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# Optimization of RHA-Based Zeolite Phase Using Hydrothermal Method for Adsorption Application

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**Abstract.** Rice husk was used as a silicate source to produce RHA-based zeolites through hydrothermal method. The synthesis of zeolite undergone different types and concentrations of alkaline solutions which were 3M and 5M of sodium hydroxide (NaOH) and 5M of potassium hydroxide (KOH), respectively. The rice husk ash and resulted zeolites were then being characterized by X-ray fluorescence (XRF), Brunauer-Emmett-Teller (BET), scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR) analyses. Analyses of XRF, SEM and BET results shown that RHA has high content of silica (83.90%) with irregular particles due to large surface area of 48.7 m<sup>2</sup>g<sup>-1</sup>. For FTIR analysis, RHA at band 1040.52 cm<sup>-1</sup> assigned to asymmetric Si-O-Si bond stretching in SiO<sub>4</sub> tetrahedron. The band at 794.71 cm<sup>-1</sup> was corresponded to the stretching vibrations quartz of Si-O-Si. Subsequently, the zeolites were applied as an adsorbent in sugarcane bagasse wastewater and tested for total suspended solid (TSS) and chemical oxygen demand (COD). The adsorption capacity of TSS, COD and colour by the previous synthesized zeolites was then observed experimentally through the effects of contact time. Results indicated that adsorption capacity for COD is 13% to 22% and ranged from 50% to 57% for TSS.

## 1. Introduction

Perlis is one of the leading producers of rice in Malaysia. It supplies more than half of the total paddy produced even though it covers only a quarter of the total paddy-planting land area in Malaysia. The process to obtain grain from paddy plant had produced abundant of rice husk as agricultural residues. Rice husk contains maximum amount of siliceous ash that used in various application such as power generation, water treatment, cement industry and fertilizer. Big fractions of rice husk were considered as waste, causing pollution to environment and disposal difficulties which one of the current major problems in Perlis state.

The burning of THs results in the formation of RHA with a content in silicon dioxide, SiO<sub>2</sub> that varies from 85% to 98% [1]. Among the various utilizations of RHA, there is a significant interest in its use in the preparation of zeolites due to the widespread industrial used. Zeolites are microporous crystalline solids that have well-defined structures. Mainly they comprise of silicon, aluminum and oxygen in their framework and cations, water or other molecules insides their pores. Zeolites known



with its unique porous structure so that it is used in a broad and various range of applications with an international market of millions tons per year. It is also extensively used to reduce colour and inorganic pollutants concentration from industrial wastewater as in adsorption and ion exchange [2].

The presence of suspended solids, high concentration of color and COD in the industrial wastewater are a major problem to many life forms. The treatment of this organic pollutant using precipitation, coagulation and flocculation are not always able to meet the limit of discharge standards. The advanced technologies able to meet the standards, but these are very complex and expensive. The formation of zeolites from RHA show special importance in wastewater for the removal of contaminants [3]. This is because of their molecular sieve properties and high thermal, mechanical and chemical stability [4].

Adsorption is one of the most effective physico-chemical removal processes for high concentrations of colour, suspended solid and COD in wastewater treatment [5]. Zeolite as adsorbent provides an attachment surface for microorganisms to bio-regenerate the adsorbent. The combinations of organic pollutants that exist in wastewater need adsorbents which have the ability to remove pollutants including organic species. It is well known that activated carbons are the most effective adsorbents for the removal of organic pollutants from the aqueous phase. Therefore, this type of adsorbent is widely applied as a commercial adsorbent in the treatment of wastewater.

## 2. Materials and method

### 2.1. Material

The raw rice husk was collected from Beras Nasional (BERNAS) paddy factory located in Simpang Ampat, Perlis. After collection and sieving the rice husk was washed with distilled water to remove muddy materials and impurities before oven-dried at 100°C for 2 hours. Rice husk ash was obtained from the combustion of rice husk at 700°C for 3 hours in furnace. The cooled RHA was then ground and passed through a sieve to obtain a volume mean diameter of 150 µm.

### 2.2. Method

Quantitative chemical analyses of RHA were accomplished by X-ray Fluorescence (XRF) using PAN analytical MiniPAL 4. The specific surface area and microspore volume of the samples were determined by Brunauer-Emmett-Teller (BET) method. Infrared spectra were recorded on a Spectrum Perkin Elmer 400 using the Attenuated Total Reflection (ATR) technique. In addition, the morphologies of the samples were observed by scanning electron microscope (SEM) with 20 kV after gold coating with  $\times 200$  and above of magnification. The samples for FTIR measurements were prepared in the range of 650 – 4000  $\text{cm}^{-1}$  to identify functional group of synthesized zeolites.

