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# EFFECT OF MICRO-STRUCTURE AND MINERALOGICAL COMPOSITION ON THE WATER DEMAND AND SUPER PLASTISIZER CONTENT OF TERNARY BLENDED SELF-CONSOLIDATING PASTE.

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## ABSTRACT

The characteristics of cement paste, mortar and the resultant concrete greatly depends on the mineralogical composition as well as the micro-structure of the cement and/or the supplementary cementitious materials. In this study, the effect of the mineralogical composition and micro-structure of pulverised burnt clay (PBC) and palm oil fuel ash (POFA) on the water demand and high range water reducing admixture requirement of paste formulated from a ternary blend self-consolidating high performance concrete was investigated. To characterise the supplementary cementitious materials, various tests were carried out. These tests include particle size analysis (PSA), X-ray diffraction (XRD), Scanning electron microscope (SEM), surface area analysis and chemical analysis (XRF). The results of the investigation revealed that POFA, OPC and PBC have a BET surface area of 23.7514 m<sup>2</sup>/g, 5.0670 m<sup>2</sup>/g and 2.9791 m<sup>2</sup>/g respectively. Although PBC has the least BET surface area, XRD results shows that it has the highest amount of quartz and glassy substances which present it as a flow enhancing material. Furthermore, paste formulated from a binary blend of OPC and PBC shows lesser flow time, lower HRWR dosage as well as water demand in comparison with paste formulated from a binary blend of OPC and POFA and also paste formulated from a ternary blend of OPC, POFA and PBC.

**Keywords:** Cementitious materials, Micro-structure, Mineralogical composition,

Self -Consolidating Paste, Ternary blend, water demand, High Range Water Reducer.

## INTRODUCTION

Self-consolidating concrete has been advocated to be among the most difficult concretes to design owing to the difficulty inherent in determining the equilibrium between its different properties such

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as optimum flowing ability and optimum segregation resistance. Nonetheless, the process of mixture proportioning and the properties of the resultant paste, mortar and concrete is further complicated due to the divergent characteristics and mineralogical composition of the constituent materials (Girish et al, 2010). Furthermore, to be able to achieve self-consolidation through greater flowability, higher stability at rest after casting and good filling ability through congested reinforcement, high volume of powder and paste is required. Thus a rational optimization process is required to deal with shrinkage and creep problems normally associated with powder and paste contents. Most importantly, high-range water reducer (HRWR) improves the flowing ability of SCC. On the other hand, excessive dosage of the HRWR create a very high fluidity that may cause segregation problems in the form of bleeding, aggregate pilling and aggregate sedimentation. (Md Safiuddin et al, 2011). Consensuses of research opinions have revealed that, appropriate supplementary cementitious materials (SCM) such as fly ash, rice husk ash, palm oil fuel ash, granulated blast furnace slag, limestone powder and silica fume have been effectively used to produce SCC with good flowing ability (Nanthagopalan and Santhanam, 2009; Md. Safiuddin, et al, 2011a; Md Safiuddin, et al., 2011b; Md Safiuddin et al, 2012; Uysal et al, 2012)

Industrial processes and research have shown that powders provide to a great extent, the highest percentage of specific surface area in any concrete mix, their packing behavior and water demand, which is very paramount in the design of self-consolidating concrete. In addition, these powders show dominant effect on the fresh properties as well as hardened properties such as strength, deformation and durability. Consequently, the knowledge of powder properties is necessary in order to ensure high degree of quality control, optimum product performance as well as improved powder production process (Hunger and Brouwers, 2009). The hardened attributes of concrete are influenced by the properties of the matrix, the aggregates, and the bond behavior of matrix and aggregates. This is largely due to the fact that the matrix is made up of powder (binder), mixing water, fines content of the aggregate and admixtures (Taylor, 1997). Effective water/binder ratio (water/cement ratio), binder reactivity, particle shape and particle size distribution of the binders are the key factors that affects the characteristics of the matrix and the bond behavior between the matrix and the aggregate (Powers, 1968).

