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Physical and Chemical Properties of Cement with Nano Black Rice Husk Ash

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Abstract. The existing of pores and micro-crack in the hardened cement paste influences the strength itself. Nano silica produced from BRHA has been used in this study. Nano BRHA replacement levels of 0%, 10%, 20% and 30% by weight of cement were applied. The TGA/DTA and XRD has been conducted for hardened cement paste powder to analyses the effect of different partial replacement of Nano BRHA. It was found that the weight loss results for $Ca(OH)_2$ seems decreasing with increasing in Nano BRHA replacement. The higher CSH percentage contained in cement paste was 10% of Nano BRHA replacement.

INTRODUCTION

Black rice husk ash (BRHA) defined as rice husk ash (RHA) that contained high amount of carbon after burning process. The other name of BRHA is rice husk char. According to Carmona et al. [1], 94% of RHA contained silica. RHA is an agricultural waste which is classified as “a highly active pozzolan” because it contains a very high amount of amorphous silica and a large surface area [2]. Yusak et al. [3] characterized RHA using X-ray diffraction (XRD) and found that the cristobalite, tridymite and quartz are the major phases present in the SiO_2 obtained from rice husk. Sarangi et al. [4] confirmed in their study that the major peak in BRHA is silica. Ramadhansyah et al. [5] in their study conducted thermal analysis on RHA. They found that there have four endothermic peaks which are corresponding to polysaccharide depolymerisation, dehydration of sugar, silica in the amorphous form and the crystallization of tridymite and cristobalite respectively.

Cement paste binder is one of the important parts of concrete. According to Yang and Jiang [6], the existing of some pores and microcracks in the hardened cement paste will influences the hardened cement paste strength greatly. Chindaprasirt et al. [7] also found in their literature review that, the presence of void acts as weaknesses in cement matrix and produces crack formation. To overcome this weakness, previous researchers found that the addition and/or replacement of silica fume and nano silica [8] in cement content strengthen the hardened cement paste. Aleem et al. [9] study on the cement pastes containing different percentages of nano silica hydrated for 28

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days and they found that the cement pastes incorporating nano silica has lower intensity of the diffraction lines corresponding to calcium hydroxide and higher intensity of diffraction lines of characteristics for CSH than those of the corresponding lines of the control mix. The higher intensity of diffraction lines for CSH gives the denser of the structure. Yu et al. [10] found in their study that the mass loss of free water and ettringite slightly increase with additions of nano silica in the cement paste and the mass loss of $Ca(OH)_2$ gradually reduced with an increasing of nano silica amount. This finding similar with Isfahani et al. [11] that the mass loss of $Ca(OH)_2$ decreased with increasing of nano silica amount. Rupasinghe et al. [12] found in their study that, the $Ca(OH)_2$ content of the mixes including nano silica is lower than that of the plain cement paste, indicating that nano silica reacts with the cement paste through a pozzolanic reaction. This study conducted in order to determine the thermal effect of cement paste containing nano silica produced from BRHA. Thus the knowledge from this study would be beneficial for future researchers on the mechanisms of cement paste containing nano silica produced from BRHA.

MATERIALS

Cement

Type I ordinary Portland cement (OPC) was used as the major binder material in this study. The cement used was supplied by Tasek Corporation Sdn. Bhd in one batch for the entire experimental works. The chemical composition of the OPC was given in Table 1. The mean particle size of the OPC is 1353 nm.

Nano black rice husk ash

Nano material was produced from the black rice husk ash. The black rice husk ash was ground using laboratory ball mill grinder. The mean particle size of nano used in this study is 66 nm. Table 1 shows the chemical composition of the black rice husk ash.

Table 1. the chemical composition of the OPC and Nano BRHA

Oxides	OPC	Nano BRHA
SiO_2	22.68	91.33
Al_2O_3	4.72	0.07
Fe_2O_3	3.5	0.07
CaO	62.27	0.45
MgO	1.89	0.28
Na_2O	-	0.01
K_2O	0.31	2.64
SO_3	4.29	0.05

Water

Water is an important material for hydration process in cement paste. To produce good quality of hardened cement paste, the water must not contain any substance that might affect the chemical reaction between cement and water. In this study, tap water supplied to the highway laboratory of Universiti Teknologi Malaysia (UTM) was used throughout the study in mixing and others purpose.

