Pitchstone Fines – A New Naturally Occurring Pozzolan from North Queensland

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ABSTRACT: Global warming presents an ever-challenging battle to humankind, as emissions arising from industrially produced greenhouse gases are predicted to alter the long-term climatic patterns of earth. Harmful environmental emissions arising during the manufacture of Portland cements (C) can be effectively reduced by incorporating siliceous-aluminous based supplementary cementitious materials (SCMs), as partial cement replacements. In Australia, mined pitchstone fines (PF), derived as waste material from expandable perlite production, are a viable SCM for reducing cement consumption using an eco-friendly approach. This paper reports on the results of an experimental investigation into the pozzolanic activity of PF. Up to 40% cement was replaced with PF in mortar mixes. In addition, PF was used to partially replace sand. Strength activity index (SAI) values for PF were evaluated using accelerated 28-day compressive strengths for all PF substitution levels, with flows and wet densities of mortar mixes reported.

1. INTRODUCTION

The evaluation of numerous siliceous rich materials as reactive pozzolans for use as supplementary cementitious materials (SCMs) in concrete mixes have been investigated in recent times by diverse pozzolanic reactivity assessment methods (Agarwal, 2006; Asgeirsson and Gudmundsson, 1979; Moropoulou et al, 2004; Raask & Bhaskar, 1975; Roszczynialski, 2002; Schlorholtz et al, 1984). Important strength development attributes of natural pozzolans and volcanic tuffs, as reported by Turanli et al (2004) and Shannag (2000), respectively, and incorporating clay brick waste materials for examining durability to chemical attack, as investigated by O'Farrell et al (2006), are just some representative examples of naturally occurring and by-product SCMs possessing advantageous physical and chemical properties of a pozzolan.

A pozzolan is defined as a silicate or aluminosilicate material possessing little or no cementitious benefit. However, in finely dispersed form combined with water and calcium hydroxide, liberated by cement hydration, a pozzolan will chemically react to form cementing compounds exhibiting cementitious properties (AS/NZS 3582.3:2002, 2002; ASTM C 125-07, 2007). Natural occurring geological minerals, including diatomaceous earths, opaline cherts, clays, shales, tuffs and volcanic ashes, have pozzolanic classification for use in concrete or in producing blended cements (AS 3582.1-1998, 1998; AS 3583.6-1995, 1995; AS/NZS 2350.0:1999, 1999; ASTM C 595-05, 2005; ASTM C 618-05, 2005).

With recent worldwide conjecture relating to harmful greenhouse gases released during large-scale manufacturing processes, natural pozzolans can be considered as a viable environmental friendly partial replacement material for reducing the amount of overall carbon dioxide (CO_2) emissions released into the atmosphere during the manufacture of cement. The use SCMs to partially replace cement in concrete mixes effectively reduces cement content, reducing emissions, as for every tonne of Portland cement (C) produced, approximately one tonne of CO_2 is generated, which accounts for 5% to 10% of all greenhouse gases generated worldwide by human activity (Anand et al, 2006; Worrell et al, 2001).

Perlite belongs to a natural occurring group of acidic, siliceous-aluminous rhyolitic rocks, which are amorphous and glassy in appearance, falling into the potential classification of natural pozzolans. Perlite can expand four to twenty times its original size upon rapid heating. Based on internal hydrated mass composition, perlite can be further categorised accordingly into either obsidian (containing less than 2% water), perlite (having between 2% to 5% water) or pitchstone (containing greater than 5% water) (Sodeyama et al, 1999).

The use of perlite powder as a pozzolanic addition in pre-mixed concrete investigated by Yu et al (2003) illustrates significant pozzolanic reactivity and improvement was noted for all compressive strengths over control equivalents at 91-days ageing, relative to C replacement amounts ranging from 10% to 40%. Additionally, Erdem et al (2007) in evaluation of perlite addition as a function of particle fineness, blended perlite with C to form mortar specimens to report compressive strengths for finer grade perlites in 20% and 30% partial substitution of C approaching control specimens at 91-days ageing. More recently, preliminary investigations by Ray et al (2007) reported that at 10% C replacement and at 28-days ageing, compressive strengths almost equivalent to the control mixes were achievable, surpassing the reported performance equivalent of fly ash pozzolanic substitutions.