Although there are a number of literatures on the performance of both POFA and PBC on the production of mortar and concrete, there is no literature that studies the effect of the Physio-chemical characteristics of the blend of POFA and PBC on the water demand as well as the HRWR requirement of paste, mortar or concrete.

## **2. MATERIALS AND METHODS**

### **2.1. Materials processing**

The materials used for this study include; pulverized burnt clay (PBC), palm oil fuel ash (POFA), Ordinary Portland cement (OPC) and polycarboxylic based high range water reducer (HRWR). The PBC used in carrying out this study consisted of fragments of fired clay bricks that were discarded

as waste in a clay brick factory Kota Tinggi area of Johor as well as demolition site in Johor Bahru, in Malaysia. The fragments were reduced to smaller sizes and then ground to a very fine powder using Los-Angelis abrasion machine. The material was then packaged in air-tight plastic bags so as to control the moisture content. POFA is a by-product of palm oil processing industry which is generated from the combustion of palm oil plant residues. The material was obtained from a palm oil processing industry located at Kluang in Johor, Malaysia. It was dried, sieved and then ground to a very fine powder. OPC type I with a brand name “TASEK” was used in this study. HRWR with a brand name “GLENIUM ACE 388(RM) SURETEC” conforming to ASTM C494 for type F and G admixtures was used.

## 2.2. Materials testing

To effectively characterize the materials, essential tests as specified by relevant standards were carried out. These tests include; particle size analysis (PSA), X-ray diffraction (XRD), Scanning electron microscope (SEM), surface area analysis and chemical analysis (XRF).

### 2.2.5. Paste formulation

Two series of paste were formulated based on the mix design of parent self-consolidating concrete. Series 1 consist of 9 different mixtures of paste that are formulated based on water to binder (W/B) ratio of 0.3 with a blend of POFA and PBC as SCM at the replacement level of 0-30% (0/0; 5/5; 10/5; 10/10; 15/15; 0/5; 0/10; 5/0; 10/0). HRWR is used in the preparation of series I paste. On the other hand, series 2 paste is also formulated based on the same W/B ratio of 0.3 and percentage replacement of OPC with the SCM, but in this case, no HRWR was used. Instead, certain percentage of mixing water was added to achieve similar flow as in series 1. For each of the series, 3L of paste was prepared using the medium sized epicyclic revolving type mixer, conforming to ASTM C305 (2012) specification. The mixture proportion of the various binder pastes is as shown in tables 1 and 2.

**Table 1: Mixture composition of series 1 paste**

Paste Numenclatur	POFA (% of B)	PBC (% of B)	W/B Ratio	Cement (kg)	POFA (kg)	PBC (kg)	Water (kg)	HRWR (% of B)
30P1	0	0	0.3	4.57	0.00	0.00	1.37	0.25-2.00
30P2	5	5	0.3	4.06	0.23	0.23	1.35	0.50-3.00
30P3	5	5	0.3	3.81	0.45	0.22	1.34	0.75-3.00
30P4	10	10	0.3	3.57	0.45	0.45	1.34	0.75-4.00
30P5	15	15	0.3	3.09	0.66	0.66	1.32	0.75-4.00
30P6	0	10	0.3	4.08	0.00	0.45	1.36	0.25-2.00
30P7	0	20	0.3	3.59	0.00	0.90	1.35	0.25-2.00
30P8	10	0	0.3	4.05	0.45	0.00	1.35	0.25-2.00
30P9	20	0	0.3	3.55	0.89	0.00	1.33	0.25-2.50

B: Binder

**Table 1: Mixture composition of series 2 paste**

Paste Numenclature	POFA (% of B)	PBC (% of B)	W/B Ratio	Cement (kg)	POFA (kg)	PBC (kg)	Water (kg)	Additional water (% of W)
30P1	0	0	0.3	4.57	0.00	0.00	1.37	30-80
30P2	5	5	0.3	4.06	0.23	0.23	1.35	40-90
30P3	10	5	0.3	3.81	0.45	0.22	1.34	50-100
30P4	10	10	0.3	3.57	0.45	0.45	1.34	50-100
30P5	15	15	0.3	3.09	0.66	0.66	1.32	50-100
30P6	0	10	0.3	4.08	0.00	0.45	1.36	30-80
30P7	0	20	0.3	3.59	0.00	0.90	1.35	30-80
30P8	10	0	0.3	4.05	0.45	0.00	1.35	50-100
30P9	20	0	0.3	3.55	0.89	0.00	1.33	60-120

