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Temperature-Programmed Reduction of Copper-Manganese Catalysts Derived from Biomass Activated Carbon

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Abstract

This study investigates the potential of bimetal impregnated catalysts supported on activated carbon derived from biomass for Selective Catalytic Reduction (SCR) of Nitrogen Oxides (NO_x) with ammonia (NH_3). The bimetal catalysts, Copper-Manganese (Cu-Mn) was deposited onto palm kernel shell (PKS) and coconut shell (CS) via impregnation method and calcined at 250 °C. Hydrogen Temperature-programmed reduction analysis (H_2-TPR) using 5% Hydrogen gas (H_2) in Argon (Ar) have been carried out to study the effect of different variables such as metal impregnation and support properties on the reduced states of the catalysts. Besides, FTIR, TGA and XRD were also used to characterize the catalysts. It was observed that impregnation of bimetals enhanced the catalyst characteristics where include important results from FTIR, TGA, XRD and H_2-TPR. Based on the results presented in H_2-TPR analysis, it was observed that the reduction peak of bimetal catalysts deposited on palm kernel shell activated carbon shifted to high temperature, about 597 °C. This demonstrates the intensity of the precursor interaction exists and a higher dispersion of bimetals on the surface of the support. In addition, the higher dispersion of bimetals was shown in XRD analysis. Hence, palm kernel shell-derived catalysts could be new and promising catalysts in SCR system.

Keywords: Activated carbon, Catalyst, SCR, H_2- TPR, XRD

1. Introduction

Nitrogen oxides (NO_x) are one of the greenhouse gases with an atmospheric lifespan of 1 to 7 days for nitric oxide and 170 years for nitrogen dioxide [1]. The origins of NO_x are categorized into mobile and stationary sources where, mobile source refers to combustion of fossil fuels from automobile system; meanwhile stationary source is referring to emission of flue gas due to combustion of coke from electrical power plants. Emission of NO_x to the atmosphere has been linked to a variety of environmental problems including rain acidification, global warming, photochemical smog, formation of ground level ozone and greenhouse effects, as well as health related issues such as

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bronchitis and pneumonia [2]–[3]. Thus, various methods have been studied in recent years to reduce the amount of NO_x emitted to the atmosphere in which one of those is catalytic technology [3].

Selective Catalytic Reduction (SCR) of NO_x to N_2 is one of the most effective and widespread treatment for the abatement of the emission of this pollutant to the atmosphere. Nowadays, NH_3-SCR technology is advantageous for stationary sources such as thermal power plant and refinery plant at temperature of more than 300 °C using metal oxides such as manganese, molybdenum, and copper supported on titanium dioxide as the catalysts [4]. Besides, TiO_2-V_2O_5 catalyst exerts high efficiency in the temperature range of 300 °C to 400 °C [5]–[6]. However, at this temperature window, the flue gases have high concentrations of contaminants, for example, SO_2 and particulate matter that may cause deactivation and poisoning of the catalyst [5]. Other transition metals have been reported as active for SCR process at temperature of more than 300 °C. The main problems in these catalysts are that still exhibit low-catalytic activities and poor resistance to catalyst poisoning [7]. Hence, there is a big interest to develop SCR catalyst which are active at lower temperatures, than 300 °C, where only small amount of particulates and SO_2 can be found in the flue gases, therefore, the deactivation and poisoning of the catalyst can be prevented [5].

Activated carbon (AC) is known for its extraordinarily large internal surface area and pore volume, which give activated carbon large adsorbent capacity. They have large surface area, up to 3000 m^2/g with pore volume range from 0.20 to 0.60 cm^3/g [8]-[9]. The surface area of activated carbon is formed by a micro-porous structure with pore diameters smaller than 2 nm [9]. Surface modification of AC can be done using bimetallic impregnation as it helps to enhance the catalyst properties to capture NO_x. Several raw materials for example cotton stalks, bituminous coals, palm kernel shell and coconut shell have been proposed to produce AC, where several studies have been done and the results indicate that the activated carbon has the potential to act as a catalytic support material. In addition, in Malaysia the wastes from palm shell and coconut shell are abundantly available and these wastes may pollute the environment. Thus, utilization of the agricultural wastes in developing countries, in Malaysia, is essential to promote greener environment and to sustain the natural resources. Apart being economically feasible, AC exhibits high catalytic activity at temperature between 100 °C – 250 °C, which fulfilled the requirements of SCR catalysts [10]. Considering the state of the art described above, the aim of this paper is to investigate the ability of carbon-supported Cu-Mn catalyst for NO_x reduction with NH_3.

