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Pore size distribution and compressive strength of waste clay brick mortar

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Abstract

This paper reports the results of an investigation of the pore size distribution of mortar that contains varying amounts of ground brick from different European brick types. Clay brick deriving from four European countries was ground to roughly cement fineness and used to partially replace cement in quantities of 0%, 10%, 20% and 30% in standard mortars. The pore volume, pore size distribution, threshold radius and strength of these mortars were tested for curing periods of up to one year. The presence of ground brick (GB) alters significantly the compressive strength of mortar and this is attributed to both the dilution effect and production of additional C–S–H gel from reaction of GB with CH. The additional C–S–H gel refines the pore size distribution of the mortar and this is reflected in compressive strength values obtained for these mixes. A critical relationship between threshold radius and compressive strength is also observed. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Mortar; Pozzolan; Ground brick; Mercury intrusion porosimetry; Compressive strength

1. Introduction

The strength of mortar or concrete is generally considered to be its most important property, although in practice, other characteristics such as durability and permeability are of equal importance. The key parameters that influence these characteristics in mortar are its porosity and pore size distribution. The pore structure develops during hydration and is influenced by the interaction between the various aggregates present in the mix and the hydrating cement paste. Porosity is a major component of the microstructure of hydrated cement paste and influences strength (compressive and flexural), permeability, durability, diffusion, fracture toughness, Young's modulus, shrinkage and creep properties of the paste [1]. Nevertheless an overall indication of the quality of the mortar or concrete can be gained from its strength, as this is directly related to the structure of the cement paste [2].

It is generally accepted that the strength of mortar is fundamentally a function of the form and distribution of void space and porosity within it [3–5] and is influenced primarily by the pore size distribution rather than by the total cumulative pore volume as has been previously reported [4,6,7]. Strength development observed during hydration is now thought by some to be controlled by two factors. At early stages of hydration maximum pore size is a key factor whilst at a later stage, porosity becomes a controlling factor [8]. This view is reflected by the behaviour of cement paste that has been shown to develop smaller total porosities and smaller threshold pore widths as curing times increase [9]. Within hydrated cement there are two distinct pore populations, gel pores and capillary pores [3,4]. Work by Kendall et al. [4] indicates that gel pores (pores within the cementing gel) are too small to initiate cracking under relatively low stress and therefore do not detract from the strength of the mortar. Capillary pores (residual pores from the hydration of cement), however, can be some millimetres in length and are sufficient to initiate cracking thus reducing the strength of the mortar [4]. Although the distinction between capillary pores and gel pores with respect to pore diameter is not well defined, it is assumed generally that gel pores have diameters in the range 1–3 nm and capillary pores in the range 10–5000 nm [10].

During cement hydration large capillary pore spaces are filled with the hydration products, thus refining the size of these pores and at the same time increasing the

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cumulative volume of very fine gel pores [8,11]. With the addition of pozzolanic materials to the mix, CH produced by the cement hydration reacts with the pozzolan and produces additional C–S–H gel which has a pore blocking effect, further refining the pore structure [12,13]. For some alumino-silicates, pozzolanic activity can be imparted by heat treatment such as the conversion of kaolinite clay (weakly pozzolanic) to metakaolin (highly pozzolanic) at 700–800°C. Other pozzolans such as PFA are produced by heating alumino-silicates to even higher temperatures (>1000°C) the pozzolanic component in this case being an amorphous glass phase. As bricks are manufactured by the calcination of alumino-silicate clays, it seems logical that bricks will possess pozzolanic tendencies when ground to suitable fineness. It has been shown [14,15] that with increasing calcination temperatures up to ≈1000°–1100°C, brick clays, when incorporated in a cementitious material, exhibit qualities similar to other recognised pozzolans such as PFA.

Table 1
Chemical composition of standard PC[a]

SiO_2	Al_2O_3	Fe_2O_3	CaO	SO_3	Free Lime
20.0	4.3	2.3	64.0	3.0	1.0

[a] Data provided by Blue Circle Industries plc.

Table 2
Mineralogical composition of standard PC[a]

C_3S	C_2S	C_3A	C_4AF
61.7	11.4	7.6	6.8

[a] Data provided by Blue Circle Industries plc.

2. Materials and methodology

2.1. Materials

The cement used was Portland cement (PC), supplied by Blue Circle Industries plc to BS12: 1996 [16] the composition of which is given in Tables 1 and 2. The sand used in the mortar mixes was DIN EN 196-1 'Normensand', a standard European sand (complying with clause 5 of EN 196-1: 1995 [17]) imported from Germany. Eight brick types were chosen initially from a variety of sources across Europe. Four were selected for a detailed investigation (referred to as B, D, L and P). Basic information on the oxide content and mineralogy of these brick types can be found in Tables 3 and 4. The bricks were ground to a specific surface of 320–350 $m^2\ kg^{-1}$ as determined by a constant flowmeter air permeability apparatus [18].

2.2. Mixing

The mortar mixes had proportions of 1 binder:3 sand. The binder consisted of cement and ground brick (GB). The water:binder (w/b) ratio was kept constant at 0.5. The cement was partially replaced by 0%, 10%, 20% and 30% of the various GB types. Each mix was assigned a code e.g. B10 represents 10% partial replacement of cement by GB type B. In all cases 0% replacement of cement in mortar is referred to as the control. The partial replacement of PC by GB in the range 0–30% was found to have little influence on the workability as

Table 3
Composition of GB types B, D, L and P[a]

GB type	SiO_2	Al_2O_3	Fe_2O_3	CaO	SO_3	TiO_2	MgO	Na_2O	K_2O	P_2O_5
B	54.83	19.05	6.00	9.39	2.90	0.97	1.77	0.50	3.15	0.20
D	69.99	10.62	4.02	8.86	0.038	0.55	1.39	1.02	2.61	0.11
L	68.79	15.23	6.28	1.79	0.127	0.85	2.02	0.26	3.71	0.07
P	72.75	15.89	4.97	0.87	0.07	0.84	1.20	0.27	2.17	0.10

[a] Data provided by the Danish Technical Institute.

