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Behaviour and Properties of Lime-Pozzolan Pastes

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ABSTRACT:

Lime pozzolan mortars have been used by numerous ancient civilizations of builders, and are now regaining popularity as environmental sustainable alternatives to cements for masonry and concrete applications. This paper studies the relationship between the composition and physical characteristics of nine pozzolans and their corresponding reactivity, water demand, setting times and mechanical properties in lime/pozzolan pastes. A diverse selection of artificial pozzolans were investigated including Ground Granulated Blastfurnace Slag (GGBS); Leca; Pulverised Fuel Ash (PFA); Calcined Clay (MetaStar); Microsilica (MS); Rice Husk Ash (RHA); Red Brick Dust (RBD); Tile and Yellow Brick Dust (YBD).

Each pozzolan was found to have a particular water demand for a given workability that was related to the particle surface area. It was concluded that the pozzolan surface area has a much greater influence on the water demand of the paste than its particle size or the lime:pozzolan ratio. It appears from this research, that the effect of the water content of the paste on the setting times surpasses the influence of the reactivity of the pozzolans. It was also demonstrated that the particle size of the pozzolan critically affects the set of the paste, both the coarsest and the finest pozzolans set the fastest; and there is a critical particle size, ranging between 63 and 250um, for which the set slows down significantly. Finally, it was also noted that pozzolanic reaction takes place at variable speed for different pozzolans, and that there can be instances when hydration can be slower than carbonation.

Keywords: Pozzolans, lime-pozzolan paste, reactivity, water demand, setting times, compressive strength.

1 INTRODUCTION

In many ancient civilizations, pozzolans were used to enhance the properties of lime, and many structures are still extant as a testament to the durability of lime-pozzolan mortars and concrete. According to Caijun, refering to Malinowski et al. [1] evidence of the use of pozzolans has been found in Galilee dating from the Neolithic period (7000 BC), the Minoan civilization (2700 to 1450 BC) [2] and Ancient Greece (1500BC) (Moropoulou et al, refering to Jiang&Roy [3]). The Roman Empire is however the most synonymous with the use of pozzolans, the name deriving from the volcanic rock found near Naples called pozzolana. The Romans also used artificial pozzolans such as brick and tile dust. According to historic records [4,5,6,7] and current research [8] the Romans preferred pure lime to the more hydraulic types, making it hydraulic with the addition of pozzolans.

The use of lime binders decreased with the development of cements in the early 19th century, however these are regaining popularity as an environmentally sustainable alternative to concrete. Their environmental credentials are based on their lower production energy consumption and the

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reabsorption of the CO₂ emitted during burning. Pozzolans enhance the properties of lime mortars and therefore increase the practicability and potential opportunities of lime. Pozzolans are often industrial or agricultural by-products and their use is often an alternative to disposal.

Pozzolans are materials with an amorphous siliceous or siliceous and aluminous content that react with calcium hydroxide in the presence of water to form cementitious hydration products (calcium silicate hydrates and calcium silicate aluminate hydrates). Pure lime hardens by carbonation, however the introduction of pozzolans alters the hardening process of the lime mortar by imparting a hydraulic set. The hydration products of lime/pozzolan pastes are similar to those found in a hydraulic lime mortars and cements [9] although their formation is considerably slower than cement pastes. Lime with hydraulic properties has faster setting times, higher mechanical strength and lower permeability and flexibility along with improved resistance to salt, frost and moisture damage [10].

The pozzolanic reaction depends on the chemical and mineralogical composition of the pozzolan, the type and proportion of their active phases, the particle's specific surface area, the ratio of lime to pozzolan, water content, curing time and temperature [9].

This research was undertaken in order to ascertain the influence of some physical properties of pozzolans on the pozzolan's reactivity, and on the setting time, water demand and mechanical performance of lime pozzolan pastes.