### 2.3. Material Preparation of RHA-based zeolite

The zeolite was synthesized using hydrothermal method modified from Bohra et al., (2013), Othman Ali et al., (2011) and Kordatos et al., (2008) [4,6]. 2 grams of RHA and aluminium sulfate,  $\text{Al}_2(\text{SO}_4)_3$  used as starting materials were individually dissolved in 30 mL distilled water containing 10 mL of different basic solution which were 3M and 5M of sodium hydroxide (NaOH) and 5M of potassium hydroxide (KOH). The mixture stirred for half an hour at 100 rpm and placed on hotplate at 80°C. Alkaline solutions is used in order to attack the silicon and aluminium contained in the starting materials and thus balanced the zeolite structure. Then, the solutions were sterilized in sealed autoclave at 124°C for 15 minutes with pressure of 0.1 MPa before centrifuged at 4500 rpm for 15 minutes in room temperature. After that, solid phase was washed with distilled water and oven dried at 110°C for 12 hours. The dry solid residue was then ground and sieved to obtain a volume mean diameter of 150 µm.

### 2.4. Adsorption

The raw wastewater was collected from sugarcane bagasse wastewater effluent for adsorption application in reducing TSS, COD concentrations and colour. The adsorption analysis started with 250 mL of conical flask were filled with 100 mL sugarcane molasses wastewater and 5 gram of zeolite

obtained at different types and concentration of alkali solution. The mixture placed on shaker at 150 rpm with contact time for 0.5, 1, 2, 3 and 4 hours at room temperature. The solution was filtrated and residual concentration of TSS, COD and colour removal were studied by evaluating the percentage of removal efficiency.

### 3. Results and Discussion Materials and method

#### 3.1. Characterozations of RHA

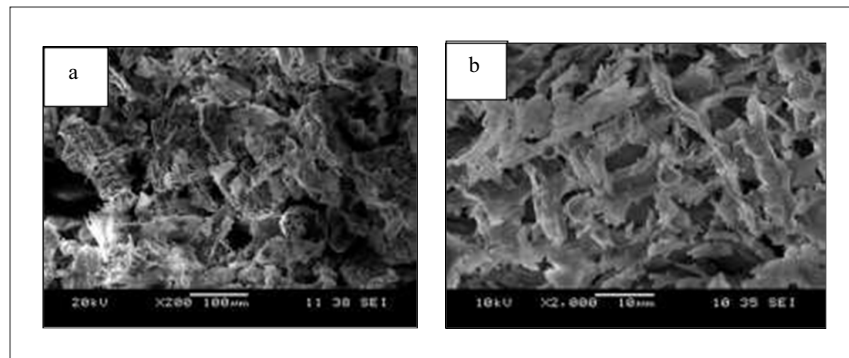
The physical properties of raw RHA were presented in table 1. The main component of RHA was 83.9 % of silica followed by of 8.82 % of potassium, 4.09 % of calcium, 0.82 % of manganese, 0.71 % of sulfur, 0.43 % of iron, 0.20 % of copper and 0.11 % of zinc. The surface area and micropore volume were 48.7 m<sup>2</sup> g<sup>-1</sup> and 1.61×10<sup>-3</sup> cm<sup>3</sup> g<sup>-1</sup>, respectively.

**Table 1.** Characteristics of RHA.

Characteristics	RHA
Chemical compositions (wt)	
Si	83.90
K	8.82
Ca	4.09
Mn	0.82
S	0.71
Fe	0.43
Cu	0.20
Zn	0.11
Specific surface area (m <sup>2</sup> g <sup>-1</sup> )	48.70
Micropore volume (cm <sup>3</sup> g <sup>-1</sup> )	1.61×10 <sup>-3</sup>

The results shown that combusted rice husk at 700°C has high of silica content and low content by some oxides in RHA because most of the impurities were eliminated during combustion. The result corresponded with Mohamed (2015) that reported the high yield of silica in ash sample was 89.00% [7]. Bohra et al. (2013) also analyzed that a major amount of silica in RHA obtained, when it leached with an acid treatment as it can remove more of impurities [4]. According to Chen et al. (2010), surface area of RHA increases with ashing temperature due to composition of organic compound [8]. While, RHA showed small in micropore volume due to large of carbon content in RHA.