METHODOLOGY

Sample preparation

A control mix was prepared using OPC. Nano BRHA replacement levels of 10%, 20% and 30% by weight of cement were applied. Table 2 shows the mix proportion used in this study. In order to mix the cement paste, all the materials were placed into bowl in the following order: cement and nano BRHA. The materials were first mixed for 30 seconds under dry condition. The water was then added into bowl and the mixing was performed for another 1.5 minutes. Then the cement paste was casted in the mould. Immediately after casting process, the specimens were kept and cover by wet cloth to avoid moisture loss. After 24 hours, the specimens were transferred for curing in the water until testing time.

Table 2. Cement paste mix proportions

Nano BRHA (%)	w/c	Water (g)	Cement (g)	Nano (g)
0 (OPC)	0.34	34	100	0
10	0.34	34	90	10
20	0.34	34	80	20
30	0.34	34	70	30

Thermal analysis

In this study, differential thermal analysis and thermogravimetric analysis (DTA/TGA) was used to analyse the thermal behaviours of cement paste powder. DTA locates the ranges corresponding to thermal decompositions of different phases in paste, whereas TGA relies on a high degree of precision in three measurements of mass change, temperature and temperature change. Simultaneous TGA and DTA were performed using the machine METTLER TOLEDO TGA/SDTA851e. At the specified day of testing, the hardened cement paste specimens were crushed become powder form (passing 75µm sieve). A total of 20 to 40mg of the specimens was used to conduct the test. The specimens was then placed on the platinum pan and heated to a nitrogen atmosphere at the temperature of 25 ˚C to 900 ˚C at 10˚C/min. The results were recorded for further analysis.

X-ray diffraction analysis

The X-ray diffraction (XRD) is a rapid analytical technique primarily used for phase identification of crystalline material. In this study, cement paste powder specimens were characterized by RIGAKU Smart lab X-ray Diffractometer. As described in BS EN 13925-1 [13], the specimens were scanned in steps of 2θ with a fixed counting time of 1 second. The range of x-ray scan was from 2θ = 10° to 90°, using copper (Kα Cu) with wavelength (λ) of 1.5406 nm as x-ray source.

RESULTS AND DISCUSSIONS

OPC and Nano BRHA

Figure 1 and 2 shows the TGA/DTA results of OPC and BRHA respectively. Generally, the result curves for OPC and BRHA shows decreasing in TGA percentage with increasing temperature. This is due to the decomposition of elements with increasing of temperature. Based on Figure 1, there are 4 phases of weight loss with increasing of the temperature. The first phase, range from 60 °C to 104 °C, the second phase range from 104 °C to 175 °C, the third phase range from 396 °C to 449 °C and lastly from 528 °C to 740 °C. According to the temperature range, the first phase is due to the elimination of moisture in sample. The second phase is due to the decomposition of most of the hydration products. The decomposition of portlandite takes place for the third phase. Lastly, for the fourth phase, this is due to the decomposition of carbonate in the material. Snyder and Stutzman [14] reported that the peak below 250 °C is the loss of free water and the decomposition of most of the hydration products while the peak between 400 °C to 500 °C is due to portlandite decomposition. The percentage of weight loss for phase 1, 2, 3 and 4 are 0.371 %, 0.558 %, 0.1725 % and 3.1539 % respectively.

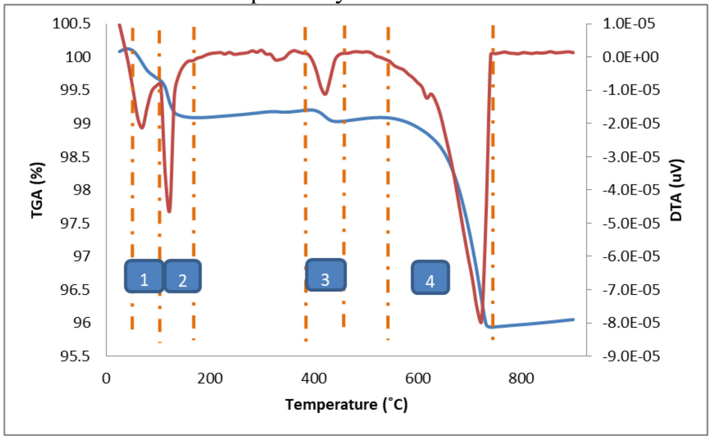


Figure 1. TGA-DTA of ordinary portland cement

The TGA result for BRHA used in this study is shown in Figure 2. It can be seen that there are 4 phases of weight loss in TGA for BRHA. First phase from 42 °C to 104 °C, second phase from 104 °C to 175 °C, third phase from 350 °C to 600 °C and lastly for the forth phase, the decomposition start from 600 °C to 731 °C. For the first phase, the weight loss is due to the elimination of moisture in the sample which is take place for 0.6289 %. As reported by Ramadhansyah et al. [5], the second phase, third phase and forth phase is the result of polysaccharide depolymerisation, dehydration of sugar and decomposition of silica in the amorphous form respectively. The weight loss of second, third and fourth phase is 0.6538 %, 0.38 % and 0.5077 % respectively.

