Characterization and Application of Mangosteen Peel Activated Carbon for Ammonia Gas Removal

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1. INTRODUCTION

Ammonia (NH₃) is a colorless gas with pungent odor which is emitted from agriculture, fertilizer industry, fossil fuel combustion, and some chemical industries (Vohra, 2020). The typical NH₃ concentrations emitted from the industrial process can range from 5 to 60 ppm (Chung et al., 2001) and ammonia from agricultural activities comprises approximately 80%-90% of total anthropogenic ammonia emissions (Xu et al., 2019). Ammonia is a threat to the environment due to its contribution to aerosol (PM₂.₅) formation that could adversely affect respiratory and cardiovascular systems, and its deposition leads to eutrophication, acidification, and loss of biodiversity (Xu et al., 2019).

Adsorption using activated carbon is a simple and low-cost method (Guo et al., 2005) to purify NH₃. Many reports have described the adsorption of ammonia gas by activated carbon (Domingo-Garcia et al., 2002; Ro et al., 2015; Vohra, 2020). This adsorption process depends on some factors such as the pore size, area, and surface chemistry (Bernal et al., 2018). Lignocellulosic agricultural wastes with their abundant availability, biodegradability, and non-toxic nature (Crini and Lichtfouse, 2018) can be used as an ideal precursor to produce activated carbon due to its high carbon content from the lignin composition (Nor et al., 2013). Several studies that used fruit peels for activated carbon productions such as durian peel (Chandra et al., 2009), rambutan peel (Ahmad and Alrozi, 2011), orange peel (Fernandez et al., 2014), and mangosteen peel (Nasrullah et al., 2019) have been conducted, and mangosteen peel has the highest lignin content of 48.63% (Devi et al., 2012). Mangosteen (Garcinia mangostana L.) is a fruit largely found in Indonesia. The production in 2018 reached 228,155 tons which increased 41.05% from the previous year (Statistics Indonesia, 2019). This mass production leads to the peel waste increase (Rattanapan et al., 2014) where, according to Foo and Hameed (2012) and this study, about 60-70% of 1 kg of mangosteen fruit is the peel. Mangosteen peel can be used as an activated carbon precursor because it has high lignin content (Devi et al., 2012) and low volatile
substances, and it has porous properties comparable to commercial activated carbon (Chen et al., 2011).

Physical activation using carbon dioxide (CO₂) has advantages such as creating narrower pores, being easy to control, inexpensive, and environmentally safe (Ahmad et al., 2013; Rangabhashiyam and Balasubramanian, 2019). However, the use of CO₂ activation for mangosteen peel activated carbon (MP-AC) is rarely reported. Mukti et al. (2015) was using steam activation, while other studies were using chemical activation (Rattanapan et al., 2014; Nasrullah et al., 2019). Although the effectiveness of mangosteen peel as an adsorbent in CO₂ and ethylene gas adsorption (Giraldo and Moreno-Piraján, 2017; Mukti et al., 2015) is accepted, none has assessed the MP-AC for the NH₃ adsorption. This study aims to characterize MP-AC prepared by CO₂ activation for NH₃ gas removal. Dynamic adsorption was used to illustrate the real conditions in the environment (Meneghetti et al., 2010; Patel, 2019) where emitted gas moves continuously through the bed. The adsorption capacities, kinetics, and isotherm were also investigated in this study. The results may provide necessary theoretical guidance of this material using physical activation method and implementation in the gas-phase dynamic adsorption.

2. METHODOLOGY
2.1 Adsorbent preparation
Mangosteen peels collected from the market were washed with distilled water and then dried at 105°C for 24 h (Li et al., 2018) before they were crushed using roll crushing machine and mortar grinder RM 200, and sieved into 10-20 mesh size. The carbonization was conducted in a tube furnace, Carbolite Gero HST 12/600 + 301 Controller, with a temperature of 700°C for 3 h under the flow of nitrogen (N₂) gas. The process continued with CO₂ activation under N₂ atmosphere at 850°C for 15, 120, and 180 min. The ramping rate for carbonization and activation process was 10°C/min. The purity of CO₂ and N₂ gases used in this study was industrial grade ≥99% from PT. Aneka Gas Industri.