Table 4
Mineralogy of GB types B, D, L and P normalised on a scale 0–100[a]

GB type	Quartz	Feldspar	Haematite	Cristobalite	Gypsum	Anhydrite	Glass content (%)
B	35	4	7	9	12	4	28
D	53	20	5	11	–	Trace	12
L	35	19	16	11	–	Trace	19
P	70	2	4	3	–	–	21

[a] Data provided by the Danish Technical Institute.

determined by slump and compacting factor measurements [19].

2.3. Specimen preparation, curing and testing

Mortar cubes (100 mm × 100 mm × 100 mm) were manufactured and cured in water at 20 ± 2°C for 7, 28, 90 and 365 days. After each curing period the compressive strength of each mix was determined using a loading rate of 180 kN/min. Each reported value of compressive strength is the average of three measured values. Analysis of variance (ANOVA) was carried out on the compressive strength results. It was found that the differences between the means were always significant at the 99% confidence level with the exception of the P10-P30 mortar strengths obtained at 90 days, the differences between the means of which were significant at the 95% confidence level. Samples, that showed no visible signs of stress, were taken from the cubes after compressive strength testing and were dried over silica gel at 40°C until constant weight was achieved. This 'gentle' drying regime was used to minimise major pore alteration as a result of hydration product destruction common with more aggressive drying techniques [20]. Due to the large surface area to volume ratio of the small samples (≈1–3 g) most of the weight loss (≈80%) occurred in the first 24 h. After drying, the samples were stored in a sealed container over silica gel to minimise any further modification of the pores by moisture ingress. The pore size distribution of each of the small solid mortar samples was then determined using mercury intrusion porosimetry (MIP). The surface tension and contact angle values of mercury used were 480 dyne/cm and 141.3°, respectively. Further details of the MIP technique used can be found in [13]. The variability of MIP values was ±4.3% (cumulative pore volume) and ±3.7% (percentage of pores smaller than 0.05 μm radius). In all cases this variability did not affect the trends observed.

The values of surface tension and contact angle have a significant influence on the resultant pore size distribution. In particular an increase in the value of the contact angle causes the pore size distribution curve to shift towards higher pore radius values [21,22] which will result in increased threshold radius values. Both the type of material being intruded and the manner in which that material has been dried prior to intrusion influence the contact angle. For example, partial replacement of PC with PFA, is reported [23] to increase the contact angle. A similar effect may also occur on partial replacement of PC with GB. Therefore caution must be exercised when directly comparing the current results with those of other researchers. However, within these constraints, the relationships that are observed are self-consistent for the specific system investigated.

3. Results

Fig. 1 shows the intruded pore volume for mixes with 10%, 20% and 30% replacement of cement with GB types B, D, L and P for curing periods of up to one year. The data are compared with the control mortar (CTR), which does not contain any GB. For all brick types the addition of GB to mortar results initially in an increase in the intruded pore volume relative to the control. The intruded pore volume increases generally with increasing replacement level and decreases with increasing curing period. However even after 365 days the interconnected pore volumes of mortars containing GB types B, D, L and P are, with the exception of GB mortar B at 10% and 20% replacement, still in excess of that of the control mortar.

The percentage of the total pore volume that is made up from pores of radii <0.05 μm was taken as a measure of pore refinement as illustrated in Fig. 2 for mortars containing GB of all four types. The pore size of 0.05 μm was chosen as this corresponds approximately with the lower end of the capillary pore size range and is easily obtainable utilising MIP [10]. The data are compared with those of the control mortar and initially (i.e. at 7 days) indicate that the GB mortars have a consistently lower proportion of pores with radii <0.05 μm than has the control. Also at early curing ages there is a decrease in the percentage of pores finer than 0.05 μm as the GB replacement level increases. As curing periods increase, for all GB types and at all replacement levels, the percentage of pores finer than 0.05 μm increases more rapidly for mortars containing GB than for the control mortar and approaches the value for the control mortar at curing periods of 90 and 365 days (B10, B30, L30 and P30). Between 7 and 28 days curing the rate of refinement of pore size for the majority of the mortars is substantially greater than that of the control mortar indicating a greater rate of production of C–S–H gel during this period. Between 28 and 90 days curing, the rate of refinement of the pore size decreases to a level similar to or less than the control except for mortars containing 30% GB, which in general still show rates of pore refinement greater than the control. After 90 days curing further refinement of pores continues gradually relative to the control for mortars containing 30% GB other than for mortar containing GB type B which appears to achieve maximum pore refinement at 90 days.

Threshold radius is the pore size at which there is a sudden increase in the number, and therefore the cumulative volume, of pores that can be intruded by mercury. It is observed as a sudden marked increase in the rate of intrusion of mercury into the sample. Fig. 3 shows the threshold radii measured for mortars containing all GB types used in the study. At early curing times (7 days) the partial replacement of PC has a