2. Experimental

2.1. Catalyst Preparation

The granular activated carbon (AC), derived from coconut shell (CS) and palm kernel shell (PKS) was purchased from a local supplier. It was left to dry in the laboratory-scale oven at 100 °C, overnight. Each AC was impregnated with manganese and copper salts with a ratio of 1:1 and 10 wt% total metals loading. After gentle agitation at room temperature for 1 hour, the sample was left in a cabinet for one day [12]. After that, the impregnated AC was washed with distilled water until the effluents attained pH 6-7, to ensure that the residual impregnation solution is removed [11]. The resulting catalysts were allowed to dry overnight in the oven [12].

For calcination process, the catalysts were heated in a controlled environment for 4 hours at 250 °C. The samples were then placed in sample bags and kept in vacuumed desiccator. Catalyst developed using CS and PKS are denoted as Cu-Mn/CS and Cu-Mn/PKS respectively.

2.2. Catalyst Characterization

Both activated carbon (CS and PKS) and their derivatives (Cu-Mn/CS and Cu-Mn/PKS) were characterized using Fourier-Transform Infrared Spectroscopy (FTIR), Thermo-gravimetric Analysis (TGA) and X-ray Diffraction (XRD).

FTIR analysis was carried out using Thermo Nicolet 380 (Thermo Scientific, USA) where the samples were mixed with KBr, and the spectra were recorded within the range of 400 cm^{-1} to 4000 cm^{-1} to identify the oxygen functional groups on the catalysts surface [10].

Besides, TGA curves of the samples were acquired using a Shimadzu DTG-60H (Shimadzu Corp., Japan) with a simultaneous DTA-TG analyser. During the analysis, 30 mg of sample was heated up to 450 °C in nitrogen stream, with heating rate of 20 °C/min [13].

Furthermore, XRD was used to analyse the crystal phase of the samples in which it was performed using D/MAX rA (Rigaku Co., Japan). The data were collected between 10° to 70° with a step size of 0.02° [14].

2.3. Hydrogen Temperature-Programmed Reduction (H_2-TPR) Experiment

H_2-TPR was performed using Thermo Finnigan TPDRO 1100 (Thermo Scientific, USA). Prior to the experiment, 20 mg of samples was placed in a quartz tube reactor and pretreated under nitrogen gas flow of 30 ml/min at 150 ^{o}C for 10 min and cooled to ambient temperature. Then, 5% H_2/Ar gas flow at 25 ml/min was introduced to the samples to determine the hydrogen consumption (using thermal conductivity detector) from 50 to 950 ^{o}C with a ramp of 10 °C/min.

3. Results and Discussion

3.1. FTIR characterization of catalysts

The FTIR spectra of PKS, CS and the derivative catalysts are shown in Figure 1 and 2 respectively. It could be seen that there were certain changes in the surface functional groups due to impregnation of bimetals catalysts. Figure 1 displays the removal of peaks at 1743 cm^{-1} which shows the presence of carbonyl (C=O) group, referring to esters compound. In addition, the absorption of sp^3 C-H at 2952 cm^{-1} and broad peak at 1115 cm^{-1} due to stretching of C-O functional group referring to prominent ester for Cu-Mn/PKS was lower compared to PKS. This indicates that oxygenated groups were used by the metal oxides [13]-[15]. Furthermore, calcination process and nitrate decomposition might partly contribute to the structure change of AC [15].

Likewise, a single peak at 2406 cm^{-1}, attributed to –OH functional group, which is carboxylic acids was seen to diminish in Cu-Mn/CS, and at 3395 cm^{-1} attributed to –OH group was less compared to CS. Furthermore, new absorption peaks were observed for Cu-Mn/CS at 1419 cm^{-1} that corresponds to the COO- functional groups and at 1739 cm^{-1} that is contributed by the presence of (C=O) functional groups. This indicates that the acidic property of PKS has been enhanced by the dispersion of catalyst which is beneficial for NH_3 adsorption during SCR reaction.

