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# Characterization of adsorbent derived from Coconut Husk and Silica (SiO<sub>2</sub>)

M Mariana<sup>1,\*</sup>, F Mulana<sup>1</sup>, S Sofyana<sup>1</sup>, N P Dian <sup>2</sup>, M R Lubis<sup>1</sup>

<sup>1</sup> Department of Chemical Engineering, Faculty of Engineering, Syiah Kuala University, Darussalam, Banda Aceh, Indonesia

<sup>2</sup> Undergraduate Student of Department of Chemical Engineering, Faculty of Engineering, Syiah Kuala University, Darussalam, Banda Aceh, Indonesia

E-mail: mariana\_hasyim@yahoo.com

**Abstract.** Activated carbon is one of the most commonly used adsorbents because of its high adsorption effectiveness. However, an adsorbent with one type of activated carbon causes a lacking of adsorption efficiency. Therefore, in this study we prepared activated carbon from two types of raw materials. The type of raw material is a combination of coconut husk and silica with several products of activated carbon that contained coconut husk and silica with mass ratio of 70:30, coconut husk with and without activation and pure silica (SiO<sub>2</sub>). In this study, raw materials were physically heated at 800 °C for 2 hours and chemically activated with H<sub>2</sub>SO<sub>4</sub> solution for 2 hours. FTIR analysis identified a chemical group of Si-O in adsorbent at 898,83 – 1442,74 cm<sup>-1</sup>. XRD analysis showed that the prepared activated carbon of combination coconut husk:silica; coconut husk with activation; coconut husk without activation and pure silica had silica (SiO<sub>2</sub>) composition of 83%; 75%; 62.4% and 63.2%, respectively. The water content in activated carbon of combination coconut:silica; coconut husk with activation; coconut husk without activation and pure silica (SiO<sub>2</sub>) was 4.41%; 3.87%; 3.21% and 2.71%, respectively and the ash content was 3.9%, 2.04%; 1.86% and 0.32% respectively. Both water content and ash content met Indonesian National Standard (SNI) No. 06-3730-1995 with the maximum of ash and water content was 10% and 15%.

## 1. Introduction

Environmental pollution occurred so far either because human activities or natural processes can change the state of the environment. These changes result in decreasing quality of the environment and cannot function properly. Most environmental pollution is caused by industrial waste. With the increase of industrial growth, the amount of waste generated either solid waste, liquid or gas also increased significantly. Liquid waste of tofu industry and batik industry contains various organic compounds such as ammonia and synthetic dyes. Methylene blue is one of the synthetic dyes contained in batik industrial liquid waste. The content of methylene blue in water that exceeds the permitted quality standards can cause human health problems in the form of nausea, vomiting, abdominal and chest pain, headaches, excessive sweating and hypertension, but it can also cause the death of aquatic biota [1,2].

\* Corresponding author : mariana\_hasyim@yahoo.com.



The nitrogen content in ammonia can cause toxic effect if the compound is in the form of nitrites [3,4]. Nitrogen from nitrite can produce methemoglobin that is a process of inhibiting the transport of oxygen in the bloodstream. According to Government Regulation No. 82 year 2001 on Water Quality Management and Water Pollution Control, the permitted ammonia content is 0.5 mg NH<sub>3</sub>/L and Regulation of Environmental Ministry of Indonesia No. 14 year 2013 also states that the amount of methylene blue allowed to be discharged into the environment is only 0.5 mg/L. Therefore, both types of waste before disposal into the environment need to be treated first. Various liquid wastes processing process has been applied, such as adsorption process using adsorbent. The advantages of adsorption process using adsorbent are cheap, easy in operation and environmental friendly [5,6].

