# A Comparative study into the performance of hot mixed and lime putty mortars with additions of metakaolin and brick dust pozzolans.

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# A Comparative study into the performance of hot mixed and lime putty mortars with additions of metakaolin and brick dust pozzolans.

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## Abstract

This paper explores the properties of mortars where the principle binder is calcium hydroxide. Two distinct forms of the binder were used, a 'hot mix' consisting of quicklime (calcium oxide) and a slaked and matured lime putty mortar.
The modifications of properties exhibited by the two binders with and without the addition of pozzolans, brick dust and metakaolin were established. This study has relevance because of the lack of empirical data on the performance of modified mortars of these types. The experimental evaluation of properties including the mortar compressive and flexural strengths and the bond strengths obtained with brick demonstrated that although the hot mixed lime mortars expressed some advantages in terms of porosity over the putty based counterparts, there is no significant difference in performance regardless of the pozzolan added.

KEYWORDS ; Hot lime, Lime Putty , Pozzolan, Brick Dust ,Metakaolin

## Introduction

The use of hot lime mortars is a relatively recent and apparently popular practice in conservation[1]. Practitioners within conservation appear to generally agree that the use of hot lime mortars (otherwise referred to as hot mixed lime) gives a mortar that expresses greater durability and adhesion with the added advantage that it may be applied in cold and damp climatic conditions. These are produced on site by gauging quicklime (CaO) with wet or damp sand, with the addition of water to create the required workability. A mortar made with quicklime exhibits a degree of expansion between the stones or bricks which improves the bond between them [3] These perceived qualities have led to an increase in the use of hot mixed lime mortars by conservators. However, very little is currently published on the fundamental physical properties and performance of these mortars compared to mortars made with slaked lime putty, with some exceptions [2]. It is now generally considered that matured lime putty was largely reserved for fine plasterwork and that the majority of ordinary construction would have been quicklime, hot-mixed on site with aggregate[1].It is also worth noting that Hot mixed lime mortars are also used and applied after the initial slaking and subsequent expansion has taken place.

There is relatively little work on the properties of hot mixed lime mortars made with the addition of pozzolans, compared to lime putty mortars. This could represent a missed opportunity to extend the use of this material and understand the possible benefits of these mortars. Testing existing materials allows good performance and aesthetic matches to be achieved [4]. For this reason, additional in-depth analysis of the performance of hot mixed limes is required so as to quantify their performance not only in terms of bond strength but also workability, ease of application, water absorption, and porosity. The experimental work set out in this paper is not designed to replicate historic lime mortars but to provide a direct comparison between two mortar types with different additives.

## Materials and Method

The laboratory procedure used in Hetherington[5] was largely followed which consists of:

- Mortar preparation and evaluation of wet properties
- Creation of Brick couplet samples [5]
- Monitoring of cure temperatures
- Evaluation of mortar hardened properties and couplet strengths

For the benefit of the reader the methods are outlined briefly in this paper.

## Materials Used

### Quicklime

A proprietary brand of 90 micron powder of high purity, commercially available quicklime was used. This was chosen for its relative ease of even distribution through the mortar and because of its reaction time. Another advantage of using a powder as opposed to lump lime is that the expansion of the lime is constant throughout the mortar, as is the heating effect of the exothermic reaction. Calcium oxide is known to increase in volume during slaking with a "ratio of change in the particle volume of CaO to $Ca(OH)_2$ is 16.9/33.2.[6]. This rapid expansion and slaking when using finely divided lime reduces the risk of "Jacking and potential failure of the masonry structure" due to expansion of the masonry joints[7].

### Sand

The sand use for the project was sharp sand and should ideally be tests for its particle size distribution and its grading curve plotted as illustrated in figure 1 to a relevant standard in this case the test procedure adopted was that stated in BS 1199[8] and 1200[9] for building sand and was found to fall within the “S” type grading envelope.

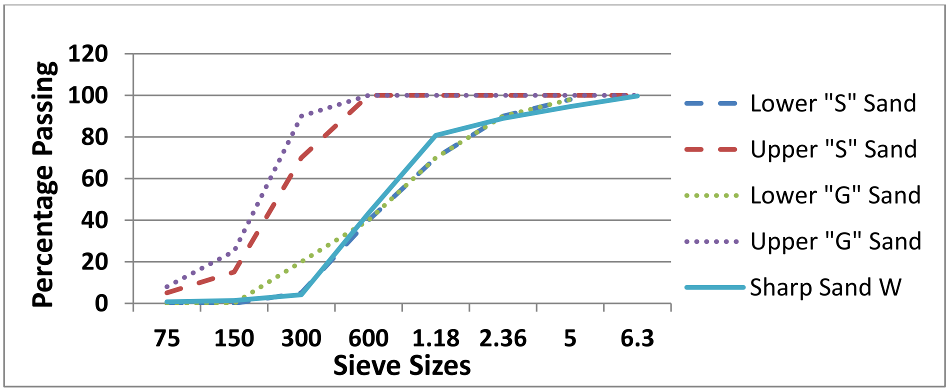



Figure 1 Grading of the sand used throughout the experiment

### Metakolin and Brick dust

The metakolin used was a proprietary brand of calcined china clay. This material is highly reactive and combines readily with calcium hydroxide to form calcium silicate hydrates and calcium alumino-silicate hydrates **[10]**. It has been used to enhance the performance of concrete. The brick dust was from a supplier of conservation materials and is a cheap and highly effective pozzolanic additive, providing a useful alternative to cement **[11].** In the presence of calcium hydroxide the reactive silica contained in these materials reacts creating products which enhance the compressive and flexural strength of lime mortars.