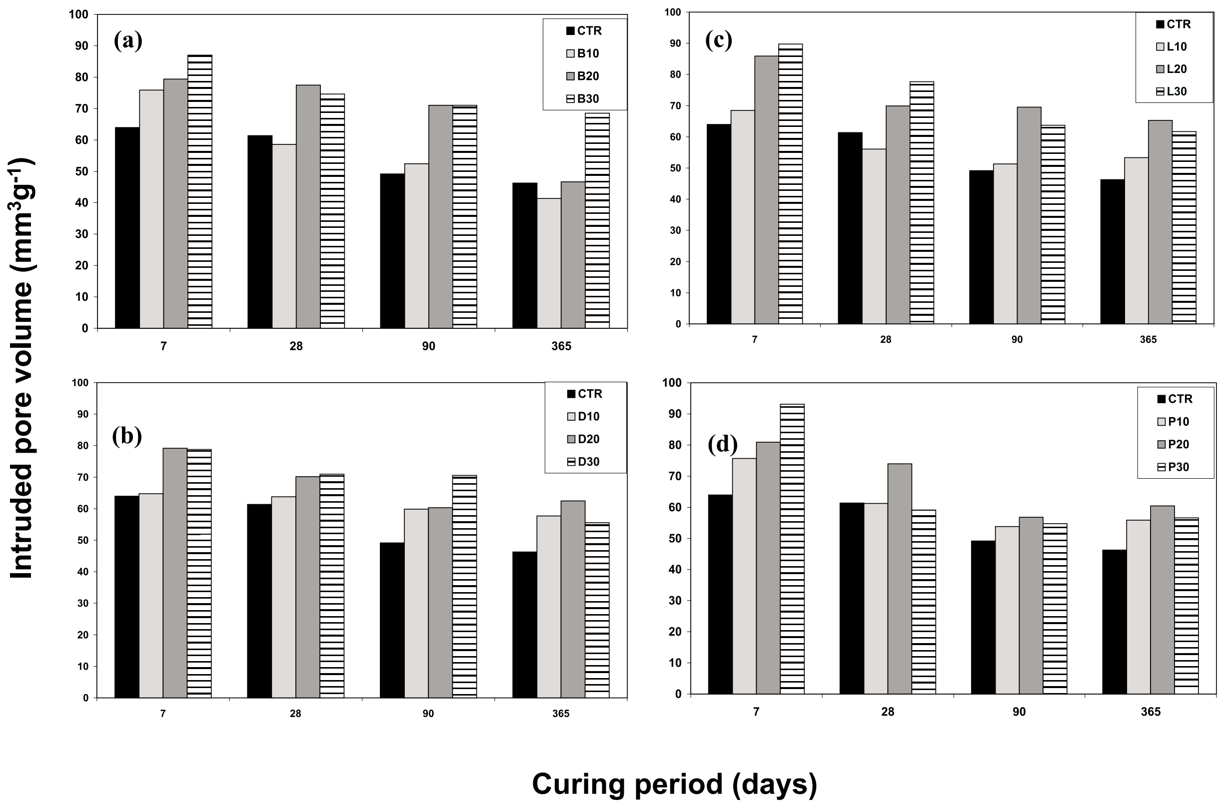

Fig. 1. Intruded pore volume of GB type (a) B, (b) D, (c) L and (d) P mortars.

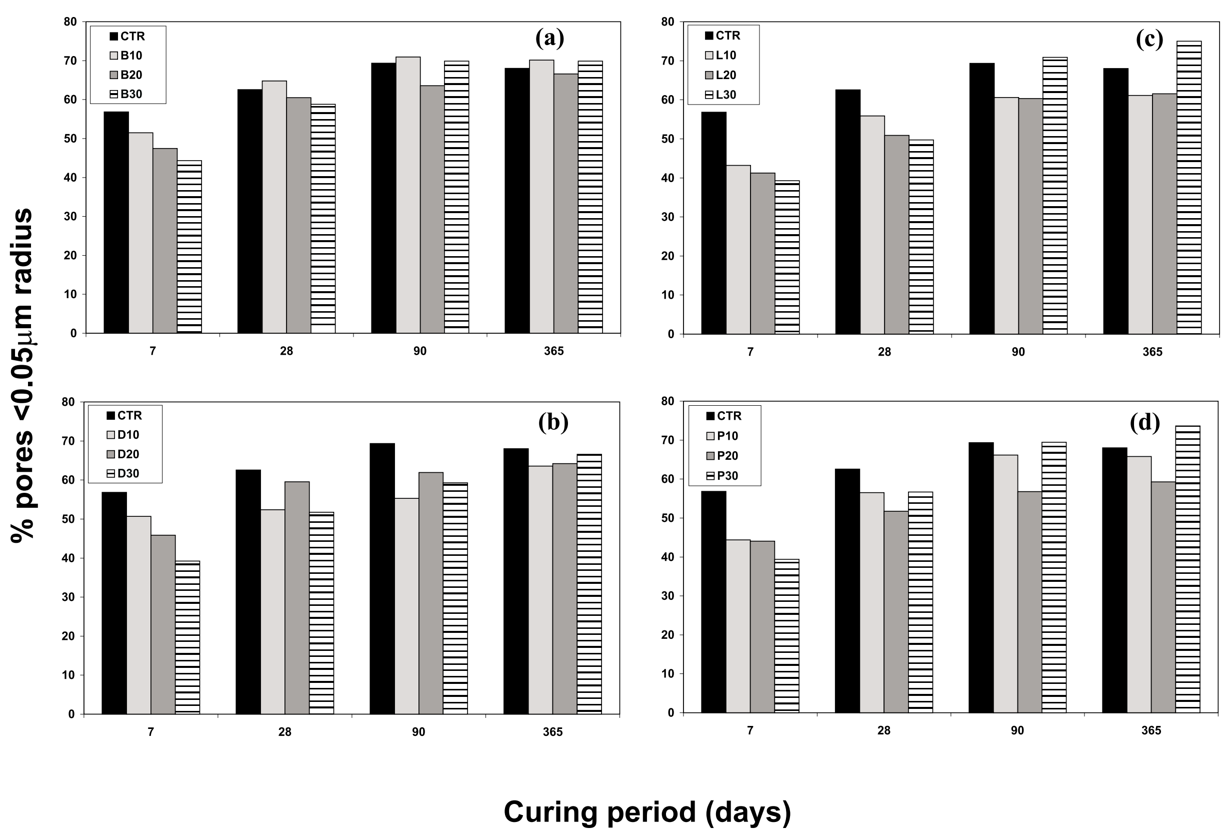

Fig. 2. Pore refinement of GB type (a) B, (b) D, (c) L and (d) P mortars.