B: Binder

### 2.2.6. Test for water demand and HRWR requirement

Two different series of paste were used to evaluate the water demand and the HRWR requirement of the various binder pastes. The first method involves the use of a standard flow cone conforming to ASTM C 939 (2010) to determine the time of flow of the respective binder paste at an incremental dosage of HRWR from 0.25 up to 4.0% by weight of binder and the second method involves determining the time of flow of the respective binder paste at an incremental quantity of water from 30 up to 120% by weight of the mixing water.

## 3. RESULTS AND DISCUSSION

### 3.1. Physical properties

Table 1 shows the physical properties of the respective powder samples in relation to that of OPC. From the physical observation, OPC and POFA show grey and dark grey colors respectively. The dark grey color of POFA is due to the presence of un-burnt carbon. On the other hand, PBC maintains the reddish colour of the parent brick from which it was obtained. The assessment of the colour of the respective powders is very important, particularly when designing coloured paste, mortar or concrete (López, Tobes, Giaccio, & Zerbino, 2009). The specific gravity of POFA is 2.42, which is lesser than PBC which has a value of 2.69. Nonetheless, both POFA and PBC have specific gravity less than that of OPC which has a value of 3.15. These lower values of specific gravity of the supplementary cementitious materials are in most cases, responsible for the reduction in the density of paste, mortar and concrete (Md Safiuddin, et al., 2012). The BET surface area of the respective binders as shown in table 3 shows that PBC has the least BET surface area, with a value of 2.9791m<sup>2</sup>/g. This reduced surface area contributes to the reduction of the water demand and HRWR requirements at higher replacements as shown in figures 3 and 4. Notwithstanding, all the materials satisfy the basic requirements of ASTM C 618. In terms of the percentage of the fine powder passing 45- $\mu$ m wet sieve, as well as 75- $\mu$ m and 150- $\mu$ m dry sieving, all materials falls well within the range specified by ASTM C311 and ASTM C430 of 66%.

**Table 3: Physical properties of powders**

Property	Description	PBC	POFA	OPC
Specific gravity		2.69	2.42	3.15
Fineness	BET surface area (m <sup>2</sup> /g)	2.9791	23.7514	5.067
	% passing 45- $\mu$ m wet sieve	96.40%	98.40%	98.60%
	% passing 75- $\mu$ m dry sieve	99.20%	99.50%	99.70%
	% passing 150- $\mu$ m dry sieve	99.50%	99.80%	99.90%
	Median Particle size, d <sub>50</sub> ( $\mu$ m)	15.70	14.8	14.3
Colouration	Visual inspection	Reddish	Dark grey	Light Grey

### 3.2. Chemical characterization

The chemical composition of the respective powders is provided in table2. The major chemical component is SiO<sub>2</sub>, which is 68.60% and 63.70% for PBC and POFA respectively. Other Pozzolanic components are Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>. The combined oxide content; SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> falls within the limit specified by ASTM C 618 for class F fly ash. In effect, these properties make them suitable materials to be used as supplementary cementitious materials for the production of different type concrete and mortar.