Perlite mined from locations around the world, when processed to its expanded form, is utilised in high volumes in the field of construction, horticultural and industrial applications. During the primary excavation processing stage, jaw crushers are utilised to pulverise the material to an acceptable particle size range through screening procedures before secondary heat processing to the expanded stage. In the former primary crushing process, a certain undesired amount of raw ore perlite fines result which are classified as waste material with no significant economic value.

In Australia, a large pitchstone (water content circa 7% to 8%) mining operation owned by Perlco Pty Ltd (PPL), located in the Nychum district in far north Queensland near the town of Chillagoe, provides vast resource of more than 100 million tonnes for use as expanded perlite grade related materials after dehydration. The current primary crushing stage of the pitchstone ore produces approximately 30% by-product fines, all less than 500-µm in size, from the initial pulverisation and final screening processes. This fine grade waste material is stored in the form of stockpiles on site, as no current viable alternative potential use has yet been identified (Ray et al, 2007).

This paper reports the pozzolanic reactive classification of as received waste PF through ASTM C 311-05 (2005) (AS 3582.1-1998, 1998; AS 3583.6-1995, 1995) strength activity index determinations at 7-day accelerated ageing (simulating that of 28-days standard ageing) for 10%, 20% and 40% mortar C replacements and aggregate (A) substitutions, using fixed water-to-cementitious material (w/cm) and water-to-cement (w/c) ratios.

2. EXPERIMENTAL DETAILS

The following raw materials were used for all experimental mix mortar designs, having typical chemical compositions, as indicated by x-ray fluorescence (XRF) analysis (Table 1):

Table 1: Chemical analysis of raw materials by XRF method.

Chemical Composition	A (%)	C (%)	PF (%)
SiO_2	98.4	20.5	68.53
Al_2O_3	0.69	4.5	12.94
Fe_2O_3	0.11	4.5	1.04
CaO	0.02	64.9	0.9
MgO	0.02	1.2	0.02
Na_2O	0.03	0.0	4.51
K_2O	0.17	0.46	2.58

Table 2: Particle size grading of water PF.

Particle Size	Retained	Passing
0.600	0.0	100.0
0.300	0.0	100.0
0.150	0.0	100.0
0.075	36.4	63.6
0.045	22.6	41.1
< 0.045	41.1	0.0

- Shrinkage limited Portland cement (C) – Blue Circle Southern Cement Pty Ltd
- Pitchstone fines (PF) – PPL
- Raymond Terrace single washed sand (A) – Rocla Quarry Products Pty Ltd.

Commercial construction grade fine aggregate sand meeting the exact grading requirements stipulated within ASTM C 778-06 (2006) (AS 1141.11-1996, 1996) was utilised, with the fine aggregate dried in ambient conditions to eliminate free water prior to use.

The particle size grading distribution determination of PF was analysed by dry sieving against AS 1141.11-1996 (1996) criteria using washing prescribed in AS 1141.12-1996 (1996), including further washing over an additional 45-µm sieve size (Table 2).

Mortar cube specimens were prepared for 0%, 10%, 20% and 40% PF substitution amounts, all subjected to an accelerated 7-day curing regime (AS 3583.6-1995, 1995), changing three variables:

1. C substituted by PF and w/cm ratio fixed at 0.48 (CPF_{wcm}).
2. C substituted by PF and w/c ratio fixed at 0.48 (CPF_{wc}).
3. A substituted by PF and w/c fixed at 0.48 (APF_{wc}).

Relative to ASTM C 618-05 (2005) (AS 3582.1-1998, 1998; AS 3583.6-1995, 1995) specified water requirement (WR) range, a fixed WR was employed for all PF substitution CPF_{wcm}, CPF_{wc} and APF_{wc} mixes, as an increase in w/cm and w/c ratios, was likely to decrease compressive strengths (Hover and Stokes, 1995).

