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# Potentials of biomass waste sources for heterogeneous catalyst production

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**Abstract.** 80% of processes in industries in developing countries generally use catalysts, both homogeneous and heterogeneous, derived from biomass. The research aimed to synthesize silica-based heterogeneous catalysts from waste of Sugar palm peel, Pineapple leaf and Orange peel, then carried out characterization of the heterogeneous catalyst used. The method started from the preparation of biomass waste, catalyst characterization including yield of SiO<sub>2</sub> in extract, crystallinity, pore size, surface area and catalyst morphology. The yield of SiO<sub>2</sub> extract from the Sugar palm peel was 12.281% ; Pineapple leaf was 34.131%, and Orange peel was 30.102%. The diffractogram showed that all SiO<sub>2</sub> catalyst synthesis had a crystalline phase; The pore size of the catalyst ranged from 3.918 to 5.6512 nm, and the surface area of the catalyst ranged from 14.2137-247 m<sup>2</sup> g<sup>-1</sup>. Morphological test results showed that the catalyst SiO<sub>2</sub> extract of Sugar palm peel was composed of small, regular pores while the catalyst SiO<sub>2</sub> extract of Pineapple leaf was composed of globular blob, and the catalyst SiO<sub>2</sub> extract of Orange peel was composed of very heterogeneous-tight-porous structures. Based on the test results, it is known that the synthesis of heterogeneous catalysts from biomass waste meets the characteristics of standard silica catalysts.

*Keyword:* Pineapple leaf, Sugar palm peel, Orange peel, Silica (SiO<sub>2</sub>), Diffractogram

## 1. Introduction

The manufacturing industry has recently been introduced to the concept of Industry 4.0 which is supported by very rapid technological advances [1]. In the industry, most processes (above 80%) involve catalysts [2]. The production of a compound generally uses a homogeneous catalyst and heterogeneous catalyst. The disadvantage of homogeneous catalysts is that they are difficult to separate from the resulting solution so that additional processes and costs are needed in the production stage [3]. Homogeneous catalysts can be replaced by heterogeneous catalysts which are more environmentally-friendly, stable at high temperatures, and have large pores [4]. However, the use of commercial heterogeneous catalysts is still dominated by commercial catalysts which has a

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disadvantage, that is, their prices are quite expensive but they remain to be excellent in the industry [5].

In connection with the focus of the development of Science Technology and Innovation towards the Industrial era 4.0, local natural resources from renewable biomass is a research potential which interests researchers to reduce environmental degradation. Concerns about sustainable development have encouraged efforts to improve the efficiency of the utilization of raw resources and reduce the production of waste generated from the agribusiness sector of agriculture and agro-industry due to improper disposal [6, 7]. The source of biomass has the potential as new materials, namely the procurement of basic materials for making heterogeneous catalysts to replace commercial catalysts in meeting industrial needs. One of heterogeneous catalysts which have been developed includes amberlyst-15 [8], amberlyst-35 [9], natural zeolite [10], mesoporous silica, alumina [11], and the use of ion exchange resins [12].

In current developments, the natural potentials which can be considered as raw materials for heterogeneous catalysts are agricultural biomass wastes such as Sugar palm peel, Pineapple leaf and Orange peel whose existence is not utilized due to lack of knowledge and technology especially in rural areas, so they tend to be ignored. In this term, the reuse of waste will cause a significant reduction of the resulting waste so that the environmental impact is reduced. This research aimed to synthesize silica-based heterogeneous catalysts from the waste of Sugar palm peel, Pineapple leaf and Orange peel and tested the characterization of heterogeneous catalysts.

## 2. Experimental Procedure

### 2.1. Preparation

In this study, samples of Sugar palm peel waste were from the kolang kaling industry, Pineapple leaf waste in Tamban, Barito Kuala, and Orange peel waste from the market, juice drink sellers, and restaurants. The samples were dried under the sun for 7 days until the water content dropped to around 12-14%. Then the dried samples were mashed with a chopper until it passed the 40 mesh sieve before heated in a furnace for calcination at 800°C for 4 hours. Furthermore, the results of calcination were crushed and sieved to pass 100 mesh.

