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Adsorption of Soluble Ammonia Using A Porous Polymer Composite Cu-(TAC) And Activated Carbon Empty Fruit Palm Oil

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ABSTRACT

This study aims to determine the stages in the process of making activated carbon, synthesis of porous polymer composites Cu(TAC) and activated carbon composites (KA-Cu(TAC) for adsorption of dissolved ammonia (NH_4OH). To know the characterization results of activated carbon and KA-Cu(TAC), as well as knowing the optimum mass, concentration, and contact time of activated carbon KA-Cu(TAC) for the adsorption of dissolved ammonia. The research stages started from Empty Oil Palm Bunches (EFB) used as biosorbents for carbon production at 500ºC. The resulting product was activated with H_3PO_4, then modified with porous polymer Cu(TAC) to make a composite. The KA-Cu(TAC) composite was synthesized by reflux method. Then MOFs, activated carbon and KA-Cu(TAC) composite were characterized by BET. The BET characterization results showed that the successfully synthesized KA-Cu(TAC) composite experienced an increase in surface area.In the NH_4OH adsorption process the optimum mass efficiency on activated carbon still increased at 8 grams while the KA-Cu(TAC) composite obtained an efficiency of 36, 6% and optimally at a mass of 4 g. At variations in concentration, the efficiency of the two samples still decreased. And the optimum time efficiency obtained in the NH_4OH adsorption process with activated carbon was 70% and the KA-Cu(TAC) composite was 86.6%, both samples were equally optimal at 75 minutes.

Keywords: Activated carbon, MOFs Cu(TAC), NH_4OH adsorption

1. INTRODUCTION

In the process of processing palm oil into oil, by-products or waste are produced, one of which is the Empty Palm Oil Sign (EFB). OPEFB contains thick and coarse filaments, easily decomposes and is not toxic. In addition, OPEFB waste contains cellulose and hemicellulose which are suitable as activated carbon[1]. OPEFB has the potential to be used as activated carbon, because of its large adsorption capacity, high surface area, low cost and can be easily regenerated[2]. To increase the selectivity and adsorption capacity, activated carbon is modified with MOFs (Metal Organic Frameworks).

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MOFs are inorganic-organic hybrid compounds formed from metal ions and organic ligands through coordinating bonds[3]. MOFs have special characteristics, including being able to produce good adsorption capacities because they have regular pore sizes, large surface areas. The adjustable structure and abundant active sites make MOF considered as the main adsorbent candidate in the adsorption process which is better than adsorption with zeolite, silica, and activated carbon [4,5].

In this study activated carbon and MOF were modified into composites for adsorption of ammonium hydroxide. Where in the synthesis of MOFs Cu (copper) is used as the main metal (knot) in coordination with an organic linker, namely Terephthalic Acid (TAC) and will form a framework called MOFs Cu (TAC)[6]. The large number of MOFs that can be synthesized with a combination of organic linkers and metal centers provides an opportunity to control the surface area, pore size, and functional groups on the surface [7].

Ammonium hydroxide (NH_4OH) adsorption in this study used activated carbon and activated carbon MOFs Cu(TAC) (KA-Cu(TAC) composite synthesized by reflux method. Modifications were made to increase adsorption power and activate the active side of activated carbon. Ammonium one of the liquid wastes produced by the palm oil industry. Increasing industrial activity has resulted in large amounts of ammonium being discharged into the aquatic environment[8]. Excessive ammonium hydroxide content in waters causes eutrophication[9] which can damage aquatic ecosystems and oxygen depletion[10]. In addition, water containing ammonium is considered polluted and toxic, so it has the potential to cause disturbances and internal diseases[11]. , concentration , and time to determine application efficiency. The advantages of using the adsorption method are; processing is relatively simple, and the efficiency is relatively high, and does not have a negative impact on the environment[12-15].