### Masonry units

The masonry units used to fabricate the tensile samples were solid Engineering bricks of high density. These masonry units were employed because of their predicable performance characteristics. One of the most important parameters that will affect the performance of the whole construction is the initial rate of suction of the units as this parameter will affect the bond strength of the brick /mortar composite. If the masonry unit has a high suction rate and the mortar being used is of low workability then the necessary mechanism of bond formation will be compromised**[12]**. Therefore *a* series of suction rates were carried out on 10 of the masonry units employed in the experiment to determine the initial rate of suction. This procedure was based on BS EN 772[13] and the results calculated using the equation below.

$$c_{w.i} = \frac{m_{so,s} - m_{dry,s}}{A_s t} \times 10^3 [kg/(m^2 \times min]$$

*Where: t* = 1min

$m_{dry,s}$ is the mass of the specimen after drying, (g);
$m_{so,s}$ is the mass of the specimen in grams after soaking for time t, (g);
$A_s$ is the gross area of the face of the specimen immersed in water, ($mm^2$);

The brick units tested according to BS EN 772[13] gave the following suction rate results (Table 1). Although the coefficient of variation appears high this is only due to the fact that the actual water absorbed by each unit is very small and therefore a small variation in the amount of water absorbed will appear large when expressed as a coefficient of variation.

| | |
|---|---|
| Average $kg/m^2/min$ | 0.18 |
| Standard Deviation | 0.08 |
| Coefficient of Variation | 44.3% |

Table 1

### Mortar preparation

All mixes were prepared in the laboratory with reference to the procedures set out in BS 4551[14] which was modified to take into account the properties of the materials being mixed, namely the heat evolved from the use of quicklime. This issue does not occur with the lime putty binder therefore the mixing procedure stated in the standard could be more closely adhered to. The procedure stated in BS 4551[14] was chosen as it provides a basis for replication of the mixing procedure and produces a volume of mortar suitable for the sample preparation required in the proposed regime.

The use of wet /damp sand to slake the quicklime is problematic in terms of controlling water content between batches which in turn will influence the performance of the mortar. A "speedy" moisture meter could be used to chart the water content of the sand at various times, but this would not address the problem of consistency; it would only report on the condition of that particular sample of sand. One way of ensuring a consistent water content for the sand is to "seed " the dry sand with a pre-determined amount of water. This would ensure that the water content was constant therefore controlling the water content and providing a mixing procedure that is in keeping with what would happen in the field. The mixing procedure consisted of placing a measured mass of sand into the mixer and adding 20% by mass of water.

The use of wet aggregate is analogous to site practice and is used to initiate the slaking process. The total amount of water required was determined prior to the main mixing procedure by performing a series of trial mixes**[5]**.

Quicklime was then incrementally added to the damped sand and the mortar was mechanically mixed together. This afforded more control over the heat evolved from the mortar and ensured that less water was lost from the system by the generation of steam. When all the quicklime had been added the remaining water was added and the mixing continued for a further two minutes.

Where mixes included pozzolans this was added dry prior to the wetting process.

The hot lime was then placed onto the spot board where any additional water was added to create a workable mixture as the workability of all the mortars would be a controlling factor and baseline for the mortars performance. The mortar at this stage is still in a "dynamic" condition and this period of rapid change last for between 5 to 10 minutes after this period the mortar is still hot but less dynamic and more predictable in its performance.

### Evaluation of mortar properties

Once the dynamic period had passed the dropping ball measurement technique was applied from BS 4551[14]. This is used to evaluate the workability (consistency) of the mortar.

The test evaluates the depth of penetration into a pre-prepared mould containing the mortar sample of a methyl methacrylate ball of a set mass and diameter falling from a set distance (Figure 2). A workable lime mortar is acknowledged as obtaining lower values in this test than a mortar made with Portland Cement normally anticipated to be, and adjusted to 10mm ± 0.5mm"**[11]**. An additional test used by conservators is to ensure that the conservation mortar be judged by its ability to maintain adhesion to the underside of an up-turned trowel the "practitioner test" (Figure 3).

|  |  |
|---|---|
| Figure 2 Dropping ball measurement | Figure 3 "Practitioner test" |

## Mortar samples

In order to evaluate the flexural and compressive strengths of the series of three mortars, prism samples (40mm by 40mm by 160 mm) were manufactured from the mixes used to construct the couplet samples. These were fabricated in accordance with BS EN 1015[23] and tested after 90 days for their flexural and compressive strengths.