### 2.2. Catalyst extraction and synthesis

Each calcined sample was mixed into a 3M NaOH solution in a ratio of 1: 4 (w/v). The leaching process was done for 12 hours at a temperature of 90°C, then the separation between the filtrate and the residue was carried out. The filtrate was then added with 1M HCl little by little until a white precipitate formed and reached a neutral pH (6.5-7). The precipitate was then washed with distilled water and dried at 110°C for 6 hours to form serogel (dry silica gel). The Serogel was then mashed to obtain white silica powder and then calcined at 700°C for 3 hours to obtain silica crystals.

### 2.3. Characterization of catalyst synthesis

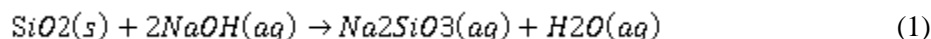
Characterization of heterogeneous catalyst synthesis included analysing the crystallographic structure of the catalyst based on amorphous or crystalline properties with X-ray Diffraction (XRD), analysing pore size of catalysts with Brunauer-Emmett-Teller (BET), and analysing the surface morphology of samples and particle size with Scanning Electron Microscopy-Energy Dispersive X-Ray Spectrometer (SEM-EDX).

## 3. Results and Discussion

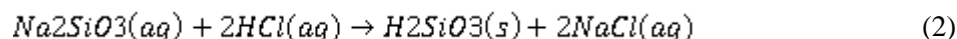
### 3.1. Silica extract yield

Based on the yield of silica extract (SiO<sub>2</sub>), the figure obtained from Sugar palm peel was at 12.281%; Pineapple leaf was at 34.131%, and Orange peel was at 30.102% in Figure 1. This showed that the

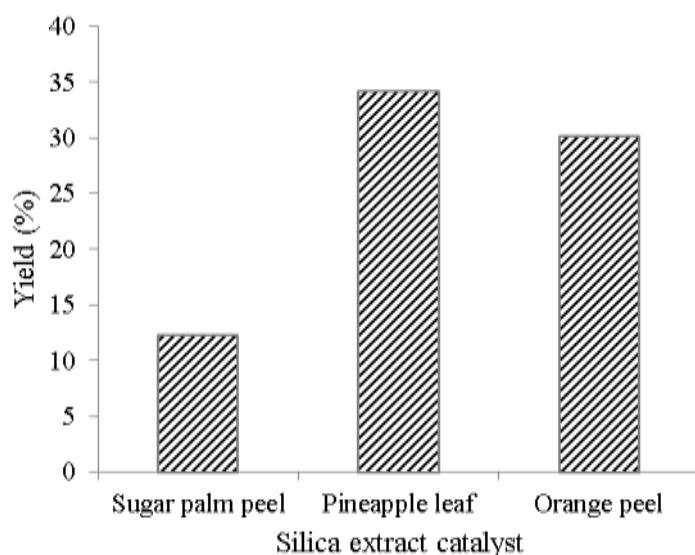
extraction process with NaOH was able to penetrate the capillaries in the sample calcination of biomass waste and silica solubility where sodium silicate was formed, as in Equation (1).



Furthermore, the addition of HCl caused the exchange of  $\text{Na}^+$  ions with  $\text{H}^+$  to produce a gel-shaped solid which would separate silica particles bound to water molecules, namely silicate acid ( $\text{H}_2\text{SiO}_3$ ) or silica hydrosol. In this process, the reaction which occurs was as in Equation (2).



This showed that the greater the concentration of NaOH in the extraction process was, the higher hydrogen yield obtained, and the greater the amount of raw material was, the greater the result was [13].