2.2 Activated carbon characterization
The Brunauer-Emmett-Teller (BET) analysis (Quantachrome Nova Ver 11.0) carried out to determine the surface area through nitrogen adsorption experiment at the temperature of 77 K (-196.15°C). Scanning Electron Microscope-Energy Dispersive Spectroscopy (SEM-EDS, Analytical SEM JEOL JSM-6510A) was used to determine the morphological and elemental composition of MP-AC. The surface functional group was also determined using FTIR spectroscopy (IRPrestige-21 Shimadzu) at the wave interval of 4,000 and 340 cm⁻¹.

Moisture content, ash content, and iodine adsorption were determined according to the Indonesian standard, SNI 06-3730-1995 (Hastuti et al., 2015). The moisture content test was done by drying 1 g of the MP-AC at 105°C until the constant mass. The MP-AC was heated in the furnace at 600°C for 4 h to determine the ash content. Meanwhile, the iodine number test was performed by mixing 0.5 g of MP-AC with 50 mL of 0.1 N of iodine solution for 15 min. The filtrate (10 mL) was then titrated with 0.1 N of sodium thiosulfate solution and starch as an indicator. The equations used were as follows:

\[
\% \text{Moisture content} = \frac{a-b}{a} \times 100\% \quad (1)
\]

\[
\% \text{Ash content} = \frac{b}{a} \times 100\% \quad (2)
\]

\[
\text{Iodine number (mg/g)} = \frac{(V_1N_1-V_2N_2) \times 126.93 \times fp}{W} \quad (3)
\]

Where; \(a\) and \(b\) are initial and final mass of activated carbon (g) respectively; \(V_1\) is the analyzed iodine volume (mL); \(V_2\) is the volume of Na₂S₂O₃ used (mL); \(W\) is activated carbon weight (g); \(N_1\) and \(N_2\) are the iodine and Na₂S₂O₃ normality (N) respectively; \(fp\) is the dilution factor; and 126.93 is the iodine amount corresponding to 1 mL of Na₂S₂O₃ solution.

2.2 Adsorbate preparation
An analytical grade stock solution of ammonia (NH₄OH) was prepared in this study to make artificial gas by aerating dilute ammonia solution (Yani et al., 2013) with an air flow of 880 mL/min for 30 min. The NH₃ gas was produced and collected in a polyethylene bag with a volume capacity of 120 L.

2.3 Adsorption experiment
Dynamic adsorption experiments were conducted in the laboratory using the Duran glass column (diameter=1.2 cm, height=40 cm). The NH₃ gas was injected into the column at a flow rate of 1.1 L/min. The outlet gas was measured by a gas sensor SKY2000-M2 at intervals of one reading per minute. The test was carried out continuously until the adsorbent reached saturated condition. The schematic diagram for the adsorption test is presented in Figure 1.
The variations used in NH₃ adsorption process were adsorbent mass (1, 3, and 5 g) and initial NH₃ concentrations (10±1, 20±1, and 40±1 ppm). Data were collected twice for each variation and the adsorption capacity was calculated using equation 4 (Choo et al., 2013):

\[ q (\text{mg/g}) = \frac{(C_0 - C_t) \text{mg/L} \times \text{Flowrate (L/min)} \times t \text{ (min)}}{\text{Adsorbent mass (g)}} \]

Where; \( t \) is the equilibrium time, \( C_0 \) and \( C_t \) are the gas inlet and outlet concentrations.

Activated carbon used in this study were disposed to the waste management and treatment facility by following applicable rules or can be regenerated using thermal, steam, and chemical processes (Reza et al., 2020).