## Dropping ball Results

The readings were taken over a 10 minute period after the initial dynamic period of the mortar and can be used to quantify the workability of each mortar mix produced thus ensuring each mortar has a comparable workability. The standard notes that "the consistence shall be adjusted to a penetration of (10 ± 0.5) mm"[14] (however this is intended for Portland cement mortars and it is well documented that conservation mortars are generally less workable. The aim of this testing is to ensure that the series of mortars have similar workabilities and this was found to be the case with consistent performance across the whole mortar set (Table 2).

| | Mortar mixes | | | | | |
|---|---|---|---|---|---|---|
| Dropping ball | Hot lime 1:3 | Lime Putty 1:3 | Hot lime Metakaolin 1:1:3 | Lime Putty Metakaolin 1:1:3 | Hot lime Brick Dust 1:1:3 | Lime Putty Brick Dust 1:1:3 |
| Test 1 | **7.2** | **8.6** | **9.1** | **9.1** | **9.6** | **8.4** |
| Test 2 | **8.1** | **9.1** | **7.9** | **8.1** | **8.2** | **8.8** |
| Test 3 | **7.8** | **8.5** | **8.5** | **8.7** | **8.9** | **9.3** |
| Average | **7.7** | **8.7** | **8.5** | **8.6** | **8.9** | **8.8** |

Table 2 Dropping ball results of each mix

**<u>Tensile Test Sample preparation</u>**

The tensile test samples were fabricated to a non-standard test that was developed to create a series of samples that are constructed in a way that reduces the influence of the operator and provides samples that express a higher degree of repeatability and therefore increases the reliability of the test [15]. This method has been found in other work[15] and is employed to provide a measure of control over the joint thickness in contrast to the stack bonding used in BS EN 1052[16]. The process of making the samples is shown in Figures 4 to 7.

Figure 4

Figure 5

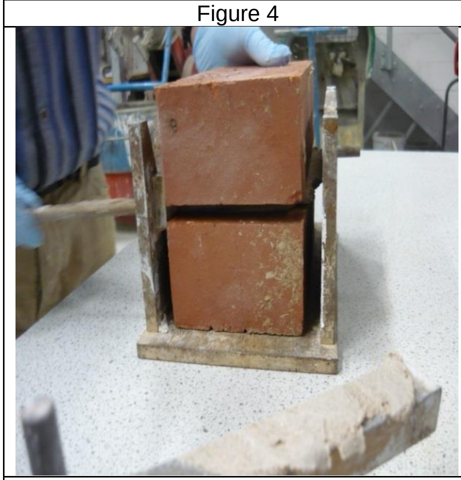


Figure 6

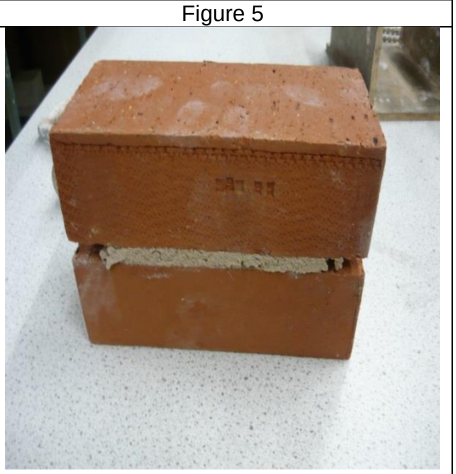


Figure 7

Figures 4-7 Illustrating the tensile sample manufacturing process

Parameters pertaining in the laboratory at the time of manufacture and conditions in the curing chamber were recorded. The laboratory temperature was 23.8 ºC with a relative humidity 43.7% and a carbon dioxide level recorded at 420ppm. Carbon dioxide content was checked to ensure that the levels did not fall during the curing period.

A series of five couplets were fabricated for each mortar mix with the samples being placed in a curing chamber for 90 days in controlled conditions prior to being tested. Temperature and humidity were maintained within the limits stated in BS EN 1015[23] (20 $^{0}$C ± 2 $^{0}$C) in this case the chamber was maintained at 65% relative humidity.

$CO_2$ levels resulted in an average reading of 420 ppm. All the samples were tested at 90 days.

The couplets were tested at 90 days using a direct tensile rig that had been developed at Sheffield Hallam University [15] and can be seen in figure 8. The calculation of the bond strength is done by dividing the load at failure in Newtons by the contact area measured in $mm^2$.

Figure 8

## Thermal Decay of Hot Lime Mortars

The thermal decay of the hot lime mortar when combined with different building units was undertaken to provide a greater understanding of the relationship between the type of building units used and the performance of the mortar.

The Building units were classified as to their suction rate performance. A low suction rate unit HD (High density engineering bricks) and medium suction rate (low density facing bricks).These units had thermocouples attached to the central portion of upper and lower bed faces prior to being fabricated into a couplets with a 10mm joint using the hot lime mortar to create samples as illustrated in Figures 9 and 10 below. The methodology for sample preparation was the same as used for the tensile test samples and the monitoring incorporated into the samples was to chart the thermal decay at the brick mortar interface.

This work was to assess the claim that hot mix mortars produced on site and applied hot may be used in adverse weather conditions where the risk of frost or low air temperatures may occur. This is in contrast to the application and use of lime putty mortars where the use and application of these mortars should be avoided if there is a risk of temperatures close to freezing.

Figure 9 and Figure 10 Brick mortar interface thermocouple set up.

The results illustrated below in figure 11 gives an indication of the likely performance of the masonry composite. The rate of temporal decay in temperature does not appear to be affected by the suction rate or density of the masonry units.  These results suggest that if these were units within a larger construction, then the overall temperature increase from the slaking process could offer protection from inclement weather.