2 MATERIALS AND METHODS

2.1. Materials

A hydrated commercial lime (CL90s) complying with EN 459-1 [11] was used. Nine pozzolans were investigated including Ground Granulated Blastfurnace Slag (GGBS); Leca; Pulverised Fuel Ash (PFA); Calcined Clay (MetaStar); Microsilica (MS); Rice Husk Ash (RHA); Red Brick Dust (RBD); Tile and Yellow Brick Dust (YBD). Their chemical composition is included in table 1. Two lime:pozzolan ratios 1:1 and 1:3 (by weight) were investigated with water content to produce a flow diameter of 165mm.

2.2. Methods

Chemical composition and amorphousness of the pozzolans

The chemical composition was determined by XRF analysis, with a ThermoFisher Scientific and Edwards Analytical using a Quant'X EDX Spectrometer and UniQuant analysis package. An elemental quantitative analysis was carried out. The samples were mixed with Hoescht wax in the ratio 0.1 wax/sample, and pressed into aluminum sample cups using a hydraulic press at 10 tonnes for 60 seconds. The amorphous character of the pozzolan was assessed by X-Ray diffraction (XRD), using a Phillips PW1720 XRD with a PW1050/80 goniometer and a PW3313/20 Cu k-alpha anode tube at 40kV and 20mA. All measurements were taken from 3 to 60 degrees (20) at a step size of 0.02 degrees/second.

Particle size and surface area of the pozzolans

The particle sizes were analysed using a Malvern Mastersizer 2000 based on the measurement technique of laser diffraction. The specific surface area was measured using a Quantachrome Nova 4200e and the BET method, a model isotherm based on adsorption of gas on a surface.

Pozzolan reactivity (Activity Index)

Pozzolanic reactivity indicates how quickly after mixing the pozzolanic material reacts with the calcium hydroxide and the rate at which the reaction takes place. In effect it is a measure of how effective the pozzolan is. Accurately deducing a pozzolan's reactivity is complex [12] and dependant on the methodology. For this reason, the reactivity was determined using two methods.

The mechanical method was undertaken according to EN 450-1 [13]. This measures reactivity by monitoring the compressive strength development of a lime: pozzolan mix in relation to a standard

lime mix at constant water content over 28 days. The same method was applied to all pozzolans. A fixed water content was not possible as the pozzolans have different water demand, thus the same quantity of water yielded a sloppy mix with some pozzolans and a dry "breadcrumb" mix with others. Therefore, in order to determine a suitable water content, the pozzolans were mixed to flow and, according to the results obtained, they were subdivided into two groups of high and low water demand. The GGBS, Leca, PFA, RBD, Tile and YBD had a lime:pozzolan:sand:water ratio of 1:1:3:1 and therefore a binder:water ratio of 0.5. The remaining three pozzolans: Meta, MS and RHA, have a higher water demand and a ratio of 1:1:3:1.5 was selected for them. The water content was therefore fixed for the two groups. The prisms 160mmx40mmx40mm were demoulded after 1 day and stored in a curing room at a temperature 20°C ±3°C under damp hessian to maintain humidity at c.95 %. The compressive test was carried out using a Zwick testing machine at a loading rate of 400 ± 40 N/s.

The chemical method followed the pozzolanic reaction indirectly by measuring the changes in the conductivity of a saturated lime and pozzolan solution. The fixation of dissolved Ca(OH)₂ by pozzolan particles forming hydration products as well as carbonation reduce the portlandite concentration in solution and consequently leads to a decrease in conductivity. A saturated solution of lime in distilled water (approx 0.5g per 75g of water) provided an electrical conductivity reading of approx 9.8mS at 20°C. 4g of pozzolan was added to the solution. The solution was continuously stirred by a magnetic stirrer and the conductivity (in Siemens) and temperature was measured at intervals over 125hours using a WTW LF 197 conductivity meter with Tetracon 325 probe.