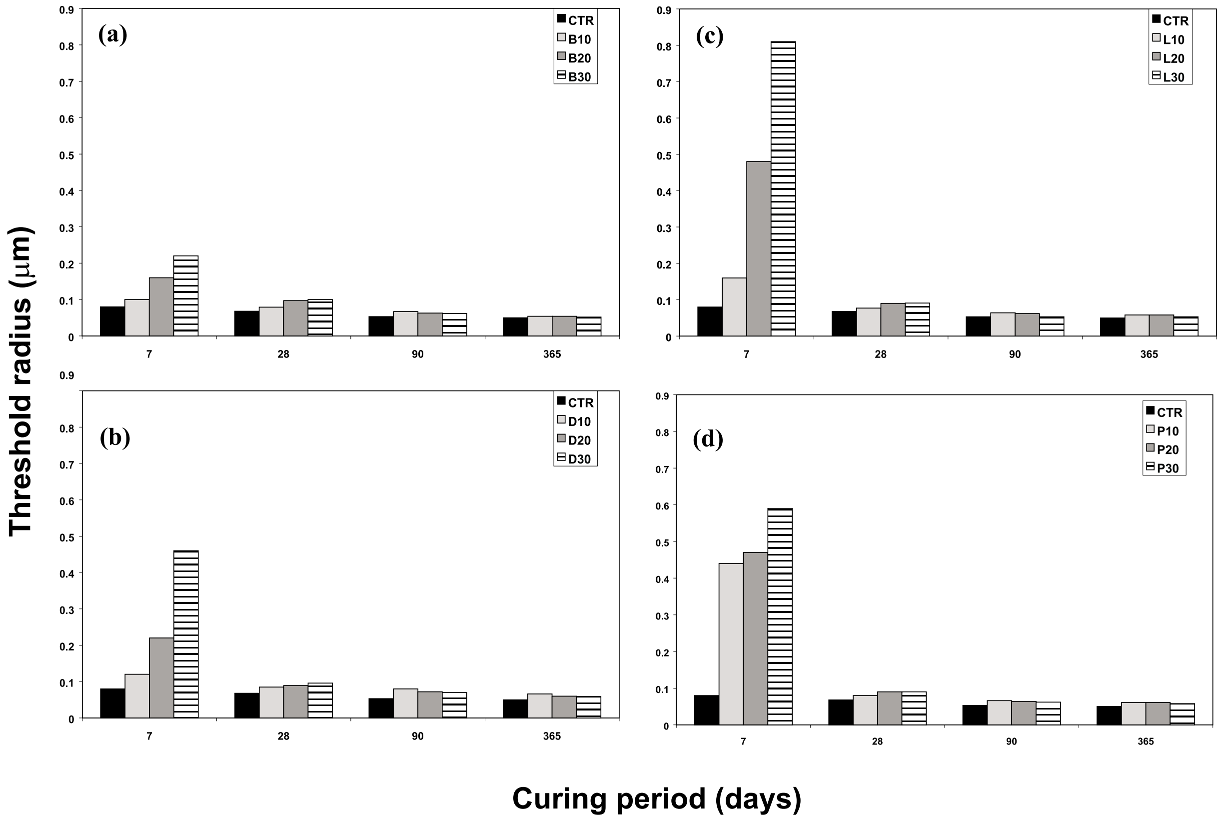

Fig. 3. Threshold radius of GB type (a) B, (b) D, (c) L and (d) P mortars.

profound effect on the threshold radius of GB mortars. The presence of GB, regardless of type (B, D, L or P), has the effect of significantly increasing the threshold radius of mortars with respect to the control mortar. It is also apparent that increasing replacement level of GB results in increasing size of threshold radius value. This is attributed to an increase in the effective water to PC ratio producing a coarser cement paste capillary pore structure at early ages. After a curing period of 28 days the effect of the GB, again regardless of type, is much less pronounced and threshold radius values tend towards those of the control. At this point, it is still the case that increasing replacement level increases the threshold radius although the effect at extended ages is very small. By 365 days curing the threshold radius values measured, irrespective of type, are in the same region as those of the control mortar.

Fig. 4 shows the compressive strength of mortars containing GB types B, D, L and P relative to the compressive strength of the control mortar. At early curing times (up to 28 days) partial replacement of PC by GB, irrespective of source, results in a significant decrease in compressive strength. The reduction in strength increases with increasing level of replacement by GB. However, with increasing age the trend is for the compressive strengths to approach, and in some cases exceed that of the control mortar. This is especially noticeable at the lower level of replacement (i.e. 10%). Another noticeable feature is the enhanced relative strength that mortars with cement replacement by GB type B exhibit relative to the other mortars (D, L and P), particularly at early ages (up to 28 days). In contrast at replacement levels of 20% and 30%, mortars containing GB type D consistently produce lower strengths than the other GB mortars at all ages of testing.

There have been many attempts to correlate compressive strength with various parameters describing the porosity and pore structure of cementitious materials. Previous work [3–5] indicated that the strength of mortar or concrete is fundamentally a function of the form and distribution of the void space and porosity within it. It is generally accepted that the existing relationship between strength and porosity can be most simply expressed in an empirical manner by a linear plot [24]. Close examination of the results presented in Figs. 1–4 reveals that similar correlations exist between the relative strengths and the pore characteristics measured for all four GB types investigated. As might be expected strength increases with decrease in total pore volume (see Fig. 5(a)) and with increase in the proportion of small pores (see Fig. 5(b)). All data for all the different mortars and curing times are plotted in