## RESULTS

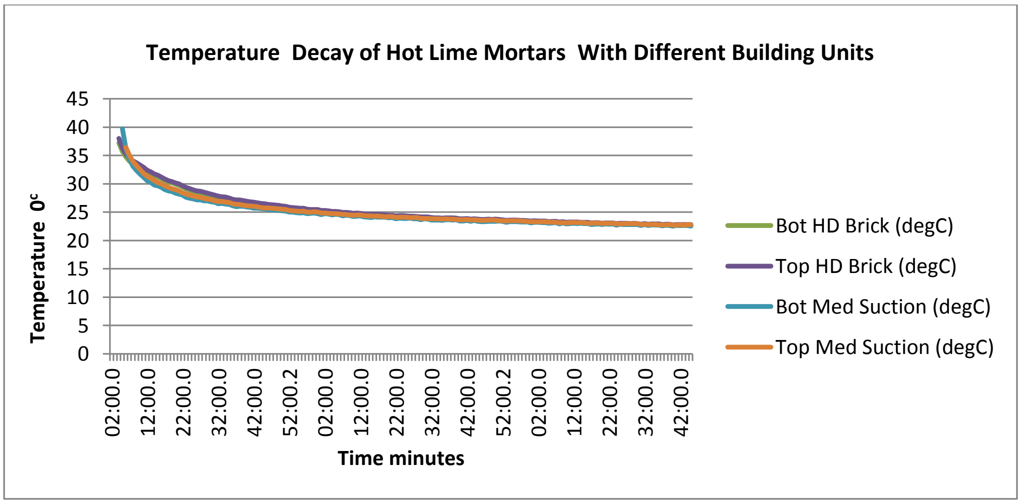



Figure 11 Thermal decay of the brick mortar interface.

**Flexural and Compressive strengths of Mortar samples**

After 90 days of cure the flexural and compressive strengths of the mortars was established in accordance with BS EN 1015[23] and is illustrated in Figures 12 and 13 with results expressed in $N/mm^2$.

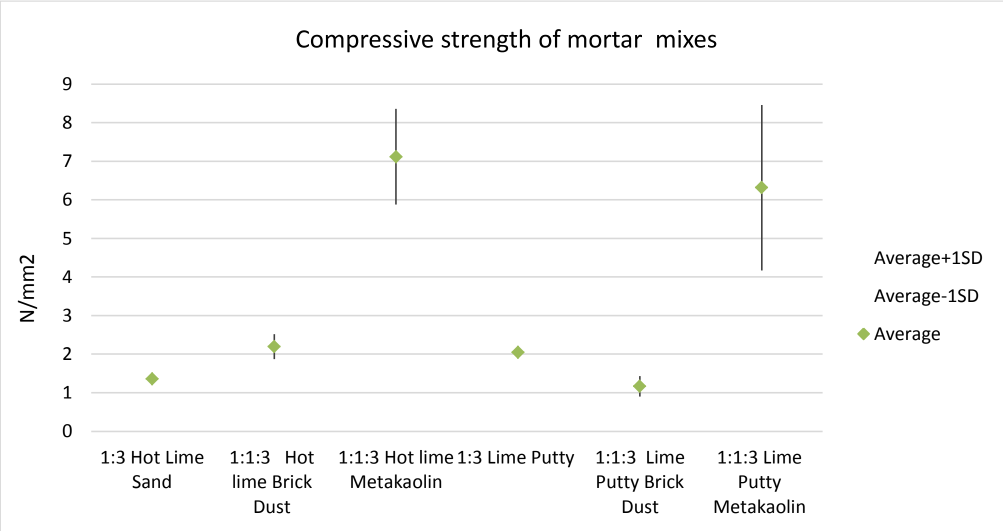



Figure 12 Compressive strength average and standard deviation range for each mortar mix

The graph illustrated in figure 12 shows the results of the compressive strength of the different mortars.

Statistical analysis demonstrates that all mixes containing metakaolin were significantly stronger than the other mixes in terms of their compressive strength. This would appear to agree with the findings that metakaolin reacted with lime more easily than brick dust [18].

There was no statistically significant difference between the strengths of the hot lime with metakaolin and the lime putty and metakaolin. Likewise the strengths of hot lime and lime putty with brick dust showed no significant strength differences. This would suggest that in terms of compressive strength, there is no functional advantage to using hot lime.

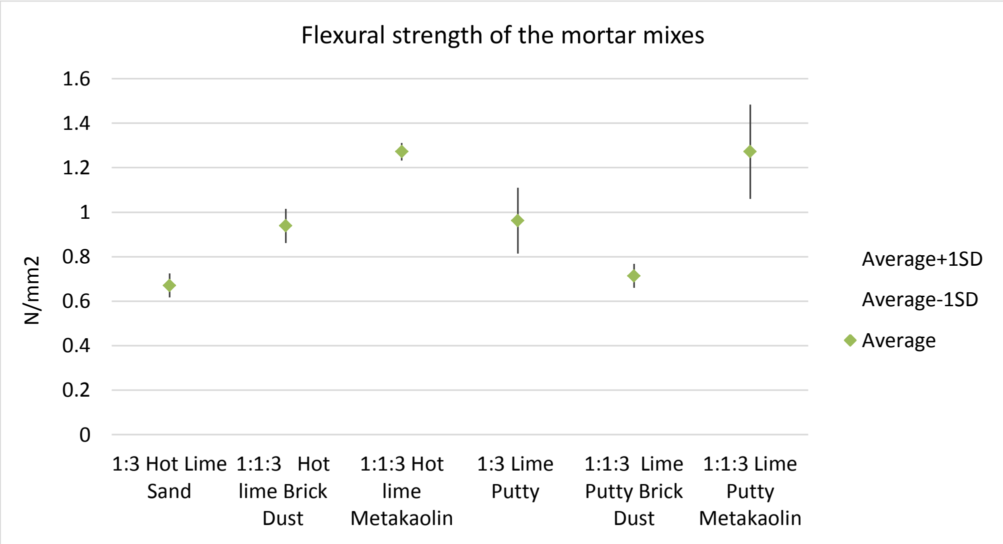



Figure 13 Flexural strength average and standard deviation range for each mortar mix