While for the morphology of the combusted RHA was studied by SEM as shown in figure 1 by ×200 and ×2000 magnification. RHA combusted at 700°C for 3 hours represented that the RHA particles were very irregular with the remains of the porous cellular structure of rice husk. It also showed less-fractured, with a skeleton like internal structure and thicker cell walls, giving fewer pores. Thus, it is leading to a high surface area.



**Figure 1.** SEM photomicrographs of RHA (a) 200× (b) 2000×.

According to Abu, Yahya, & Neon (2016), the uneven and highly roughened when the RHA was not un-leached with acid [9]. In addition, it showed greater degree of roughness than those that have been leached with dilute acids, presumably due to the hydrolysis of some organic components by the acids.

### 3.2. Zeolite Characteristics

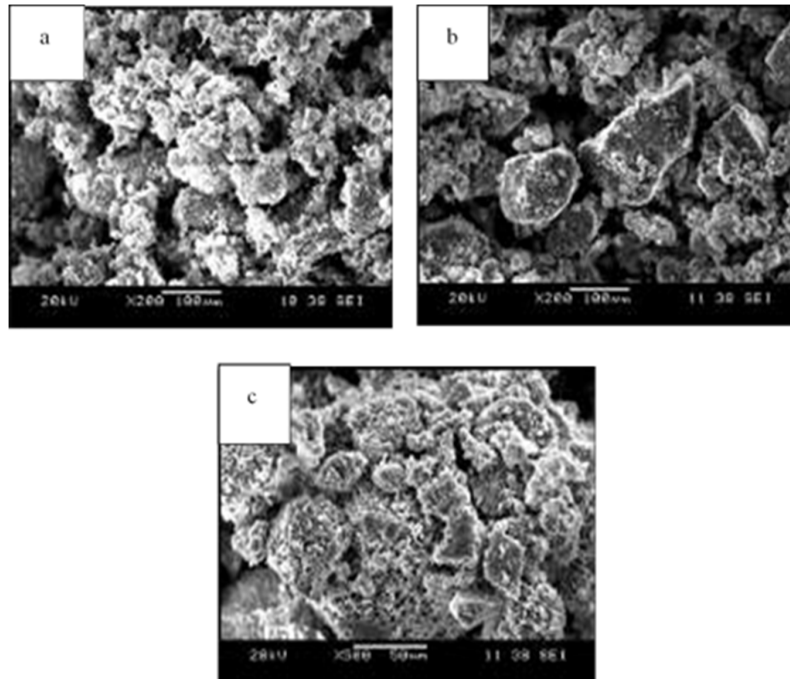
The chemical properties of zeolites formed are shown in table 2. Zeolite1 (Z1), Zeolite2 (Z2) and Zeolite3 (Z3) represented the zeolites obtained after dissolved in different type and concentrations of basic solutions which is 3M and 5M of NaOH, and 5M of KOH, respectively. The silica and alumina contents remains after dissolved in Z2 were 35.8 wt % and 11.0 wt % by weight which is lower than Z1 and Z3 with 69.0 % of Si and 15.0 % of Al and 48.7 % of Si and 11.0 % of Al, respectively.

From this results, 5M of NaOH was completely dissolved at high concentration when compared to 5M of KOH and 3M of NaOH. The Z1 showed high remaining of Si and Al than Z2 and Z3, largely due to the effects of low alkali concentration in leaching of both starting materials as Si and Al source.

**Table 2.** Characteristics of RHA-based zeolites.

Chemical Compositions (wt%)	Zeolite1 (Z1)	Zeolite2 (Z2)	Zeolite3 (Z3)
SiO <sub>2</sub>	69.0	35.8	48.7
Al <sub>2</sub> O <sub>3</sub>	15.0	11.0	11.0
SO <sub>3</sub>	8.55	3.65	25.7
K <sub>2</sub> O	3.46	47.5	3.99
MnO	0.32	0.34	0.63
Fe <sub>2</sub> O <sub>3</sub>	0.31	0.43	1.03