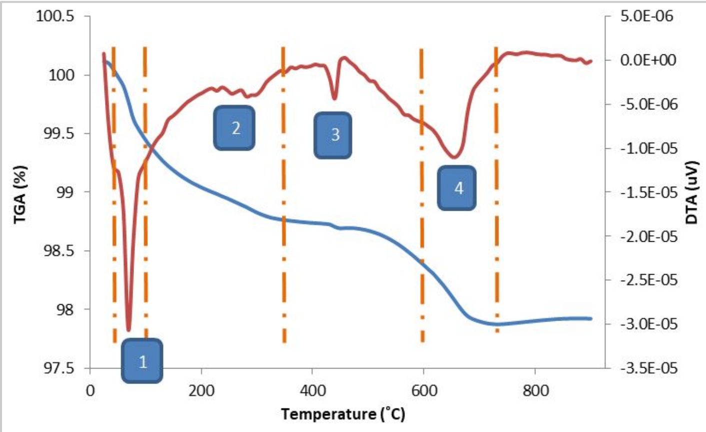


Figure 2. TGA-DTA of black rice husk ash

The result for x-ray diffraction (XRD) of ordinary Portland cement (OPC) is shown in Figure 3. The figure shows the phase mineralogy composition of element in OPC. The main components in OPC are Alite and Belite. It can be seen that, most of the intensity peak of the diffractogram are alite and belite. According to Poulsen et al. [15], the hydraulic properties of Portland cement are mainly related to alite and belite which are impure forms of the calcium silicates Ca_3SiO_5 (C_3S) and Ca_2SiO_4 (C_2S) respectively. Both of these silicates will react in cement hydration process to strengthen it structure.

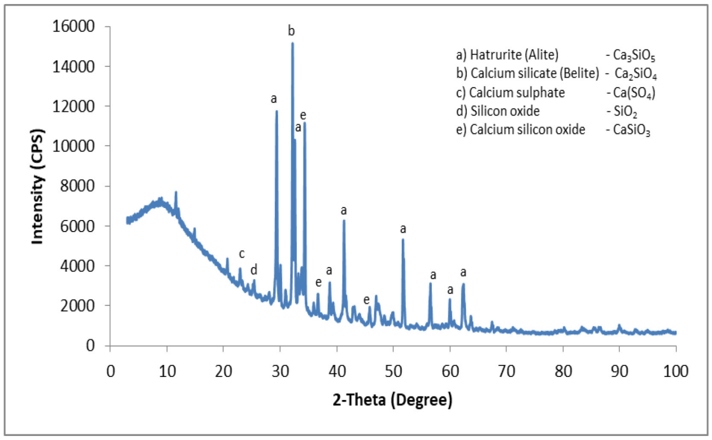


Figure 3. XRD of ordinary Portland cement

Figure 4 shows the XRD analysis result of the black rice husk ash. It shows that the highest intensity peak located at 21.84° 2θ is silicon oxide (SiO_2). It indicates that the major composition in black rice husk ash material is silica.