Coconut husk as raw material combined with silica was used in this research to produce activated carbon. Coconut husk, an agricultural solid waste that has not been used optimally, contains lignin, cellulose and porous structures and through controlled combustion process coconut husk can be converted to ash which is a source of amorphous silica so it is suitable for use as an adsorbent. The ability of adsorption increases with the increase of silica content in the adsorbent. Silica (SiO<sub>2</sub>) is inert and has good adsorption and ion exchange properties, easily modified with certain chemical compounds [7]. To increase the silica content in the activated carbon product, some pure silica needs to be added in the preparation of the adsorbent to produce adsorbents with good adsorption capability and ion exchange. Various studies related to the addition of silica in a particular adsorbent have been made to increase the adsorbent reactivity. Silica and bentonite were utilized to produce adsorbents for adsorption of Fe metal ion in batik liquid waste [8]. In this study, we attempted to produce activated carbon by using two types of raw materials that are derived from the biomass of coconut husk and pure silica.

The purpose of this research is to produce the activated carbon from the combination of coconut husk and silica with certain mass ratio. In this initial study, the produced activated carbon was characterized before being used in the adsorption process of ammonia and methylene blue. The characterization was necessary to know the reactivity of the adsorbent by knowing the type of functional groups, the amount of silica, water content and ash content in the prepared activated carbon.

## 2. Methodology

### 2.1. Materials and tools

The materials used in this study were coconut husk and pure powder silica (SiO<sub>2</sub>) (Merck), H<sub>2</sub>SO<sub>4</sub> 98 v% (Merck) and aquades. While the tools used were desiccator, muffle furnace, pH meter, +80-100 mesh mechanical sieve, analytical balance, filter paper and electric oven.

### 2.2. Adsorbent activation and characterization

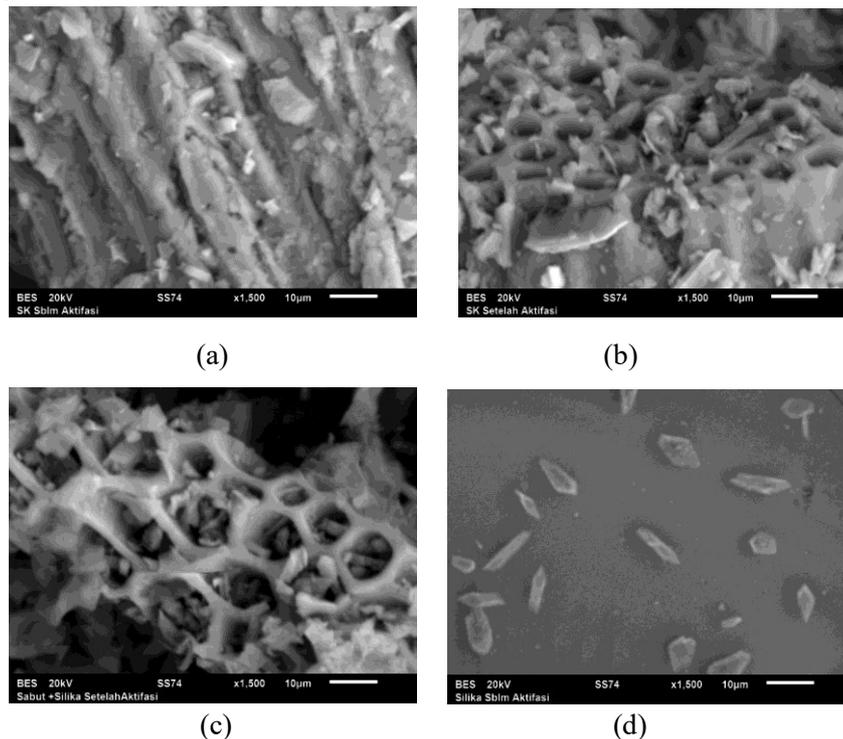
Coconut husk weighed as much as 10 g then was carried out the carbonization process using muffle furnace to form carbon [9]. The pure silica was then dissolved by mixing in water with a mass ratio of 10:1, then the activated charcoal from coconut husk and pure silica were mixed and stirred using a magnetic stirrer for 2 hours. After the stirring and filtration process, the wet activated charcoal was heated in the oven for 1 day to remove water content.