Water demand of the lime/pozzolan pastes

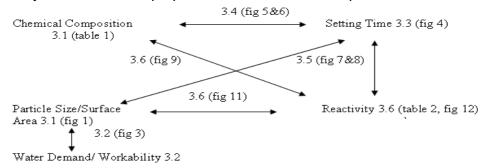
This was assessed by measuring flow in accordance with EN 459-2 [14] for lime:pozzolan ratios of 1:1 and 1:3. The water content was adjusted to produce a flow a diameter of 165mm after 15 jolts on the flow table. Mixing was in accordance with EN 459-2 [14] except for the addition of the pozzolan which was added after 1 minute and the mixing stopped for 30 seconds.

Setting of the lime/pozzolan pastes

The Vicat test [14] determines the rate of stiffening of the lime mortar by dropping a needle from a fixed height and measuring its penetration into a sample. In this research, the stiffness of the mortar is related to the formation of hydrates and the rate of carbonation. The initial and final setting times (at 35mm and 0.5mm respectively) are standard references which provide comparative data between samples. The Vicat test was carried out on lime:pozzolan samples, in the ratios 1:1 and 1:3, with the water content required in order to produce a flow of 165mm.

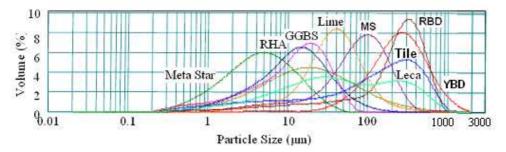
3 RESULTS AND DISCUSSION

The interdependency of pozzolan properties and their corresponding influence on workability, setting times, reactivity and mechanical properties and their relationship is abbreviated in the chart below.



3.1. Physical properties and composition of pozzolans

The particle size distribution and surface area of the pozzolans are presented in Figure 1. The microsilica particles flocculated during the laser analysis on account of their extremely small size, therefore the MS particle size result is not reliable.



	Surface Area
Pozzolan	m2/g
GGBS	2.65
Leca	1.28
Meta	18.33
MS	23.69
PFA	4.09
RBD	4.29
RHA	13.70
Tile	4.16
YBD	0.31

Figure 1. Particle size distribution of pozzolans and surface area of pozzolans in m²/g.

By comparing the particle size and surface area of the pozzolans, an indication of the shape and surface texture of their particles can be obtained. The PFA, RBD and tile pozzolans have a similar surface area and a different particle size indicating that the PFA, with the lowest particle size, has a smoother surface and/or more regular geometry than the tile and RBD.

The chemical composition and amorphousness are included in Table 1. The amorphous character of the pozzolan is represented on the XRD by a broad hump which that cannot be accurately measured, however the degree of amorphousness of the pozzolan is indicated by the size of the hump [15].

Table 1. Chemical and mineral composition of the pozzolans analysed by XRF and XRD respectively (chemical analysis as weight percentage by oxides).

				T	1	1	T		T	T
Pozzolan	SiO2	Al2O3	CaO	Fe2O3	SO3	TiO2	MnO	K2O	MgO	P2O5
GGBS	34.14	13.85	39.27	0.412	2.43	0.54	0.25	0.26	8.63	
	Totally amorphous									
Leca	52.78	24.39	3.59	11.42	0.397	0.886	0.37	2.82	2.7	
	Medium sized hump									
Meta	51.37	45.26		0.521				2.13	0.55	
	Large amorphous hump									
MS	92.1	2.13	1.1	1.62	0.287			1.32	1.05	0.23
	Large amorphous hump									
PFA	65.32	24.72	0.947	4.84	0.378	0.914		1.37	0.68	0.37
	Medium to small amorphous hump									
RBD	48.24	22.15	10.31	6.67	6.94	0.91		2.97	1.17	0.26
	Very low amorphous content									
RHA	93.84	1.93	0.68	0.29			0.12	1.38	0.45	1.11
	Large amorphous hump									
Tile	46.61	21.47	11.34	7.19	7.62	0.96		3.05	1.12	0.2
	Very low amorphous content									
YBD	43.9	44.94	0.36	2.11		0.4		1.27	6.28	0.26
	Very low amorphous content						•			

3.2. Water demand

The results indicate that the water demand of the paste depends on the pozzolan's particle size, its specific surface and the lime:pozzolan ratio of the mix. However, the results indicate that the surface

area of the pozzolan has a much greater influence on water demand than its particle size or the lime:pozzolan ratio.