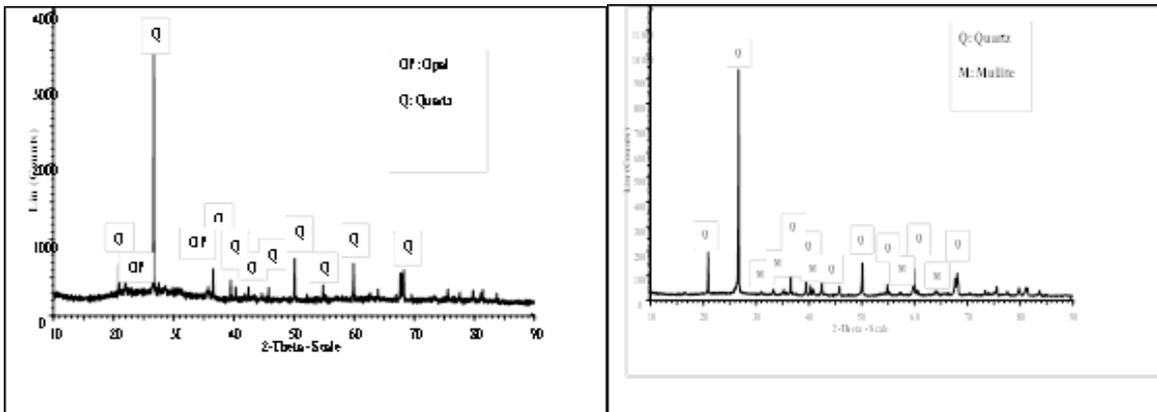
**Table 2: Chemical composition of powders**

Oxide composition	PBC (%)	POFA (%)	OPC (%)
SiO <sub>2</sub>	68.6	63.7	16.4
Al <sub>2</sub> O <sub>3</sub>	20.6	3.68	4.24
Fe <sub>2</sub> O <sub>3</sub>	4.66	6.27	3.53
CaO	0.34	5.97	68.3
K <sub>2</sub> O	3.99	9.15	0.22
P <sub>2</sub> O <sub>5</sub>	-	4.26	-
MgO	0.34	4.11	2.39
SO <sub>3</sub>	-	1.59	4.39
Cl	-	0.5	-
TiO <sub>2</sub>	0.63	0.3	0 < LLD
Na <sub>2</sub> O	0.32	0 < LLD	-
Mn	-	0 < LLD	0.15
CO <sub>2</sub>	0.1	-	0.1
SiO <sub>2</sub> + Fe <sub>2</sub> O <sub>3</sub> + Al <sub>2</sub> O <sub>3</sub>	93.86	73.65	

### 3.3. Characterization by X-ray diffraction (XRD)

The X-ray diffraction pattern of the respective powders is as shown in figure 1. The major mineral that presents a crystalline structure in the three respective powders is Quartz. PBC shows higher quartz intensity of about 10,000 counts in comparison to POFA which has 3500 counts, this is due to the fact that sand granules are used to stabilize brick during firing to prevent surface cracks. Although the two SCM are similar in terms of their quartz content, PBC contain crystals of mullite and Quartz, with an approximate mullite intensity of about 1200 counts. The X-ray diffraction pattern of PBC has been shown to have close similarities with that of fly ash (FA) (Hassan et al., 2013). These similarities arise because the FA particles are formed from clay during heating just like the brick powder (Bensted and Barnes, 2002). Thus, PBC, just like FA performs an important role in the reduction of water demand. On the other hand, POFA contains crystals of Opal which are

formed from the calcination of organic constituents of the palm oil fibers and shells (Hassan, et al., 2013).

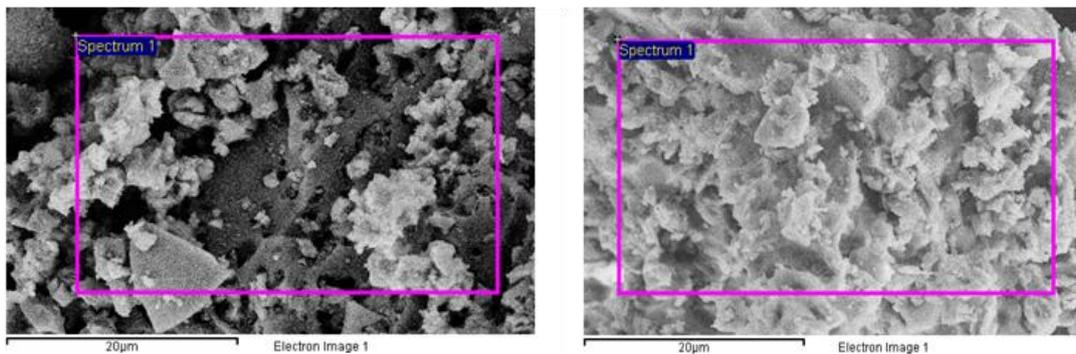


(a) (b)

