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# Thermogravimetric analysis on rice husk ashes-based geopolymer paste

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**Abstract**. Geopolymer is a material produced from the reaction of rice husk ashes and alkaline solution. These rice husk ashes are not widely used yet due to unstable chemical content than fly ashes. However, the high Si content in rice husk ashes are believed to contribute for better thermal properties such as in coating application. This paper will discuss the studies done to rice husk ashes geopolymer in aspects of thermal stability via conducting thermogravimetric analysis. Thermogravimetric analysis is carried out to determine the optimum temperature for each sample. This work shows the reactions of geopolymer paste with different molarity of NaOH solutions.

## 1. Introduction

Geopolymer is a binding material produced from reaction between rice husk ash (RHA) and alkaline activator (AA) solution. RHA is known as a cheap raw product material with good material properties that can be utilized for many applications [1]. Apart from that, RHA is also known as one of the most available wasted biomass resources. Due to its low cost price and biodegradable characteristic, RHA has become one of the most potential materials to be applied for engineering application. Combination of different alkaline activators such as sodium silicate ($Na_2SiO_3$) and sodium hydroxide (NaOH) can create synergistic effects and highly potential in dissolving the rice husk ash to produce a geopolymer binder [2-3]. Geopolymers have been known to have high mechanical properties, low shrinkage, low permeability and good thermal properties [2-4]. To investigate a process that involves many potential factors to obtain optimum result is tiresome and time consuming, hence works from other researchers are referred to. Many have agreed that the three most influencing factors in geopolymer materials are ratio solid to liquid, ratio Na2SiO3 to NaOH and NaOH molarity [5-8].

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The objective of this study is to analyze the important factors that mostly control the weight loss of RHA based geopolymer material. Furthermore, to assist in clarifying the effects of each factor on the material properties, a testing called thermogravimetric analysis is done.

## 2. Experimental work

### *2.1. Preparation of material*

The material used is rice husk ash (RHA), which is grounded to a finer size of particles. Then RHA particles is sieved to the average size of 50 microns. Table 1 shows the sample designation based on the composition made for each sample. The chemical composition of the rice husk ash is tabulated in Table 2, which is obtained from the X-ray Flourescence (XRF) analysis. The chemical composition for sodium silicate ($Na_2SiO_3$) is comprised of 57.8% $SiO_2$, 11.9% $Na_2O$ and also 30.3% $H_2O$. Sodium hydroxide (NaOH) pellets with 98% purity are used after being diluted using distilled water to obtain 4M, 8M and 12M.

**Table 1.** List of samples

| Sample | Ratio RHA to AA | Ratio $Na_2SiO_3$ to NaOH | NaOH Molarity |
|---|---|---|---|
| S01 | 1.0 | 0.5 | 4 |
| S02 | 1.5 | 0.5 | 4 |
| S03 | 1.0 | 2.5 | 8 |
| S04 | 1.5 | 0.5 | 8 |
| S05 | 1.0 | 0.5 | 12 |
| S06 | 1.5 | 0.5 | 12 |

**Table 2.** Chemical composition of rice husk ash

| Compound | Concentration Unit (%) |
|---|---|
| $Al_2O_3$ | 3.00 |
| $SiO_2$ | 87.40 |
| $K_2O$ | 0.49 |
| CaO | 1.40 |
| $Cr_2O_3$ | 0.27 |
| MnO | 0.19 |
| $Fe_2O_3$ | 1.49 |
| NiO | 0.07 |

### *2.2. Mixing process*

Initially, the alkaline activator (AA) solution is prepared by combination between NaOH and $Na_2SiO_3$. The preparation of geopolymer solution is done by mixing the RHA particles with the designated ratio of alkaline activator solution. The mixtures are then mechanically stirred for 5 to 10 minutes until they are homogeneous. The homogeneous mixture of geopolymer solution are then cast onto moulds with the size of 70mm x 50mm x 12mm. Finally, samples are left cured at 75°C for 24 hours in the oven.

