Fabrication Of Carbon Aerogels From Coir For Oil Adsorption

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Abstract. Coir, known as coconut fibers, are an abundant cellulosic source in Vietnam, which are mostly discarded when copra and coconut water are taken, causing environmental pollution and waste of potential biomass. In this research, carbon aerogels from chemically pretreated coir were successfully synthesized via simple sol-gel process with NaOH-urea solution, economical freeze-drying, and carbonization. The samples, including pretreated coir, coir aerogels, and carbon aerogels, are characterized using FTIR spectroscopy, SEM, XRD spectroscopy, and TGA. The carbon aerogels exhibit low density (0.034–0.047 g/cm^3), high porosity (97.63–98.32 %), and comparable motor oil sorption capacity (22.71 g/g). The properties of carbon aerogels are compared with those of coir aerogels, indicating such better values than those of coir aerogels. Coir-derived carbon aerogels is a potential replacement for the hydrophobically-coated cellulose aerogels in term of treating oil spills.

1. Introduction

Coconut is considered the tree of life that nearly all parts of the plant are valuable in industry [1]. The tree of life is grown in over 90 nations, especially in Asian and Pacific regions [1]. In Vietnam, coconuts are among perennial crops with large harvesting area. Nowadays, the total area under coconut is about 175,000 ha, in which Mekong Delta accounts for roughly 80 % of the total area [2]. The coconut husks, which are the source to obtain coir, are mostly wasted [2] when coconut water and copra are already taken. The husks are burnt, left on the sea, or landfill, causing environmental pollution. To address the problem, coir can be extracted from the husks to be further applied in industry. Coir have many applications; they can be used in reinforcing composite materials in construction [3,4], in environmental remediation [3,5], or in biotechnology [3,6]. Its composition is presented in table 1. Among the solutions, the transformation of coir to aerogels to be utilized in adsorption of liquids is a considerable option [3,7–9].
Oil spillage which occurs frequently every year is a part of the petroleum exploration, production and transportation business [8,9]. Oil-contaminated water will bring about extremely harsh hazards to the environment and to public health. Among the existing technologies (e.g. containment, skimming, microorganism utilizing [8]), adsorption is considered to be greatly effective for the removal of oils and organic pollutants from water [8,9,11] as other aforementioned methods are reviewed to be ineffective [8]. Traditional sorbents are polyethylene or polypropylene, which are proved to treat the oil well; however, they are controversial of its plastic content and non-biodegradability [8].

Aerogel is a gas-filled porous solid network of bonded particles, fibers, or both, exhibiting very low density, low porosity, and high specific surface area [12]. Since about 40 years after the introduction, it has been massively studied and developed because of its wide ranges of useful applications [13]. High specific surface area helps the aerogel be a perfect sorbents for metal ions, oil, organic liquids etc. [12]. Many reports have shown cellulose aerogels and carbon aerogels are those chosen one [14,15]. As the awareness about green products and sustainability draws such interest, agricultural waste has risen to be a promising candidate to produce the aerogels. A number of reports have shown such optimistic views towards the transformation of the waste into aerogels with good adsorption ability [5,11,15–18]. In 2014, Li et al. [16] converted winter melon directly into carbon aerogels for adsorption of oils and organic solvents. The materials had low density of 0.048 g/cm³, high water contact angle of 135°, and can adsorb organic liquids at 16–50 times of its own weight. Later, Jing et al. [11] was able to fabricate biomass carbon aerogel using corn bracts. The product was applied in selective oil/water separation with high specific surface area (675.9 m²/g), superhydrophobicity (water contact angle > 150°), and high sorption capacity (77.7–143.6 g/g) towards toluene, hexane, soybean, engine oil, diesel etc.