Dry activated charcoal was then immersed in 500 ml of sulfuric acid solution for 24 hours. After that it was filtered and rinsed with distilled water until reaching a neutral pH and followed by calcination process using muffle furnace at 800 °C for 2 hours [10]. After the carbonization process, the activated carbon was sieved with a size of +80-100 mesh and the fine activated carbon was stored in a desiccator. The characterization of samples was performed by XRD D-6000 (Shimadzu) to identified composition of adsorbent, SEM typed JSM 6510 LA (JEOL) to know morphology of adsorbent, and FTIR Prestise -21 (Shimadzu) to identified functional groups.

### 3. Results and Discussion

#### 3.1. Morphology of adsorbents

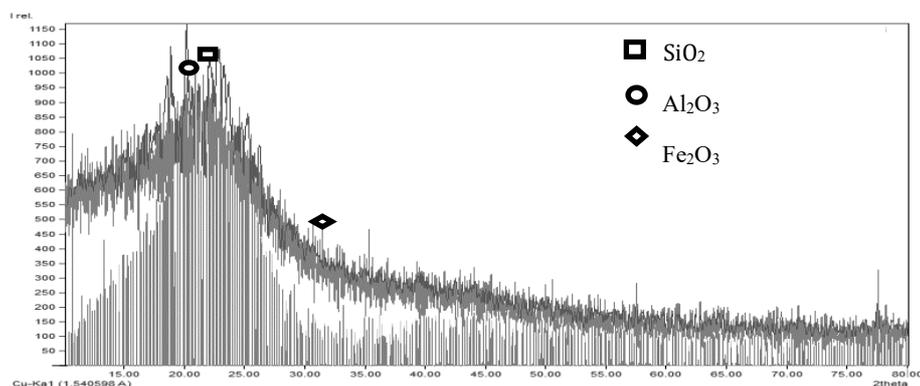
Figure 1(a) and 1(b) is SEM image of activated carbon from coconut husk with activation process and without activation process, respectively. While, figure 1(c) and 1(d) is SEM image of activated carbon activated carbon from coconut husk:silica with mass ratio of 70:30 and pure silica ( $\text{SiO}_2$ ), respectively. SEM results show that the activated carbon from combination of coconut husk:silica with mass ratio of 70:30 has larger pore than that of from coconut husk only and pure silica. The SEM results also showed the presence of impurities particles attached to activated carbon without activation process as seen in figure 1(a).