### *2.3. Thermogravimetric analysis*

The thermogravimetric analysis was done using Pyris 1 TGA instrument and samples were prepared according to the required standard. The samples were prepared to be weighed less than 5 mg cube. The instrument was set to heat from 30°C to 500°C at 20.00°C/min.

## 3. Results and discussion

This paper analyses the thermal behaviour on geopolymer samples and the influencing factors that are affecting the weight loss of the geopolymer samples. A well-combined sodium silicate and sodium hydroxide were used as the alkaline activator (AA). The reason of using these two solutions because they are cheaply available and conveniently to be used [6, 9-10]. There are a total of six samples that were thermally tested as shown in Table 3.

**Table 3.** Percentage of weight loss and mass ratio for all samples

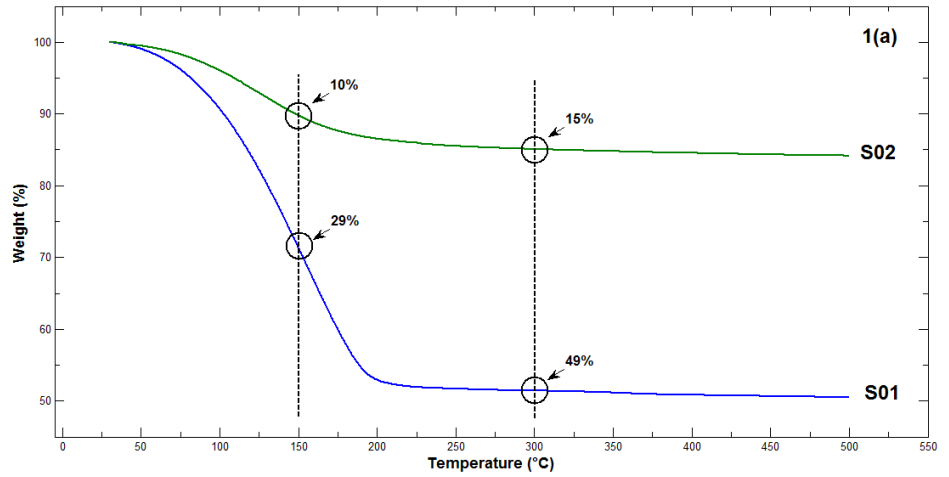
| Sample | Percentage of Weight Loss (%) | | Molar Ratio | | | |
|---|---|---|---|---|---|---|
| | 150°C | 300°C | $Na_2O$/ $SiO_2$ | $SiO_2$/ $Al_2O_3$ | $H_2O$/ $Na_2O$ | water/ solid |
| S01 | 29 | 49 | 0.105 | 60.169 | 20.736 | 1.933 |
| S02 | 10 | 15 | 0.074 | 56.791 | 20.719 | 1.408 |
| S03 | 32 | 64 | 0.108 | 72.593 | 10.669 | 1.027 |
| S04 | 19 | 49 | 0.044 | 56.791 | 30.543 | 1.251 |
| S05 | 25 | 54 | 0.206 | 60.169 | 8.853 | 1.489 |
| S06 | 18 | 38 | 0.146 | 56.791 | 8.853 | 1.109 |

In the TGA test, the weight loss was measured while the geopolymer samples were gradually exposed to the increasing temperature. Figure 1 represents the results of the TGA analysis on the percentage of weight loss of the geopolymer samples. It shows in overall that the decreased in weight starts before 100°C as recorded in the TGA analysis, which can be associated with the loss of evaporable water in the geopolymer as claimed by Al Bakri *et al.* [7]. Furthermore, all the geopolymer samples stabilized the weight loss between 200°C to 500°C.