2. EXPERIMENTAL

2.1. Chemicals, Equipment and Instrumentation

The tools used in this study were glassware, burettes, statives, clamps, analytical balances, 200 mesh sieves, ovens, furnaces, hotplates and reflux equipment. For characterization analysis using Brunnaeur-Emmelt-Teller (BET). The main material used in this research is Palm Oil Blank Signs. The chemicals used are NH_4OH, H_3PO_4, HCl, TAC (terephthalic acid), HF (fluoric acid), HNO_3 (nitric acid), $Cu(NO_3)_2$(copper (II) nitrate) Phenolphthalein indicator (pp), Mineral-free Aquadest , Filter Paper and Whatman No.1 Filter Paper.

2.2. Research Procedure

2.2.1 OPEFB Activated Carbon Synthesis

In this study, activated carbon was made from OPEFB in a furnace at 500^{o}C for 2 minutes, then activated carbon was activated with H_3PO_4.

2.2.2 Synthesis of MOFs Cu(TAC)

Then the synthesis of MOFs Cu(TAC) was carried out by refluxing a mixture of copper (II) nitrate solution, hydrofluoric acid, nitric acid, terephthalic acid, and distilled water with a ratio of 3:2:1:5,9:200 at 105oC for 8 hours.

2.2.3 Modified Activated Carbon with Cu(TAC) MOFs

Modification of activated carbon MOFs Cu(TAC) was carried out by immersing a mixture of terephthalic acid, ethanol and activated carbon with a ratio of 1:10:0.6 for 24 hours. Then the mixture was mixed with MOFs Cu(TAC) solution and refluxed at 105^{o}C for 8 hours. Product activated carbon, MOFs Cu(TAC), and MOFs Cu(TAC) activated carbon composites were characterized by Brunnaeur-Emmelt-Teller (BET).

2.2.4 NH_4OH adsorption

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NH_4OH adsorption was carried out by adding 30 ml of NH_4OH to a mass variation of 0.5 g, 1 g, 2 g, 4 g and 8 g. For activated carbon the concentration used was 0.3 M and 3 M MOFs Cu(TAC) activated carbon composite into mass variations of 0.5 g, 1 g, 2 g, 4 g, and 8 g. Then to determine the concentration efficiency, concentrations of 0.1 M-0.5 M were used for activated carbon, 1 M-5 M for MOFs Cu(TAC) activated carbon composites. Then determining the efficiency of time used is 15, 30, 45, 60, and 75 minutes.

3. RESULTS AND DISCUSSION

3.1. Brunnaeur-Emmelt-Teller (BET) Characterization

Brunnaeur-Emmelt-Teller (BET) characterization is used to determine the pore type, pore volume, surface area, and adsorption isotherm curve. From Figure 1, it is found that the type III isotherm curve does not have an unlimited number of layers on the surface of the adsorbent (multilayer).