In order to make the best use of potential waste coir, this research aims to fabricate carbon aerogels from waste coir via a facile synthesis process involving pretreatment, sol-gel process, freeze-drying and carbonization. The related samples were characterized, evaluated, as well as compared with each other to comprehend the study. Previous studies also attempted the carbonization of coir-based cellulose aerogel; however, the approach to oil adsorption application was not mentioned. Fauziyah et al. [5,7] successfully made cellulose aerogels from coir for oil and methylene blue treatment. The group later developed cellulose to carbon aerogels to research in the catalytic field but environmental remediation [19]. This paper studies the adsorption ability of coir-based carbon aerogel towards oil, giving a new solution to address the oil spillage.

2. Experimental section

2.1. Materials

Coir is purchased in Tien Giang Province, Vietnam. Sodium hydroxide (NaOH), urea, and hydrogen peroxide (H₂O₂) are purchased from China. Ethanol (EtOH), and motor oil 5w30 are purchased from Vietnam. All solutions are made with deionized water.

2.2. Experiment

The first stage is the pretreatment of original coir (OC). OC is firstly blended into smaller pieces (1–3 cm). Secondly, the fibers were treated with alkali solutions. The fibers and NaOH 3% solution are stirred in a chamber at 80°C. After that, delignified coir (DC) is obtained. The fibers are further bleached with 1% NaOH-10% H₂O₂ solution. When bleaching was finished, the coir was washed till pH 7 and dried to get bleached coir (BC). To assist the preparation of aerogels, BC are blended one more time into smaller fibers (below 5 mm).

The second stage comes the synthesis of coir aerogels and carbon aerogels. Foremost, the pretreated coir is taken with desired amount (0.5 g, 0.75 g, 1 g, 1.25 g, and 1.5 g) then dispersed and dissolved in
25 mL of the solution of 7:12:81 wt% of NaOH:urea:water by a magnetic stirrer (2000 rpm). After 30 minutes, the suspension is put in the freezer for a day. The frozen gel was then thawed for 20 min at room temperature before EtOH is gently introduced to the suspension for gelation. The mixture is left for a day. Subsequently, the coir gel was washed with deionized water for several times until pH of the last liquid is 7, then, freeze-dried to obtain coir aerogel (DA). To make carbon aerogel (CA), DA is inserted into a tubular furnace of the carbonization equipment. The carbonization is conducted at 800°C with a heating rate of 5°C/min, and 100 mL/min of N2 flow.

2.3. Characterization

The density of the aerogels is measured by mass divided by volume with the measurement of the volume of a cylinder. The porosity of the aerogels ($\psi$) is determined by equation (1) based on their density ($\rho_a$) and density of cellulose fibers ($\rho_b = 1.528$ g/cm$^3$ [5]), or density of carbon ($\rho_b = 2$ g/cm$^3$ [16])

$$\rho = \left(1 - \frac{\rho_a}{\rho_c}\right) \times 100\%$$

(1)

The surface area of carbon aerogels was determined by nitrogen physisorption measurements, performed by BET Sorptometer. Scanning electron microscopy (SEM) was performed by FE-SEM S4800 Hitachi to study the morphology of the aerogels. XRD spectroscopy was performed by D8 Advance. The Cu-Kα radiation source ($\lambda = 0.1541$ nm) was applied. The scanning step was 0.02° with the rate of 0.6 °/min in the 2θ range from 5° to 80°. Crystallinity index (CI) is determined by the method of Segal et al. [20] via equation (2), where $I_{002}$ is the intensity of the peak at 2θ around 22° (for cellulose I), or the intensity of the peak around 16° (for cellulose II), and $I_{am}$ is the intensity of the peak at 2θ around 18° (for cellulose I), or the weakest intensity of between the peaks 18° and 22° (for cellulose II), both have the same arbitrary unit.

$$CI = \frac{I_{002} - I_{am}}{I_{002}} \times 100\%$$

(2)

FTIR spectra of the samples were generated by MIR/NIR Frontier. The spectroscopy was performed in the range from 400 to 4,000 cm$^{-1}$ with a scanning speed of 1 cm$^{-1}$s$^{-1}$. TGA was performed by TGA/DSC 3+ thermal analyzer. The aerogels were heated up in from room temperature to 900°C at a rate of 5°C/min in N2. The maximum oil adsorption capacity of the aerogels is calculated using equation (3).