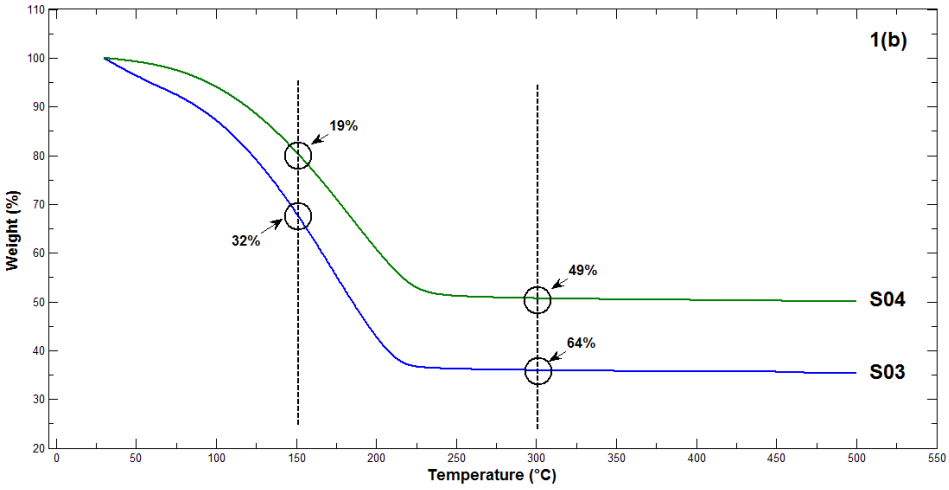
Meanwhile Figure 2 shows the pattern of degradation of the RHA based geopolymer samples are similar, which means that the samples start to degrade before they reach 100°C. The decomposition of the RHA based geopolymer samples took place during 100°C to 250°C. The decomposition process of the samples stop as the temperature when higher than 250°C.

### *3.1. Effect of ratio solid to liquid and NaOH molarity*

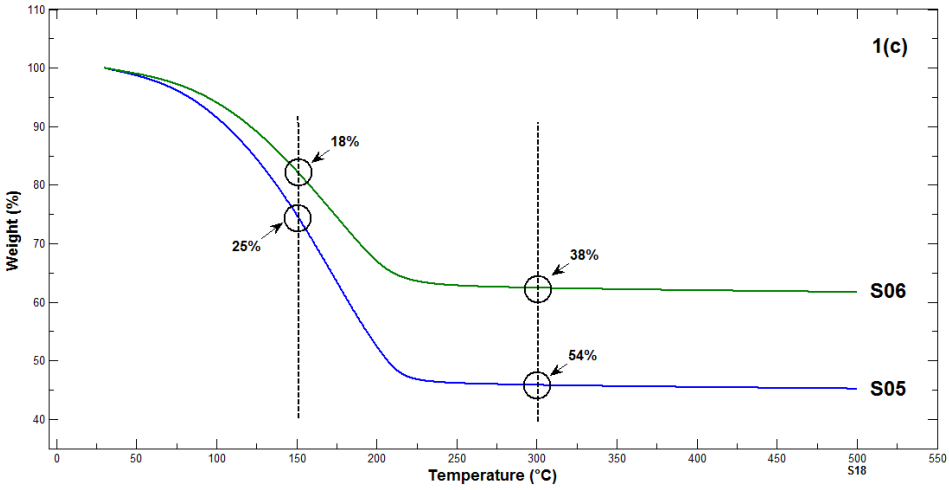
According to the Figure 1(a), which is 4M for both samples, at 150°C, S01 losses 29% and S02 losses only 10%. As the temperature goes to 300°C, S01 sample losses its weight for about 49% while for S02 sample, the weight loss is about 15%, which is increased by 5% only. Furthermore, in Figure 1(b) for 8M samples, at 150°C: S03 losses 32% and S04 losses 19% and at 300°C: S03 losses 64% and S04 losses 49%. However, the increment trend for both samples to lose weight as the temperature went from 150°C to 300°C is similar. Lastly, Figure 1(c) is for 12M samples, at 150°C: S05 losses 25% and S06 losses 18% and at 300°C: S05 losses 54% and S06 losses 38%. For 12M samples, S05 has higher losses than S06 in weight as the temperature went higher.