The trend established by the compressive strengths was reflected in the flexural strengths (Figure 13) with no significant difference expressed between the respective sets of results.

The results of the bond strength tests on the couplets are illustrated in the graph in Figure 14 These results suggest that the hot lime mortars offer no significant difference from their lime putty counterparts although it appears that the hot lime mortars provided a more consistent set of results with the overall average coefficient of variation for the hot lime mortars of 16%, compared to a coefficient of variation of 33% for Lime putty bond strength results. This is in part due to the additional mixing of the mortar due to expansion of the calcium oxide during slaking and is also observed in the variation of the compressive and flexural strengths of the respective mortars .The bond strength results illustrated in figure 14 follow a similar pattern to the compressive and flexural strengths with the exception of both the lime putty and hot mixed lime mortars containing brick dust. Both of these mortars illustrated greater bond strengths than all the other mortars but also were the most variable in each respective set .

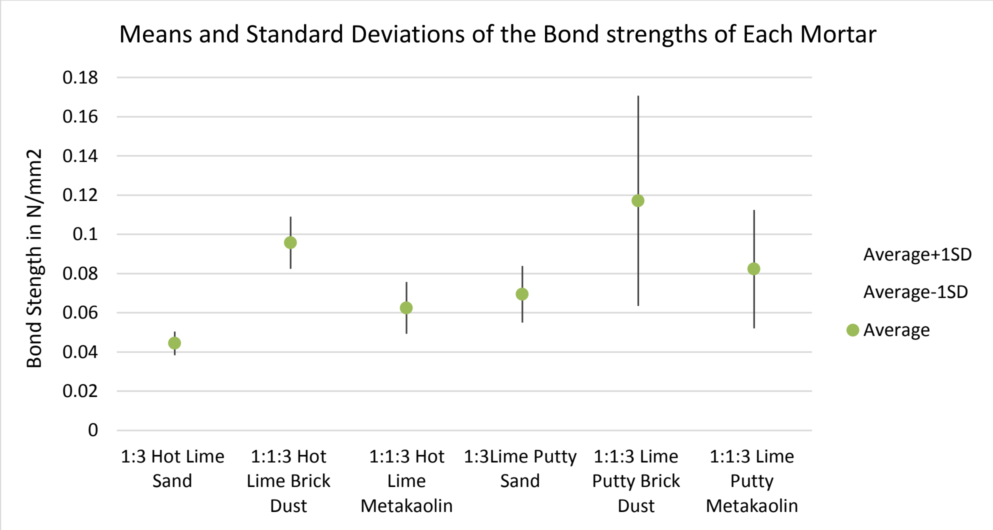
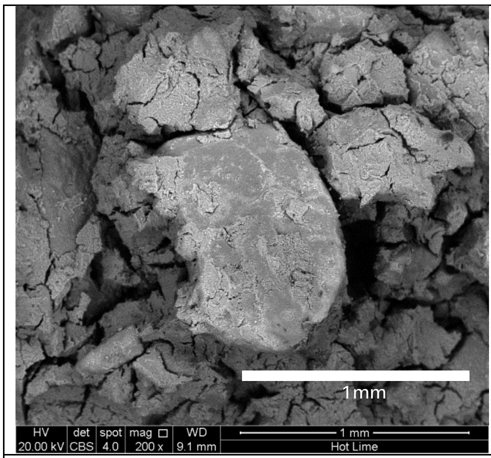
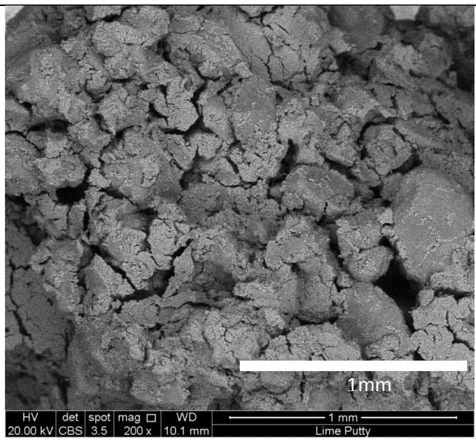



Figure 14 means and standard deviations of the bond strengths.

**SEM and MIP analysis of the mortar samples.**

In order to understand the physical differences in the mortars more fully each type was analysed by electron microscope using a QUANTA 650 Scanning Electron Microscope manufactured by FEI. Assessment of the mortars pore size and distribution by Mercury intrusion porosimetry using a Pascal 140/240 from Thermo Fisher Scientific. The samples were all tested at a maturity of 90+ days. While an understanding of pore size cannot on its own predict the durability of mortars or any other building materials it does go some way to giving an indication of its likely performance [18].

