Scale-up preparation and testing of activated-carbon-cloth as carbon-filter prototypes for ammonia-vapor adsorption

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Abstract. This study is a scaled-up experiment for carbon cloth made of the pristine-palm-kernel shell studied previously at the laboratory level. Prototypes of activated carbon filter (cylindric net, 40 cm height & 18 cm diameter) were prepared from the activated palm-kernel carbon cloth. The ammoniac adsorption capacity test was carried out within six closed rooms with the individual dimension of 120 cm x 90 cm x 220 cm. The ammonia vapor samples were determined by UV-Vis spectrophotometric method. Each room contained 2.8 x 10^-5 M of ammonia vapor at ambient temperature, (28±1)°C atmospheric pressure. The activated carbon mean-weight of (179±5) g or equivalent to (0.1±0.003) g per cm². The optimum time of adsorption was 60 minutes and the mean adsorption capacity of the carbon filter was 1.78 x 10^-5 M per 1720 cm² of carbon cloth. The carbon filter prototype absorbed 60.6% of ammonia vapor in the closed room. The adsorption remained constant, although the absorbent and time were expanded.

1. Introduction
The ammonia vapor has a stinking smell, poisoning and irritating. The ammonia poison effect depends on its emission concentration level in the air (National Research Council, 2008). It is possible to detect the odor in the concentration range of 5-53 ppm and has also been observed to have the ability to spread to other rooms at 50 ppm in 10 minutes [1].

Activated carbon has been widely used adsorbent in water and gas, including for ammonia vapor adsorption [2-5]. Using granular carbon is required a porous container. Carbon particles become dense that is not easily penetrated by gas. The fine carbon particles will block the gas stream when it is used in a column reactor. Therefore, immobilizing carbon particles on the cloth became an alternative to spread the particles and more practical in uses [6]. However, activated charcoal made of carbon fibers is relatively expensive and often involves a complex procedure [7].

The immobilization technique of active substances onto solid supports has been widely reported [8-9]. The immobilizing of activated charcoal and activated charcoal doped with TiO₂ on the glass surface has been investigated [10-11]. However, the immobilization technique of activated charcoal on a cloth is very little found in the literature. Most literature discussed a reverse way: the active component was immobilized on charcoal, very rare the inversely [12-14].

We have conducted laboratory-scale research on the preparation of activated charcoal from palm kernel shells. The activated carbon was fabricated by a simpler method using a new prototype furnace designed palm shell itself as a source of fuel and heat. Activated charcoal products have been characterized and compared with commercial activated charcoal. The activated charcoal product is then immobilized on the fabric as the support material to wider the uses and more practical applications.
This study aims to scale up the experiment by repeating several experimental works, including activated carbon preparation, characterization and immobilization on cloth. The carbon filter prototype (CFP) made of palm-kernel carbon cloth was tested for ammonia vapor adsorption in simulated large rooms. The scale-up experiment was set up to close to real environmental testing, which is crucial before the commercialization process.

2. Material and Methods

2.1 Manufacture of palm kernel activated carbon
Dried palm kernel shell (PKS) weighed to be 16 kg was washed, sun-dried. The PKS was soaked into 25% NaOH for 24 h, subsequently sun-dried before carbonized within a pyrolysis chamber. The heat source was the burning PKS itself, other than PKS filled in a pyrolysis chamber. The combustion equipment is made of two different sizes of hollow metal cylinders. The inner (smaller size), which was the pyrolysis tube, was shielded from the air and the outer was the combusting chamber, where PKS was continuously added as fuel to produce the heat for pyrolysis. Combustion was adiabatic took 3 hours and the temperature reached around 800°C-1000°C, which was controlled by continuous addition of PKS as the fuel to simulate the real application in the community. The powder activated carbon was characterized according to the Indonesian National Standard (SNI) 06-3730-1995. The surface and surface area analysis were carried by Scanning Electron Microscope (SEM) and Brunauer-Emmett-Teller (BET). Brunauer-Emmett-Teller (BET) Sorptomatic 1800 and scanning electron microscopy (SEM) JEOL JSM-6510LA analyses and the activated carbon level for each referred to ASTM D3175-11, ASTM D3175-02, ASTM D3174-11 and ASTM D4607-94, respectively.