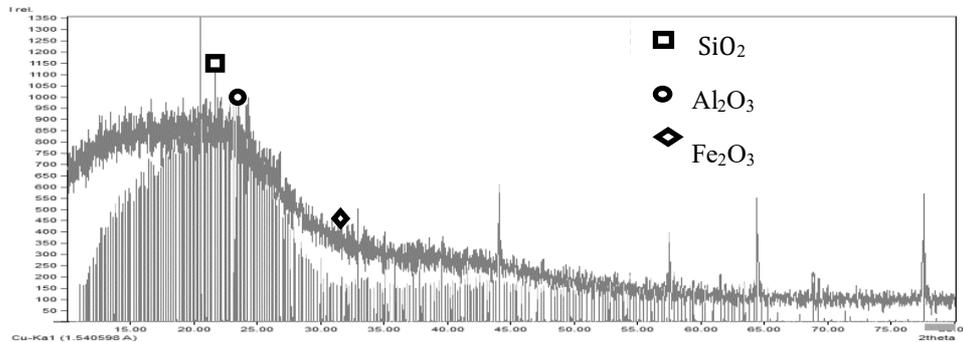
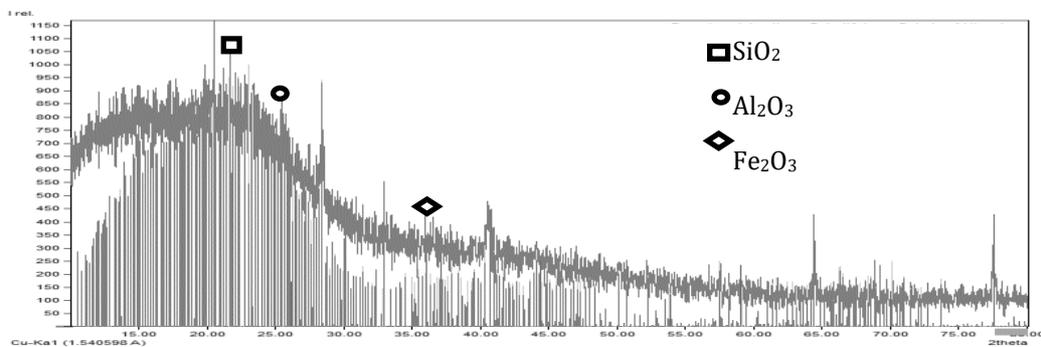
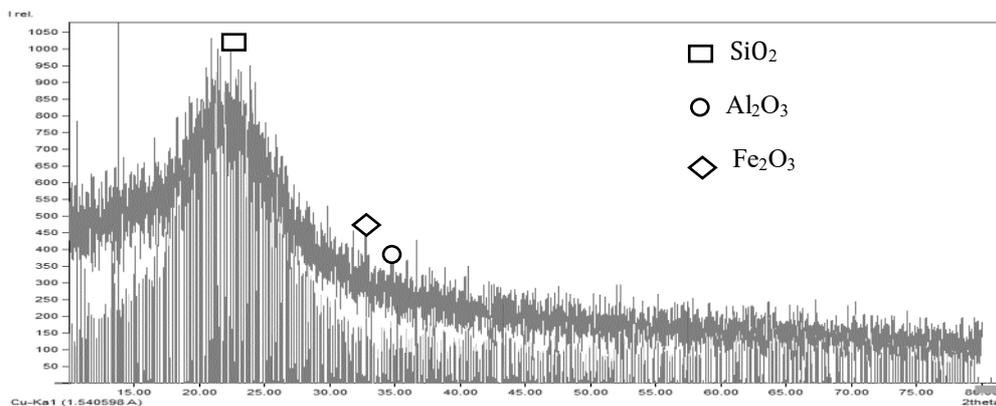


**Figure 1.** SEM image of activated carbon from coconut husk without activation process (a) coconut husk with activation process (b), coconut husk:silica with mass ratio of 70:30 (c), and pure silica (d). the highest magnification was 1500 X.

#### 3.2. Composition analysis using X-Ray Diffraction (XRD)

The XRD analysis results of four types of adsorbents are showed in figure 2 to 5.



**Figure 2.** XRD profile of activated carbon from coconut husk:silica with mass ratio of 70:30**Figure 3.** XRD profile of activated carbon from coconut husk without activation process**Figure 4.** XRD profile of activated carbon from coconut husk with activation process**Figure 5.** XRD profile of pure silica adsorbent

Figures 2 to 5 showed the XRD diffractogram of activated carbon from coconut husk and silica.  $\text{SiO}_2$  peaks of XRD diffractogram of the activated carbon from coconut husk:silica with mass ratio of 70:30, activated carbon from coconut husk without activation process, activated carbon from coconut husk with activation process, pure silica adsorbent were identified at  $2\theta$  around  $22.54^\circ$ ,  $21.98^\circ$ ,  $21.96^\circ$ ,  $22.24^\circ$ , respectively. The XRD analysis results shown in figures 2 to 5 interpreted the composition of  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$  of all samples as summarized in Table 1.