According to the flow test, finer pozzolans [Figure 1] have a greater water demand [Figure 2]. This agrees with the well known relationship of finer particles requiring a greater water content to provide a given workability

The influence of the surface area of the pozzolan on the water demand of the paste is included in figure 3. As expected, a greater amount of water was required by the pozzolans with a higher surface area [Figure 1]. However, the surface area of the pozzolan has a much greater influence on water demand than its particle size (RHA and GGBS have a similar particle size however RHA has a greater surface area and a significantly higher water demand). Finally, the surface area of the pozzolan has a much greater influence on water demand than the amount of pozzolan in the mix; this is demonstrated by the bigger difference in water demand between the ratio 1:1 and 1:3 for a pozzolan of low surface area than for one of a higher surface area [Figure 3].

As it can be seen from Figure 2, there is a clear linear relationship between water demand and pozzolan content: the replacement of lime by pozzolan lowered the water demand of all lime:pozzolan pastes with the exception of Metastar. This is likely due to the great water demand of the Metastar due to its small particle size and high specific surface.

It was also noted, that each pozzolan needed a specific amount of water in order to achieve a flow of 165mm. An equation was formulated in order to calculate the water demand of each pozzolan (WDP) [Equation 1]. This was based on the water demand of the lime (water: lime ratio for the lime to flow 165mm is 0.862), the amount of lime and pozzolan, and the total water content of the paste.

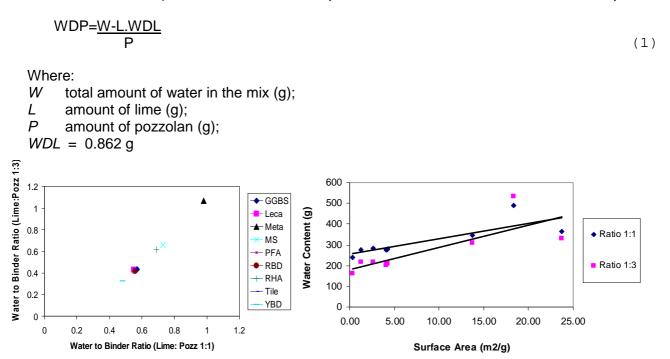


Figure 2. Water demand of pozzolans in lime:pozzolan pastes of ratios 1:3 and 1:1

Figure 3. Influence of pozzolan surface area and lime: pozzolan ratio on water demand

The WDP (expressed as grams of water per gram of pozzolan) was calculated according to equation 1 as an average of two ratios (1:1 and 1:3). The difference between the two ratios was typically under 10% except for the Leca and YBD. This was probably due to non-homogenous character of these pozzolans, including a wide range of particles sizes. According to equation 1, GGBS has a water demand of 0.28g, Leca 0.27g, Meta 1.1g, PFA 0.25g, RBD 0.26g, RHA 0.53g, Tile 0.26g and YBD

0.12g. Mixes 1:2 lime:RHA and 1:2 lime:PFA were tested for flow using the water demand calculated with the equation. These produced flows of 162/165 and 165 respectively, thus validating the equation.

According to the results obtained, the pozzolans were divided into two groups of high (Meta, MS and RHA) and low (GGBS, Leca, PFA, RBD, Tile and YBD) water demand respectively.

3.3. Setting time

The results evidenced that the setting time of lime-pozzolan pastes is highly sensitive to the water content of the paste. It became clear that a small increase in the water content (5%) significantly slowed down the setting times. The great influence of water content on setting time was also evidenced in the Metastar paste. Here, the setting time had to be measured in a 1:3 rather than a 1:1 paste because it set faster, providing values comparable to those of the other pozzolans in 1:1 pastes, due to the great water requirement of this pozzolan.

All the pozzolans reduced final setting time of the lime paste by at least 40% [Figure 4]. In addition, the pozzolans speed up the initial set of the lime paste except for PFA and MS [Figure 4]. This suggests that, for these two pozzolans, at early stages, the pozzolanic reaction is slower than the rate of carbonation, however after 22 hours the rate of pozzolanic reaction overtakes the rate of carbonation of the paste.