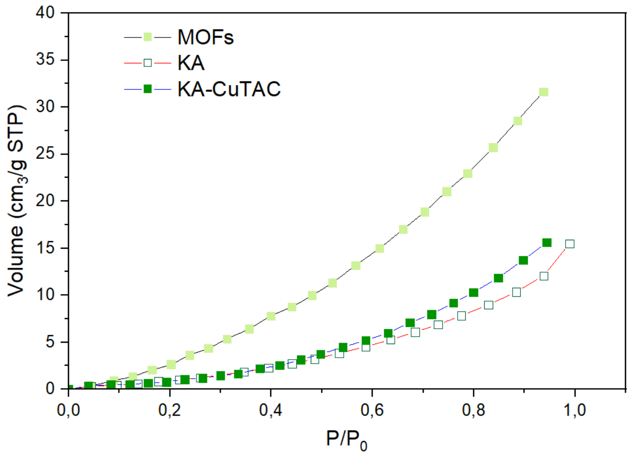

Figure 1. Isotherm KA, MOFs Cu(TAC) and KA-Cu(TAC) Composite Curves

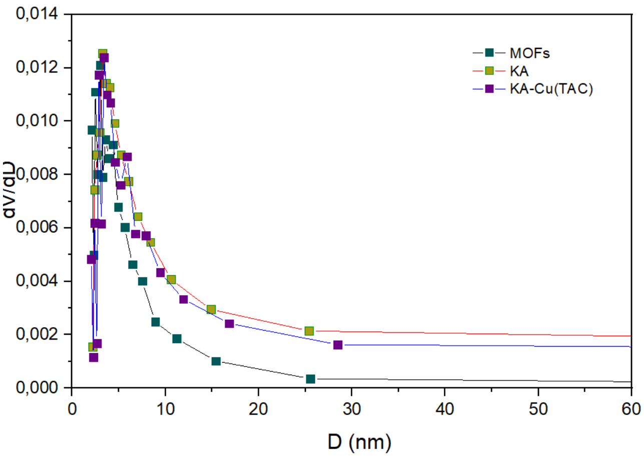

Figure 2. BET Analysis of KA, MOFs Cu(TAC), and KA-Cu(TAC)

Table 1. Pore properties of KA, MOFs Cu(TAC), Composite and KA-Cu(TAC)

Sample	Surface Area (m2/g)	Pore Volume (cm3/g	Pore Size (nm)
KA	14,89	0,039	10,47
MOFs Cu(TAC)	48,40	0,068	5,61

KA MOFs Cu(TAC)	19,06	0,037	7,69

Figure 2. shows the large variation in pore size between 2-50 nm in activated carbon, MOFs Cu(TAC) and KA-Cu(TAC) and this indicates that the pore size in the sample is not homogeneous. Based on table 1 MOFs Cu(TAC) has the largest surface area and larger pore volume compared to KA and KA-Cu(TAC. However, based on the isotherm curve in figure 1, KA-Cu(TAC) has increased in surface area from 14,891 $m2/g^{-1}$ to 19.06 $m2/g^{-1}$ This is because KA reacts with MOFs Cu(TAC) during the reflux manufacturing process.

3.2 NH_4OH Adsorption

Determination of the absorption efficiency of the adsorbent mass variation can be seen in the figure 4. It can be seen in the graph that the mass concentration efficiency of activated carbon for NH_4OH absorption increases with increasing mass, the increase causes the amount adsorbed to increase. On activated carbon, optimum efficiency is obtained at a mass of 8 grams. With an efficiency value of 62.3%. Whereas for KA-Cu(TAC) the optimum efficiency was obtained at a mass of 4 grams with an efficiency value of 36.6%. The difference in the concentration of KA and KA-Cu(TAC) affects the efficiency of absorption. It can be seen that KA-Cu(TAC) with a concentration of 3 M NH_4OH is optimal at 4 grams, but for activated carbon the absorption efficiency at 8 grams also still increases.

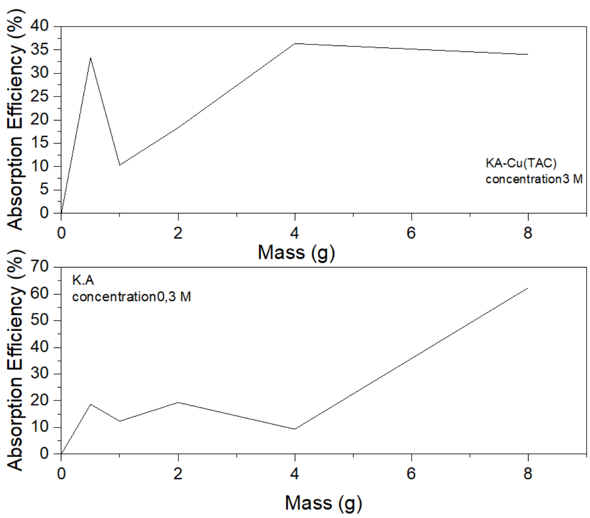

Figure 3. NH_4OH Absorption Efficiency with Mass Variation

Determination of absorption efficiency of variations in adsorbent concentration can be seen in the figure 5. In the graph it can be seen that the efficiency values of activated carbon and KA-Cu(TAC) composites both decrease. the effect of concentration variations on the absorption efficiency of NH_4OH. The difference in the concentrations of the two samples in adsorbing NH_4OH affects the absorption efficiency where for activated

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carbon the concentration increases until it is optimum and then the efficiency decreases. As for KA-Cu(TAC), the higher the concentration the lower the absorption efficiency of NH_4OH.