## SEM results

| | |
|---|---|
|  |  |
| Figure 15 Hotlime mortar | Figure 16 Lime putty mortar |

The Hot Lime mortar shown in Figure 15 exhibits an open pore structure with many large fissures due to the expansion and subsequent contraction of the mortar matrix.

In contrast the lime putty mortar in figure 16 shows a greater number of smaller crack and fissures. This trend of larger fissures in the hot lime mortars compared to the lime putty mortars could be observed irrespective of the additive .The addition of brick dust caused an interesting morphological change with a significant reduction in the surface topography. This is even more marked in the metakaolin samples (Figures 19 and 20). At enhanced magnification, needle like structures can be observed on the lime putty with brick dust (Figures 21 and 22). EDS analysis (Figure 23) confirmed these to be calcium aluminium silicate hydrate crystals which act to reduce the size of the pores in the mortar.

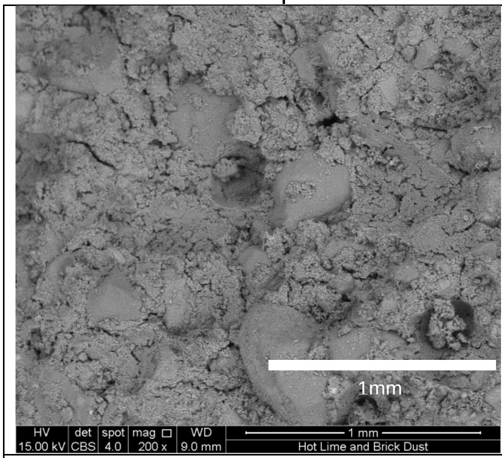



Figure 17 Hot lime Brick dust
Scale bar 1mm

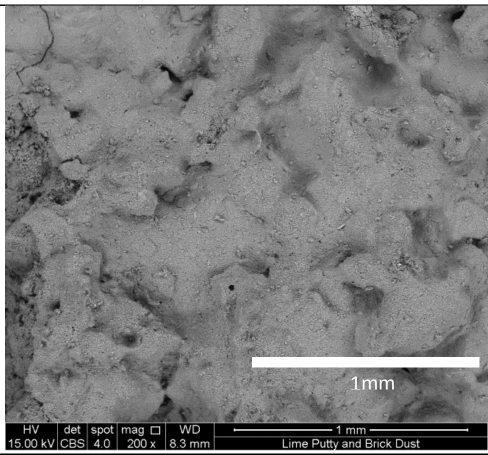



Figure 18 Lime putty Brick dust
Scale bar 1mm

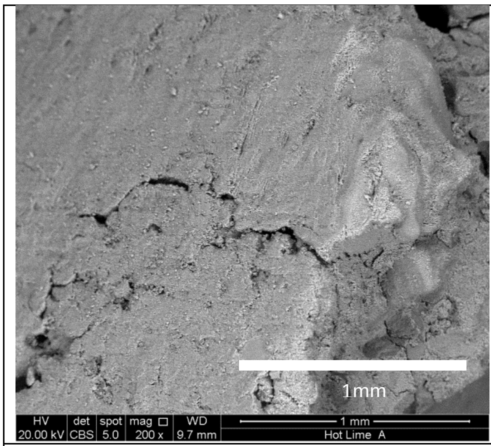



Figure 19. Hot Lime metakaolin
Scale bar 1mm

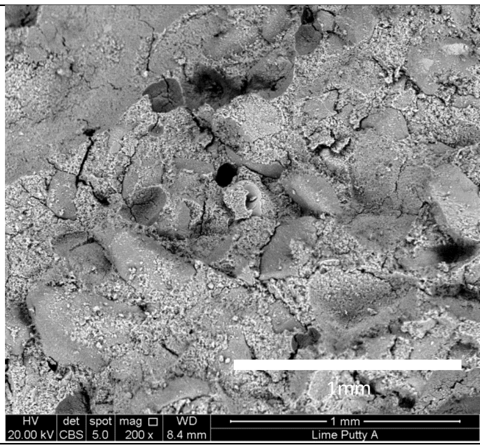



Figure 20 Lime putty metakaolin
Scale bar 1mm

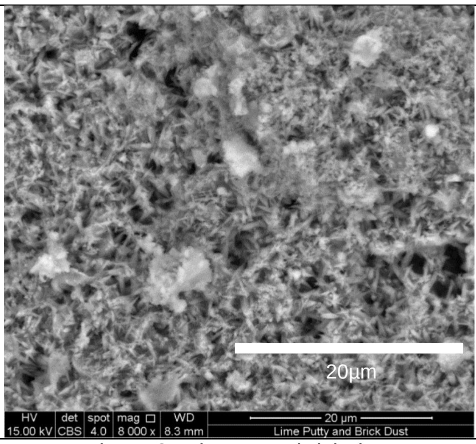



Figure 21 Lime putty brick dust

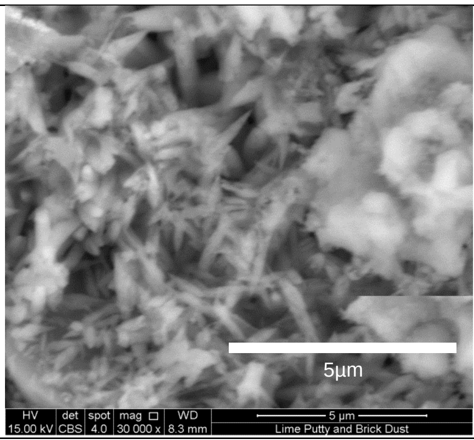



Figure 22 Lime putty brick dust

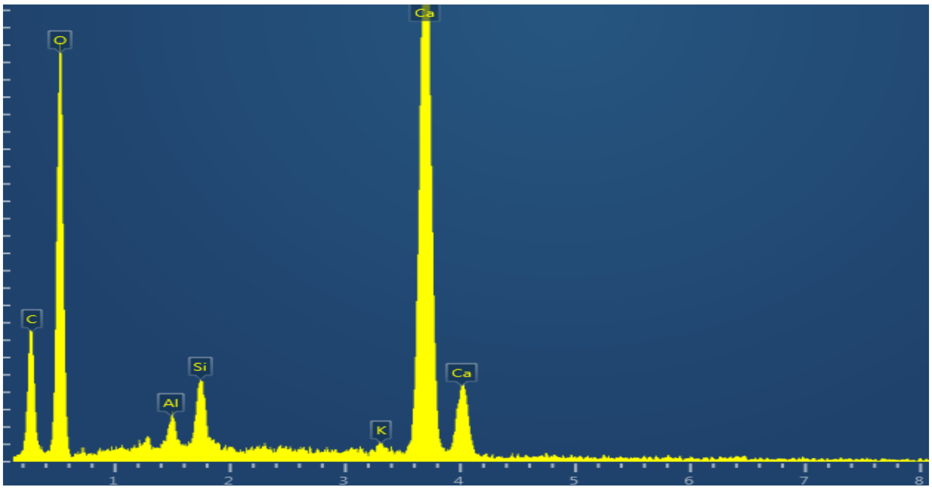



Figure 23 EDS analyses of the Lime putty brick dust sample

## <u>**Mercury Intrusion Porosimetery**</u>

The overall porosity of the samples can be seen in figure 24, with each of the respective hot lime mortars having greater porosity than their lime putty counterparts. The hot mixed lime mortars expressed overall greater porosity that the lime putty mixes.