3. RESULTS AND DISCUSSION

3.1 BET analysis and activation time selection

Microporosities and surface area are important properties that characterize carbon adsorbents (Saputro et al., 2020). The results of BET analysis at various activation times are shown in Figure 2.

Figure 2 shows that the surface area increased from 39.06 m²/g to 588.41 m²/g with activation times of 15 and 120 min, respectively, and then decreased to 535.62 m²/g with longer activation time (180 min). In the activation process, superheated CO₂ diffuses into the inner precursor which burns the blockage of the byproducts, expands the pore, and increases the surface area (Lan et al., 2019; Yuliusman et al., 2017). The activation reaction can be seen below (Cheremisinoff dan Ellerbusch, 1978).

\[ \text{CO}_2 + \text{C}_x \rightarrow 2\text{CO} + \text{C}_{x-1} \]

According to Yang and Lua (2003), the increase in activation time increases the BET surface area. However, it can also result in the excessive carbon-\( \text{CO}_2 \) reaction, thus causing the expansion of the pores and some pore walls to collapse. Therefore, the surface area decreases in 180 min, and 120 min was chosen as the optimum activation time used in this study. The surface area obtained is smaller compared to the previous MP-AC studies using steam and chemical activation (Mukti et al., 2015; Nasrullah et al., 2019), probably due to the temperature and activating agent being used (Gebreegziabher et al., 2019). However, the surface area of activated carbon usually ranges 300-2,000 m²/g (Saputro et al., 2020), meaning that the result is still in the suitable range.

The isotherm graph obtained (Figure 3) showed the BET type 1 according to IUPAC classification, which usually indicates that the material is micro-shaped (Ambroz et al., 2018) with a relatively broad range of pore size distributions including wider and
narrower micropores (<~2.5 nm) (Giraldo and Moreno-Pirajan, 2018).

3.2 Characterization of mangosteen peel activated carbon

The results of moisture, ash content, and iodine number compared to other studies are shown in Table 1.

Based on Table 1, the moisture content (6.07%) is higher than other studies’ which use chemical activation, and lower than that of using physicochemical activation. This content indicates the hygroscopic nature which can affect the adsorption capacity (Hastuti et al., 2015). Meanwhile, the ash content which indicates the mineral content in activated carbon (Hastuti et al., 2015) has the highest value (9.8%) of all. According to Rangabhashiyam and Balasubramanian (2019), it can be influenced by the pyrolysis temperature and the activation method. The physical activation has a lower efficiency in reducing the mineral content than chemical activation, so the ash content becomes relatively higher.

Table 1. Characterization of MP-AC

| Activation/temperature (℃) | Moisture content (%) | Ash content (%) | Iodine number (mg/g) | References                  |
|---------------------------|----------------------|-----------------|----------------------|-----------------------------|
| Physical CO₂/850          | 6.07                 | 9.8             | 1153.69              | This research               |
| Chemical ZnCl₂: 600       | 4.8                  | 1.45            | N/A                  | Nasrullah et al. (2019)     |
| Chemical ZnCl₂            | 1.07                 | 5.68            | 820                  | Rattanapan et al. (2014)    |
| Physicochemical KOH-CO₂/828 | 9.08              | 1.63            | N/A                  | Ahmad and Alrozi (2010)     |

The iodine number obtained is 1153.69 mg/g, higher than that of Rattanapan et al. (2014). This iodine number shows adsorption ability, illustrates the porosity of activated carbon, and its higher value attributed to the presence of micropores as already proven in the BET isotherm graph. The result is also included in the range of suitable activated carbon (500-1,200 mg/g) (Saka, 2012).