**Figure 1.** Silica extract yield from calcination results of biomass waste

### 3.2. Characterization of silica extract catalyst synthesis

Based on the results of diffractograms in the Joint Committee for Powder Diffraction Standards (JCPDS) No. 46-1045 in the International Center for Diffraction Data (ICDD) database, they showed that all silica catalyst synthesis had a crystalline phase having a characteristic peak of silicon oxide ( $\text{SiO}_2$ ), presented in Table 1.

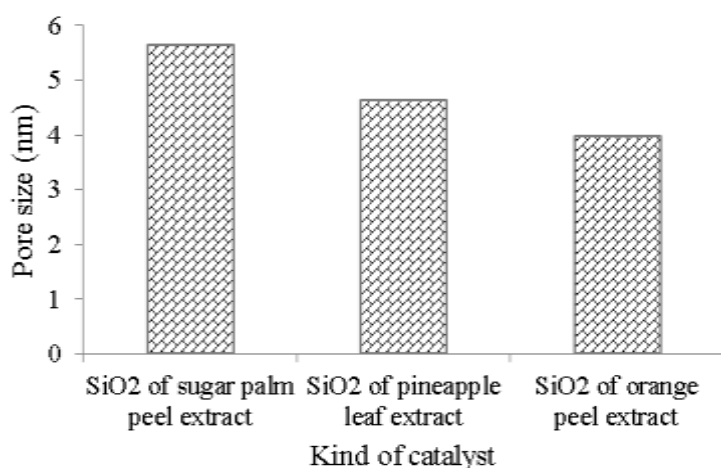
**Table 1.** The peak diffractogram of the synthesis of silica extract catalysts.

Silica extract source	Phase form	Peak diffractogram			Information
		$2\theta$	$2\theta$	$2\theta$	
$\text{SiO}_2$ standard	Crystal	$20.860^\circ$	$26.640^\circ$	$45.793^\circ$	JCPDS No. 46-2045
$\text{SiO}_2$ sugar palm peel extract	Crystal	$20.673^\circ$	$26.522^\circ$	$45.250^\circ$	research
$\text{SiO}_2$ pineapple leaf extract	Crystal	$21.397^\circ$	$26.532^\circ$	$45.660^\circ$	research
$\text{SiO}_2$ orange peel extract	Crystal	$20.883^\circ$	$25.917^\circ$	$45.574^\circ$	research

Table 1 showed that the peak pattern of diffractogram of Sugar palm peel extract, Pineapple leaf extract and Orange peel extract in crystalline phase with a characteristic peak of silicon oxide ( $\text{SiO}_2$ ) was in accordance with XRD standard silicon oxide structure ( $\text{SiO}_2$ ) JCPDS card No. 46-1045 at  $2\theta = 20.860^\circ$ ,  $2\theta = 26.640^\circ$ , and  $2\theta = 45.793^\circ$ . This showed that the synthesis of silica extract catalyst from

calcined biomass waste at 700°C formed the sharp and narrow peak position of diffractogram with a high enough intensity to include the crystalline silicon oxide ( $\text{SiO}_2$ ) phase produced.