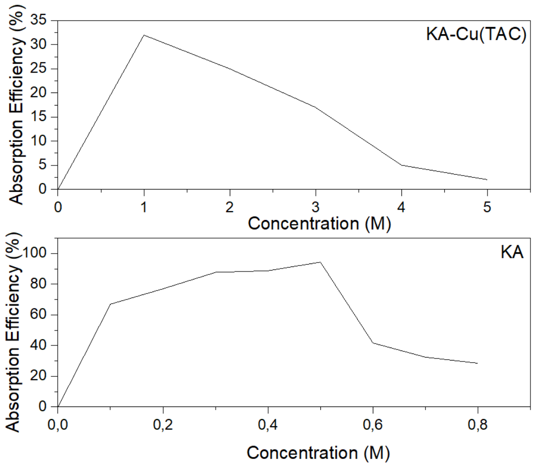

Figure 4. NH_4OH Absorption Efficiency of Concentration Variations

Determination of the absorption efficiency of the adsorbent contact time can be seen in the figure 6.

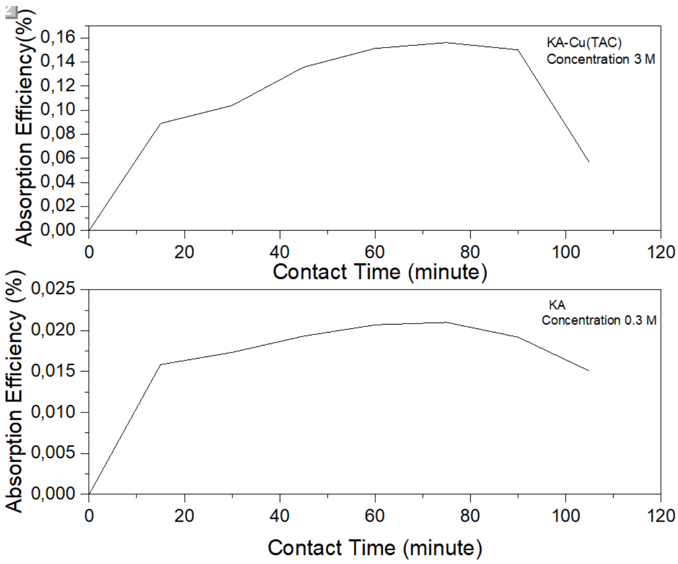

Figure 5. Time Variation Absorption Efficiency

Figure 6. shows the absorption efficiency of activated carbon and KA-Cu(TAC) for time variations. Where the optimal efficiency of the two samples is equally optimum at the contact time of 75 minutes, with an efficiency value of 70% for activated carbon and for the KA-Cu(TAC) composite, an efficiency of 86.6% is obtained. The efficiency value of the KA-Cu(TAC) composite is greater than that of activated carbon. This can be affected because of the different concentrations used. In this study[1] a higher efficiency value was obtained for activated carbon modified with FeCu because the presence of FeCu was able to make the active side of activated carbon regardless of the availability of pores. Modification of carbon in fact can increase the effectiveness of absorption. This was proven in previous studies and in this study, where activated carbon

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composites modified with MOFs Cu(TAC) were able to absorb high concentrations of NH_4OH, because KA-Cu(TAC) has an adjustable structure and abundant active [16].

4. CONCLUSION

Synthesis of activated carbon from OPEFB was made at 500°C and activated with H_3PO_4. MOFs Cu(TAC) and KA-Cu(TAC) Composites were successfully synthesized by reflux method. The BET characterization results showed that the successfully synthesized KA-Cu(TAC) composite experienced an increase in surface area. The mass variation efficiency for activated carbon is still increasing, while for the KA-Cu(TAC) composite the optimum efficiency is at 4 grams of mass. Efficiency of concentration variations showed a decrease in both samples, and for optimum time efficiency in both samples it was optimal at 75 minutes.

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