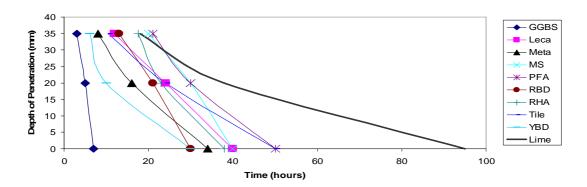


Figure 4. Setting time of lime and lime:pozzolan pastes at ratio 1:3, except for Metastar with a ratio of 1:1 (each value is an average of 3 results).

3.4. Setting Time of pozzolans vs Chemical Composition

The results demonstrated that an increase in the silica content of the pozzolan slows down the initial setting time of the lime/pozzolan paste. It was evidenced that pozzolans with high silica content (MS, PFA and RHA) were slower to initially set [Figure 4]. These high silica pozzolans are the finest, therefore, their slower setting may be partially due to their higher water demand. However, the same relationship between silica content and setting time is maintained for the five remaining pozzolans (of variable silica content, particle size and water demand). In particular, the silica content of the pozzolan slowing down the initial set of the paste became clear with the GGBS, PFA, RBD, Tile and Leca pozzolans, as these have very similar water and alumina contents which eliminate other variables that affect setting [Figure 5]. The results also suggest that increasing amounts of Ca and Mg in the pozzolan lead to a faster initially set of the paste [Figure 6].

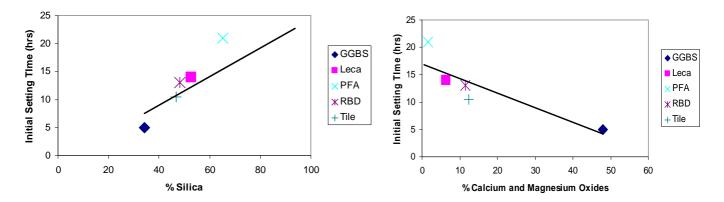


Figure 5. Relationship between the silica content of the pozzolan and the initial setting time of the paste.

Figure 6. Relationship between the calcium and magnesium content of the pozzolan and the initial setting time of the paste.

With the exception of GGBS, the YBD and Metastar (pozzolans with highest Al_2O_3 content 45%) initially set the fastest. Although it can be argued that the low water demand of the YBD and the high specific surface/small particle size of the Metastar contribute to their fast initial set, the fast initial set of the red brick dust, tile dust and Leca (also with a significant alumina contents 21-24%) further supports the trend. This agrees with previous research stating that aluminates, despite contributing little to strength, are responsible for the early set of a paste [16].

According to the Vicat test, the GGBS paste sets significantly faster than any other paste. It is the fastest to develop an initial set and takes only 7 hours to fully set. GGBS has a lower alumina content (13.85%), however, it is the only pozzolan that is totally amorphous and has a high calcium content (39.27%). Therefore, these may be responsible for the early set of the GGBS paste. This suggests that the rate of amorphousness and the calcium content of the pozzolan have a greater effect than the alumina content of the pozzolan on the setting of the paste.

3.5. Setting Time of pozzolans vs particle size

In order to investigate the effect of particle size on setting times, the YBD was divided into six size factions and 1:1 lime:pozzolan pastes prepared with each fraction at fixed water contents.

It is widely reported that as particle size decreases, the rate of reaction increases [16],[17]&[18]. This is attributed to an increase in the reaction speed due to smaller particles having a larger surface area available for reaction. However, according to this research, both the largest and the smallest pozzolans set the fastest: the results in figure 7 show that, both the coarsest yellow brick dusts (particles sized above 250um) and the finest brick dusts (particles sized under 63um) set faster than the intermediate particles (sized between 63um and 250um). A theory suggested by [20] in relation to cements would explain the shortest setting time of coarser pozzolans as follows: a lime/pozzolan paste sets as hydration products link particles of lime and pozzolan together. Larger particles have smaller surface areas therefore need fewer bridges and consequently set faster.

