

SYNTHESIS OF CELLULOSE AEROGEL FROM KAPOK FIBER FOR CLEANING THE WASTE OF LUBRICANT OIL

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Abstract

Cellulose aerogel is known to be superior in removal of oil pollutants and organic solvent. This research developed the synthesis of cellulose aerogel from kapok fiber, application as an adsorbent for lubricating oil waste and its reusability. Chemical delignification was carried out by immersing the kapok fibers in NaOH 6% solution at 100°C for 1h to obtained a cellulose of 65.5% (SNI-2009). The effect of urea concentration on adsorbent products was studied in the range of 8-16w%. Whereas the effect of cellulose was studied at a concentration range of 0.5-1.5w%. The gelation stage was carried out by adding cellulose in a NaOH/urea solution accompanied by stirring (1000rpm) for 15minutes. The mixture was cooled at 0 °C for 48h to form a gel. The formed gel is dried by the freeze drying method at -45°C. The cellulose aerogel from kapok fiber with a density of 0.11g/cm³ and porosity of 92.46% had an absorption capacity of up to 11.987g/g in the first cycle. The use of each aerogel cellulose sample to absorb lubricant oil waste reached a maximum of three cycles.

Keywords : adsorbent ; aerogel ; cellulose ; kapok ; waste oil

INTRODUCTION

The conventional utilization of kapok in Indonesia is generally for the production of mattresses, pillows and other similar products. Along with the development of technology to satisfy consumers and also because people in Indonesia prefer spring beds, cotton mattresses are no longer produced in large quantities. On the other hand, kapok production in East Java reached 25,288tons/year (BPS, 2017). Kapok fiber basically has a high cellulose content of around 70-75% (Rojas, 2016). The high cellulose content showed the potential of kapok fiber as a cellulose material that can be used for wider applications such as absorbent, oil/water separation, thermal insulation, and biomedical applications, as well as many other fields. Cellulose is a renewable and biodegradable natural polymer (Long et al., 2018).

The cellulose can be used for preparation of aerogels, i.e. cellulosic I fibrils disintegrated from the cotton fiber, are very interesting as nano-sized building blocks due to their high aspect ratio, high specific surface area and very attractive mechanical properties. Microscopically, aerogels are composed of tenuous networks of clustered nanoparticles, and the materials often have unique properties, including very high strength-to-density and surface-area-to-volume ratios (Cervin et al., 2012). Aerogel, which has a three-dimensional pore structure and is very light, is

excellent in removing impurities in oil or in organic solvents (Cheng et al., 2017; Long et al., 2018; Yang & Cranston, 2014; Zhu et al., 2019). In addition, cellulose for the synthesis of aerogel from biological sources are also abundant, biodegradable, renewable, low cost and environmentally friendly (Shi et al., 2019; Zhu et al., 2019). Aerogel has large porosity, high elasticity, and also lightweight material which has a density of up to 0.004g/cm³ where the solid phase volume is only a few percent of the total volume (0.2-20%)(Nguyen et al., 2014).

The several methods have been developed for aerogel adsorbent synthesis. The porous cellulose aerogels were used as adsorbents to remove dye, oil, and organic solvents (Bao et al., 2009) because it is hydrophobic and oleophilic (Abdullah et al., 2010). Cellulose aerogel as an absorber of waste oil is generally prepared by the sol-gel method. There are several parameters which influence the hydrolysis and condensation reactions (sol-gel process), including the activity of the metal alkoxide, the water/alkoxide ratio, solution pH, temperature, nature of the solvent, and additive used. Modifications were developed related to coatings or templates for hydrophobic surface modification. Cellulose has complex intermolecules where hydrogen tissue bonds cause cellulose to not dissolve in water and organic solvents such as ethanol. On the other hand, cellulose macromolecules are amphiphilic, which are compounds that have a combination of hydrophilic and lipophilic properties