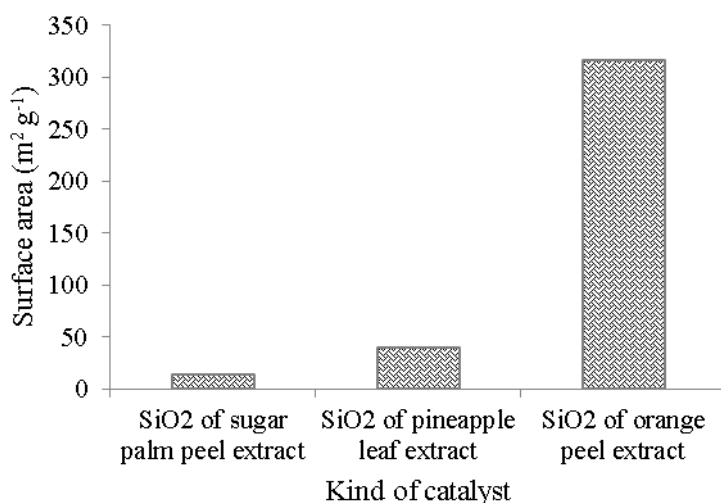
Furthermore, the pore size of the catalyst obtained from the adsorption isotherm described the type of adsorption of the solids produced. The pore size results of  $\text{SiO}_2$  extract of Sugar palm peel,  $\text{SiO}_2$  extract of Pineapple leaf and  $\text{SiO}_2$  extract of Orange peel with the Brunauer Emmett Teller (BET) method could be seen in Figure 2.



**Figure 2.** The catalyst pore size of the  $\text{SiO}_2$  of Sugar palm peel extract,  $\text{SiO}_2$  of Pineapple leaf extract and  $\text{SiO}_2$  of Orange peel extract.

Figure 2 showed that the catalyst pore size of the  $\text{SiO}_2$  of Sugar palm peel extract,  $\text{SiO}_2$  of Pineapple leaf extract and  $\text{SiO}_2$  of Orange peel extract ranged around 3.918-5.6512 nm. These results were in line with studies of mesoporous silica nanoparticles ( $\text{SiO}_2$ ) catalysts which produced pore sizes ranging from 2.4 to 6.5 nm [14]. The catalyst pore size produced, including mesoporous, was below 50 nm, because the smaller the pore size, the better the selectivity to the product. This showed that the activation and calcination process at 700°C could change the size of the crystal pore, so there was evaporation of water in the crystalline pore of the catalyst  $\text{SiO}_2$  of Sugar palm peel extract,  $\text{SiO}_2$  of Pineapple leaf extract and  $\text{SiO}_2$  of Orange peel extract.

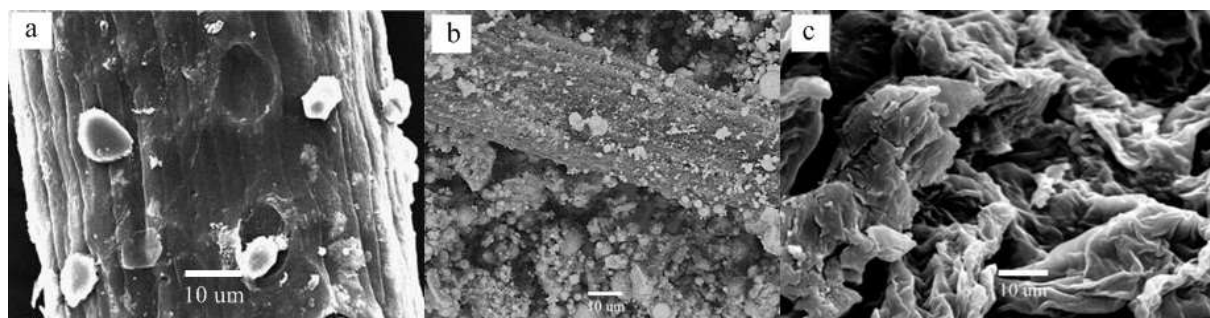
While the surface area of the catalyst produced from the  $\text{SiO}_2$  of Sugar palm peel extract,  $\text{SiO}_2$  of Pineapple leaf extract and  $\text{SiO}_2$  of Orange peel extract could be seen in Figure 3.



**Figure 3.** The catalyst surface area of the  $\text{SiO}_2$  of Sugar palm peel extract,  $\text{SiO}_2$  of Pineapple leaf extract and  $\text{SiO}_2$  of Orange peel extract.