**Table 1.** Chemical composition in each adsorbents

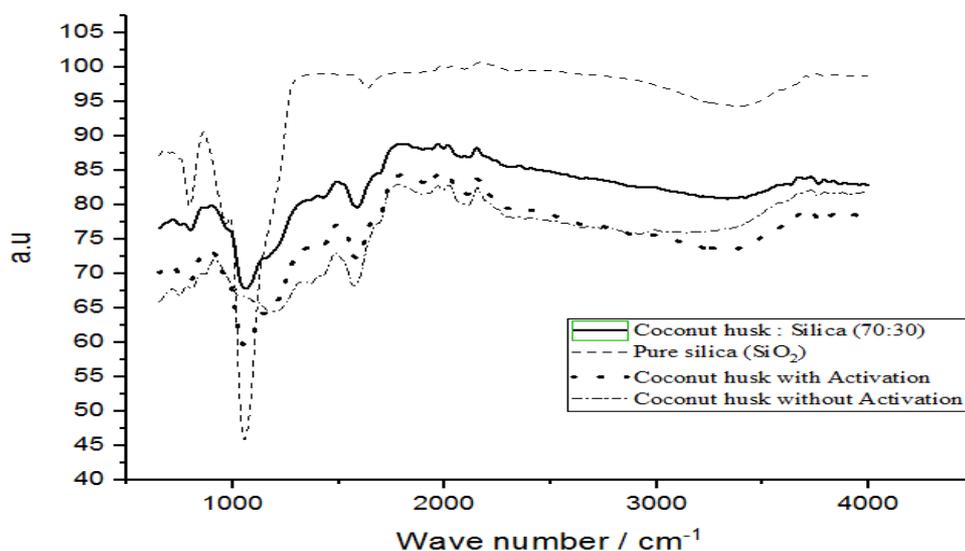
Type of adsorbent	Composition (weight %)		
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>
coconut husk : silica (70:30)	83	14.7	2.3
coconut husk without activation process	62	21.9	15.7
coconut husk with activation process	75	16.2	6.8
pure silica without activation process	63.2	16.7	20.1

Table 1 showed that composition of SiO<sub>2</sub> in the activated carbon from coconut husk:silica with mass ratio of 70:30 was higher than composition of SiO<sub>2</sub> in pure silica adsorbent and composition of SiO<sub>2</sub> in activated carbon from coconut husk with activation process. The higher of SiO<sub>2</sub> content in the sample induced the reactivity of the activated carbon from coconut husk:silica with mass ratio of 70:30 was better than the reactivity of pure silica adsorbent and activated carbon from coconut husk with activation process [11]. Silica plays an important role in the adsorption process because it has a good adsorption and ion exchange capability [12].

The content of SiO<sub>2</sub> from activated carbon from coconut husk with activation process was higher than that of the activated carbon from coconut husk without activation process. The activation of adsorbent with sulfuric acid affected the release of impurities on the adsorbent surface easily so that the active site pores inside the sorbent became broader [13].

### 3.3. Functional groups analysis of prepared activated carbon

Table 2 and figure 6 showed that the activated carbon from coconut husk:silica with mass ratio of 70:30, activated carbon from coconut husk without activation process, activated carbon from coconut husk with activation process, pure silica adsorbent has a Si-O functional group at peak of 1175 cm<sup>-1</sup>, 1202 cm<sup>-1</sup>, 1162 cm<sup>-1</sup>, 1178 cm<sup>-1</sup>, respectively. These results confirmed that the spectra pattern and wavenumber located at 1175 cm<sup>-1</sup> of the activated carbon from coconut husk:silica with mass ratio of 70:30, is the closest to the standard wavenumber reference of 1022.27 cm<sup>-1</sup> for Si-O functional groups [14]. Figure 6 shows FTIR spectra of 4 types of adsorbents.

**Figure 6.** FTIR spectra of 4 types of adsorbents

**Table 2.** FTIR functional groups of each adsorbents

Type of Adsorbents	Functional Groups	Wavenumber / $\text{cm}^{-1}$	
		Measured	References [14]
coconut husk : silica (70:30)	Si-O-Si	1175	898,83-1442,75
coconut husk with activation process	Si-O-Si	1162	898,83-1442,75
coconut husk without activation process	Si-O-Si	1202	898,83-1442,75
pure silica without activation process	Si-O-Si	1178	898,83-1442,75

### 3.4. Water content & ash content

Characterization of adsorbents is used to determine the physical and chemical properties of the adsorbent that later would be useful to be used in the adsorption process. In this study, we characterized the water content and ash content of the four types of adsorbents as shown in table 3.