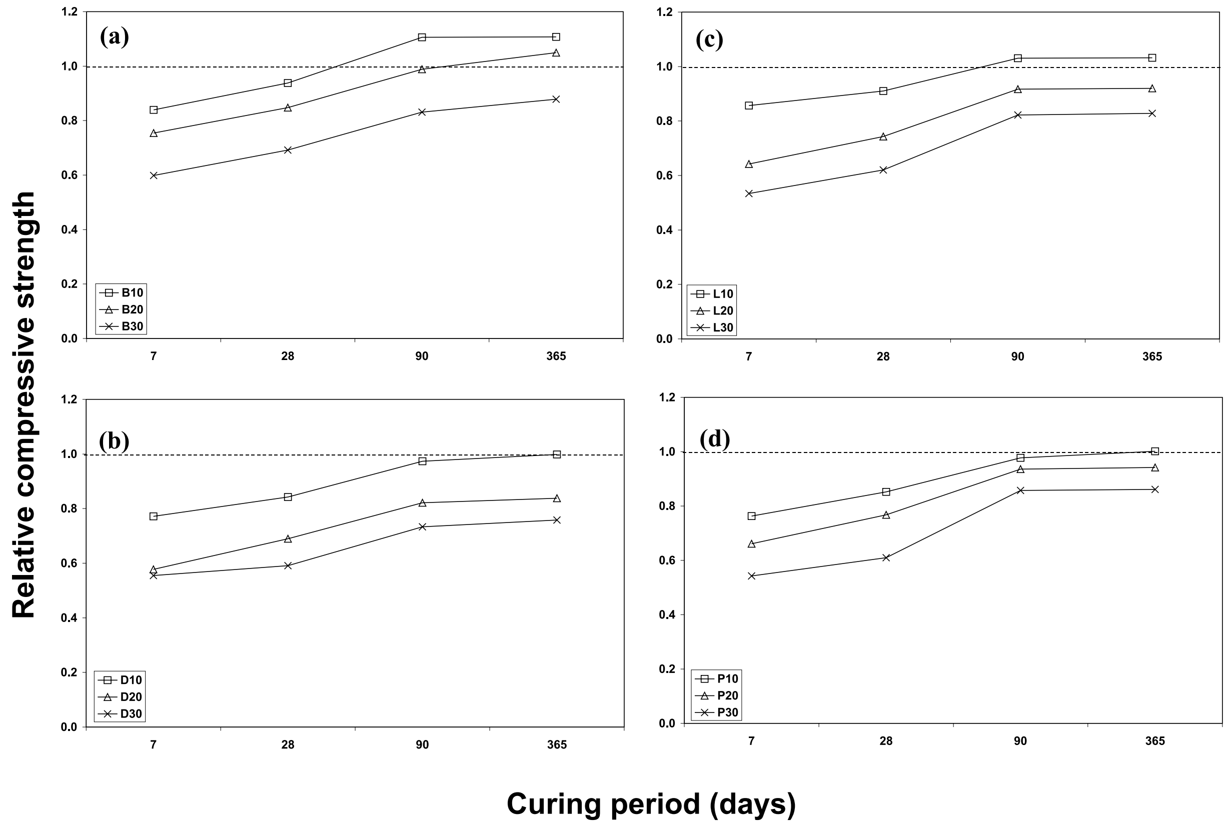

Fig. 4. Relative compressive strength of GB type (a) B, (b) D, (c) L and (d) P mortars.

Fig. 5 and although there is wide scatter, the linear trends are clear.

The above relationships indicate the clear correlation between compressive strength and pore size distribution. There is an additional aspect of porosity that has received little discussion in the literature and that is threshold radius. If compressive strength is plotted against threshold radius (μm), as in Fig. 5(c), another interesting relationship is noted. For threshold radii down to 0.1 μm) compressive strength is not very sensitive to threshold radius and there is only a small increase in compressive strength for a large decrease in threshold radius. However, as the threshold radius decreases below 0.1 μm (in this instance equivalent to a compressive strength of roughly 40 MPa) strength now increases very sharply with a small decrease in the threshold radius. This seems to indicate a critical relationship between threshold radius and compressive strength with a definite threshold radius value below which compressive strength increases at a significantly greater rate. It should be noted here that 0.1 μm is the upper value of pore diameter for cementitious gel, which was reported by Odler and Rossler [25] to range between 1 and 100 nm. The actual size of the upper limit of gel porosity is, however, a contentious issue and has been quoted by other workers as 3–4 nm [3,4,10]. Thus MIP, whilst a good analytical tool for collecting comparative data, has inherent uncertainties associated with it for the determination of absolute pore size values [26–28].

From the results presented above GB type B is the most beneficial in improving the performance of mortar. However GB type B exhibits an unusually high SO_3 content of 2.9% (see Table 3). The effect of additional sulphate was also investigated by intentionally dosing a GB of low SO_3 content (GB type L) with gypsum at levels equivalent to 2%, 3% and 4% SO_3 by mass. Making small changes in the sulphate content of the ground brick as detailed above produced relatively small non-systematic changes in pore size distribution and relative strength. It was considered that the effect of specimen variability on these results was of the same order as the effect of varying the sulphate content of the ground brick by these relatively small amounts. Therefore the porosity values and the relative strength values reported are the mean of the values at each of the three different sulphate contents and represent a mean sulphate content of $3(\pm 1)\%$ SO_3. This is equivalent to the natural sulphate content of GB type B (2.9% SO_3) . Data are only available, for this investigation, up to 90 days curing.