3.3 FTIR and SEM-EDS analyses

The surface functional group of MP-AC is characterized using FTIR as seen in Figure 4.
The FTIR result (Figure 4) shows many peaks which representing the complex nature of MP-AC (Nasrullah et al., 2019). The broad peak in 3,600-3,200 cm\(^{-1}\) indicates the presence of hydroxyl (O-H) group influenced by CO\(_2\) as an activating agent, and the peak in 2,400-2,300 cm\(^{-1}\) shows a formation of nitrile (C≡N) (Ahmad et al., 2013). The small peak that occurs in 1,267.23 cm\(^{-1}\) and 1,120.64 cm\(^{-1}\) indicates the MP-AC contains a lack of C-O stretching as oxygen functional group in lactonic groups, alcoholic groups, and carboxylate moieties (Chen et al., 2011; Nasrullah et al., 2019). Moreover, the peaks in range of 1,620-1,400 cm\(^{-1}\), 1,760-1,690 cm\(^{-1}\), 3,100-2,850 cm\(^{-1}\), and <900 cm\(^{-1}\), indicates C=C stretching, carbonyl group C=O, hydrocarbons C-H, and aromatic bond C-H, respectively (Ahmad et al., 2013).

The results of SEM analysis before and after the adsorption process can be seen in Figure 5 as follows.

Figure 5. SEM image (a) before; (b) after NH\(_3\) adsorption

Before adsorption (Figure 5(a)), morphology shows presence of circular pores in different size and crevices after the carbonization and CO\(_2\) activation. It is shown that the MP-AC has porous nature that might affect the adsorption process (Nasrullah et al., 2019). After the adsorption (Figure 5(b)) there is an increase of impurities on MP-AC surface compared to the initial sample. This is because the adsorbate moves into the carbon pores during adsorption and results in the pore blockage (Basrur and Bhat, 2018). The surface elements of MP-AC determined using EDS is presented in Table 2.

Table 2. EDS analysis result of MP-AC before and after NH\(_3\) adsorption

| Sample            | % mass | C    | N    | O    | Other compounds |
|-------------------|--------|------|------|------|----------------|
| Before adsorption | 71.84  | 16.07| 8.77 | 3.32 |                |
| After adsorption  | 70.62  | 16.94| 9.43 | 3.01 |                |

Based on Table 2, the major compounds of MP-AC surface are carbon (71.84%), nitrogen (16.07%), and oxygen (8.77%). The N value of activated carbon is higher than dried mangosteen peel (1-2.67%) (Devi et al., 2014; Giraldo and Moreno-Pirajan, 2018; Nasrullah et al., 2019), probably due to the use of N\(_2\) as the inert atmosphere during the pyrolysis and cooling processes (Ahmad et al., 2013). Meanwhile, after the adsorption, there is no significant difference to these three major compounds.

3.4 Adsorption of NH\(_3\)

3.4.1 Effect of adsorbent mass and adsorbate concentration

The breakthrough curves of NH\(_3\) adsorption at different adsorbent mass and adsorbate inlet concentration are presented in Figure 6.

Based on Figure 6, initially, NH\(_3\) is fully adsorbed and adsorption process decreases with approaching equilibrium and gradually becomes constant. Figure 6(a) shows that for adsorbent mass of 1, 3, and 5 g, the equilibrium time was 16, 23, and 25 minutes respectively, indicating that saturation time becomes longer with the addition of adsorbent mass. This is due to the increasing surface area and available adsorption sites (Patel, 2019). Meanwhile, shown in Figure 6(b), smaller inlet concentrations produce the
longer breakthrough curves. According to Ding and Liu (2020) the decreasing inlet concentration decreases the amount of gas molecule passing the adsorbent, so the active sites are exhausted slowly.

![Figure 6. NH₃ breakthrough curve at different (a) adsorbent mass (10 ppm inlet concentration), and (b) different inlet concentration (adsorbent dosage 1 g/27.5 L).]