**Table 3.** The water content and ash content of four adsorbents

Parameter	Indonesian National Standard (SNI) No. 06-3730-1995	Sorbents	Results (%)
Water content	Maximum 15%	coconut husk : silica (70:30)	4.41
		coconut husk without activation process	3.21
		coconut husk with activation process	3.87
		pure silica without activation process	2.71
Ash content	Maximum 10%	coconut husk : silica (70:30)	3.90
		coconut husk without activation process	1.86
		coconut husk with activation process	2.04
		pure silica without activation process	0.32

The water content can affect the adsorption capability of adsorbent. The higher water content causes the smaller capability of adsorbent to adsorb adsorbate. In this study, the water content of each samples was below 15% that meets the Indonesia national standard. These results indicated that the characterization result of the water content of all adsorbents was much lower than the standard one so that all adsorbents has potential to be used in adsorption process of ammonia and methylene blue of liquid wastewater.

The characterization of ash content is carried out to determine the amount of metal oxides or mineral salts and impurities contained in the adsorbent. The ash content obtained from each adsorbent was below 10%, which means that the adsorbent used in the research is feasible to use and meet the

standard. The highest ash content was found in the activated carbon of the combination type of coconut husk: silica (70:30) which was 3.90%, followed by the activated carbon of coconut husk with activation process, the activated carbon of coconut husk without activation process and pure silica was 2.04%, 1.86 % and 0.32%, respectively. The high ash content found in the activated carbon of the combination type of coconut husk: silica (70:30) and the activated carbon of coconut husk with activation process due to the use of chemicals as activating agents often results in contamination of the produced activated carbon. Generally activators leave undesired materials, such as oxides which are not soluble in water at the time of washing process [15].

### 3.5 Surface area

Characterization of activated carbon coconut husk and silica was done by BET method to determine the surface area. The result show that the specific surface area of activated carbon coconut husk : silica (70:30) was 52.924 m<sup>2</sup>/g with pore volume of 0.0948 cm<sup>3</sup>/g and pore size of 71.712 nm. The specific surface area of activated carbon from coconut husk with activation was 3.0438 m<sup>2</sup>/g with pore volume of 0.00751 cm<sup>3</sup>/g and pore size of 98.6295 nm, The specific surface area of activated carbon without activation was 2.8119 m<sup>2</sup>/g with pore volume of 0.00753 cm<sup>3</sup>/g and pore size of 107.1203 nm, and the specific surface area of pure silica (SiO<sub>2</sub>) was 23.0385 m<sup>2</sup>/g with pore volume of 0.1688 cm<sup>3</sup>/g and pore size of 293.24 nm. Those BET data also showed that the activated carbon of coconut husk : silica (70:30) can be classified as mesoporous size. According to IUPAC, mesoporous diameter lies in the range of 2-50 nm. Activated carbon of coconut husk : silica (70:30) had a higher ratio of surface area to pore volume compared to pure silica (SiO<sub>2</sub>) and coconut husk only. This makes the activated carbon of coconut husk : silica (70:30) more reactives.

## 4. Conclusion

1. The XRD analysis confirmed that the highest silica composition of 83 % was observed in the activated carbon from coconut husk: silica (70:30). The silica composition in the activated carbon of coconut husk with activation process, the activated carbon of coconut husk without activation process and pure silica was 75 %, 62.4 % and 63.2 %, respectively.
- 2 The activated carbon of coconut husk with activation process had higher silica composition compared to the activated carbon of coconut husk without activation process. FTIR spectra identified the presence of Si-O fungsional group in all type of prepared adsorbent. SEM image showed that the activated carbon from coconut husk:silica (70:30) had larger and broader pores compared to the activated carbon of coconut husk with and without activation process.
3. The specific surface area of the activated carbon from coconut husk:silica with mass ratio of 70:30 was higher than the specific surface area of pure silica (SiO<sub>2</sub>) and coconut husk only.

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