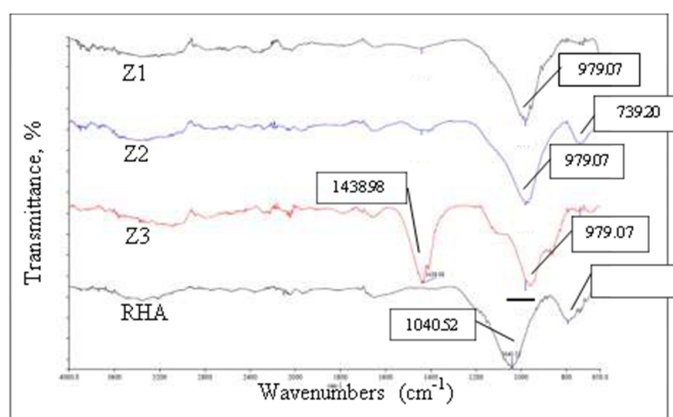
SEM photomicrographs of Z1, Z2 and Z3 morphologies were then compared, as shown in figure 2. The morphology of zeolite in figure 2(a) composed a various sizes of agglomerated particles with octahedral shapes. The particles of zeolite were very irregular with some pores in figure 2(b) compared to figure 2(a). The zeolite that dissolved with 5M KOH appears as compact fibrous aggregates with fibers of divergent radial arrangement as shown in figure 2(c).



**Figure 2.** SEM photomicrographs of zeolite synthesized. (a) Z1, (b) Z2 and (c) Z3.

From SEM results, the concentration and type of alkali used to attack Si and Al affected the morphology and particle size of zeolite formed. This typical of morphology for zeolites were shown by other researchers as well [10]. The increase in basicity of the reaction mixture, the particles became more irregular as spotted in figure 2(b) [11]. While in figure 2(a) shown lower concentration of alkalinity formed an octahedral morphology [12].

The FTIR spectroscopy used to examine the functional group of the RHA and synthesized products as shown in figure 3. For RHA, the band at  $1040.52\text{ cm}^{-1}$  represented asymmetric siloxane bond, Si-O-Si stretching in  $\text{SiO}_4$  tetrahedron. The band at  $794.71\text{ cm}^{-1}$  was corresponded to the stretching vibrations quartz of Si-O-Si. The broad band in 5M of potassium hydroxide (KOH) solution shown significantly at  $1438.98\text{ cm}^{-1}$  was assigned to the asymmetric stretching vibration of Si-O or Al-O. The band at  $979.07\text{ cm}^{-1}$  for these three types and concentration of alkali solution corresponded to the internal asymmetric stretching vibration of Si-O-T where T is equal to Si or Al. While, the presence of adsorption modes at  $739.20\text{ cm}^{-1}$  was due to the external symmetric stretching of Si-O-T.



**Figure 3.** FTIR Spectra of synthesized products.

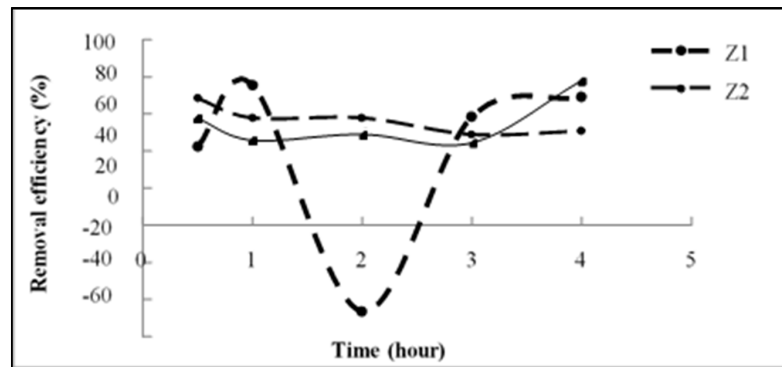
The FTIR spectra of RHA at 1040.52 cm<sup>-1</sup> corresponded to Othman Ali et al., (2011) that stated the signal band at 1090 cm<sup>-1</sup> was assigned to the asymmetric stretching vibrations of tetrahedral SiO<sub>4</sub> while at 794.71 cm<sup>-1</sup> nearest to 795 cm<sup>-1</sup> is due to symmetric stretching vibrations of the same units, SiO<sub>4</sub> [6]. In addition, Wang et al., (2018) also stated the bands at 1080, 1003 and 799 cm<sup>-1</sup> were assigned to symmetrical and asymmetrical stretching vibrations of Si-O-Si and Si-O-Al tetrahedra structure [13]. The peaks at 1044 cm<sup>-1</sup> and 796 cm<sup>-1</sup> attributes to O-Si-O stretching and bending vibrations of un-leached silica [9].