Mortar mix designs (Table 3) were taken from standardised proportions listed under ASTM C 311-05 (2005) (AS 3582.1-1998, 1998; AS 3583.6-1995, 1995), with the cementitious material-to-sand ratio (cm/s) fixed at 1:2.75 for all PF substitutions.

Preparation of mortar mixes were carried out in accordance with ASTM C 305-99 (1999) (AS 2350.12-2006, 2006) and modified accordingly with an additional step in that prior to adding any C

Table 3: Mix design proportions of mortar specimens.

Specimen	C (grams)	PF For C (grams)	A (grams)	PF For A (grams)	Water (grams)
$0CPF_{wc}$	500	- (0%)	1375	-	242
$10CPF_{wcm}$	450	50 (10%)	1375	-	242
$20CPF_{wcm}$	400	100 (20%)	1375	-	242
$40CPF_{wcm}$	300	200 (40%)	1375	-	242
$10CPF_{wc}$	450	50 (10%)	1375	-	218
$20CPF_{wc}$	400	100 (20%)	1375	-	194
$40CPF_{wc}$	300	200 (40%)	1375	-	145
$10APF_{wc}$	500	-	1238	137 (10%)	242
$20APF_{wc}$	500	-	1100	275 (20%)	242
$40APF_{wc}$	500	-	825	550 (40%)	242

to water, PF substitutions were allowed to mix with water for an additional 30 seconds on speed setting 140 ± 5 revolutions/min.

All flows were measured in accordance with the requirements listed under ASTM C 1437-01 (2001) (AS/NZS 2350.18:2006, 2006) and wet densities were determined using a modified test method developed adhering to principles listed in ASTM C 138/C 138M-01a (2001) (AS 1012.5-1999, 1999), using the rod consolidation procedure.

50-mm cube mortar specimens were cast in accordance with ASTM C 109/C 109M-05 (2005) (AS/NZS 2350.11:2006, 2006) specifications, using the hand tamping compaction technique and meeting initial 24 hours moist environment curing criterion stipulated (AS/NZS 2350.11:2006, 2006; ASTM C 511-05, 2005). The specimens were then subjected to a 7-day accelerated ageing regime based on AS 3583.6-1995 (1995), with compressive strengths determined for 0%, 10%, 20% and 40% PF substitutions (AS 3583.6-1995, 1995; AS/NZS 2350.11:2006, 2006; ASTM C 109/C 109M-05, 2005).

Compressive strengths on all cast 50-mm cube test specimens were determined using a calibrated Tinius Olsen Super L 600 kN hydraulic universal testing machine. Vernier callipers were used to measure the dimensions of the specimen cubes prior to compressive strength testing, at a level of precision within 0.1 mm.

3. RESULTS & DISCUSSION

3.1 CPF_{wcm} (Cement Replacement & w/cm Fixed)

Flow

The degree of flow measured in mortar equates to the amount of workability or consistency present in a cementitious system, with the rheological abilities to fill volumetric shapes for a given amount of work. Flow is affected by water content, particle shape, particle size and cementitious material content.

For a fixed w/cm ratio system, the degree of flow was observed to decrease with increasing PF substitutions over C replacements (Table 4). For $10CPF_{wcm}$, $20CPF_{wcm}$ and $40CPF_{wcm}$, the flow fell by 14%, 30% and 59%, respectively, relative to the control ($0CPF_{wc}$). This decreasing trend of flow indicates a reduced availability of water resulting in less degree of workability, suggesting that water is readily adsorbed by PF in preference to C.

Table 4: ***Flow (F), wet density (ρ), compressive strength (f_c) and strength activity index (SAI) data for accelerated 7-day ageing data specimens.***

Specimen	F (%)	ρ (kg/m³)	f_c (MPa)	SAI (%)
$0CPF_{wc}$	36	2140	27	100
$10CPF_{wcm}$	31	2100	23	85
$20CPF_{wcm}$	25	2110	19	70
$40CPF_{wcm}$	17	2050	11	41
$10CPF_{wc}$	-	2090	13	48
$20CPF_{wc}$	-	1940	8	30
$40CPF_{wc}$	-	1730	4	15
$10APF_{wc}$	28	2140	22	81
$20APF_{wc}$	-	2040	18	67
$40APF_{wc}$	-	1770	17	63

Wet Density

Another way of monitoring the degree of workability or consistency present in a cementitious system is through consolidation measurements involving wet densities. With increasing PF substitutions, the corresponding wet densities were theoretically expected to decrease, as the higher density C (3150 kg/m³) was partially substituted with lower density PF (2300 kg/m³).