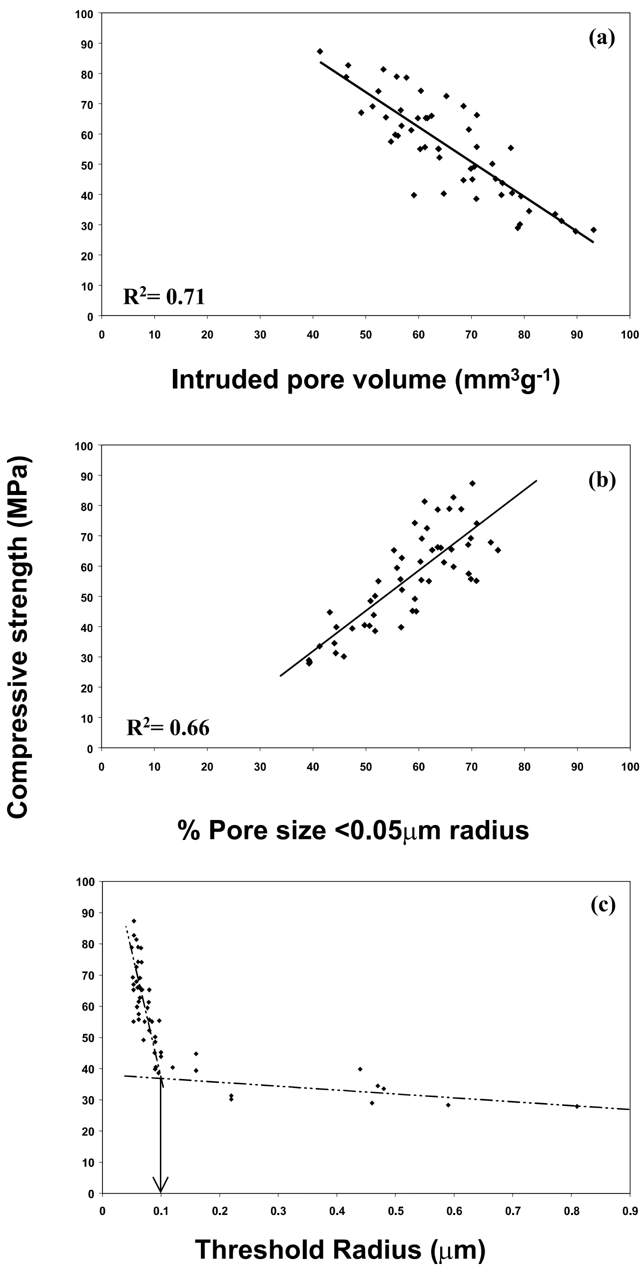

Fig. 5. Relationship between compressive strength and (a) intruded pore volume, (b) pore refinement and (c) threshold radius of GB types B, D, L and P.

Fig. 6 shows the changes in the characteristics of the pores with curing time for GB type L mortar both with and without additional sulphate. In general (see Fig. 6(a)) interconnected pore volume increases with increase in cement replacement level and decreases with increase in curing time. The increase in intruded pore volume with increase in cement replacement level is however greater for the mortar without added sulphate. Thus, overall, at low replacement levels the intruded pore volume is greater for mortars with added sulphate and at high replacement levels the intruded pore volume is greater for mortars without added sulphate, although in the latter case the 90 day data do not comply with the trend.

Differences are also apparent in the pore fineness – curing time relationship between GB mortars with and without additional sulphate (see Fig. 6(b)). The principal and consistent difference is apparent at short curing periods and particularly at 28 days where there tends to be a greater proportion of pores with a radius <0.05 μm for GB mortars with additional sulphate than those without it. At 90 days this difference is noticeably diminished. Therefore after 90 days the effect of additional sulphate, with respect to pore refinement, is negligible.

Fig. 6(c) shows the threshold radius values measured. At early curing times (7 days) increasing replacement level results in a marked increase in the threshold radius observed and this is true for mortars both with and without additional sulphate. The increase in threshold radius with increase in GB content is, however, much greater for mortar without added sulphate. As curing times increase to 28 days there is a significant decrease in threshold radius values especially at 20% and 30% replacement levels. Also there is little distinction between mortars with or without added sulphate and this is also the case after 90 days curing. In addition after 90 days curing increasing replacement level has little effect on threshold radius values. Thus the primary effect of sulphate in GB mortars is the lowering of the initial threshold radius values regardless of replacement level. This is attributed to the accelerating effect of sulphate on the hydration reactions.

Relative compressive strengths for GB mortars with additional sulphate are compared with those without in Fig. 7. What is immediately noticeable is that at early curing times (up to 28 days) the relative strengths of mortars containing additional sulphate are greater than those for mortars without added sulphate. As curing times increase further (up to 90 days) this strength enhancement is not maintained and at all replacement levels the relative compressive strengths of mortar without additional sulphate now exceed those with it. There is however no simple relationship between this consistent fall in relative strength at 90 days for mortars with added sulphate and the changes in pore fineness shown in Fig. 6.

This suggests that the sulphate may be influencing the cement hydration and the nature of the cementitious gel that is formed. Although the inclusion of sulphate clearly modifies the porosity and pore fineness (and hence the compressive strength) it does not change the fundamental relationship between these parameters and compressive strength. Fig. 8 shows the relationships between the compressive strength and the characteristics of the pores as measured by the total pore volume, pore