$$Q_m = \frac{m_f - m_i}{m_i}$$

(3)

where $Q_m$ is the maximum oil adsorption capacity of the aerogel (g/g), $m_i$ (g) and $m_f$ (g) are the aerogel weights before and after the oil adsorption test. To model the adsorption kinetics, two common pseudo-first and pseudo-second order models, represented by equation (4) and (5), are used.

$$\ln\left(\frac{Q_m}{Q_m - Q_t}\right) = k_1t$$

(4)

$$\frac{1}{Q_t} = \frac{1}{Q_m k_2} + \frac{t}{Q_m}$$

(5)

where $Q_t$ are the oil adsorption capacity of the aerogel (g/g) at time t (s), respectively; $k_1$, and $k_2$ is the rate constant of the pseudo-first, and pseudo-second order model, determined from least-squares method for the graph.

3. Results and discussion

3.1. Morphology, density and porosity

DA is well-formed with sol-gel process and freeze-drying method, without any structural collapse. After pyrolysis, the three-dimensional solid structure is still maintained, and the mass reduction in the carbonization step, from coir to carbon, is about 78 %, while the volume reduction is determined to be...
around 70%. Figure 1 shows the morphology and structure of DA and CA while figure 2 illustrates the density and porosity of them. The images show that DA have porous structure with randomly arranged fibers, and the fibers are 11.0–15.0 μm in term of diameter. After carbonized, the aerogels still keep the well-built structure but the size of the fibers is reduced to 4.8–8.6 μm. This is because of the loss of chemical elements when cellulose was pyrolyzed. There are also layers at some part of the fibers, as noted in figure 1(b) and 1(f). It seems they are the result of the pretreatment process, in which they outer skin of the fibers were partially destroyed; or the result of the swelling and regeneration of cellulose in sol-gel process with NaOH-urea solution. With increasing coir content, it can be seen that the fibers occupy more space and stay closer to one another in the whole volume of the aerogel. It is discernible in the images of both coir and carbon samples, and corresponds well to the trend of density and porosity of the aerogels.

![Figure 1. FE-SEM images of the coir aerogels at various fiber contents: (a) 0.5 g, (b) 1 g, and (c) 1.5 g; and the corresponding carbon aerogels: (d) 0.5 g, (e) 1 g, and (f) 1.5 g](image)

![Figure 2. Density and porosity of coir aerogels and carbon aerogels.](image)

When it comes to the density and porosity, DA possesses low density of 0.040–0.063 g/cm³, and high porosity of 95.86–97.40 % while those values for CA are 0.034–0.047 g/cm³, and 97.63–98.32 %, respectively. CA has lower density than DA does, which is relevant to the basic difference between the two types of aerogels. Both of them witness the same trend regarding the properties that if more fibers
are added, the density will be increased but the porosity will be decreased. The specific surface area of the carbon aerogel is representatively measured for the sample of 0.5 g of fibers. The result shows the value of 289.4 m$^2$/g, which is higher than coir carbon aerogel (150.85 m$^2$/g) [19], green-leaf carbon aerogel (47.28 m$^2$/g) [18].

### 3.2. XRD and FTIR spectra

Chemical composition and its change in each pretreatment step are analyzed by XRD and FTIR spectroscopy. XRD spectra are illustrated in figure 3. It can be seen that the first three spectra [figure 3(a–c)] show the characteristic peaks of cellulose I, at 16.3°, 21.9°, and 34.0° [5,18,21,22], indicating that the cellulosic structure of the fibers is not changed after the pretreatment [21]. Besides, it is known that hemicellulose and lignin are in amorphous form in nature but cellulose is both crystalline and amorphous [21]. Therefore, changes in crystallinity of the coir can also attribute to changes in their chemical composition. Those characteristic peaks of DC and BC are sharper and clearer than that of OC, especially 15.0° and 21.9°, implying the cellulose content in pretreated coir is improved [21]. Furthermore, the crystallinity indices of the coir are shown in table 2. The increase in CI in the coir after each pretreatment stage shows the progressive elimination of the non-crystalline contents, hemicellulose and lignin [21,22]. The spectrum of DA shows characteristic peaks of cellulose II at 12.0°, 20.6°, and 22.3°, which are distinguished from the those of cellulose I [5,19,23]. The bleached fibers have CI of 57.2, which decreases after dissolution with NaOH-urea to 15.2. This might be caused by the crosslinking of cellulose [5]. So the sol-gel process of pretreated coir and NaOH-urea solution is confirmed to be successful. After carbonization, it can be observed most of the peaks of cellulose are weakened and broadened, or disappeared [figure 3(e)]. The broad bands from 20° to 25° and from 40° to 45° attribute to the existence of amorphous carbon [18,19]. This means the cellulose structure was destroyed and amorphous carbon formed [18].

