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Thermal Analysis and Pozzolanic Index of Rice Husk Ash at Different Grinding Time

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Abstract

Thermo-gravimetric and differential thermo-gravimetric analyses were performance to study the effect of temperature on the mineralogical compositions of rice husk ash subjected to different grinding time. Eight rice husk ashes with different grinding time, i.e. coarse original rice husk ash (RHA0), RHA1, RHA2, RHA3, RHA4, RHA5, RHA6, and RHA7 were used for the study. The TGA/ DTA analysis and X-ray fluorescence (XRF) was used through this investigation. On the other hand, the pozzolanic activity index of the RHA was assessed in accordance with ASTM C 311-11a. From the experiment, it was found that the greater the weight loss, the less the crystallinity of the RHA. In addition, there are no significant differences in chemical compositions of the rice husk ashes with different grinding time. Furthermore, when the grinding time increased from 1 hour and 30 minutes to 5 hours, there was a significant drop in the pozzolanic index.

Keywords: Rice husk ash, grinding, thermal analysis, X-ray fluorescence, pozzolanic index;

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1. Introduction

The burning of rice husk under controlled temperature produces a highly reactive RHA [1]. Reactivity is enhanced by increasing the fineness of the reacting materials [2]. Prasad et al. [3] reported that the reactivity of RHA is attributed to its high content of amorphous silica and its porous nature. According to Rukzon and Chindaprasirt [4], the properly burnt and ground RHA are also factors affecting the blended cement of concrete. In addition, Chandrasekar et al. [5] stated that RHA, being porous in nature, has an extremely high surface area while its average size remains fairly high. In addition, thermoanalytical techniques, including differential thermal analysis and derivative thermogravimetry, have all been used successfully over the years [6, 7]. A number of thermal, mechanical and chemical methods have been used to activate the reactive potential of pozzolanic materials such as RHA, FA, POFA, etc. [8]. Most of the previous studies focused on the effect of the RHA grinding of cement paste, concrete, or mortar. However, very limited information is available on the thermal effects of RHA grinding. Therefore, in this investigation, the results of RHA grinding time on the thermal analysis are presented. The knowledge in terms of thermogravimetric analysis (TGA), derivative thermogravimetric (DTA), chemical analysis, and pozzolanic activity index would be beneficial for understanding the mechanisms, as well as for future applications of these materials.

2. Materials

Rice husk samples were collected from Nibong Tebal district of Penang (Malaysia). The rice husk samples were prepared by burning at 700°C for six hours in Gas Furnace with a heating rate of 10°C/min. After the burning process was completed, the ash was left to cool inside the furnace and removed on the following day as shown in Fig. 1. The burned ash will then be taken through grinding process and it was executed using the laboratory ball mill with porcelain balls [9]. Ball mills are advantageous because they operate in a simple manner: any hollow cylindrical or conical container can be rotated on the mill at different speeds [10]. The rice husk ash was ground into eight lots; RHA0: the original rice husk ash, RHA1: the rice husk ash was obtained using ball mill grinding until 30 minutes, RHA2: grinding until 60 minutes; RHA3: grinding until 90 minutes; RHA4: grinding until 120 minutes; RHA5: grinding until 180 minutes; RHA6: grinding until 240 minutes and RHA7: grinding until 300 minutes.

Fig. 1. Rice husk ash in Gas Furnace before (left) and after burning (right)

3. Methods

3.1. Thermal Analysis

Generally, DTA locates the ranges corresponding to thermal decompositions of different phases in specimen, while TGA simultaneously measures weight loss due to decompositions. However, a change in TGA slope is reflected as a marked peak in DTA [7]. In this investigation, thermogravimetric analysis and differential thermal analysis were applied in order to observe the reactions taking place during the thermal treatment of the samples. The analysis was carried out using Shimadzu model TA-60WS. At the specified testing, a total of 20 - 25 mg of the samples were taken in a platinum pan and heated in nitrogen atmosphere at a temperature range between 20°C to 1200°C with controlled heating rate 10°C/min. On the other hand, the pozzolanic activity index of the 15% RHA blended cement concrete was evaluated in accordance with ASTM C 311-11a [11].

