RHEOLOGICAL BEHAVIOUR OF FRESH MORTAR FORMULATED FROM SELF-COMPACTING HIGH PERFORMANCE CONCRETE INCORPORATING RICE HUSK ASH

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ABSTRACT

This study investigated the effect of pore structure of rice husk ash (RHA) on rheological properties of mortar formulated from self-compacting high performance concrete. Firstly, properties relating to pore structure of RHA were evaluated by scanning electron microscopy image, measurement of specific surface area, determination of pore volume and pore size in RHA particles, and by water demand test. Subsequently the effect of mean particle size and content of RHA on saturation dosage of superplasticizer and rheological properties of mortar was investigated. These properties were evaluated for reference and silica fume (SF) containing samples as well. The results show that the RHA was a porous material including macro and meso pores. Pore size distribution, namely macro pore content, played a key role in influencing other properties of RHA. Depending on particle size and content, blending RHA or SF with cement resulted in a significant change of rheological properties of mortar.

Keywords: **Rice husk ash; pore structure; self-compacting high performance concrete; rheological property.**

INTRODUCTION

Self-compacting high performance concrete (SCHPC) is considered as a new generation of self-compacting concrete (SCC) with high strength and good durability of high performance concrete (HPC) [1]. To meet criteria for SCHPC, the rheological properties at low water binder ratio (w/b) are of the most importance. RHA is classified as a highly reactive pozzolan. It possesses a very high silica content similar to that of SF [2]. SF is a dense material whereas RHA is a porous material [2, 3]. With the given amorphous silica content, variation in mean particle size (MPS) of RHA results in variation of pore volume, and pore size; a different specific surface area (SSA) and therefore a different pozzolanic reactivity and different water absorption of RHA [4, 3]. These parameters dramatically influence properties of fresh concrete containing RHA at low w/b.

RHA is a very good replacement for SF in respect of compressive strength and durability of concrete [4, 1]. Apart from a function as a mineral admixture, RHA has been used to improve viscosity and thus segregation resistance of SCHPC [5, 6, 1]. However, the pore structure of RHA as a main factor influencing the rheological properties of SCHPC has not been studied in detail. Especially, shear thickening behaviour of SCHPC containing RHA has not yet been investigated before.

This study investigated the effect of pore structure of RHA on rheological properties of mortar formulated from SCHPC. Firstly, properties relating to pore structure of RHA were evaluated by scanning electron microscopy (SEM) image, measurement of specific surface area (SSA), determination of pore volume and pore size in RHA particles, and by water demand test. Subsequently, the effect of MPS and content of RHA on saturation dosage of superplasticizer and rheological properties of mortar was investigated. These properties were evaluated for the reference and SF containing samples as well.

EXPERIMENTAL PROGRAM

Materials

Portland cement (CEM I 52.5R), RHA, SF and natural sand with a maximum particle size of 2 mm were used in this study. Chemical composition and physical properties of the Portland cement, RHA and the natural sand have been given in a previous study [5]. RHA was produced by burning rice husk, an agricultural waste, in a simple incinerator prototype in Vietnam which was designed based on the principle of the atmospheric bubbling fluidised bed [7]. The obtained ash was ground in a ball mill. RHA was used with five different MPSs of 5.7, 7.7, 12.0, 15.6, and 22.6 μm. The SF (containing 96.6% SiO_2) had MPS of 0.35μm; SSA of 18.9 m^2/g and density of 2.26 g/cm^3. In addition, a polycarboxylate-based superplasticizer with density of 1.08 g/cm^3 and 40% solid content by weight was used.

Mixture proportions

The mortars were formulated from their corresponding SCHPCs. The SCHPC mixtures were designed based on the absolute volume of the constituent materials with a constant paste volume of 38.5 % (including 2% of air), a constant fine aggregate volume of 30.4 % and a coarse aggregate volume of 31.1 %. The cement content was replaced with RHA/SF by weight but the paste volume was kept constant [5]. The proportions of sand, cement, RHA/SF and water of mortars were identical to their corresponding concretes. For all mixtures the superplasticizer saturation dosage (SSD) was used. The mixture proportions are shown in Tab. 1.