Wet densities were seen to progressively decrease when compared to $0CPF_{wc}$ with increasing PF substitutions (Table 4). A decrease of 2% was noted for both $10CPF_{wcm}$ and $20CPF_{wcm}$, while $40CPF_{wcm}$ showed a decrease of 4%. These decreasing wet densities possibly illustrate an increasing uptake of water by PF with increasing substitution levels, additionally reflected by the difficulties experienced during consolidation of a fixed volumetric quantity, further suggesting that water is adsorbed by PF in preference to C.

Strength Activity Index (SAI)

The strength activity index (SAI) is a measure of the pozzolanicity of a SCM and is measured as the replacement strength relative to the control, in percent. For an SCM to be classified as a pozzolan, the SAI must be greater than 75% at 28-day ageing (that is, the strength of the blended cement must not be less than 75% of the control strength) (AS 3582.1-1998, 1998; AS 3583.6-1995, 1995; ASTM C 618-05, 2005).

The SAI of $10CPF_{wcm}$ (10% PF substitution and w/cm fixed) was found to meet the minimum permissible 75% limit with a value of 85% (Table 4). For $20CPF_{wcm}$ and $40CPF_{wcm}$, the SAI values were calculated at 70% and 41%, respectively, with both falling under the acceptable range (Figure 1). As noted for flow and wet densities, a decreasing trend with increasing PF substitutions is seen, with difficulties again observed in the degree of compaction present with increasing PF substitutions for cube specimens, also indicating that the water is more readily adsorbed by PF in preference to C.

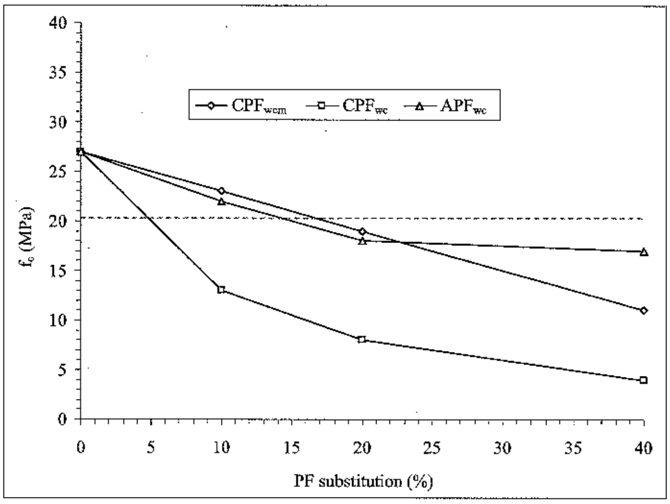

Figure 1: ***Compressive strengths (MPa) versus PF substitutions (%) for accelerated 7-da6 ageing (dashed line = possolanic SAI 75% limit).***

3.2 CPF_{wc} (Cement Replacement & w/c Fixed)

Flow

For a fixed w/c ratio system, the flow was observed to decrease significantly with increasing PF substitutions compared to $0CPF_{wc}$ (Table 4). For $10CPF_{wc}$, $20CPF_{wc}$ and $40CPF_{wc}$ no flow was observed, as all C replacement mixes were observed to be too dry for any adequacy of workability. This absence of flow signifies a reduced availability of water for C, again suggesting that water is readily adsorbed by PF in preference to C for the decreasing water contents used for this series of mixes in keeping the w/c ratio fixed (Table 3).