3.2. Fluorescence (XRF)

The chemical compositions of RHA were determined by X-ray fluorescence (XRF). In XRF test, a 10 g sample in dried powder form (passing a 45 µm sieve) was compacted by pressing the sample into a 35 mm diameter pellet at a load of 20 tonnes for 10 seconds. The samples were placed into the platinum gold-fusion crucible. The crucible was then placed in the furnace using platinum-tipped tongs at a temperature of 1100 °C, and left to fuse. After the sample was prepared, the sample was characterized using RIGAKU RIX3000 wavelength XRF.

4. Results and Discussions

4.1. Thermogravimetric Analysis (TGA) and Derivative Thermogravimetric Analysis (DTA)

The results of the thermogravimetric (TGA) and differential thermogravimetric (DTA) analyses obtained at 10^{o}C/min for RHA samples at different grinding time are graphically presented in Fig. 2 (a-b). Generally, the test results show that the thermogravimetric curves decreased with increasing temperature. This is due to the carbonisation and decarbonation of rice husk ash. Carbonization is the decomposition of rice husks at temperatures greater than 300°C; this releases combustible gas and tar. Decarbonation is the combustion of fixed carbon in the rice husk char at higher temperatures in the presence of oxygen [12].

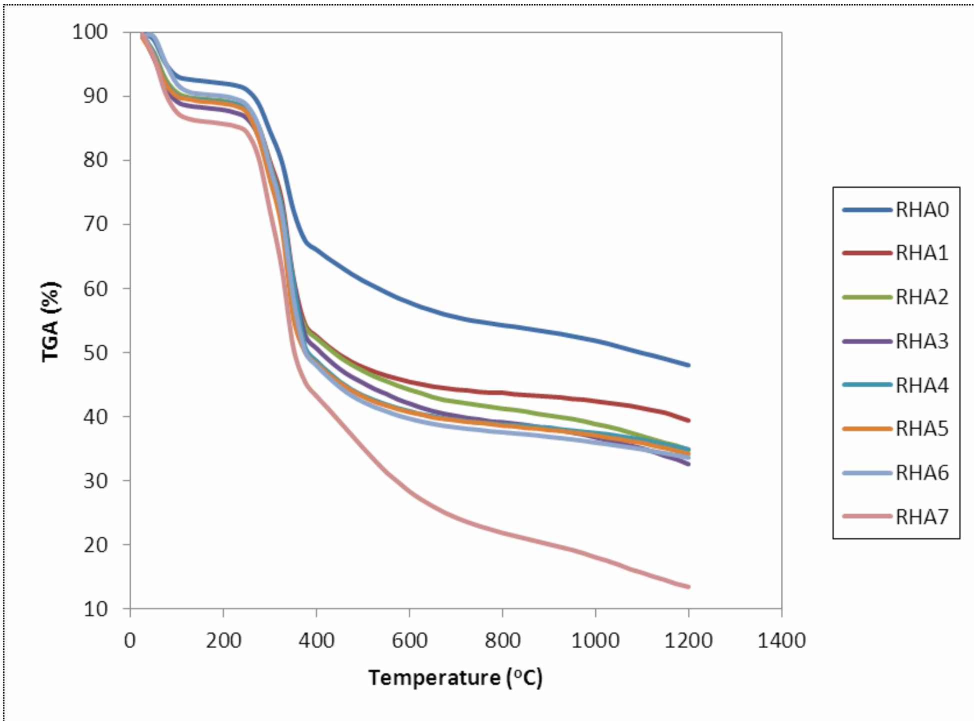

Fig. 2(a). TGA curves of rice husk ash fired to various grinding times

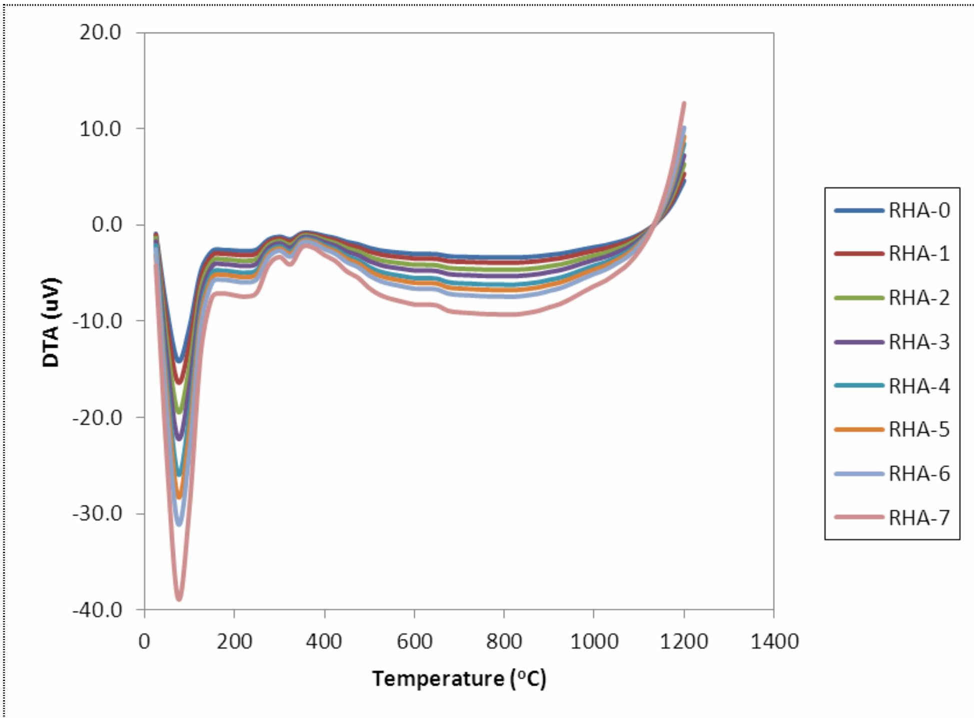

Fig. 2(b). DTA curves of rice husk ash fired to various grinding times

4.2. Relationship between TGA/DTA, temperature and weight loss