2.2 Activated Carbon Cloth Preparation
Calico cloth (100 cm x 80 cm) was placed on a flat surface. The calico cloth was smeared truly with polyvinyl acetate glue prepared with a concentration of 80 wt% in ethanol. A grinder roller (1 meter long; 7 kg) was used to control the glue-layer thinness. The two type of activated carbon (granular; 80 mesh and powder; 100 mesh) was spread on the surface of glue and was flattened by a grinder roller. The activated carbon cloth (ACC) was dried until constant weight. The dried ACC was randomly sampled for quality control. The sample was weight before and after rotated in a rotary evaporator with 40 rpm for 30 minutes to identify carbon active lost during the friction test. ACC sample was also observed by a light microscope to determine the surface homogeneity of attached activated carbon particles.

2.3 Carbon Filter Prototype (CFP) Preparation
The stainless steel mosquito net was rolled up to form a cylindrical shape with a dimension of 11.4 cm x 40 cm. Dried ACC was cut with a dimension of 40 cm x 50 cm and was flipped (zigzag). The zigzag flipped ACC was inserted into the cylindrical net before closed with PVP pipe caps at both sides. The CFP was prepared for both granular and fine ACC, which six CPFs for each type, as shown in figure 1.

2.4 Ammonia adsorption test
Six plastic rooms with each dimension of 120 cm x 90 cm x 220 cm were prepared and all rooms were airtight. Several CFP was placed in those room with a variety of control (0, 1 calico cloth only, 1, 2 and 3 CPFs). A piece of round filter paper (Whatman, Ø 125 mm) was wetted with 4.5 ml concentric ammonia solution (density = 0.91g/ml, purity = 25%). Along with CFP, the filter paper was hung in the middle of the airtight plastic room. The temperature within the room was monitor by using a thermometer. At 30 minutes of the time interval, the ammonia concentration in the room was sampled by using 20 ml syringe that connected to a smooth plastic hose. The sample was kept in a 100 ml vial containing 50 mL water and a rubber stopper that make an airtight container. The samples were wrapped with wet cotton and kept in a Styrofoam box before UV-Vis analysis, as illustrated in figure 2.
Figure 1. CFP component; (a) flatted ACC, (b) flipped ACC, (c) inserted ACC in cylindrical net, (d) CFP.

Standard curves were prepared using the reaction of NH₄Cl with NaOH, as described previously [15]. Stock solutions of 0.8 M NaOH and 1.58 M (84.66g / L) NH₄Cl were prepared to dilute in several concentrations. For example, a mixture of 2 mL NH₄Cl + 4 mL NaOH + 19 mL water = 25 ml is equivalent to 1073.06 ppm NH₃. Dilutions are based on this calculation as well as for larger multiples of volume. Pipettes of 10 mL of standard solution were put into a test tube, added with 4 drops of Nessler's solution, shaken and let stand for 10 minutes. Enter the sample that has been given Nessler's solution into the cuvette and the adsorption of the solution is measured at a wavelength of 420 nm [16].

Figure 2. (a) schematic ammonia vapor adsorption, (b) schematic sampling & temperature recording, (c) plastic rooms for ammonia adsorption simulation
3. Result and Discussion
The mass of carbon produced from pyrolysis reaches 42.86% of the weight of dry palm shells. The yield was considered higher than the results of a previous study (Dada, Inyinbor & Oluyori, 2012). The powder activated carbon fulfilled the Indonesian National Standard (SNI) and ASTM except for the ash content parameter, which is higher and it is as measured in table 1, except for the ash content value, which is 31.12 wt%, slightly higher than the Indonesian National Standard, SNI (≤ 25 wt%). The activated carbon was discovered to have produced more micropore than other raw materials [17-18].

Porosity analysis of BET shows that the characteristics of activated charcoal from palm kernel shells, as shown in Figure 3. BET-surface-analysis adsorption-model tends to follow model III, which is a weak interaction and the adsorption model commonly occurs in graphite. The BET and surface areas are displayed in table 2, which are considered low compared to commercial water filter activated charcoal. Water filter activated carbon (Hexagon®) was made of coconut shells, which has higher adsorption capacity.