(Cervin et al., 2012). Hydrophobic repair is done by modifying the surface using two methods, namely physical and chemical coating methods. In the physical coating method, a commercial water repellent is used to spray into dry aerogel allowed to dry for one day at room temperature (Long et al., 2018). The chemical method is done by adding methyltrimethoxysilane or trimethylchlorosilan solvents to cellulose aerogel. Cold technology developed using trimethyl-chlorosilane (TMCS) as plasma for hydrophobic modification on the aerogel surface (Lin et al., 2015) or functionalized with methyltrimethoxysilane (MT-MS) to enhance its hydrophobicity and oleophilicity (Nguyen et al., 2013). Different solvent systems caused considerable differences in the properties of aerogel, such as internal surface area, morphology, porosity and mechanical stability (Pircher et al., 2016). Superhydrophobic and oleophilic oil sorbent was studied by the incorporation of silica nanoparticles onto kapok fiber via sol-gel method and subsequent hydrophobic modification using hydrolyzed dodecyltri methoxysilane (DTMS) (Wang et al., 2012). Cellulose from kapok fiber showed very good performance in its application to separate oil from water, with an increase in oil absorption capacity to 46.6% compared to raw fiber. This research was used kapok fiber for preparation of aerogel with sol gel method for lubricant oil waste adsorbent without modification using DTMS. Chemical modification of cellulose can produce hydrocolloid products, for example hydroxyalkylcellulose which is obtained through the reaction of cellulose with alkali, so that the hydroxy groups present in cellulose will be substituted by alkyl groups. Hydrocolloid is a polymer that is soluble in water, capable of forming colloids and able to thicken a solution or form a gel from that solution (Zhu et al., 2019). The sol-gel method is carried out by reacting cellulose with alkali, so that the hydroxy groups present in cellulose will be substituted by alkyl groups to produced hydroxy-alkylcellulose.

The regenerated cellulose treatment process is generally through a viscose process that uses a mixture of carbon disulfide and sodium hydroxide (NaOH) as the solvent. The viscose process is not preferred because it produces toxic waste that is difficult to treat, thus potentially polluting the environment. The cellulose dissolution method using NaOH and urea developed by Zhang et al (2014) is claimed to be less toxic and more economical than the viscose process. The presence of urea as a support for NaOH hydrates to penetrate into the cellulose crystal region and cause an increase in the dissolved cellulose fraction. This condition causes the cellulose molecules to close to each other so that the porosity is smaller when dried. NaOH/urea aqueous solutions (Qin et al., 2013) were proved to be a stable and more homogeneous reaction medium for preparing cellulose ether

with a more uniform microstructure (Zhou et al., 2004). The hemicelluloses, precipitated in ethanol after an acidification step had a very high brightness and low lignin content compared with the hemicelluloses obtained by the direct precipitation in ethanol without the acidification (Z. Liu et al., 2011). Associated with cellulose and urea that form cross-linked, soaking in ethanol can support the coagulation process that causes cellulose to bind to one another.

The synthesis of cellulose aerogel followed several important steps which include preparation of cellulose fibers, cross cellulose chains to obtain polymer tissue, and drying methods. Aerogel is a sol-gel product which is dried to avoid collapse of pores where in the material it contains 90-99% air so it has high porosity. High porosity in cellulose aerogel is expected to be an absorbent with good absorption power. To minimize pore collapse, the drying method used is freeze drying. The freeze drying method is carried out by freezing samples whose ambient pressure is reduced such that it allows frozen water in the sample to be sublimated directly from the solid phase to the gas phase, creating minimal strength in the pore walls in the air thereby preventing the porous structure from collapsing (Pircher et al., 2016)(Pierre, 2011). An ideal absorbent is expected to possess advantages of low cost, green, high absorption capacity and excellent reusability (Bao et al., 2009; Li et al., 2019; Lin et al., 2015).