Figure 3 showed that the SiO<sub>2</sub> catalyst of Orange peel extract had a surface area of 247 m<sup>2</sup> g<sup>-1</sup> greater than the surface area of the SiO<sub>2</sub> catalyst of Sugar palm peel extract 14.2137 m<sup>2</sup> g<sup>-1</sup> and SiO<sub>2</sub> catalyst of Pineapple leaf extract with a surface area of 39.3118 m<sup>2</sup> g<sup>-1</sup>. This showed the catalyst produced from biomass waste had potential as a supporting catalyst in the production process. These results were in line with studies which examined the surface area of silica gel supports, ranging from 14.29 to 299.9 m<sup>2</sup> g<sup>-1</sup> [15]. From the observations, the surface area of silica obtained in this study was comparable to studies which reported silica, alumina and aluminosilicate with modified conventional depositional treatment resulting in a surface area of 12–67 m<sup>2</sup> g<sup>-1</sup> [16].

Furthermore, the morphology of the catalyst solids produced by the analysis of the Scanning Electron Microscopy (SEM) size of 10 μm could be seen in Figure 4. It showed that SiO<sub>2</sub> of Sugar palm peel extract which was composed of regular small pores was in line with research related to the characterization of Sugar palm peel [17]. The SiO<sub>2</sub> catalyst of Pineapple leaf extract was composed of globular blob was in line with research examining microporous activated carbon Pineapple leaf fiber [18] and SiO<sub>2</sub> catalyst of Orange peel extract composed of very heterogeneous tightly porous structure was in line with research related to the modification of Orange peel adsorbent [19].



**Figure 4.** SEM morphology of catalyst solids (a) SiO<sub>2</sub> of Sugar palm peel extract, (b) SiO<sub>2</sub> of Pineapple leaf extract and (c) SiO<sub>2</sub> of Orange peel extract.

The morphological results of the catalyst solids with SEM supported the diffractogram data in the International Centre for Diffraction Data (ICDD) database that the SiO<sub>2</sub> catalyst of Sugar palm peel extract, SiO<sub>2</sub> catalyst of Pineapple leaf extract and SiO<sub>2</sub> catalyst of Orange peel extract had high crystallinity. This showed that the calcination temperature of 700°C for 4 hours created a complete hydrothermal process causing the SiO<sub>2</sub> catalyst of Sugar palm peel extract, the SiO<sub>2</sub> catalyst of Pineapple leaf extract and the SiO<sub>2</sub> catalyst of Orange peel extract to be more crystallized with an open pore shape so that the surface area increased. This was influenced by the fact that calcination temperature determined the characteristics of the peak, based on the diffractogram data and pore size, surface area and the catalyst solids morphology produced during the hydrothermal process [17]. From the catalyst characterization test, it was known that the catalyst extract obtained from Pineapple leaf waste, Sugar palm peel waste and Orange peel waste met commercial silica catalyst standards so that it could be applied to the bio-diesel industry such as fatty matter and crude glycerol in increasing added value.

#### 4. Conclusion

The yield of silica (SiO<sub>2</sub>) obtained from each Sugar palm peel is 12.281%; Pineapple leaf is 34.131%, and Orange peel is 30.102%. The diffractogram results show that all silica (SiO<sub>2</sub>) catalyst synthesis has a crystalline phase having a characteristic peak of silicon oxide (SiO<sub>2</sub>) with catalyst pore size ranging from 3.918 to 5.6512 nm and catalyst surface area ranging from 14.2137 to 247 m<sup>2</sup> g<sup>-1</sup>. The morphological results of the catalyst show that the SiO<sub>2</sub> catalyst of the Sugar palm peel extract is composed of small and regular pores. The SiO<sub>2</sub> catalyst of Pineapple leaf extract is composed of globular blob and the SiO<sub>2</sub> catalyst of Orange peel extract is composed of very heterogeneous tightly

porous structure. Based on the results of the characterization test, it shows that the synthesis of heterogeneous catalysts from biomass waste meets the characteristics of standard silica catalysts.

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