A typical graphical illustration of the relationship between TGA/DTA curves and temperature is demonstrated in Fig. 3. As reflected in Fig. 3, the reference specimen corresponds to original rice husk ash (RHA0). Furthermore, The TGA/DTA patterns of the specimen exhibit four endothermic peaks. The first endothermic peak, located between 100 and 350°C, is the result of polysaccharide depolymerization. Hwang and Wu [13] reported that burning RHA below 400°C depolymerizes polysaccharides. The second major endothermic peak, observed at 350 to 600°C, corresponds to the dehydration of sugar [13]. The third endothermic peak located in the range of 650°C to 850°C corresponds to silica in the amorphous form. Similar results were also reported by several researchers [13, 14]. The last endothermic peak detected between 900°C to 1200°C, is attributed to the crystallization of tridymite and cristobalite. Shinohara and Kohyama [15] stated that RHA heated at 900°C to 1200°C contain 62% cristobalite and 17% tridymite. From Fig. 3, several parameters, such as the temperature at different grinding time, and the mass loss in each process, may be evaluated. The weight loss for each sample in each temperature and grinding time are tabulated in Table 1. Each reaction of RHA corresponds to the previous heat treatment. The peak intensity for reaction 3 (temperature range of 650–850°C) decreases gradually compared with reaction (1), (2), and (4), respectively. These indicate that the reaction clearly can be used as an indicator to determine the temperature history of RHA. Conversely, for the sample heated at 900 to 1200°C, the increase in crystallization is correlated to higher temperature. Based on Table 1, when RHA are heated from 100 to 600°C, the organic matter like cellulose, lignin etc. decomposes into carbon. Therefore, a large number of silica formed was observed in the heated of RHA at 600 and 700°C. Further increase in temperature (800–1200°C) causes of crystallized. This indicated that the greater the weight loss, the less the crystallinity of the RHA.