(a)




(b)




(c)

**Figure 1.** TGA graphs for every molarity: (a) 4M of NaOH molarity, (b) 8M of NaOH molarity, (c) 12M of NaOH molarity

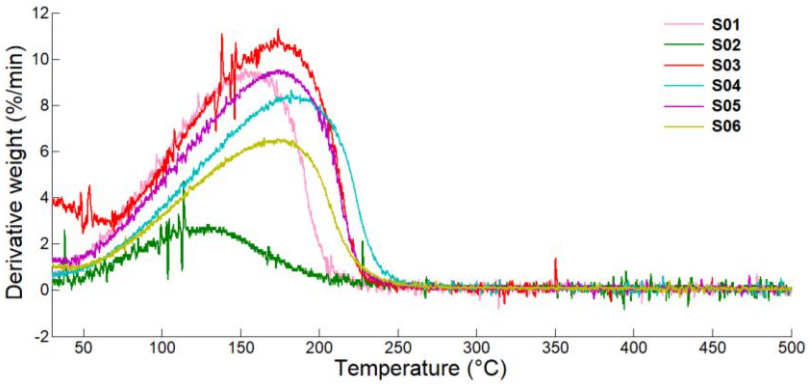



**Figure 2.** DTGA graphs for every molarities of RHA based geopolymer

The calculated ratio of solid to liquid is based on mass ratios (kg RHA per kg AA) as well as the ratio of $Na_2SiO_3$ to NaOH (kg $Na_2SiO_3$ per kg NaOH) for all samples. Ratio 1.0 of solid to liquid contents about 50% of RHA whereas in ratio 1.5 of solid to liquid contents 60% of RHA. That means these 1.5 ratio contents have more RHA than 1.0 ratio, thus the bonding structure of geopolymer more compact and less porous. S01, S03 and S01 are 1.0 ratio of solid to liquid whereas S02, S04 and S06 are 1.5 ratio of solid to liquid. During fabrication, geopolymer samples with the ratio 1.0 have better workability and flow ability during the mixing process because of its low RHA content than ratio 1.5. Geopolymer with low RHA content is much easier to stir since it has more water content than solid. Since the samples with ratio 1.0 solid to liquid have the highest water content, they probably require longer curing time or increase of curing temperature to release water binders and to reduce adsorbing more moisture [11-13].

### *3.2. Effect of ratio water/solid and Si/Al*

The molar ratio is a ratio of main compounds available in every sample. Factors such as high ratio of Si/Al and low water content samples were found to produce better thermal properties [1-2, 11]. S03 has highest ratio of Si/Al and lowest ratio water/solid. But according to the weight loss graph, S03 has the most highest in weight loss about 32% from 150°C to 300°C than other samples. For instance, S02 and S04 have higher ratio water/solid than S03 and they have similar value of ratio Si/Al that is less than S03. However, some researchers have claimed that the ratio of water/solid, ranging between 1.35 and 1.50, shows better results but it has low workability and caused inhomogeneous geopolymer [1].

## 4. Conclusion

This paper discusses the thermal behaviour for the RHA geopolymer samples, which is also influenced by the Si/Al content. Sample S03 has the lowest ratio water/solid and highest ratio $SiO_2/Al_2O_3$, thus is potentially a better thermal stability RHA geopolymer. Despite that, S03 has the highest weight loss during thermal analysis compared to S02 that is able to maintain 75% of its weight. Moreover, the ratio water/solid for S02 is still in the range of 1.30 and 1.50 that researchers have claimed to be the better workability geopolymer [1, 15]. Still, all geopolymer samples in this study show good thermal stability because the amount of Si/Al in every sample is high.

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