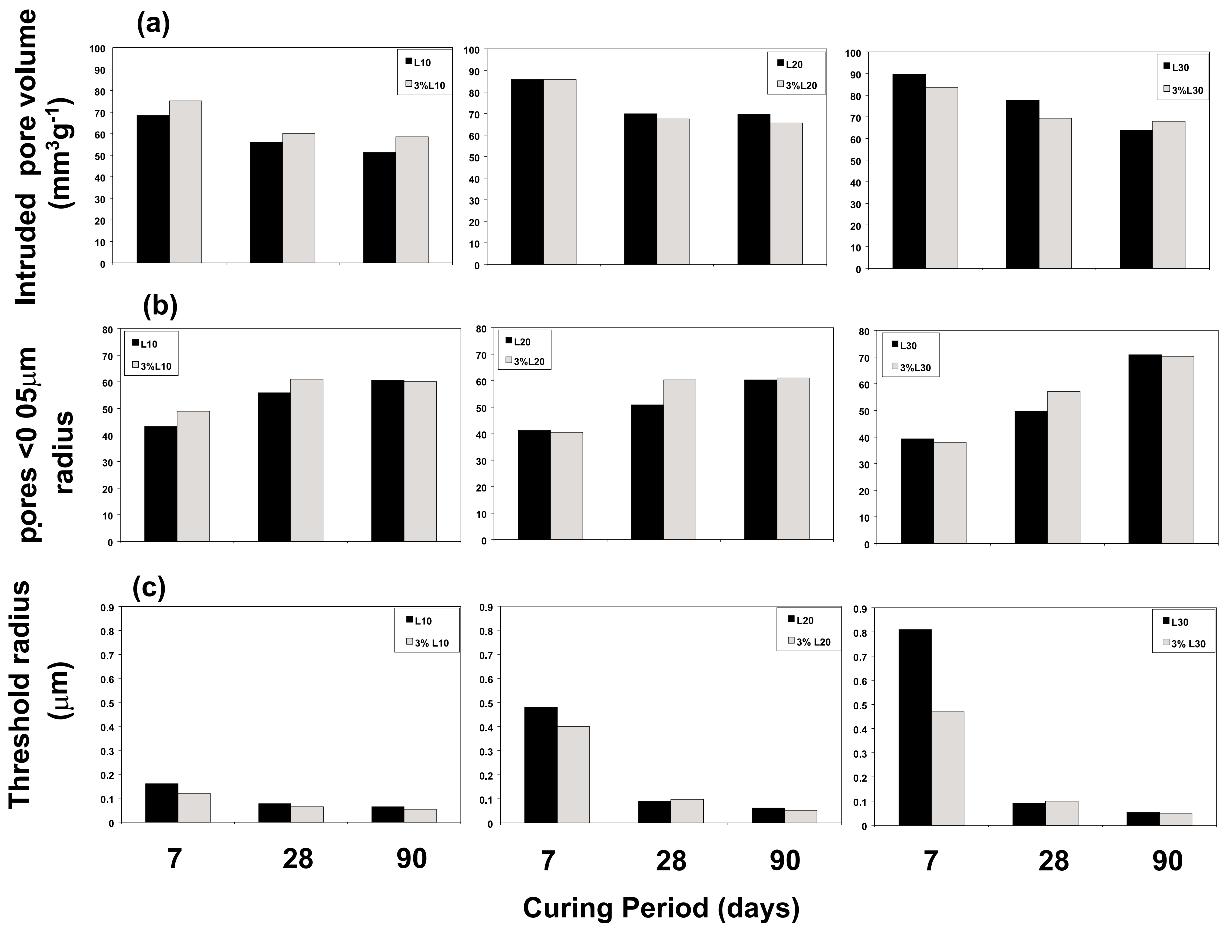

Fig. 6. Intruded pore volume (a), pore fineness (b) and threshold radius (c) of GB type L mortar with and without 3% additional sulphate (SO_3).

refinement and threshold radius. The results are similar to those for GB mortars without added sulphate. Also the relationship between threshold radius and strength (see Fig. 8(c)) is similar to that for mortars without added sulphate (see Fig. 5(c)).

The performance of the GB mortars containing additional sulphate and their effect on the physical characteristics of mortar seem to characterise two distinct phases. Prior to a curing period of 28 days, the effect of the additional sulphate seems generally beneficial in all aspects investigated for these mortars. Also general relationships between key dependent variables remain the same. At curing periods in excess of 28 days, the beneficial aspects of additional sulphate in GB mortar are no longer apparent.

4. Discussion

Increasing substitution of cement with GB in mortar results, at early ages (up to 28 days), in progressively decreasing compressive strengths relative to the control. However between 0 and 90 days, relative strengths (i.e. strength of GB mortar relative to control mortar) increase substantially with increase in curing time such that beyond 90 days the strengths of some GB mortars, particularly at the lower replacement levels, exceed those of the control (see Fig. 4). This is particularly the case for mortars containing ground brick type B.

The increase in relative strength corresponds to increasing pore refinement and decreasing threshold radius (Figs. 2 and 3, respectively) and is believed to be caused by additional C–S–H gel produced from the pozzolanic reaction between GB and portlandite (CH) from the hydrating cement. Previous work on the determination of the hydraulic index of fired brick clay used to produce GB type B has shown that the pozzolanic activity of the GB is very sensitive to the brick clay firing temperature [29]. It should be noted here that some of the CH produced from the hydrating cement, especially with the addition of GB type B (which has a high sulphate content) will be consumed in the production of additional ettringite during hydration. The ettringite formed in this manner will also contribute to the early strength of the GB mortar [30]. Additional C–S–H gel formed as a result of the pozzolanic reaction

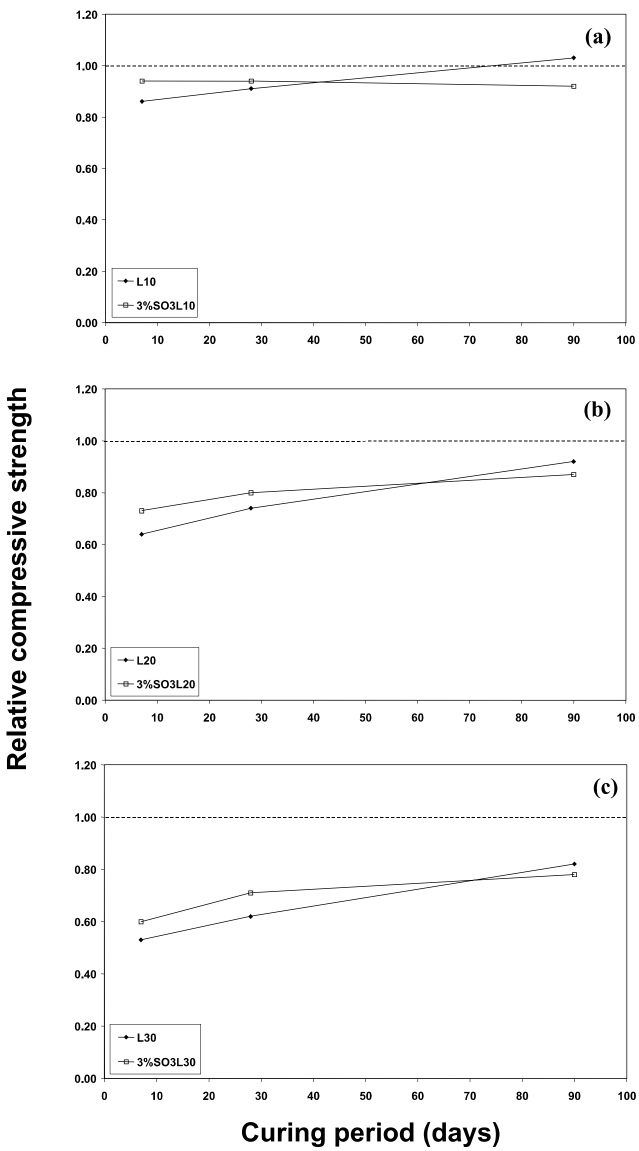

Fig. 7. Relative compressive strength of GB type L mortar with and without 3% additional SO_3 at cement replacement levels of (a) 10%, (b) 20% and (c) 30%.