**Figure 1: X-ray diffraction pattern of (a) POFA, (b) PBC.**

### 3.4. Morphological and microscopic analysis

Figure 2 (a) and (b) shows the SEM results of the SCM. POFA consist of irregularly shaped particles that are well dispersed. The irregular shape coupled with the loose particle parking is responsible for the higher surface area, higher water demand and consequently, higher dosage of HRWR. On the other hand, PBC of irregular particles that are densely packed thereby accounting for the reduced surface area. The nature of the particle parking contributes to the water demand and HRWR requirement.



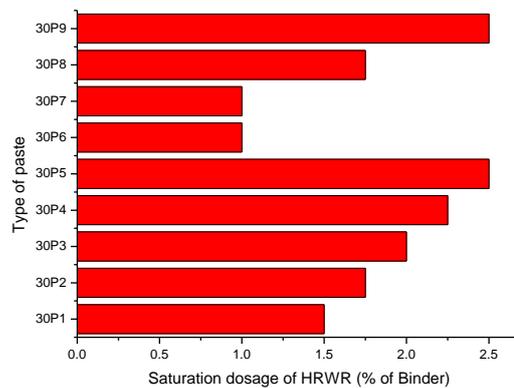
(a) (b)

**Figure 2: Scanning electron microscopy of (a) POFA, (b) PBC.**

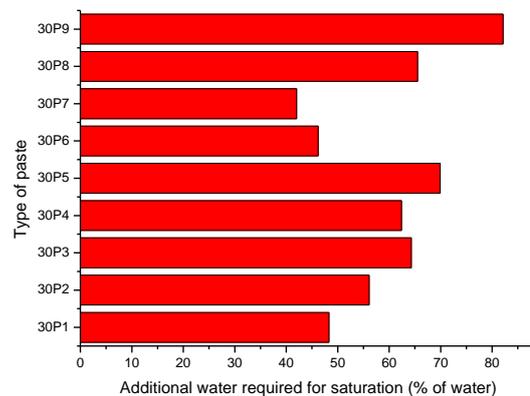
### 3.5. Water demand and HRWR requirement

Figure 5 and 6 shows the result of the saturation dosages of HRWR and the water demand corresponding to the saturation dosages as determined by the flow cone test. The saturation dosages increases from 1.5 to 2.5 as the percentage of the blend of supplementary cementitious materials increases from 10 to 30%. Also, the water demand corresponding to the saturation dosages increases from 48.3% to 69.9%. This pattern is primarily exhibited by the ternary blended pastes

30P1-30P5. A different pattern of behavior was exhibited by binary blended pastes 30P6 and 30P7 as well as 30P8 and 30P9. For the binary blended paste 30P6 and 30P7, containing 10% and 20% of PBC respectively, the saturation dosages of HRWR remain constant at 1% while the water demand decreases from 46.2% to 42%. This behavior is an indication of the fact that PBC act as a water reducer, suitable for the production of self-consolidating paste, mortar and concrete (Heikal, Zohdy, & Abdelkreem, 2013). On the other hand, binary blended paste 30P8 and 30P9, containing 10% and 20% POFA, the saturation dosages of HRWR increases from 1.75% to 2.5%. Also the water demand increases from 65.52% to 82.11%. This characteristic is attributed to the high carbon content as well as high specific surface area of POFA. Furthermore, the characteristics exhibited by PBC and POFA are basically due to their particle shapes, sizes and fineness as well as the morphological and microscopic characteristics. Nonetheless, POFA has been advocated to be a very good Pozzolanic material for the production of paste, mortar and concrete (Kroehong, Sinsiri, Jaturapitakkul, & Chindaprasirt, 2011).



**Figure 3: HRWR requirement for various binder pastes.**



**Figure 4: Water demand required to achieve saturation without HRWR**

## CONCLUSION

From the result of the study, it can be concluded that the physical characteristics, chemical characteristics as well as the morphological properties of binder can greatly affect the water the fresh characteristics of paste, mortar and concrete.

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