Table 1. Mortar mixture proportions

w/b	Sand (vol.-%)	SF (wt.-%)	RHA (wt.-%)	SSD(wt.-%)	MPS of RHA (μm)
0.26	44	0	0	2.00	-
0.26	44	10	0	2.00	-
0.26	44	20	0	2.75	-
0.26	44	0	10	2.50	15.6
0.26	44	0	10	2.25	7.7
0.26	44	0	10	2.00	5.7
0.26	44	0	20	2.75	5.7

Experimental methods

The Laser diffraction (LS 230, Beckman Coulter), the Brunauer-Emmett-Teller method (ASAP 2000, Micromeritics), and the Barrett-Joyner-Hanlenda (BJH) method (SA 3100, Beckman Coulter) were used to determine MPS, SSA, pore volume and pore size in RHA particles, respectively. Additionally, the porous structure and morphology of RHA particles were analysed by an ultra high resolution scanning electron microscope (Nova NanoSEM 230).

The water saturation demand of RHA, SF and cement was determined according to Puntke method [8]. Slump flow and flow time of fresh mortar were determined by using mini-cone slump flow described by EFNARC [9], T25 time which is spread time for a diameter of 25cm, corresponding to T50 time measured in SCC [10] respectively. The SSD of mortar was determined by testing the mini-cone slump flow. The SSD is defined as the superplasticizer dosage beyond which slump flow does not increase significantly.

Rheological properties of the mortars were measured by a Viskomat NT with a basket probe (Schleibinger, Germany). The measurements were started 15 minutes after water addition. During the measurement, temperature of the samples was kept constant at 20°C by a water bath. In order to eliminate the effects due to thixtropy, the measurement was started at maximum rotational velocity of 12 rpm for 80 seconds, and then the velocity decreased in 14 steps of each 20 seconds. Immediately after measurement, a maximum velocity and about 70% of the maximal velocity were applied again to detect segregation and/or thixotropy [11]. If segregation occurs, the torques at these points are significantly higher than former values due to blocking. The calibration proposed by Vogel was applied to calculate shear stress and shear rate from torque and rotational velocity respectively [12]. Since flow curves obtained from

all tests expressed linearity at lower than a given shear rate, however, non-linearity at higher ones, yield stress and plastic viscosity were calculated in accordance with the Bingham model with shear rates below 0.5 1/s and excellent linear regression ($R^2 \geq 0.99$) was considered. Whereas to evaluate shear-thickening behaviour, the modified Bingham model was applied with higher shear rates. This model is presented by: $\tau = \tau_0 + \mu.\dot{\gamma} + c.\dot{\gamma}^2$, where τ: shear stress (Pa); τ_0: yield stress (Pa); μ: plastic viscosity (Pa.s); $\dot{\gamma}$: shear rate (1/s) and c: second order parameter (Pa.s^2). The parameter c/μ corresponds to index n in Herschel-Bulkley model, and therefore decribes shear thickening behaviour [11].

RESULTS AND DISCUSSION

Microstructure, specific surface area, fineness and water demand of RHA

The pore structure on surface and morphology of RHA particles are shown in Fig.1. By NanoSEM imaging, it is obvious that the RHA was a porous material including macro and meso pores. In general, due to grinding RHA particle size reduced, macro pores collapsed resulting in a lower total pore volume. Whereas SSA increased with grinding time or lower particle size regardless of less pore volume. Water demand of RHA decreased with smaller particle size (Tab.2) and was higher than those of SF (0.59±0.00) and the Portland cement (0.34±0.02), excepting for RHA5.7.
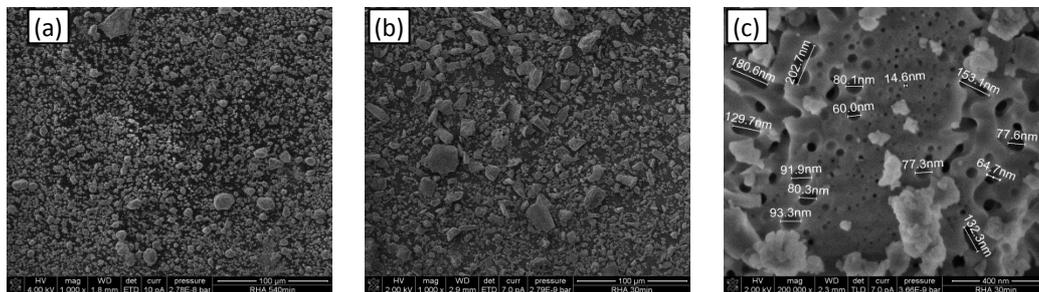

Figure 1. Nano SEM images of RHA 5.7 and RHA 15.6: a) morphology of RHA5.7 particle; b) morphology of RHA 15.6 and c) surface of RHA 15.6 particle with macro and meso-pores.