This research aims to study the effect of cellulose concentration and urea concentration on the absorption capacity of cellulose aerogel adsorbent from kapok fiber and to determine its reusability. The application of cellulose aerogel as an adsorbent to remove oil pollutants was generally carried out for oil spills or organic materials, in this research the adsorption of lubricant waste would be carried out in order to measure the power and adsorption capacity of cellulose aerogel. Nguyen et al. (2014), used a type of cooking oil and lubricating oil with a temperature of 70°C which has a lower viscosity than a dirty lubricant oil with a room temperature of around 30°C used in this study. The performance of cellulose aerogel adsorbent is related to the density, viscosity, and surface tension of the oily liquid to be adsorbed. Thus, the aerogel morphology, namely the pore structure, density, and surface moisture should be of concern. Reusability of the oil absorbent was a critical criterion for its potential pilot-scale application (H. Liu et al., 2017; Shi et al., 2019). In this study, lubricant oil waste was selected as a model adsorbate to determine the circulating life of cellulose aerogel from kapok fiber.

RESEARH METHODS

Material

Kapok fiber as the main raw material for the preparation of cellulose aerogel was obtained from

Putri Bahama Kapok Natural Store, Kedung Asem No.4 Surabaya. Kapok fiber was crushed to around 150mesh size and then cellulose content was analyzed according to SNI 2009. Chemicals such as NaOH, Na₂S₂O₃, H₂SO₄, Ethanol 99% were obtained from the World Chemistry Store. KI 1N and KMnO₄ from Indo Kimia store, Jalan Tidar 278, Surabaya. Lubricant oil waste was obtained from a motorcycle repair shop.

Equipment

Magnetic stirrer, freezer, oven, freeze dryer

Procedure

Preparation of kapok fiber

The delignification stage was carried out physically and chemically. Physically, kapok fiber was crushed to a small size then analyze cellulose-lignin by the Chesson Method. The increased concentration of NaOH tend to increase the level of lignin decomposition. Lignin decomposition reaches 9.53% with 6.0% NaOH concentration (Permatasari et al., 2014). Whereas chemical delignification was done by soaking kapok fiber into a 6% NaOH solution with a kapok fiber ratio of 1:10. The treatment is carried out for 1 h at a temperature of 100°C then the pulp was washed with demineralized water and dried. Dry pulp was weighed for further analysis of the Kappa Number to determine the level of lignin remaining in the pulp. Cellulose content in kapok fiber from the analysis results obtained 65.5%.

Preparation of cellulose aerogel

Gelation

The gelation stage was carried out by mixing samples with variations in the concentration of cellulose 0.5; 0.75; 1; 1.25; 1.5% (w/v) in NaOH/urea solution with a variation in urea concentration of 8; 10; 12; 14; 16%. (wt%) in beaker glass. The mixture was stirred using a magnetic stirrer for approximately 15minutes at a speed of 1000rpm to get a homogeneous mixture. The mixture was cooled at 0°C for 48h to form a gel. The gel formed is left in the open air until it reaches room temperature.

Coagulation

The coagulation step was carried out by soaking the gel in 10ml of 99% ethanol for 24h. Soaking the gel in ethanol solvent aims to support the coagulation process in which cellulose crystals bind to each other. Gel samples containing ethanol were put in a beaker glass for the solvent exchange process by adding demineralized water. The ethanol trapped in the gel is replaced by demineralized water. The immersion process was carried out for 48h. Furthermore, the sample was frozen at -15°C for 12h and then carried out the drying stage (Siebert et al., 2018) with a freeze dryer at -45°C and a pressure of around 17Pa for the formation of aerogel.

Coating

The aerogel cellulose coating stage was carried out by spraying a commercial water repellent spray ReviveX® Nubuck which was used to spray into a dry aerogel and then allowed to dry for one day at room temperature. The use of water repellent spray was controlled so that no lacking or no excessive. If the amount of water repellent spray was lacking, the hydrophobicity of cellulose aerogel was also bad, whereas if used excessively could cause aerogel cellulose absorption of oil to be reduced, because it is coated by water repellent spray that can cover the pores of cellulose aerogel. Related to this case, in this study the spray distance to the sample is set at around 15cm (Nguyen et al., 2013).