![Figure 3. XRD spectra of (a) OC, (b) DC, (c) BC, (d) DA, and (e) CA.](image)

![Figure 4. FTIR spectra of (a) OC, (b) DC, (c) BC, (d) DA, and (e) CA.](image)

| Sample           | Crystallinity index |
|------------------|---------------------|
| (a) Original coir| 47.0                |
| (b) Delignified coir | 51.1               |
| (c) Bleached coir | 57.2                |
| (d) Coir aerogel  | 15.2                |
The chemical composition of the aerogels is again confirmed by FTIR spectra (figure 4). It can be observed that the first four spectra [figure 4(a–d)] present the existence of cellulose as typical peaks. The bands at around 3500, 2900, 2080, 1650 and 1450, 1080, and 890 cm\(^{-1}\) are those characterize FTIR spectrum of cellulose [18,21,22,24]. In term of the pretreatment of coir, the band at 1750 cm\(^{-1}\), corresponding to lignin [21], and 1510 cm\(^{-1}\), corresponding to hemicellulose [22], of OC are observed to be shortened in that of DC and OC. The characteristic bands of cellulose at roughly 3500, 2900, 2080 cm\(^{-1}\) become clearer in the spectra of the treated coir than in that of the untreated one. Also, the spectrum of DC and BC result in the reduction of the intensity of the peaks from about 1610 cm\(^{-1}\) to 1100 cm\(^{-1}\) and improved the peak at 1726 cm\(^{-1}\), weakening the peak at 1033 cm\(^{-1}\) (aromatic C–H of lignin [24]). This may be due to breaking of some α–ether interlinks of lignin [24]. These features confirm the removal of lignin and hemicellulose and the increase of cellulose constituent in the fibers. Besides, figure 4(c) shows the change of the group of peaks 1610 to 1100 cm\(^{-1}\) more clearly, thus it demonstrates that lignin was more removed in BC than in DC. Figure 4(d) shows additional peaks at 3314 cm\(^{-1}\) (presenting the N–H stretching [19]), and 1644 cm\(^{-1}\) (indicating the functional group of urea, RHN–CO–NHR [5]). It can be implied that NaOH-urea solution contributes, to an extent, to the physical and chemical change in the structure of coir. From figure 4(e) of CA, it can be seen that all of the bands of cellulose are significantly weakened or disappear, especially 3500, 2900, 1080, and 890 cm\(^{-1}\). Some characteristic bands of carbon are detected, which are 1725 and 1580 cm\(^{-1}\) (C=C in aromatic rings), 1225 cm\(^{-1}\) (C−O), 925 and 755 cm\(^{-1}\) (C−H) [19]. This attributes to the fact that cellulose decomposed and was transformed into carbon through pyrolysis [22]. Furthermore, the color change from light yellow to black also illustrates coir were completely pyrolyzed to form carbon.