It was also noted that there is a critical particle size (63-250um) for which the set slows down significantly [Figure 7]. This means that there is a particle size detrimental to set: when the pozzolan particles are sized between 63 and 250um, the pozzolans do not benefit from either of the aforementioned effects that would speed set (neither a larger surface is available for reaction nor fewer hydration bridges will suffice for the paste to set). A similar analysis undertaken on the RBD also supported the existence of a similar critical particle size.

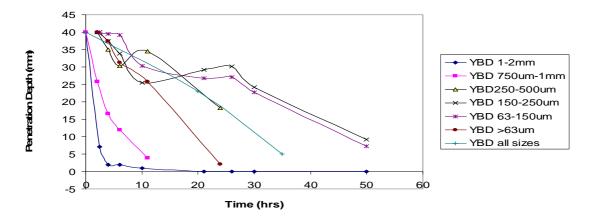


Figure 7. Influence of particle size on setting time: degree of set (depth of penetration) vs time for the YBD paste. Critical particle size that retards set.

The relationship between setting time and particle size of the pozzolan is represented by plotting the final setting time against the square root of size D90 (90% of the particles under this size) [Figure 8]. According to the results in the trendline, the D90 value at which the final setting time is maximum is c.225um ($\sqrt{D90}$ =15), which corroborates the YBD findings above.

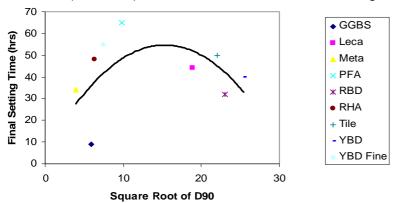


Figure 8. Relationship between pozzolan particle size and final setting time.

3.6. Reactivity of the pozzolans (Activity Index

As it can be seen from the results below, both the mechanical and chemical activity indices position the pozzolans in the same order of reactivity (from higher to lower): Meta, GGBS, MS, RHA, Leca, PFA, YBD, Tile, RBD.

Mechanical Activity Index

The pozzolanic index measured by the mechanical method, expressed as the ratio of the compressive strength of the lime/pozzolan mix to a standard lime/sand mix, is listed in Table 2. In all cases, the addition of the pozzolan increased the compressive strength of the lime mix. Meta and GGBS produced the highest mechanical activity index, followed by the high-silica pozzolans RHA and MS with little separation between PFA, Leca and the brick dusts.

Compressive strength and hence the mechanical activity index, is not only dependant on the amount of hydration products formed but also on their type and microstructure [21], with silica hydrates contributing more to strength than alumina hydrates. This agrees with the results obtained [Figure 9], as it was evidenced that increasing silica content results in a higher mechanical index.

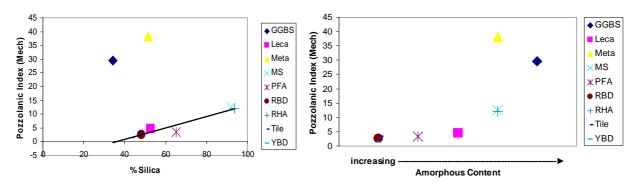


Figure 9. Relationship between silica content and reactivity (mechanical index).

Figure 10. Relationship between increasing amorphous content and reactivity

The silica content above refers to both crystalline and amorphous silica. However, crystalline silica reacts little with lime and its pozzolanic contribution is considered negligible thus, in order to be reactive, silica must be present in an amorphous form. Therefore, the rate of amorphousness (Table 1) was loosely categorised into 5 groups ranging from totally to slightly amorphous. According to the results, a clear relationship between increasing amorphous content and increasing reactivity was established for the nine pozzolans [Figure 10].

According to the results obtained, reactivity, measured as strength development, increases with decreasing particle size [Figure 11]. The filler effect of fine pozzolans, filling the space between large particles and thus increasing packing and density, may be partially responsible for the high strength of the finer pozzolans GGBS, Meta and RHA.