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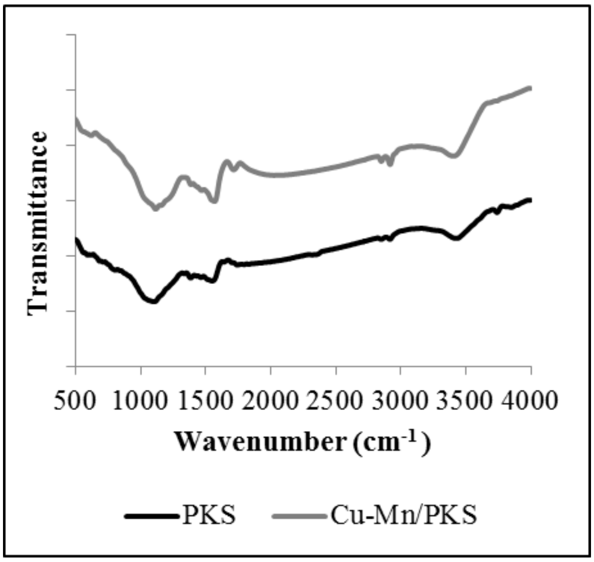


Figure 1. FTIR spectra of the PKS and Cu-Mn/PKS.

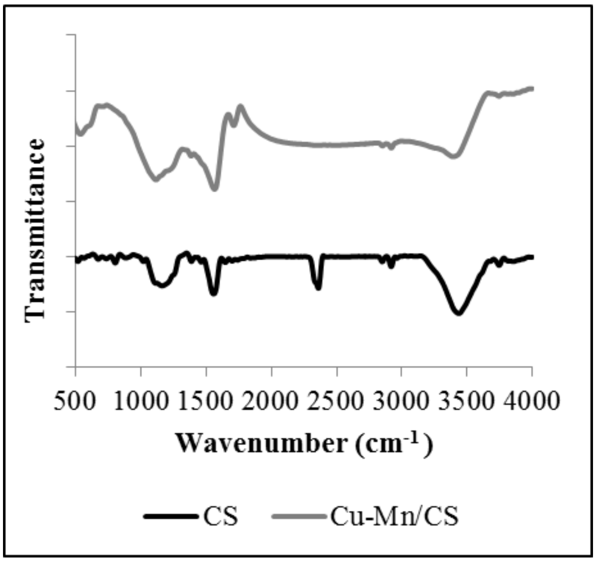


Figure 2. FTIR spectra of the CS and Cu-Mn/CS.