### 3.4.2 Adsorption capacity

The adsorption capacity value is obtained from the breakthrough graph (Figure 6) which is calculated using Equation 4. The average NH₃ adsorption capacity on MP-AC is 0.41 mg/g. The value is lower compared to commercial activated carbon which ranges from 0.6 to 4.7 mg/g (Ro et al., 2015). This result correlates with the EDS result that the N compounds after and before adsorption do not show significant differences. The NH₃ adsorption may be affected by several factors such as surface functional group, pore size and structure, and surface area (Kang et al., 2020; Yeom and Kim, 2017). Suspected in this study, acidic oxygen functional groups such as –OH, –NH, –C=O, –COOH, and metal ions is the main factor affecting adsorption capacity (Ro et al., 2015; Kang et al., 2020). According to the FTIR result, some of acidic surface oxygen groups are present such as –OH and –C=O. However, the nitrile group is also found, thus increasing the basic nature of the activated carbon (Ahmad et al., 2013). This finding might indicate that the surface of MP-AC is less acidic in nature due to the use of physical activation method (Nowicki et al., 2015) thus affecting the adsorption capacity. Additionally, further research is needed to enhance the NH₃ adsorption capacity and the use in numerous environmental applications.

### 3.4.3 Adsorption kinetics

Lagergren pseudo-first and pseudo-second order kinetic models are evaluated to describe the mechanism of NH₃ adsorption due to their good applicability in most cases (Ghasemi et al., 2014). The equations of pseudo-first order and pseudo-second order can be expressed as follows (Lagergren, 1898; Ho and McKay, 1999);

\[ q_t = q_e[1 - e^{(-kt)}] \]  \hspace{1cm} (6)

\[ q_t = \frac{k_2q_e^2t}{1+k_2q_e^2t} \]  \hspace{1cm} (7)

Where; \( q_e \) and \( q_t \) (mg/g) are the adsorption capacity at equilibrium and time \( t \), respectively; \( k \) and \( k_2 \) is the pseudo first and second order rate constant, respectively.

The results of kinetic parameters for each kinetic model are presented in Table 3.

For determining the appropriate kinetic model, calculated \( q_e \) and \( R^2 \) values should be taken into account (Unugul and Nigiz, 2020). According to the results in Table 3 and Figure 7, both pseudo-first order and pseudo-second order kinetic models show high and good \( R^2 \) values. However, from the calculated \( q_e \) value, the pseudo-first order kinetic model is close to the experiment data. This indicates that the pseudo-first order kinetic model is more suitable to describe the adsorption mechanism where NH₃ adsorption is
physically controlled. The physisorption occurs due to the Van der Waals forces (Guo et al., 2005) and in the pore surfaces which contain hydroxyl group as a preferred site to bind NH$_3$ (Yeom and Kim, 2017).

Table 3. Kinetics parameter of NH$_3$ adsorption on MP-AC

| Mass (g) | Co (ppm) | qe exp (mg/g) | Pseudo-first order | Pseudo-second order |
|----------|----------|---------------|--------------------|---------------------|
|          |          |               | $k_1$ (min$^{-1}$) | $qe_1$ (mg/g) | $R^2$ | $k_2$ (g/min.mg$^{-1}$) | $qe_2$ (mg/g) | $R^2$ |
| 1        | 10       | 0.413         | 0.205              | 0.404              | 0.971 | 0.394 | 0.516             | 0.986 |
|          | 20       | 0.303         | 0.565              | 0.290              | 0.991 | 1.909 | 0.347             | 0.999 |
|          | 40       | 0.316         | 0.793              | 0.309              | 0.996 | 2.507 | 0.370             | 1.000 |
| 3        | 10       | 0.363         | 0.231              | 0.345              | 0.988 | 0.619 | 0.417             | 0.992 |
|          | 20       | 0.450         | 0.201              | 0.475              | 0.996 | 0.259 | 0.650             | 0.992 |
|          | 40       | 0.448         | 0.306              | 0.474              | 0.993 | 0.415 | 0.637             | 0.987 |
| 5        | 10       | 0.427         | 0.134              | 0.441              | 0.994 | 0.204 | 0.586             | 0.986 |
|          | 20       | 0.493         | 0.124              | 0.581              | 0.989 | 0.106 | 0.857             | 0.983 |
|          | 40       | 0.515         | 0.169              | 0.612              | 0.989 | 0.135 | 0.908             | 0.984 |