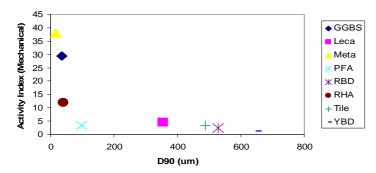


Figure 11. Relationship between particle size and activity index (mechanical)

Chemical Activity Index

The chemical method, in which the changes in conductivity of a lime/pozzolan in solution was measured over time, depicted four phases of activity similar to the four stages defined by [22]. The results are shown in Figure 12. During the first 3-5 hours (phase 1), the conductivity reduced significantly by between 10%-30%. The rate of this decrease was typically between 3 and 10% per hour. This was followed by a latent period (phase 2) where there was a gradual reduction in conductivity, the length of which depends on the reactivity of the pozzolan varying from approximately 20 to 90 hours, with a rate of decrease of less than 1% per hour. The latent period was followed by a significant drop in conductivity of between 40 and 70% (phase 3), at a drop-rate of over 1.5% per hour, extending over a period of approximately 20 hours. Following this rapid decrease, the reaction appears exhausted and a constant conductivity was maintained (phase 4).

The rate of change of conductivity during phase 1 and the duration of phase 2 are used to quantify the rate of pozzolanic activity of the material [18], [22], & [23]. The results evidenced that pozzolans

Meta, RHA and MS have the highest activity index followed by PFA, YBD, Tile and RBD. GGBS and Leca do not have a distinct phase 2 or 3 and their conductivity continuously decreases over time therefore, their reactivity cannot be determined using this method.

Table 2. R	eactivity of Pozzolans
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	Activity Index				
Pozzolan	Mechanical Method	Chemical Method (conductivity)			
	(strength ratio)	Phase 1 % drop	Latent Period (hrs)		
GGBS	29.5	-	-		
Leca	4.6	-	-		
PFA	3.4	3.4	65.0		
Meta	38.1	9.8	18.0		
MS	12.5	4.3	39.5		
RBD	2.5	2.5	91		
RHA	12.0	6.8	55.0		
Tile	3.3	3.5	90.0		
YBD	1.2	3.9	67.0		

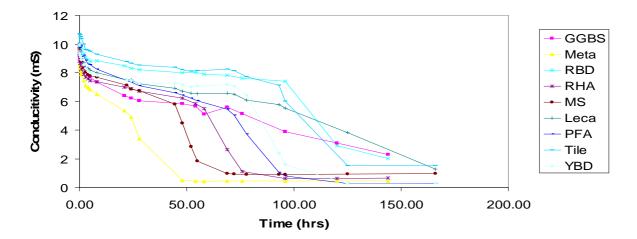


Figure 12. Pozzolanic activity as change in conductivity over time (chemical activity index).

4 CONCLUSION

The behaviour and properties lime/pozzolan pastes are determined by complex relationships of interdependent variables including pozzolan surface area, particle size, chemical composition and amorphousness. These govern the water demand of the lime/pozzolan paste which in turn affects hydraulic set, mechanical properties and reactivity

This paper concludes that the setting time of lime-pozzolan pastes is highly sensitive to the water content of the paste; and that a small increase in water content significantly slows down the set. According to this research, both the coarsest and the finest pozzolans set the fastest; and there is a critical particle size, ranging between 63 and 250um, for which the set slows down significantly.

This paper also concludes that the surface area of a pozzolan has a much greater influence on the water demand of the paste than its particle size or the lime:pozzolan ratio. The research also concludes that each pozzolan has a specific water demand, and that the pozzolans studied can be

clearly divided into two groups of high (Meta, MS and RHA) and low (GGBS, Leca, PFA, RBD, Tile and YBD) water demand respectively.

Finally, it can be concluded that the pozzolans speed up the final set of lime by at least 40%; and that, with the exception of PFA and MS, they also accelerate the initial set of the lime paste. These suggest that pozzolanic reaction takes place at variable speed for different pozzolans, and that there can be instances when hydration is slower than carbonation.

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