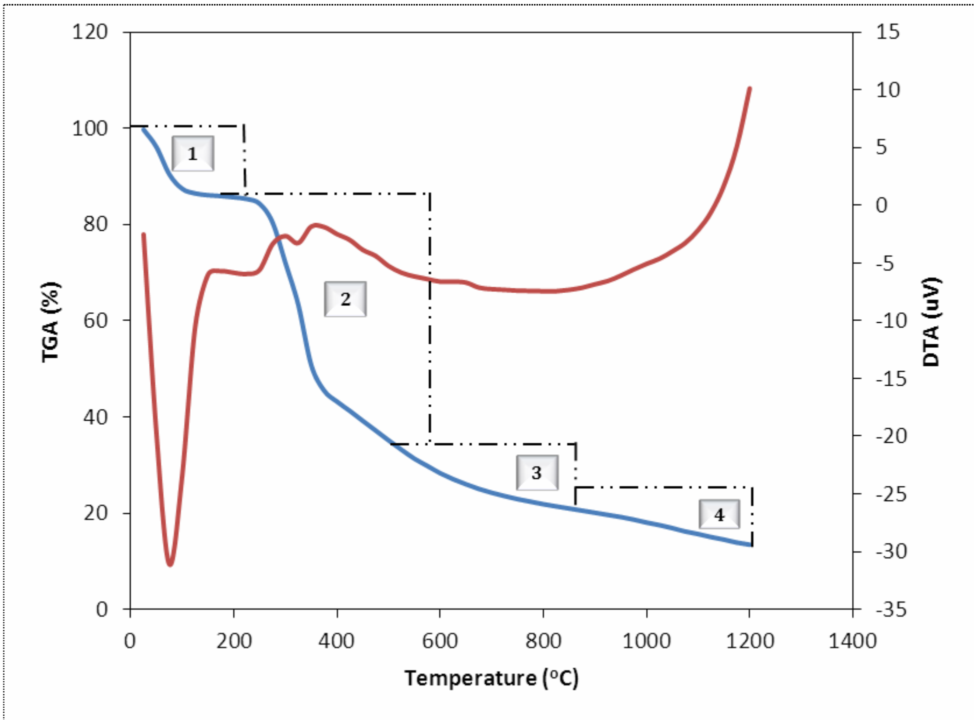

Fig. 3. TGA/DTA curves of rice husk ash fired to various temperature regimes

Table 1. Loss of weight of RHA at different temperature and grinding time

Temperature range (°C)	Loss of weight (%)							
	RHA0	RHA1	RHA2	RHA3	RHA4	RHA5	RHA6	RHA7
100 – 350	10.61	11.11	10.84	10.63	12.08	10.55	13.89	10.34
350 – 600	49.38	48.20	49.51	43.56	45.36	42.19	42.83	47.28
650 – 850	3.28	3.03	2.36	6.05	4.42	2.82	8.06	2.72
900 – 1200	3.70	4.10	4.01	6.06	4.68	5.00	12.27	3.11

4.3. Chemical compositions

The chemical compositions of the rice husk ash were analyzed using an X-ray Fluorescence spectrometer. The XRF determines the oxide compounds of the OPC and RHA such as calcium oxide (CaO), silica (SiO_2), alumina (Al_2O_3) and iron oxide (Fe_2O_3). Furthermore, minor compounds like MgO, SO_3, K_2O and Na_2O were also determined. However, other compositions found in this research are due to geographical factors, year of harvest, sample preparation, and equipment used. On the other hand, unburnt carbon existing in RHA was calculated by Loss on Ignition (LOI). The chemical compositions of rice husk ash subjected to different grinding time are tabulated in Table 2. According to the chemical compositions, the RHA is considered as silica-rich materials contain mainly of silica content (SiO_2). It shows that RHA consists in the range of 92.08 to 93.10% SiO_2, and contains 3.20 to 4.89 % loss on ignition which is an indication of its carbon content. The amount of CaO of the rice husk ash at different grinding time varied between 0.38 and 0.60%. Conversely, RHA used in this study could also be classified as class N as prescribed in ASTM C618–08a [16], where the total sum of SiO_2+AL_2O_3+Fe_2O_3 is more than the minimum requirement stated by ASTM C618 [16]. Lastly, there are no significant differences in chemical compositions of the rice husk ashes with different grinding time [9].

Table 2. Chemical compositions of RHA at various grinding time

Oxides	RHA0	RHA1	RHA2	RHA3	RHA4	RHA5	RHA6	RHA7
SiO_2	93.0	93.10	92.70	92.99	92.08	93.0	93.07	92.90
Al_2O_3	0.20	0.25	0.10	0.23	0.14	0.26	0.30	0.18
Fe_2O_3	0.13	0.23	0.10	0.26	0.75	0.15	0.20	0.43
CaO	0.49	0.42	0.40	0.41	0.39	0.38	0.60	0.41
MgO	0.73	0.85	0.90	0.73	0.65	0.50	0.73	0.35