Table 2. Parameters of RHA types relating to their pore structure

Parameters	Different types of RHA				
Grinding time (minute)	15	30	45	90	540
Mean particle sizes (μm)	22.6	15.6	12.0	7.7	5.7
Average pore width (BJH absorption, nm)	78.72	67.00	53.52	58.77	31.85
Total pore volume (BJH absorption, cm^3/g)	0.1169	0.1224	0.1030	0.1059	0.0799
Macro pore volume (vol.%)	61.26	49.88	40.56	45.69	22.37
Specific surface area (BET absorption, m^2/g)	23.69	22.36	22.94	23.05	25.21
Water demand (Puntke method, cm^3/g)	0.79±0.02	0.71±0.01	0.64±0.02	0.61±0.02	0.57±0.01

Effect of RHA on SSD, slump flow and flow time of mortar

In Tab. 1, SSD of mortar is presented. It can be seen that an increase in SSD of mortar was observed with larger MPS of RHA and higher content of RHA. The mortar incorporating larger RHA grain had a larger SSD than those of the reference and the SF incorporating samples.

Slump flow and T25 time of mortar as a function of MPS and content of RHA are illustrated in Fig. 2 and Fig. 3 respectively. The slump flow of mortar increased, whereas the T25 time of mortar decreased with smaller MPS and with lower content of RHA regardless of a decreased SSD. Compared to SF containing sample and the reference sample, RHA containing samples had lower slump flow and longer T25 time.
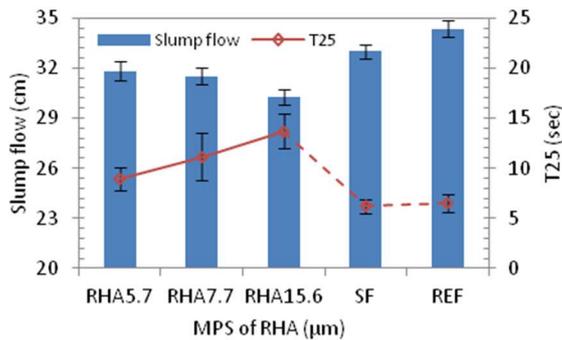
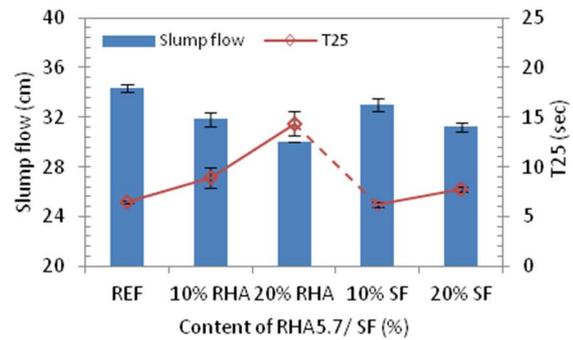
Figure 2. MPS of RHA vs. slump flow and T25   *Figure 3. RHA/SF content vs. slump flow and T25*

Effect of RHA on yield stress and plastic viscosity of mortar

In Fig. 4 and Fig. 5 rheological measurements for the mortar made with RHA or SF are plotted against MPS of RHA and RHA or SF content. It is clear that increasing content and MPS of RHA resulted in dramatically higher yield stress and viscosity of mortar. The mortars containing RHA had higher yield stress and viscosity than those of SF containing sample and the reference sample. Increasing level of yield stress and viscosity due to a higher RHA content was comparably higher than that due to a higher SF content (Fig. 5). These results were closely associated with the slump flows and the T25 times above.
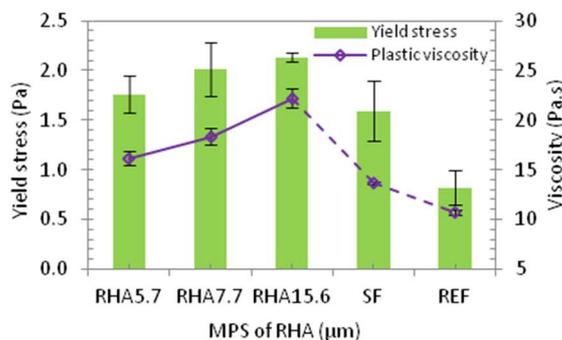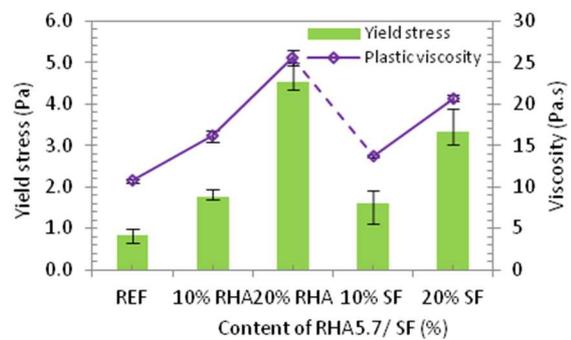