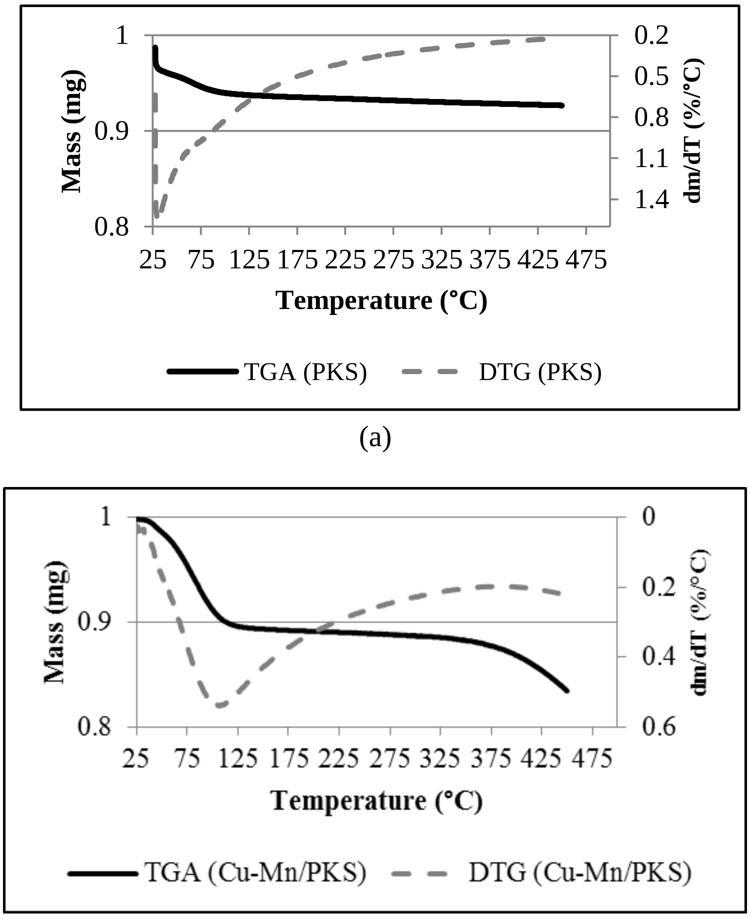
3.2. TGA characterization of catalysts

Figure 3 and 4 show the TGA curves of PKS, CS and the SCR catalysts synthesized from the two. The TGA curve for PKS in Figure 3(a) has a noticeable mass reduction peak around 55 °C with about 6% mass reduced. Meanwhile in Cu-Mn/PKS as shown in Figure 3(b), the mass loss due to water shifted to a higher temperature range at around 110 ^{o}C where the reduced mass ranges from 2% to 8%. In addition, the decomposition of organic matters occurred at around 180 °C to 400 °C, and a sudden reduction of mass occurred at 361 °C to 450 °C indicating that the manganese have not yet fully decomposed [16].

In Figure 4(a), mass reduction of about 1% to 3% occurred in CS sample at around 55 ^{o}C and 110 ^{o}C. Moreover, no peaks were observed in DTG curve at temperature 150 °C onwards. However, in Figure 4(b), two distinct weight loss regions were observed in Cu-Mn/CS. The first region occurred between 25 °C to 110 °C, followed by 110 °C onwards. A relative weight losses of 8% to 13% were observed at the first regions, corresponding to a loss of water. In the second region, a large weight loss of 17% was observed due to the decomposition of nitrates via equation (1) [17].

$$2Cu(NO_3)_2 \rightarrow 2CuO_2 + 4NO_2 + O_2 \quad (1)$$

Both activated carbons are able to act as catalysts when surface modification is done and have the ability to withstand high temperature and it is suitable to be used in SCR system. However, for Cu-Mn/PKS catalyst, the thermal stability might occur late as sudden reduction in mass occurs at the end of the analysis.



(a)

(b)

Figure 3. TGA-DTG curves of (a) PKS (b) Cu-Mn/PKS.

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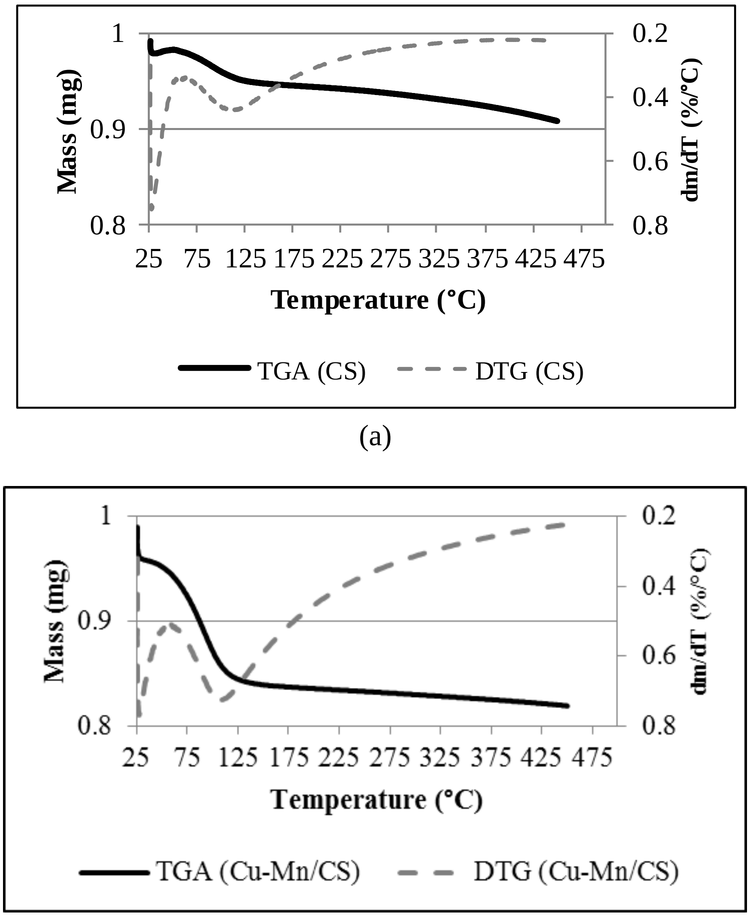

(a)

(b)

Figure 4. TGA-DTG curves of (a) CS (b) Cu-Mn/CS.