Characterization

The characteristics of cellulose aerogel were examined for their porosity and density. The application of cellulose aerogel as an absorbent for lube oil was carried out by determining the amount of absorption capacity. The first step of aerogel cellulose is weighed first to get its initial mass. Next, the cellulose aerogel was immersed in oil for about 1 hour. Cellulose aerogel that has been submerged and then removed and weighed again. The next step of cellulose aerogel was squeezed slowly until no drip oil from cellulose aerogel again. The step was repeated until the effectiveness of reusing cellulose aerogel is obtained.

Density and Porosity

The density of cellulose aerogel is known by using the equation (Wang et al., 2012) :

$$\rho = m / v \quad (1)$$

where m is the sample mass (g) and V is the sample volume (cm³). While the aerogel porosity is calculated, based on the saturation (or imbibition) method (Anovitz & Cole, 2015), by equation:

$$P = \left[1 - \left(\frac{\rho}{\rho_c} \right) \right] \times 100\% \quad (2)$$

where ρ_c is the bulk cellulose of density from literature which is 1.528g/cm³ (Shi et al., 2019).

Oil absorption capacity

The oil absorption capacity method is calculated using the formula (Liu, 2016) :

$$Q = \frac{m - m_o}{m_o} \quad (3)$$

where Q_t (g/g) is the oil absorption capacity of the aerogel at a certain time t (min), m_o (g) is the weight of the aerogel before absorption, and m (g) is the weight of the aerogel after absorption.

Reusability

To test the reusability of aerogel was done by immersing the aerogel in an organic solvent (ethanol)

and measuring its weight before and after drying. The testing procedure was repeated several times.

Reusability in the adsorption operation, cellulose aerogel was dried in an oven vacuum at 50°C after the adsorption cycle (Lin et al., 2015). After the adsorption process was complete the adsorbent was squeezed by pressing to remove the oil from the aerogel adsorbent.

RESULT AND DISCUSSION

Cellulose aerogel products from kapok fiber are physically cylindrical, white-yellowish with pores visible on its surface. Figure 1 showed the aerogel cellulose products prepared with five different kapok cellulose concentrations. The use of cellulose concentration of 0.5% has small and fine pores in large numbers while the concentration of cellulose 1.5% has large pores but the number of pores is small.

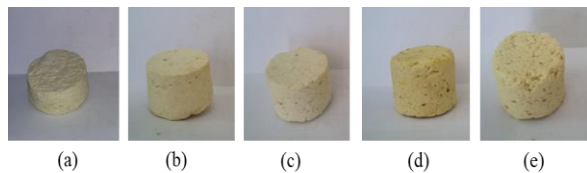


Figure 1. Cellulose aerogel from kapok fibers prepared by cellulose concentration of (a) 0.5 (b) 0.75; (c) 1 (d) 1.25 (e) 1.5% at urea concentration of 12%.

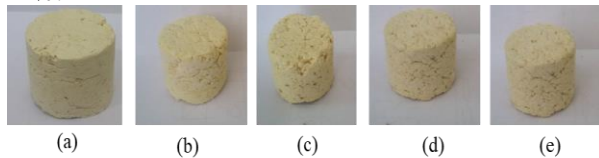


Figure 2. Cellulose aerogel from kapok fibers prepared by urea concentration of (a) 8 (b) 10 (c) 12 (d) 14 (e) 16% at cellulose concentration of 1.5%

To improve hydrophobicity of cellulose aerogel, surface modification was needed through physical coating using water repellent. Water repellent was sprayed throughout the aerogel surface and then allowed to dry at room temperature. To ensure the hydrophobicity of cellulose aerogel, it was tested by dripping water on its surface. Cellulose aerogel refused to absorb water after coating using water repellents as indicated by the formation of water droplets on the surface of the aerogel. It can be state that cellulose aerogel have been hydrophobic.