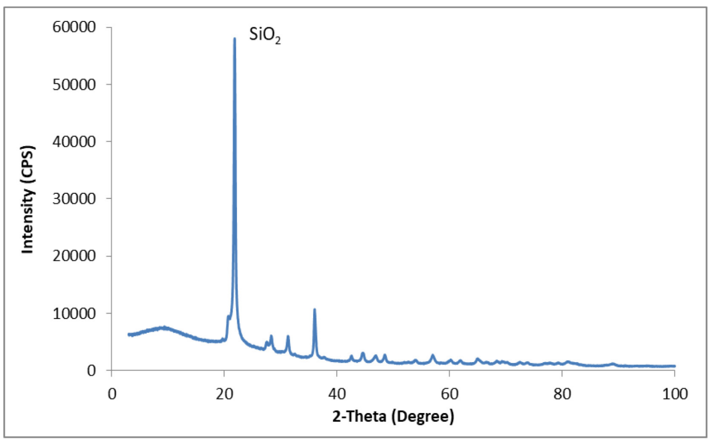
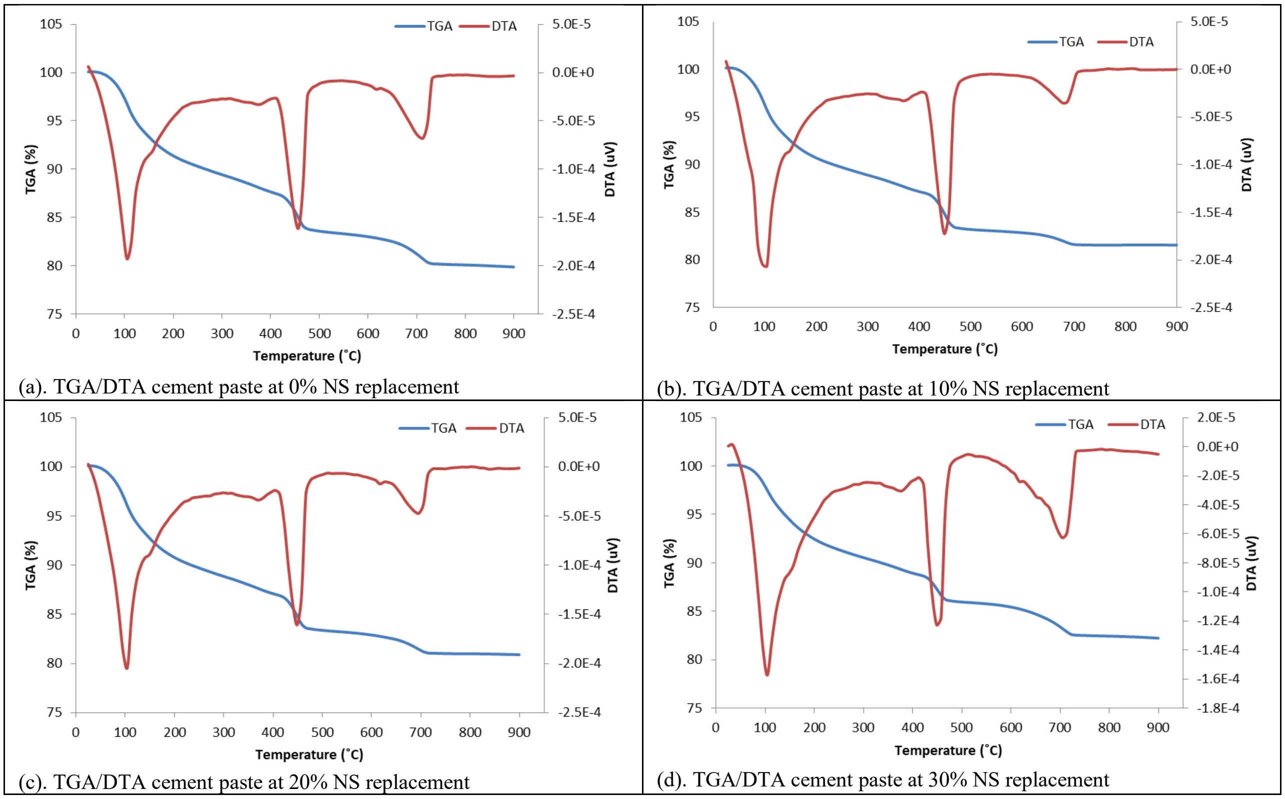


Figure 4. XRD of black rice husk ash

Thermal analysis

The results from TGA/DTA tests performed to the cement paste with various percentages of nano BRHA are shown in Figure 5. The decomposition of the components at high temperature was measured in this test. The main solid compounds of the hardened cement paste are $Ca(OH)_2$, anhydrous cement particles and calcium silicate hydrate (CSH) gel [16]. The weight loss percentage of the element decomposition was calculated based on DTA graph and it is shown in Table 3. The DTA curves illustrates that the hydrates present in a Portland cement are CSH gel and $Ca(OH)_2$ [17]. At temperature ranging from 30°C to 300°C, the decomposition of CSH gel contains in cement-blended-nano silica paste was occurred [18-21]. Then, the decomposition of $Ca(OH)_2$ occurred at 400°C to 500˚C [22]. Lastly, as reported by previous researchers, the decomposition of calcium carbonates ($CaCO_3$) take place at ranging from 600˚C to 750˚C [23]. It can be seen that the higher CSH gel percentage contained in cement paste with 10% of NS replacement. The results also indicated that the weight loss of $Ca(OH)_2$ is decreasing with increasing percentage of nano BRHA replacement. The addition of mineral mixtures in cement resulted in the formation of decreased amount of $Ca(OH)_2$ in the hydration product. On the other hand, the reaction between nano BRHA with $Ca(OH)_2$ resulting in increasing of CSH and decreasing in $Ca(OH)_2$. It can be said that the higher CSH gel percentage in cement paste is 10% of NS replacement.