The band at 1441 cm<sup>-1</sup> indicated to asymmetric stretching vibration of Si-O or Al-O [12]. While, the absorption band at 1440 cm<sup>-1</sup> indicate the amount of amorphous material in the sample gradually decreases due to the progressive phase transformation of amorphous phase into crystalline phase [14]. For the zeolites synthesized at band 979.07 cm<sup>-1</sup> and 739.20 cm<sup>-1</sup> were corresponded to 991 cm<sup>-1</sup> and 747 cm<sup>-1</sup> that due to internal asymmetric and external symmetric stretching vibration of Si-O-Si or Si-O-Al.

### 3.3. Effects of contact time

The study on effect of removal efficiency of the experimental parameter with contact times, 0.5, 1, 2, 3 and 4 hours by three zeolites formed. It was observed that as soon as the contact time increases, the adsorption capacity of TSS and COD also increased. This is due to the process of organic matter that reacted with adsorbents inside the sample as it comes into contact.

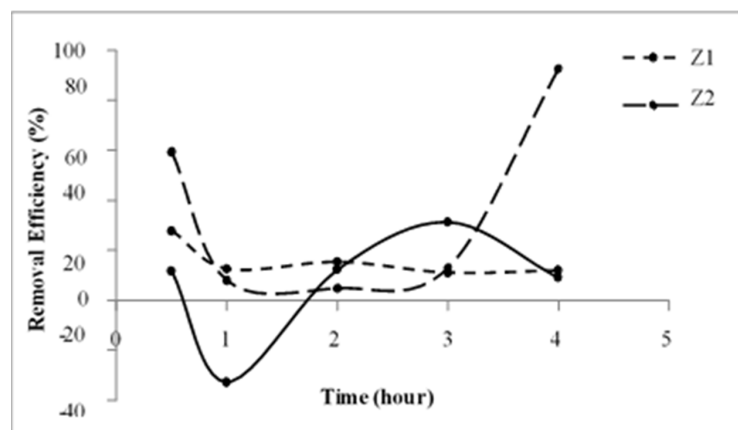
The removal efficiency of TSS at different contact time is given in figure 4. The figure 4 shows the performance of Z2 was 57 % better than 40 % of Z1 and 53 % of Z3 in 0.5, 1, 2, 3 and 4 hours, respectively. Hence, zeolite with high concentrations of NaOH was more effective in TSS removal. The average of removal efficiency was 50 % in reducing the amount of TSS in sugarcane bagasse wastewater.



**Figure 4.** Effect of contact time in TSS adsorption capacity.

The removal efficiency of TSS was 57% by natural zeolite for 6 hours contact time at 26°C. Delkash et al. (2015) in their study revealed that 82% of TSS was removed under optimized condition. They also concluded that zeolites with smaller particle size are remarkably more efficient to reduce TSS compared to the zeolite with coarser particle.

Figure 5 illustrates the percentage of COD removal by zeolite adsorbents. As shown in the figure 5, sugarcane bagasse wastewater with high COD concentration experiences a considerable decrease after being treated with the zeolites in adsorption method. The ability of Z2 to reduce COD concentration is 22% more than 13% of Z1 and 20% of Z3. In fact, zeolite at high concentration of sodium hydroxide has the negligible ability in the COD removal compared to lower concentrations. Results obtained in this study also aligned with Augusto & Santos (2011) whom discovered the removal for COD was in range of 13.64 - 27.61% lower than using the conventional activated carbon at 24.20 - 36.48%.



**Figure 5.** Effect of contact time in COD adsorption capacity.

#### 4. Conclusions

Synthesized of RHA-based zeolite using hydrothermal method was successfully prepared with rice husk ash and aluminium sulphate as starting materials. RHA with high silica content and large porosity was also proved to be effective adsorbent in wastewater treatment. Synthesis in different type and concentration of alkali was performed to affect the elemental composition, morphology and functional group of zeolites formed. Zeolite 2 that formed from the dissolving in 5M of NaOH show the increase in NaOH solution produced more irregular particles with some pores compared to Zeolite 1 and 3. The adsorption capacity of TSS and COD by synthesized zeolites was observed

experimentally through the effect of contact time. It shown decreased from the initial concentration, but have not great effect with the contact time, 0.5, 1, 2, 3 and 4 hours. Results indicated that it was able reduced 56% TSS and 19% COD concentrations from sugarcane bagasse wastewater.

### 5. Acknowledgement

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