3.3. XRD characterization of catalysts

The x-ray diffraction patterns of SCR catalysts derived from PKS and CS are shown in Figure 5. A flat peak of CuO was observed at 2Θ = 32° and sharp peaks at 2Θ = 35° and 38° referring to Cu_2O [18]-[19]. The presence of CuO was expected due to the presence of divalent ions in the precursor salt [20]. The reduction processes of CuO can occur on activated carbons that are depicted in equations (2) and (3):

$$4CuO + C \rightarrow 2Cu_2O + CO_2 \quad (2)$$

$$2Cu_2O + C \rightarrow 4Cu + CO_2 \quad (3)$$

In addition, a broad peak was observed at 25° indicating the presence of MnO_2, and it has high dispersion on the surfaces of PKS-derived catalysts. Higher peaks representing MnO_2 crystalline planes were observed compared to a study by [21]. Furthermore, only sharp dispersion peaks of MnO were observed at 36° in Cu-Mn/CS, indicating the presence of MnO_2 [22]-[23].

Dispersion of metal oxides on the surface of the catalysts is an important parameter that relates to the reactivity of the catalysts. Based on Figure 5, weak dispersion peaks were observed on the PKS-

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derived catalyst compared to CS-derived catalyst, which indicates that the metal oxides are highly dispersed, thus PKS is favorable for SCR [22]-[23].

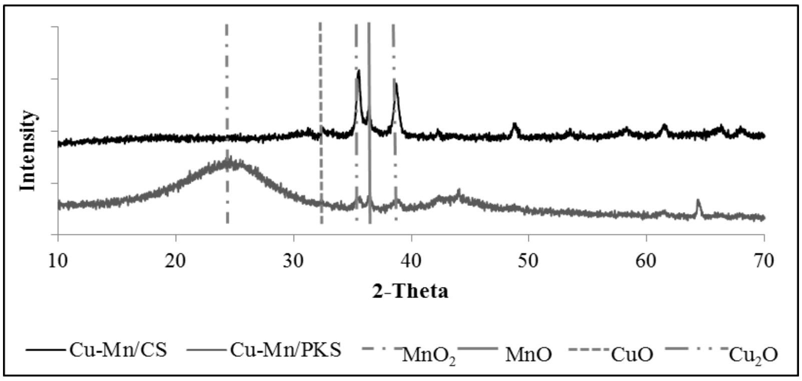

Figure 5: XRD patterns of Cu-Mn/PKS and Cu-Mn/CS

3.4. H_2-TPR Experiment

H_2-TPR experiments were carried out with both Cu-Mn/PKS and Cu-Mn/CS, and the results are shown in Figure 6 and Figure 7 respectively.

Hydrogen Temperature-Programmed Reduction (H_2-TPR) was used to determine the most efficient reduction temperature for the impregnated catalyst, as the oxidized catalyst is submitted to a programmed temperature rise while a reducing gas mixture is flowed over it.

The reduction peak at temperature range between 250 °C to 275 °C is usually assigned to the reduction of Cu^{+2} ions to Cu^{+1} ions and reduction of CuO clusters while the high-temperature peak at 275 °C to 300 °C indicating that the oxide starts to reduce from Cu^{+1} ions to Cu ions. Furthermore, the reduction of CuO may occur directly as in equation (4) or sequential reduction as in equations (5a) and (5b) [24]-[25]:

$$CuO + H_2 \rightarrow Cu + H_2O \tag{4}$$

$$2CuO + H_2 \rightarrow Cu_2O + H_2O \tag{5a}$$

$$Cu_2O + H_2 \rightarrow Cu + H_2O \tag{5b}$$

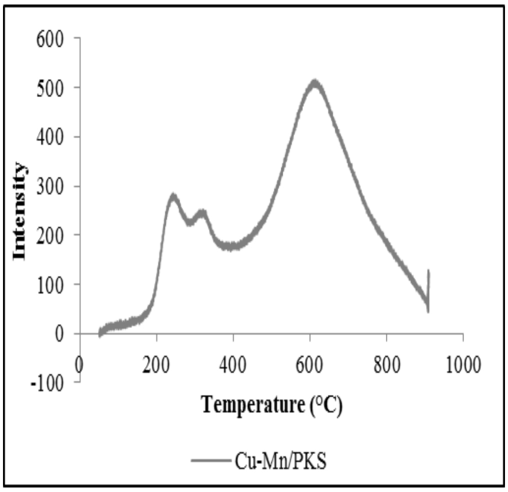

Figure 6: H_2-TPR profiles of Cu-Mn/PKS

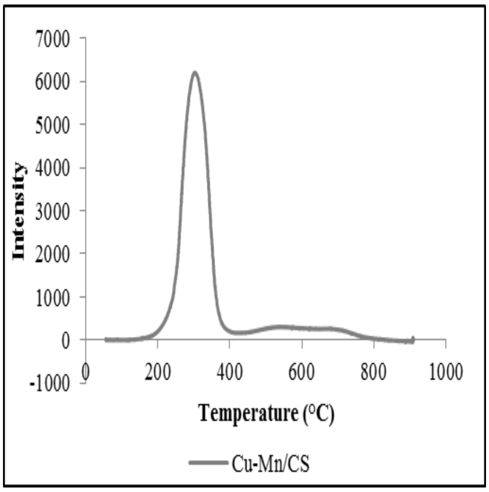

Figure 7: H_2-TPR profiles of Cu-Mn/CS

In Cu-Mn/CS, only one reduction peak was observed at 294 °C as shown in Figure 7 where the reduction of CuO may occur as in (5a) as the XRD pattern showed the presence of both oxides. For Cu-Mn/PKS as shown in Figure 6, two small reduction peaks at 228 °C and 300 °C and one reduction peak at 597 °C can be observed. The reduction peak at 228 °C was contributed by the reduction of copper nitrate to copper oxide. The subsequent peaks are associated to the reduction of manganese via $MnO_2 \rightarrow Mn_3O_4 \rightarrow MnO$ [16]-[26]. The presence or absence of Mn_3O_4 cannot be ascertained from XRD analysis as these metals may be in ionic form and its substitutes into the carbon or copper [27].

The activities of the catalysts are expected to be enhance when the AC was impregnated with bimetals. The shifting of the metals reduction peaks to higher temperature demonstrates the intensity of the precursor/support interaction exists and a higher dispersion of bimetals on the surface of the support [28]. Based on XRD analysis, higher dispersion of bimetals on the surface was observed and the support interaction increased in the following order based on XRD and H_2-TPR analysis: Cu-Mn/CS < Cu-Mn/PKS. This analysis proves that high dispersion of metals on the surface of the catalyst boosting their activities.

4. Conclusion

The catalytic activity of the activated carbon could be greatly improved by impregnation of bimetals copper and manganese. From the FTIR analysis, the distribution of oxygen functional groups increased after bimetals were impregnated onto the AC. Furthermore, in XRD analysis, weak dispersion of metal oxides was observed on the surface of PKS, which proved that the metal oxides are highly dispersed on this support. In addition, in H_2-TPR analysis, the metals reduction peaks impregnated on PKS are at higher temperature compared to peaks in CS. This is due to strong interaction between the catalyst support and metal oxides. This result is in an agreement with XRD analysis. In conclusion, both biomass-porous carbons have the potential to be applied in SCR system. It was proven that impregnating bimetals onto activated carbon was able to boost the catalysts characteristics especially in PKS-derived catalysts. Hence, bimetallic impregnated catalysts based on PKS carbon support could be a new and promising catalyst.

However, it is also noted that further detailed analysis of the matter present within the activated carbon (with or without metals catalysts) should be done. Therefore, the author suggest that all analysis especially FTIR, TGA and XRD analysis must be done on both samples which is raw activated carbon and bimetals catalysts. Besides that, surface area, pore diameter and pore volume of raw activated carbon and catalysts must be conducted for next study so that the effect of impregnating bimetallic catalysts onto the carbon-support can be observed.

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