(a). TGA/DTA cement paste at 0% NS replacement

(b). TGA/DTA cement paste at 10% NS replacement

(c). TGA/DTA cement paste at 20% NS replacement

(d). TGA/DTA cement paste at 30% NS replacement

Figure 5. Thermogravimetric and derivative analysis of cement paste with Nano BRHA

Table 3. Result of thermogravimetric analysis

Descriptions	NS (%)	0	10	20	30
Dehydrat./ decompos. of CSH gel, (%)	30°C -300°C	10.65	11.25	11.21	9.54
Decompos. of $Ca(OH)_2$, (%)	400°C -500°C	4.15	4.04	3.80	3.05
Decompos. of carbonate phase, (%)	600°C -750°C	2.85	1.30	1.94	2.94
Residue, (%)	≥ 750°C	82.35	83.41	83.05	84.47

X-ray diffraction analysis

To further study on the thermal analysis of the cement paste, the x-ray diffraction (XRD) analysis has been conducted. The percentage replacement used for cement paste in this analysis is 0%, 10%, 20% and 30%. The XRD analysis for all cement paste with different percentage of nano BRHA replacement is shown in Figure 6. It was observed that the element produced in each of the cement paste with different percentage of nano BRHA replacement is similar. The different is only in the intensity of the elements. The results also shows that the intensity of the $Ca(OH)_2$ decrease as nano BRHA decreasing form 0% to 30%. This is probably due to the consumption of $Ca(OH)_2$ during pozzolanic reaction between $Ca(OH)_2$ and nano BRHA. When the $Ca(OH)_2$ reacts with nano BRHA, it will produce CSH and lower the amount of $Ca(OH)_2$. Based on Table 3, the cement paste with 0% nano BRHA replacement shows a higher peak of $Ca(OH)_2$ where other mix indicate lowest $Ca(OH)_2$. On the other hand, the CSH intensity at 0% and 10% nano BRHA replacement shows almost similar peak in comparison to other mixture. However, CSH intensity at 20% and 30% replacement is decreases as increasing nano BRHA content. The

CSH is one of the elements to strengthen the cement paste or concrete. The increasing amount of CSH was increase the strength of the hardened cement paste.

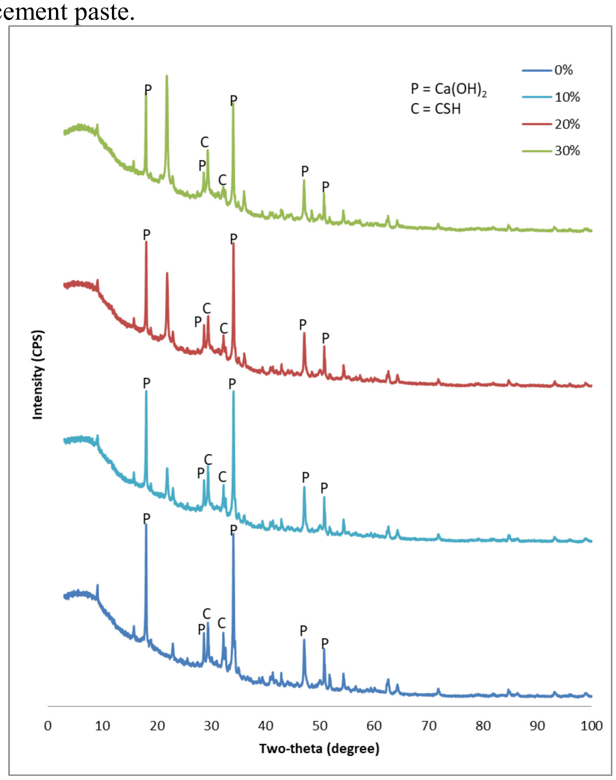


Figure 6. XRD of cement paste at various percentage of nano BRHA replacement

CONCLUSIONS

In the present study, the effect of cement paste containing different percentage of nano BRHA has been studied using TGA/DTA and XRD. It can be conclude that the cement paste containing 10% nano BRHA shows a better pozzolanic reaction due to the highest weight loss of CSH. The XRD reveal the decreasing of $Ca(OH)_2$ with increasing of nano BRHA replacement and supported the result from TGA.

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