Water repellent contained alkoxy silane (SiH_4) which reacted with hydroxy groups from cellulose. This physical coating also caused a decrease in bonds between the C and O atoms. When the transformation of cellulose formed hydrophilic to hydrophobic, trimethyl-silane radicals replace hydrogen hydroxyl groups and are connected to the cellulose chain with C-O-Si bonds (J. Liu et al., 2016).

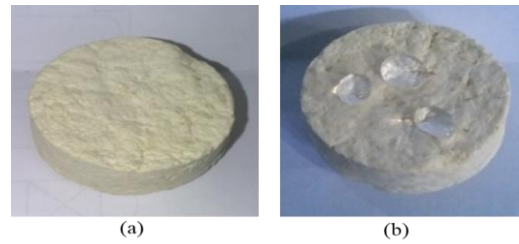


Figure 3. Cellulose aerogel from kapok fibers prepared by (a) without coating, and (b) coating with water repellent

Effect of cellulose and urea concentration on the aerogel density

Figure 4. showed the effect of cellulose and urea concentration on aerogel density. Density of cellulose aerogel increases with increasing concentrations of urea and cellulose. The results showed that the density of cellulose aerogel is relatively small in the range of 0.1152-0.1494 gr/cm^3 , which proves that the aerogel prepared from cotton fiber is mild.

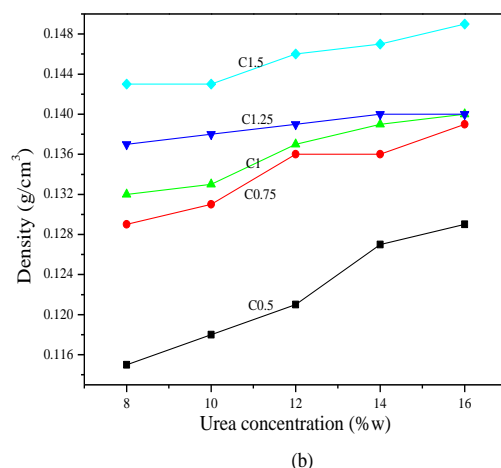
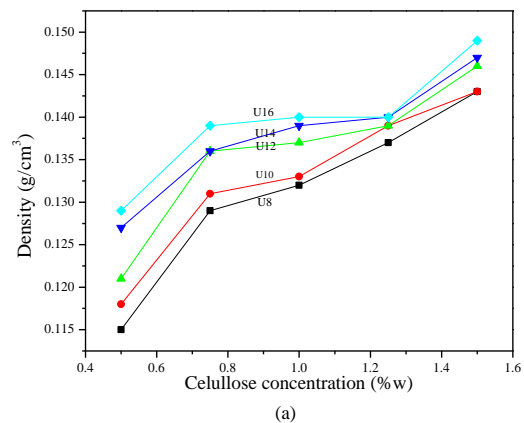


Figure 4. The effect of a) cellulose concentration and b) urea concentration on aerogel density

The large number of pores formed during the drying process caused cellulose aerogel to have a light mass. In addition, the high concentration of cellulose also influenced the density of the aerogel cellulose.

This was consistent with Yang's report (2014), which showed that the greater the concentration of cellulose, the greater the aerogel mass obtained, so that the resulting density will also be greater.

The aerogel porosity is the ratio between the volume of the cavity to the volume of aerogel cellulose. Almost all concentrations of porosity were greater than 90%, which means that the synthesized cellulose aerogel had a relatively large pore volume of around 90%, which is almost the same as the aerogel volume. The highest porosity value was 92.459% in the sample cellulose concentration: urea 0.5: 8%, while the lowest porosity was 90.22% with cellulose concentration: urea 1.5: 16%.