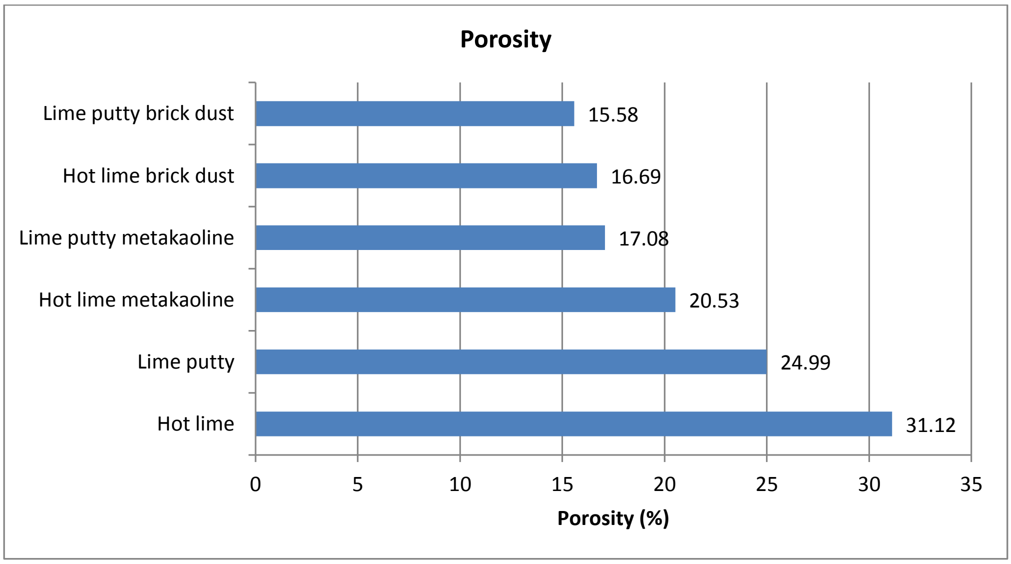



Figure 24 Overall porosity of the mortar samples

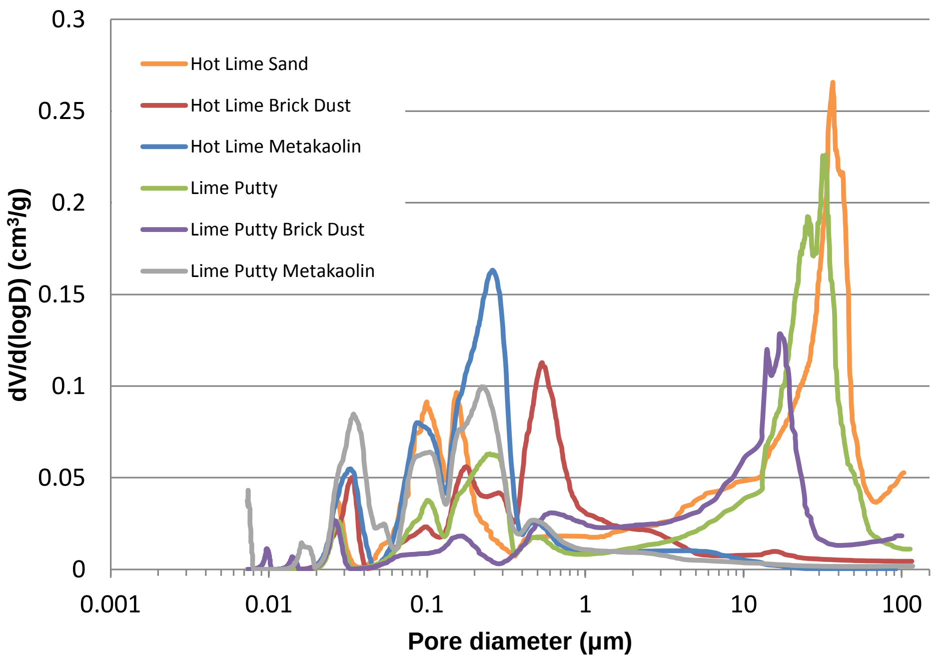



Figure 25 Pore Size and distribution in mortar samples

Figure 25 illustrates the pore size distribution and graphically illustrates the difference between the mortar samples with and without pozzolanic additives. Mortars with no pozzolan expressing a greater percentage of larger open pores than the mortars containing metakaolin and brick dust. Further comparison of results suggests that the mortars containing metakaolin have a greater percentage of finer pores than the mortars containing brick dust.

## DISCUSSION

The proposed mechanism of protection from inclement weather conditions offered by Hot lime has been shown by the increase in local temperature by several degrees for an extended period of time after manufacture.

The samples containing brick dust whether fabricated from either lime putty or hot lime displayed increased bond strength. Nezerka et al[17] observed that pastes containing brick dust did not retain water, which may influence the brick/mortar bond which develops partly due to the mechanical interlocking of cement [binder]hydration products into the surface pores of the bricks[19]. For a physically strong bond strength to occur there has to be movement of binder materials to the brick mortar interface. The effects of transport on the composition of the interfacial zone were opined to determine the development of bond strength[19] . In this case it is proposed that the presence of brick dust results in a greater degree of movement at this critical interface, and this has a role in facilitating a greater degree of bond strength.

The stronger tensile bond of the brick dust mixes is in contrast to the higher compressive strength tests exhibited by the metakaolin agreeing with the findings of Nezerka et.al[17] that metakaolin reacted with lime more easily than brick dust. The results found in this work highlight the difference between the mechanisms involved in producing a good mechanical bond. A mortar with good compressive strength may not necessarily create a good brick-mortar bond.

However the addition of pozzolans can be seen to have an influence on porosity with water sorption being higher in mortar with metakaolin[20] which could be of detriment to long term durability. A significant factor that affects the porous structures of lime mortars is the continuing chemical reactions that take place over the life of the mortar. The formation of hydration products contributes to a reduction in pores size[17]. Other work has demonstrated that metakaolin alters the pore structure of lime and cement based pastes and reduces water diffusion[21]. This work showed that there was a considerable increase in the volume of micro pores in the mortars which contained the pozzolanic additives. Calcium silicate hydrate crystals were observed in lime putty mixes containing brick dust and while this phenomenon was not observed on the hot mixed lime mortar sample this does not yet exclude the possible presence of calcium aluminium silicate hydrate within the Hot mixed lime mortar.