Figure 7. Adsorption kinetics of NH$_3$ on MP-AC at 20 ppm inlet concentration and adsorbent mass of (a) 1 g, (b) 3 g, and (c) 5 g

3.4.4 Adsorption isotherm

In this study, to describe the interaction between adsorbate and adsorbent at the equilibrium, the Freundlich and Langmuir isotherm were investigated. The linear equation of Freundlich and Langmuir isotherm model is shown in Equation 8 and 9 respectively as follows (Freundlich, 1906; Langmuir, 1917):

$$\log q_e = \log K_F + \frac{1}{n} \log C_e$$ (8)

$$\frac{C_e}{q_e} = K_L + \frac{1}{q_L}$$ (9)
\[ R_L = \frac{1}{1 + K_L C_0} \]  

(10)

Where; \( q_e \) is the adsorption capacity at equilibrium (mg/g adsorbent), \( K_L \) is the Freundlich constant, \( K_L \) is the Langmuir constant (L/mg), \( n \) is the constant related to the adsorption energy of heterogeneity adsorbent site, \( C_e \) is the concentration of contaminants in equilibrium (mg/L), and \( R_L \) is equilibrium parameter.

The values of isotherm parameters for both Freundlich and Langmuir isotherm models are presented in Table 4.

**Table 4. Isotherm parameters of NH\(_3\) adsorption on MP-AC**

| Mass | Freundlich | Langmuir |
|------|------------|----------|
|      | 1/n        | N        | \( K_f \) (mg/g)(L/mg)\(^{1/n} \) | \( R^2 \) | q (mg/g) | \( K_L \) (L/mg) | \( R_L \) | \( R^2 \) |
| 1 g  | 0.195      | 5.1      | 0.15                           | 0.615       | 0.297      | 820.63           | 0.09     | 0.992       |
| 3 g  | 0.153      | 6.5      | 1.22                           | 0.644       | 0.481      | 611.00           | 0.12     | 0.994       |
| 5 g  | 0.146      | 6.8      | 1.10                           | 0.877       | 0.557      | 544.21           | 0.13     | 0.999       |

Based on determination coefficient \((R^2)\) from Table 4, the Langmuir isotherm is more suitable to describe the NH\(_3\) adsorption process with the \( R^2 \) of 0.999. The similar result has also been reported in previous studies for Methylene Blue adsorption (Foo and Hameed, 2012; Nasrullah et al., 2019), and in the NH\(_3\) adsorption on corn cob activated carbon (Gebreegziabher et al., 2019). Langmuir isotherms indicates the monolayer adsorption where there is no interaction in adsorbate molecules (El maguana et al., 2020), and the carbon surfaces have homogeneous structures and identical active sites (Kutluay et al., 2019). Moreover, the equilibrium parameter \((R_L)\) value is in the range of \( 0 < R_L < 1 \), suggesting that the NH\(_3\) adsorption using MP-AC is favorable (Hamzaoui et al., 2018).

**4. CONCLUSION**

MP-AC prepared from physical activation using CO\(_2\) at 850°C for 120 min shows good porosity with surface area of 588.41 m\(^2\)/g, 6.07% moisture content, 9.8% ash content, and iodine number of 1153.69 mg/g. MP-AC can be used as an adsorbent material to remove NH\(_3\) with adsorption capacity of 0.41 mg/g which is lower than commercial activated carbon (0.6-4.7 mg/g). The pseudo-first order kinetic and the Langmuir isotherm are best fitted to the experimental data. Consequently, mangosteen peel may be potentially used as an activated carbon precursor with further modification for NH\(_3\) adsorption and various environmental applications.

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