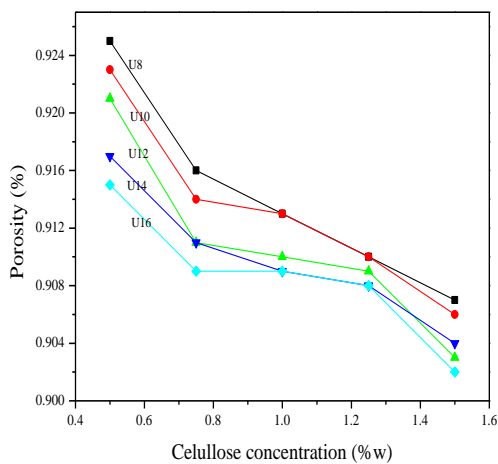


Figure 5. The effect of cellulose concentration on aerogel porosity at urea concentrations in the range of 8 to 16 %.

Porosity decreased with increasing concentrations of urea and cellulose in each sample. In this case urea played a role in supporting NaOH in order to penetrate into the cellulose crystals and then react to form hydroxyalkylcellulose. Figure 5. showed the effect of cellulose concentration on aerogel porosity. comparison of each variable of cellulose concentration to porosity obtained that the cellulose concentration of 0.5% had a higher porosity, this means that cellulose aerogel formed had large amount of porosity.

Adsorption Capacity

It had been described previously that the absorption capacity of the aerogel is quantitatively affected by the concentration of cellulose, the composition of the crosslinker agent (NaOH/Urea), and the type of oil used. If the concentration of cellulose in the solution increases, the possibility of dissolved cellulose fraction also increases so that it will form smaller porosity. The composition of NaOH/Urea affected the gelation stage, if the concentration of urea in the solution increases, the gelation time would be faster so that small pores were formed in the aerogel. Another factor that also plays a role was the use of water repellent to improve the hydrophobicity of cellulose aerogel. Figure 6. showed effect of a) cellulose and b) urea concentration on

adsorption capacity and density of aerogel. Adsorption capacity tend to decrease with increasing concentrations of urea and cellulose. For the first cycle, the largest absorption capacity was obtained at cellulose concentration: urea (0.5: 8)% which was 11.987g/g, while for the smallest capacity was 4.847g/g at (1.5: 16)%. The absorption ability of the aerogel was relatively small when compared with other studied report of cellulose aerogel (Nguyen, 2014) where the absorbing capacity ranges from 18-20g of absorbed oil/g cellulose to dry aerogel and even then it may be due to the different types of oil used as spill oil and new lubricant oil

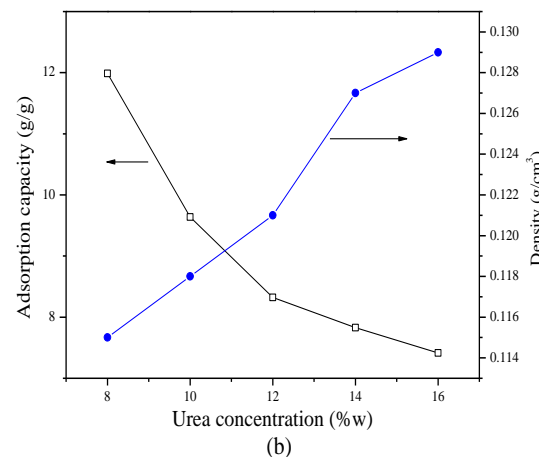
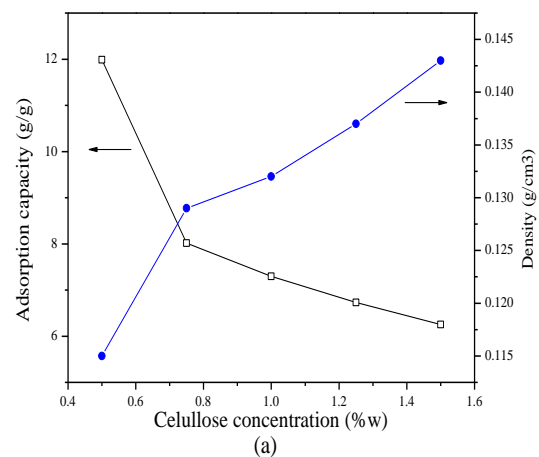