Further analysis of the results echo a similar trend observed in the literature where bimodal distribution of pores sizes was found in lime putty mortars without any pozzolanic additives [22]. The peaks were observed between 0.02nm and 0.8nm and the second set of peaks observed between 10 nm to 100 nm which was attributed to the formation of hydration products.

The peaks shown in (Figure 25 ) between 10 μm and 100 μm for the hot lime, lime putty and lime putty and brick dust can be explained by expansion and subsequent contraction of the former mortar creating larger shrink voids and shrinkage cause by the latter two of these mortars having excess water available for evaporation.

## Conclusions

The three Hot lime mortar mixes (hot mortar) were gauged in the following proportions 1:3 hot lime sand the second mix was a 1:1:3 hot lime brick dust ,sand and the third mix was a 1:1:3 hotline, metakaolin sand .These mixes were replicated using lime putty as a replacement for the hot lime to provide a direct comparison between the two different binder conditions.

In terms of overall porosity the hot lime mortars were consistently more porous than the lime putty mortars with the same pozzolanic additives.

The results of the compressive and flexural strength tests revealed that both the hot mixed lime and the lime putty mixes containing metakaolin gave greater flexural and compressive strength results than any of the other mortars, but were not significantly different from each other. The results of the compressive and flexural strengths of the other mortars showed that the lime putty brick dust and hot lime brick dust were also similar as were the mortars that contained no pozzolanic additives.

The results of the bond strength tests illustrated that although the mortars containing brick dust were weaker in compressive strength, they resulted in a higher brick mortar bond strengths, attributed to the brick dust allowing water to transport cementitious material (lime) to the brick mortar interface where the formation of hydration products then served to promote a greater bond strength.

Overall the results obtained indicated that although the hot mixed lime mortars expressed some advantages in terms of porosity over their lime putty based counterparts and it has previously been observed that the hot lime mix method gave slightly higher mechanical values at proportions of 1:3[2]. However, an in-depth evaluation of the test results obtained from these experiments indicates that no significant difference in bond strength, flexural strength or compressive strength was observed between the two mortar systems.

## Acknowledgements

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