Na_2O	0.02	0.02	0.15	0.02	0.15	0.05	0.20	0.02
K_2O	1.30	1.35	1.60	1.35	1.70	1.20	1.20	0.72
SO_3	0.15	0.15	0.50	0.08	0.09	0.10	0.20	0.10
LOI	3.98	3.63	3.55	3.93	4.05	4.36	3.50	4.89

4.4. Pozzolanic activity index of the resulting material

As prescribed by ASTM C 311-11a [11], pozzolanic activity index was evaluated by comparing the compressive strength of two concrete samples, one consisting of a standard mix of Portland cement and sand (reference), and the other having 15% of RHA replacement with superplasticizer. According to ASTM C 311-11a [11], the pozzolanic activity index of a pozzolan containing concrete after 28 days of curing should exceed that for the reference concrete by 75%. In this investigation, the pozzolanic activity indices of rice husk ash cement concrete at various grinding time are calculated and demonstrated in Fig. 4. The highest pozzolanic indices were obtained using the rice husk ash that was subjected to 90 minutes of grinding time and these indexes are 31.94% and 38.25% higher than those of unground rice husk ash at 7 and 28 days, respectively. A pozzolanic activity index of 10.61% was achieved at the age of 56 days when the rice husk ash median particle size was reduced from 17.96 to 6.65µm. When the grinding time was increased from 1 hour and 30 minutes to 5 hours, there was a significant drop in the pozzolanic activity indices at 7 days. However, at 28 days, the indices are not affected significantly. The lower values of the 7 and 28-days pozzolanic activity index for the rice husk ash after 5 hours-grinding may be due to the high specific surface of the rice husk ash.

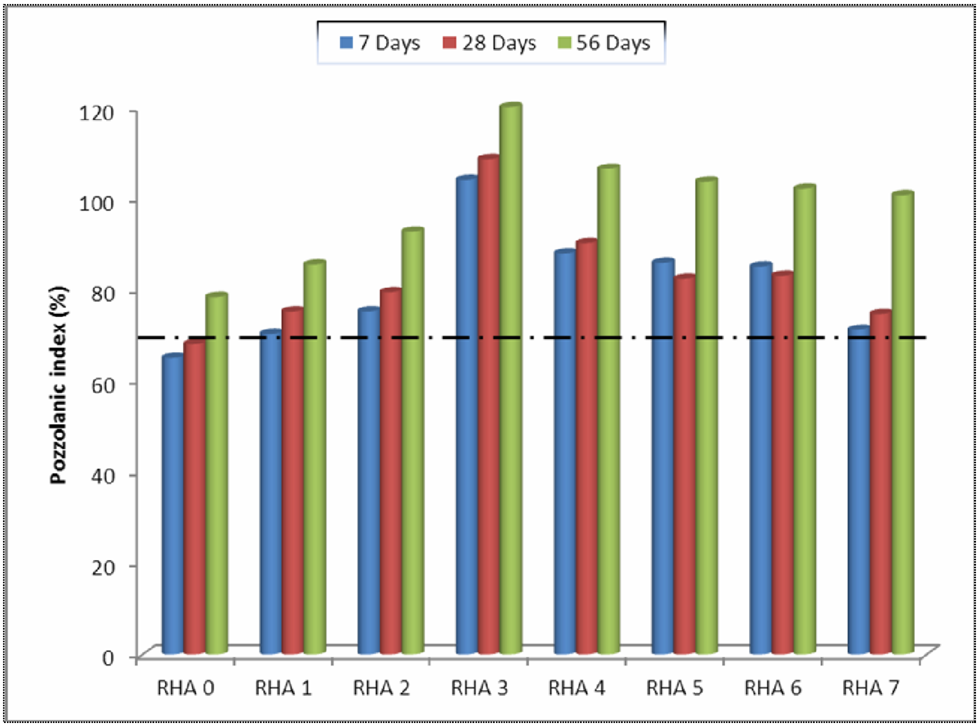

Fig. 4. Pozzolanic activity index of the rice husk ash cement concrete at different grinding time

5. Conclusion

a. Thermal analysis studies reveal that the greater the weight loss, the less the crystallinity of the RHA.
b. There are no significant differences in chemical compositions of the rice husk ashes with different grinding time.
c. The pozzolanic activity index was obtained using the rice husk ash when subjected to 90 minutes of grinding time. On the other hand, when the grinding time increased from 1 hour and 30 minutes to 5 hours, there was a significant drop in the pozzolanic index.

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