Figure 6. The effect of a) Cellulose and b) Urea concentration on adsorption capacity and density

Reusability

The reuse of aerogel cellulose adsorbents was carried out several times. Figure 7. showed the correlation of a) cellulose and b) urea concentration with adsorption operation in three cycles. The aerogel adsorbent had damaged and collapsed in its pore structure after being used in the third cycle. So that the reuse of the adsorbent after going through three adsorption cycles becomes less effective because, in addition to reducing the absorption capacity, the adsorption capacity between the second and third

cycles is relatively unchanged. Cellulose aerogel which is prepared with 0.5% cellulose concentration in the first cycle as oil adsorbent produces an adsorption capacity of 11.987g/g while in the second cycles the adsorption operation has a capacity of only 7.48g/g. There was a decrease of 4.5g or about 38% of the adsorption capacity in the first cycle. Cellulose aerogel in these conditions has the highest porosity of 92.459%. This showed that the higher the porosity of cellulose aerogel, the higher the absorption capacity.

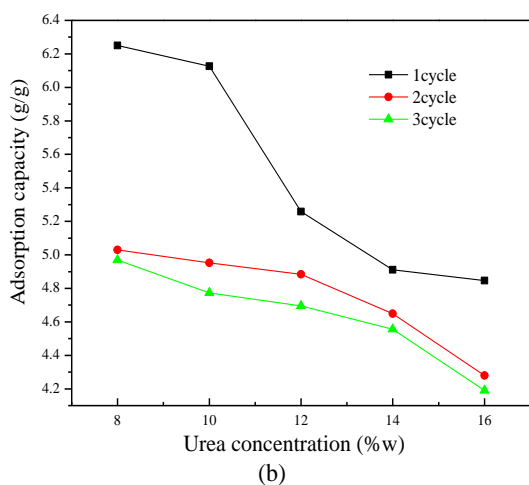
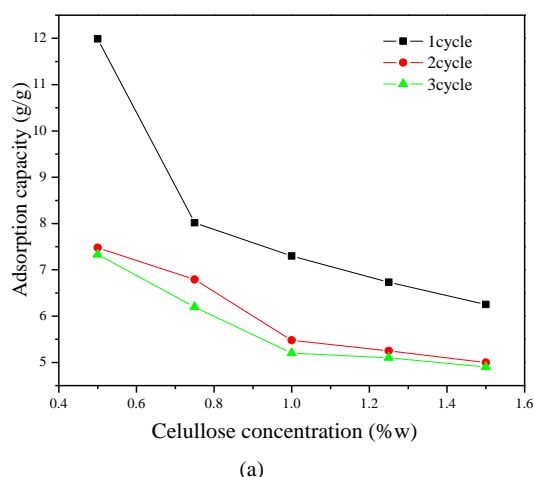


Figure 7. The correlation of a) Cellulose and b) Urea concentration on adsorption capacity in the three adsorbent reuse cycles

While the use of aerogel adsorbents from the second cycle to the third cycle the damage is only about 1.94%. On the other hands aerogel cellulose adsorbents were prepared with 1.5% cellulose concentration, the decrease in capacity from the first cycle to the second cycle is only about 12%, with a smaller number of aerogel pores so that the tendency to maintain the pore structure when squeezing the treatment is greater. The reusability observation results in this experiment showed that adsorbent disinter-gration was more influenced by the extortion power compared to the composition of cellulose urea.

CONCLUSION

Cellulose aerogel from kapok fiber used as absorbent had been successfully synthesized with a light mass, cylindrical, and yellowish white the aerogel density was higher with increasing cellulose and urea concentrations in the gel cross-linket, it was inversely proportional to its porosity. Aerogel cellulose with the highest porosity in this study was 92,459% with a ratio of cellulose: urea concentration (0.5: 8 w%), and absorption capacity reaching 11,987g/g for 1h. Cellulose aerogel from kapok fiber have the ability to reusable three times with a decrease in the value of absorbing capacity per cycle.

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