Table 1. Quality analysis of powder palm kernel activated carbon.

| Parameters                  | Data   |
|-----------------------------|--------|
| Water content (wt %)        | 5.7    |
| Ash content (wt %)          | 13.8   |
| Volatile content (wt %)     | 31.12  |
| Iodine adsorption (mg g⁻¹)  | 1133   |

Table 2. BET data analysis.

| Parameters                  | Data   |
|-----------------------------|--------|
| SinglePoint BET (m²g⁻¹)     | 10.13  |
| MultiPoint BET (m²g⁻¹)      | 12.94  |
| Langmiur area (m²g⁻¹)       | 49.6   |
| Average pore Radius (Å)     | 15446  |
| BJH adsorption;             |        |
| surface area (m²g⁻¹)        | 24.832 |
| Pore Volume (1.012 cc g⁻¹)  | 1.012  |
| Pore Radius Dv(r) (Å)       | 867.793|
| BJH desorption              |        |
| surface area (m²g⁻¹)        | 67.825 |
| Pore Volume (1.012 cc g⁻¹)  | 0.983  |
| Pore Radius Dv(r) (Å)       | 16.923 |

SEM image of activated carbon surface confirmed as porous material at 250x magnification. The porosity is distributed almost in the whole activated carbon surface, as displayed in figure 4.
Immobilization of activated carbon on a calico cloth to prepare activated carbon cloth (ACC) gave slightly different weight carbon stacked on the cloth depending on the combination of particle size and adhesive concentration. The stable-activated carbon cloth was obtained from the preparation of 120 mesh carbon particles and 80% (v/v) adhesive. This finding was resumed based on the weight loss during the friction test in the rotary evaporator, which is less than 10% carbon practice weight loss. The homogenous particle appearance from the microscope images, as shown in figure 5.

The ACC was prepared in large size, flipped up zigzag and inserted in the cylindrical net, which is named carbon filter prototype (CFP) as shown in Figure 4(a-d). The CFPs were placed in plastic rooms to adsorbed ammonia vapor a close room at ambient temperature (28±1°C) and 1 atm, as shown in Figure 2. Ammonia 4.5 mL concentrated ammonia was poured in filter paper hung in the middle of the closed rooms with the individual dimension of 120 cm x 90 cm x 220 cm (Figure 5a-c). The initial concentration of ammonia in the rooms was 2.8 x 10⁻⁵ M or equal to 0.5 ppm. This amount is considered low since ammonia smell can be detected at 5-53 ppm and the poison effect as indicated by
a slight irritation, is felt at a low level of 30 ppm and severe poisoning will be at 500 ppm (National Research Council, 2008).

The activated carbon mean-weight was (179±5) g in each CFP, equivalent to (0.1±0.003) g per cm² of ACC. As shown in Figure 6 (a & b), the optimum time of ammonia adsorption on CFP was 60 minutes, although sampling from the room was continued for several hours and the last sampling was 24 hours. The mean adsorption capacity of the carbon filter was 1.78 x 10⁻⁵ M per 1720 cm² carbon cloth. Each CFP carbon adsorbed 60.6% of ammonia vapor in the closed room, but the adsorption remained constant, although the absorbent and time have been expanded.

The effect of carbon particle size was also studied since preparing the finer size will have cost consequences. In fact, as shown in Figure 6 (a & b), the discrepancy in ammonia adsorption was not significant. The ammonia concentration reasonably caused the not obvious effect in very low, thereby not being longer detected with UV-Vis spectrophotometer. The ammonia detection limit by Nessler’s reagent was 0.02 mg/L-2.0 mg/L [19].

![Figure 6. Ammonia vapor adsorption on CFP made of powder and granular AAC.](image)

4. Conclusion
Activated carbon made of palm-kernel-shell was successfully prepared using a new pyrolysis chamber and the quality full filled the Indonesian Industrial Standard except the ash content, which was slightly higher. The structure was porous and the isothermal linear graph seems to follow type III, adsorption model. The preparation of PKS immobilization was affected by carbon particle size and adhesive concentration. The optimum time of adsorption was 60 minutes and the mean adsorption capacity of the carbon filter was 1.78 x 10⁻⁵ M per 1720 cm² of carbon cloth. The carbon filter prototype adsorbed 60.6% of ammonia vapor in the closed simulated room. The adsorption remained constant although the absorbent and time was expanded.
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