3.3. TGA diagrams

Thermal behaviors of the coir and aerogels are studied with TGA. The thermograms are shown in figure 5(a–d), including OC, DC, BC, DA. All of the samples are observed likely the same trend of decomposition. They lose small quantity of moisture till 110\(^{\circ}\)C, degraded substantially from 250\(^{\circ}\)C to 400\(^{\circ}\)C, in which hemicellulose, cellulose, and lignin are decomposed [5,21]; then, level off gradually. The moisture of the samples is observed to be 5–8 %. It is clearly witnessed that the substantial degradation of OC occurs more slowly and ends at a higher temperature, in which DC, BC, and DA degraded quickly from 250–355\(^{\circ}\)C whereas the temperature range of OC was determined to be 260–400\(^{\circ}\)C. It is because of the higher amount of lignin in the fibers [5]. Lignin has complex structure and needs higher temperature (250–700\(^{\circ}\)C [21]) to be decomposed than hemicellulose (220–300\(^{\circ}\)C [21]) and cellulose (275–400\(^{\circ}\)C [21]). Char content of the samples ranges from 18 \% to 24 \%. Figure 5(d) also indicates that if coir aerogels are carbonized at 800\(^{\circ}\)C, the mass of resulting carbon aerogels equal to about 20 \% of the original coir aerogels, implying a mass reduction of 80 \%.

![Figure 5. TGA curves of (a) OC, (b) DC, (c) BC, and (d) DA.](image-url)
3.4. Oil adsorption test

Figure 6 illustrates the oil sorption of the aerogels with respect to time. DA and CA of 0.5 g of fibers were used to test the ability because they have high porosity and low density among the studied range. It can be observed from the curves that both kinds of the aerogels adsorbed the oil very quickly at the first 10 s, then, reached the equilibrium stage of adsorption after about 20–40 s. Owing to the highly porous structure of the aerogels, the oil is quickly transferred into the interior of the fibers via capillary force [25]. The maximum oil capacity of coir aerogel is relatively high, 15.20 g/g. This is due to the abundance of air space inside the aerogel, which are for the oil to be captured within. The maximum oil capacity of carbon aerogel is 22.71 g/g, which is comparative to 16 g/g of coffee-cotton aerogel [17], 20.5 g/g of paper-waste cellulose aerogel [9]. It is larger than that of coir sample, which can be explained by the larger porosity and high specific surface area, as the two factors that significantly contribute to the sorption ability [12]. In practice, cellulose aerogel has to be hydrophobically coated to perform well in oil-water mixture, which was time-consuming (up to a day [17]) and costly (e.g., the price of MTMS, HDMS). But with carbonization, the fabrication time and cost can be saved to an extent.

Figure 6. Oil adsorption kinetics of coir aerogel and carbon aerogel.

The analysis of pseudo-first order and pseudo-second order kinetic model is depicted in table 3. The $R^2$ values for both pseudo-first order and pseudo-second order models are also calculated. Since the $R^2$ values for the pseudo-second order model is closer to 1 than those of the pseudo-first order, it can be concluded that the oil adsorption kinetics of the both coir aerogels and carbon aerogels follows pseudo-second order model more accurately.

Table 3. Data of pseudo-first order and pseudo-second order kinetic models.

|                      | Coir aerogel | Carbon aerogel |
|----------------------|--------------|----------------|
| Pseudo-first order model | $R^2$        | 0.3607         | 0.1194         |
|                      | $k_1$        | 0.0002         | 0.0001         |
| Pseudo-second order model | $R^2$  | 0.9999         | 0.9999         |
|                      | $k_2$        | 1.6554         | 1.2193         |

4. Conclusion

The pretreatment of coir is evaluated to be relatively successful with the increase of cellulose constituent in the fibers, displayed in the FTIR, and XRD spectra. Carbon aerogels were successfully fabricated from pretreated coir via sol-gel process, freeze-drying, and carbonization. They can maintain the porous structure of DA after carbonization. CA have low density of 0.034–0.047 g/cm$^3$, high porosity of 97.63–98.32 %, and high specific surface area of 289.4 m$^2$/g. The carbon samples show lower density and higher porosity than those of coir aerogel, which contributes to a higher oil sorption capacity (22.71 g/g compared to 15.20 g/g, respectively). The research shows a possibility of transforming coir into carbon aerogels for effective oil adsorption. For the practical adsorption application, instead of time-consuming
and costly hydrophobic silane-coating of cellulose aerogel, carbonization can be used as a potential alternative.

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