Figure 4. MPS of RHA vs. yield stress and viscosity   *Figure 5. RHA /SF content vs. yield stress and viscosity*

Effect of RHA on shear thickening behaviour of mortar

The flow curves for mortars containing RHA or SF and the reference mortar are provided in Fig. 6 and Fig. 7. The results indicate that shear-thickening behaviour ($c/\mu > 0$) was observed from all samples regardless of binder composition. Compared to the reference mortar, addition of RHA or SF decreased the shear thickening degree. Increasing MPS of RHA led to a greater shear thickening response (higher c/μ value). Whereas increasing RHA or SF content resulted in lower shear thickening degree or greater shear thinning degree. The flow curve for mortar made with SF expressed linearity ($c/\mu \approx 0$). The decreasing level of shear thickening response owing to a higher SF content was clearly higher than that owing to a higher RHA content (Fig. 7).
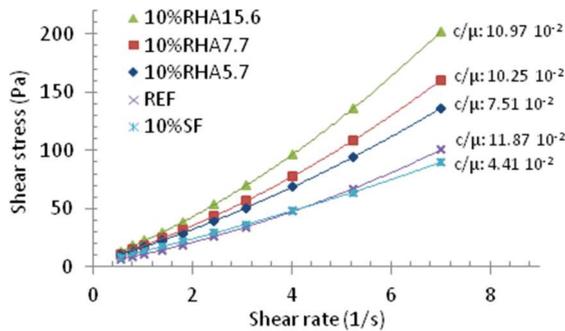

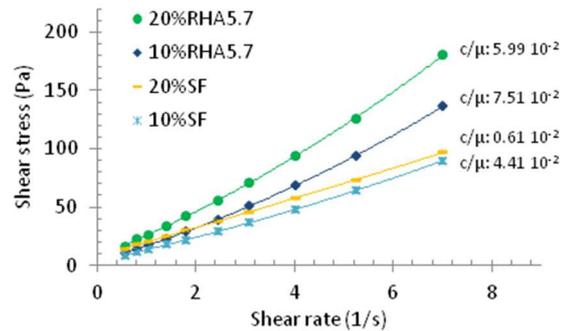Figure 6. MPS of RHA vs. shear thickening   Figure 7. RHA/SF content vs. shear thickening

Discussion

RHA is a porous material including macro (>50nm), meso (2-50nm), and micro pores (<2nm). It is documented that when the RHA is predominantly mesoporous, it is considered as a mesoporous material [13]. In this study, macro and meso pores were both observed from the investigated RHA by application an ultra-high resolution SEM (Fig. 1b). It can be found that the pore size distribution, namely macro pore content, plays an important role in influencing other parameters of RHA such as total pore volume, SSA and water demand (Tab. 4). During grinding, the macro pores collapse thereby total pore volume decreases and thus the water demand decreases. The SSA, however, increases with lower total volume. It could be due to a reduction in MPS and formation of new open pores (micro pore) [3].

Addition of RHA results in a significant change of rheological properties of mortar depending on MPS and content of RHA used. Similar to SF, the RHA has very high SSA and its particles are chemically highly reactive and may have an affinity for multi-layer adsorption of superplasticizer molecules [14]. Using RHA or SF as replacements for cement leads to an increase in SSA of the binder (cement+RHA/SF) thus to a decrease in free water compared to the mortar made without RHA or SF. As a result, the mortar incorporating RHA or SF has the higher SSD, yield stress, and plastic viscosity and the lower slump flow. This effect is more pronounced, when larger MPS and higher content of RHA are used.

Incorporating larger grains of porous RHA lowers the free water and increases the particle size of the granular mixtures of paste in mortar. That results in a greater shear thickening degree of mortar. Increasing content of RHA (5.7μm) which has a smaller grain size than cement (7.07μm) decreases shear thickening response possibly due to better polydispersion [15] and better packing density of granular mixtures. Different from RHA, SF particles are spherical and dense and hence SF with ''ball bearing effect'' and lower water demand increases the slump flow and decreases viscosity and degree of shear-thickening [16] (Fig. 6, 7) of mortar. The lower shear thickening response obtained from RHA/SF containing mortar compared to the reference mortar can possibly be attributed to the reduction in slump flow [11].