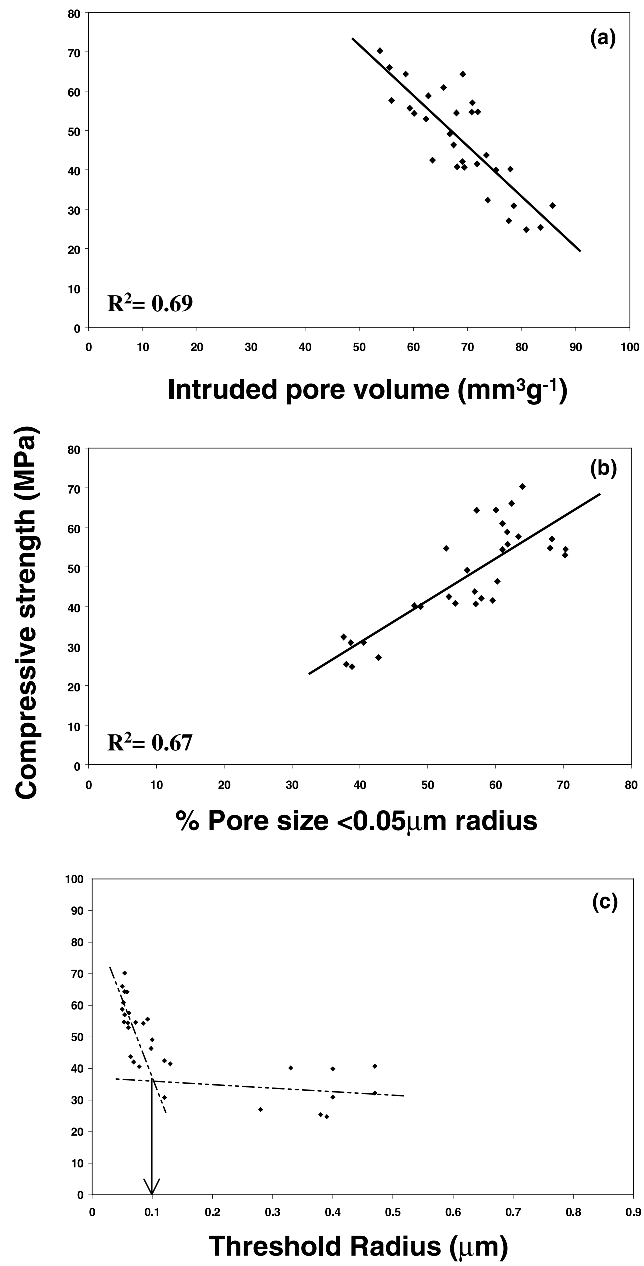

Fig. 8. Relationship between compressive strength and (a) intruded pore volume, (b) pore refinement and (c) threshold radius of GB type L mortar with additional SO_3.

will have the effect of infilling pores, thus reducing total porosity and increasing pore refinement [12,13]. Due to the infilling effect, additional C–S–H gel will reduce the amount and connectivity of capillary porosity within the mortar, which has been identified by Kendall et al. [4] as being instrumental in initiating failure. Essentially, reduction in pore volume produces an increase in elastic modulus, and an increase in pore fineness produces a decrease in average flaw size. The C–S–H gel that partially fills the capillary porosity also contains pores, but these are considered too small to initiate cracking [4]. Rossler and Odler [24], who investigated cement paste made with different w/b ratios and hydration times, state that the relationship between porosity and strength can best be expressed in the form of a linear plot. This is the case for mortars containing GB as a cement replacement material, in that clear empirical linear relationships are established between compressive strength and total pore volume, and compressive strength and pore fineness (Figs. 5(a) and (b), respectively). This not only demonstrates that as pore fineness increases strength increases and as pore volume decreases strength increases but it also shows the effect which this additional C–S–H gel has on compressive strength development.

The incorporation of sulphate (3% SO_3 as gypsum) in GB, which contains negligible natural sulphate (brick type L, natural sulphate $SO_3 = 0.13\%$), produces an increase, at the early curing stages (0–28 days), in the relative strength although this is not maintained at extended ages (see Fig. 7). This increased rate of strength development is believed to be a result of the formation of additional reaction products, which produce the increased pore refinement that is observed in GB mortar with added gypsum (see Fig. 6). The additional reaction product may be C–S–H gel, ettringite, monosulphate (which all contribute to early compressive strength), a combination of the three or another phase. It may also partly explain the rather greater strength gains for mortar containing GB type B (which has a high natural sulphate level, $SO_3 = 2.9\%$) relative to those for mortars containing the other brick types, particularly at early ages (see Fig. 4). However, when considering the GB mortars without added sulphate, the variation observed in performance cannot be explained solely by the presence of sulphate, as the difference in compressive strength is not systematic with the observed SO_3 content. Another key factor that influences the amount of cementitious gel formed from pozzolanic activity, and hence the strength contribution from this process, is the amount of glass phase present. The glass content of the GB types investigated increases in the order $D < L < P < B$ (see Table 4). Inspection of the strength values of mortar shows that at 365 days (when any pozzolanic reaction will be at an advanced stage), the strengths attained are in the order $D < L < P < B$. This provides evidence that the glass content of the brick is a principal factor, which determines the effectiveness of GB as a pozzolan.

5. Conclusions

The following conclusions may be drawn from the work:

1. The addition of GB to mortar as partial cement replacement influences the pore size distribution, threshold radius and compressive strength of the mortar. The type of GB also has a marked effect on these physical characterisations. At short curing times increasing GB contents in mortar result in increased intruded pore volume, reduced percentage of fine pores, increased threshold radius and reduced compressive strength relative to GB free mortar. At long curing times these four parameters more closely approach those of the control mortar.

2. Glass content of the GB appears to be a key factor influencing the performance of GB mortar.

3. The incorporation of additional gypsum results in enhanced early compressive strength and increased pore refinement although the effect is not maintained. The early enhancement is almost certainly due to acceleration of hydration and formation of additional hydration products.

4. There is a critical relationship between compressive strength and pore threshold radius of mortar. As threshold radius approaches gel pore size compressive strength increases rapidly, indicating that pore size, rather than pore volume, is a limiting factor of compressive strength development.

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