volume and the pore size and distribution all contribute to freeze-thaw durability (Litvan, 1980, Litvan, 1981, Maage, 1984, Robinson, 1984)

Field studies such as Waldum & Anda, (2000) and laboratory studies such as by Marie-Victoire & Bromblet (2000) and Maurenbrecher *et al*. (2000) have contributed some to our understanding of frost durability of lime mortars. All point out that lime mortars, which are not fully carbonated, are more vulnerable to freeze-thaw damage. Testing of fully carbonated laboratory mixed mortars has not been completed.

Durability in practice

For durability the ability of moisture to move within the mortar and the accessibility of a mortar to damaging agents are an important parameters. In these respect porosity plays a major role. The pore distribution and changes of pore distributions over time can be evaluated using microscopic techniques. This includes the estimation of a binder-moisture ratio at the time of production of a mortar, the establishing of the use of additives such as air-entraining agents, the formation of cracks, the change of porosity as a result of leaching, effects of transport of soluble material resulting in reprecipitation or recrystallisation. Also microscopic techniques are a powerful tool to study interfaces between old repair material and, between aggregates and pastes. These interfaces are often the places from where durability failure may originate.

Another important durability parameter is frost resistance of mortars. For lime mortars the susceptibility to frost damage especially of young uncarbonated mortars is notorious. Microscopic techniques can be deployed to evaluate carbonation progress as a function of time and composition (porosity) of the mortar resulting in recommendations for the application of lime mortars in frost prone regions. Although carbonated lime mortars show high frost resistance it is known as well that some restoration measures, like the application of very dense repair jointing mortars or the use of water repellent may influence the moisture conditions in masonry in an unfavourable way increasing the frost susceptibility of lime mortars.

The durability of structures may be essentially impaired by expansive reactions of salts; these are crystallisation and hydration. As damage caused by salt often looks similar to that of frost damage a distinction between the two of them is needed to take considered repair measures. Using petrographical methods and possibly SEM-techniques it is possible determine the nature of the salts if present.

Application of Porosity Data to Repair Mortars

In any consideration of using the porosity of a historic mortar to design a repair mortar it must be fully realized that there are limitations. There are numerous techniques and sophisticated pieces of equipment, which can determine porosity with tremendous accuracy and some degree of precision. The challenge comes in understanding what is being measured. The porosity of mortar is not the same at one week, 1 year, or 100 years. Add to this the changes associated with all the secondary processes that have been discussed above, come to the decision as to what porosity is to be replicated is not simple. While it may be possible to formulate a lime mortar with similar total porosity values as the historic mortar it is not possible to replicate the texture of 1000 years of dissolution-reprecipitation.

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