CONCLUSIONS

Based on the experimental results, the following conclusions can be drawn.

➢ The pore size distribution of RHA plays the key role in influencing pore volume, SSA-BET and water demand.

➢ Increasing mean particle size and content of RHA increases saturation dosage of superplasticizer, yield stress, plastic viscosity, and decreases slump flow of mortar. It can be explained in detail by the water absorption into the pore structure of RHA particles.

➢ The greater degree of shear thickening of mortar is observed with a larger mean particle size of RHA possibly due to the higher water absorption into RHA particles and the larger particle size of granular mixture of paste in mortar.

➢ The mortar containing 20% RHA has a lower degree of shear thickening compared to that of sample containing 10% RHA (5.7μm). It may be due to the better polydispersion and the better packing density of granular mixtures.

➢ Compared to RHA, blending SF with cement decreases the yield stress, the plastic viscosity, and shear-thickening behaviour and increases slump flow of mortar possibly due to the lower water demand; the ball bearing effect and the better filling effect.

ACKNOWLEDGEMENTS

REFERENCES

1. Safiuddin, M., Development of self-consolidating high performance concrete incorporating rice husk ash, University of Waterloo, Canada, 2008, pp. 326.

2. Mehta, P. K., Rice husk ash - a unique supplementary cementing material, Advances in concrete technology, 1994, pp. 419-444.

3. Van, V. T. A., Rice husk ash as a pozzolanic admixture in ultra-high performance concrete, FIB institute, Bauhaus universität, Weimar, 2013, pp. 120.

4. Nguyen, V. T., Rice husk ash as a mineral admixture for ultra high performance concrete, Materials and Environment, Delft university of technology, 2011, pp. 139.

5. Le, T. H., Siewert, K., Ludwig, H. M., Synergistic effects of rice husk ash and fly ash on properties of self-compacting high performance concrete, symposium on Ultra high performance concrete and Nanotechnology for High performance construction materials, 2012, pp. 287-295.

6. Memon, S. A., Shaikh, M.A., Akbar, H., Utilization of rice husk ash as viscosity modifying agent in self-compacting concrete, Construction and building materials, Vol. 25, no. 2, 2011, pp. 1044-1048.

7. Armesto, L., Bahillo, A., Veijonen, K., Cabanillas, A., Otero, J., Combustion behaviour of rice husk in a bubbling fluidised bed, Biomass and Bioenergy, Vol. 23, no. 3, 2002, pp. 171-179.

8. Puntke W., Wasseranspruch von feinen kornhauf-werken, beton 52, 2002, pp. 242–248.

9. EFNARC, Specification and guidelines for self-compacting concrete, 2002, pp.

10. Rizwan, S. A., High performance mortars and concretes using secondary raw materials, Maschinenbau, Verfahrens-und Energietechnik, Vol. Doctor-Engernieur, Technischen Universität Bergakademie Freiberg, Freiberg, 2006, pp. 114.

11. Feys, D., Verhoeven, R., Schutter, D., G., Fresh self compacting concrete, a shear thickening material, Cement and Concrete Research, Vol. 38, no. 7, 2008, pp. 920-929.

12. Vogel, R., Eine messzelle für spezialmörtel, http://www.vogel-labor.de/ Accessed 4 Apr. 2012.

13. Srivastava, V. C., Mall, I. D,. Mishra, I. M., Characterization of mesoporous rice husk ash (rha) and adsorption kinetics of metal ions from aqueous solution onto rha, Journal of Hazardous Materials, Vol. 134, no. 1-3, 2006, pp. 257-267.

14. Nehdi, M., Mindess, S., Aitcin, P. C., Rheology of high-performance concrete: Effect of ultrafine particles, Cement and Concrete Research, Vol. 28, no. 5, 1998, pp. 687-697.

15. Barnes, H. A., Shear-thickening (``dilatancy'') in suspensions of nonaggregating solid particles dispersed in newtonian liquids, Journal of Rheology, Vol. 33, no. 2, 1989, pp. 329-366.

16. Yahia, A., Shear-thickening behavior of high-performance cement grouts - influencing mix-design parameters, Cement and concrete research, Vol. 41, no. 3, 2011, 230-235.