Wet Densities

The wet densities were unexpectedly seen to decrease significantly, with increasing PF substitutions relative to $0CPF_{wc}$, since theoretical wet density results for the CPF_{wc} series were expected to increase with the amount of water decreasing for increasing PF substitution levels (Table 4). Decreases of 3%, 10% and 19% were noted for $10CPF_{wc}$, $20CPF_{wc}$ and $40CPF_{wc}$, respectively. This decreasing trend can partly be explained by the partial substitution of the higher density C with lower density PF. However, in comparison to the CPF_{wcm} series and as far less water was available for this series of mixes with increasing C replacements, poor consolidation of the mortar resulted and hence, a lower apparent density was observed.

Strength Activity Index (SAI)

No calculated SAI values were found to meet the permissible 75% limit (Table 4). For $10CPF_{wc}$, $20CPF_{wc}$ and $40CPF_{wc}$, the SAI values were calculated at 48%, 30% and 15%, respectively, all well under the minimum acceptable range (Figure 1). As evident with corresponding flow and wet density measurements, a decreasing pattern with increasing PF substitutions was noted, with the difficulty in degree of specimen hand compaction again significantly increasing with increasing PF substitutions, supporting the fact that water is probably adsorbed by PF in preference to C.

3.3 APF_{wc} (Aggregate Replacement & w/c Fixed)

Flow

For a fixed C amount and w/c ratio system, the flow was seen to decrease markedly with increasing PF substitutions, when compared to $0CPF_{wc}$ (Table 4). Only one flow was recorded for $10APF_{wc}$, decreasing by 22% compared to control. No flow was evident from $20APF_{wc}$ and $40APF_{wc}$, as these were observed to be too dry for displaying any signs of workability. The absence of flow once more reflects a reduced availability of water for C, again strongly suggesting water is readily adsorbed by PF in preference to C relative to a fixed C content and w/c ratio system (Table 3).

Wet Densities

Wet densities were also observed to markedly decrease with increasing PF substitutions over control (Table 4). No appreciable decrease was noted for $10APF_{wc}$ compared to $0CPF_{wc}$, whereas for $20APF_{wc}$ and $40APF_{wc}$, the wet densities decreased by 5% and 18%, respectively. Due to the rising amount of aggregate substitution for this series, apparent densities were expected to decrease as PF has a lower density than that of higher density A (2500 kg/m^3). In comparison to the CPF_{wcm} series, constant water was available for increasing PF substitutions, with a low degree of consolidation observed for $10APF_{wc}$. Beyond this PF substitution level, no actual consolidation was physically possible, strongly suggesting that yet again, PF is readily adsorbing water in favour of C (Table 3).

Strength Activity Index (SAI)

Calculated SAI values for 10%, 20% and 40% PF substitutions are displayed for representative purposes only, as no C was in fact replaced to assess pozzolanic activity directly against PF substitutions (Table 4). The SAI values of $10APF_{wc}$, $20APF_{wc}$ and $40APF_{wc}$, were calculated at 81%, 67% and 63%, respectively (Figure 1). As seen with corresponding flow and wet density measurements, a decreasing pattern with increasing PF substitutions was noted, with compaction difficulty in cube specimen preparation increasing markedly with increasing PF substitutions, definitely supporting the case that water is readily adsorbed by PF in preference to C.

4. CONCLUSIONS

Pitchstone fines have shown promising attributes as a SCM, for classification as a possible pozzolan, when used as a partial C replacement, while retaining the obligatory requirements of compressive strength. In this particular instance, a PF addition level of 10% was found to conform to the ASTM standards, based on the strength activity index (SAI) measurements. A further feature uncovered in terms of workability, has shown PF readily adsorbs water in preference to C, for fixed water content systems.

The use of PF for the partial replacement of C has the potential to help minimise the overall impact of harmful greenhouse gas emissions created during the manufacturing process of C, by assisting directly in minimising C consumption. As the PF are currently a waste by-product material generated from an industrial process, the use of this material as a SCM additionally helps mitigate the environmental impact at the mine site. Pitchstone fines, therefore, used as a SCM, show significant potential for reducing costs with energy consumption and minimising environmental impact, in the manufacture and application of construction materials.

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