Aerogel materials from sugarcane bagasse: the effect of additives

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ARTICLE INFO

Research Paper

Received: December 21, 2023
Revised: January 18, 2024
Accepted: January 31, 2024

Keywords

Agricultural waste
Biomass
Cellulose Aerogel
Sugarcane bagasse

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ABSTRACT

Cellulose nano fibers (CNF) are being considered as potential material for producing aerogel, an advanced materials for filtration technology. In this study, CNF were extracted from the sugarcance bagasse (a common agricultural waste) using ultrasonic technology and then fabricated with different additives including polyvinyl alcohol (PVA), polyethylene glycol (PEG) and graphene oxide (GO) to formulate aerogels. Various advanced techniques including polarized and scanning electron microscopy, fourier transform infrared spectroscopy (FTIR) and texture analyzer were applied to elaborate on the physicochsemical properties of the products. The obtained results revealed that aerogel had low bulk density (0.032 kg/m^3; 0.035 kg/m^3 & 0.041 kg/m^3) and a high porosity (96.89%; 97.06% & 98.61%) when combined with PVA, PEG and GO, respectively. Among investigated samples, aerogel fabricated with GO had the highest mechanical resistance as well as the best elasticity.

Cited as: Le, V. T. T., Nguyen, T. H., Vu, C. M. D., & Nguyen, P. T. (2024). Aerogel materials from sugarcane bagasse: the effect of additives. *The Journal of Agriculture and Development* 23(6), 50-56.

1. Introduction

Sugarcane is a common agricultural product of most tropical countries. While this plant plays an important role in improving the income of famer, the sugar industry also releases a high amount of sugarcane bagasse (approximately 30% of raw material). According to research many studies (Somerville et al., 2010; Mahmud et al., 2021), this waste currently creates pressure on landfills and pollution on incineration. Although sugarcane bagasse could be utilized for bioconversion purposes, such as bioethanol, xylitol, and specialty enzymes but these approaches still have not yet been widely applied due to the limit of technology and economics constraints (Chandel et al., 2012).

Indeed, sugarcane bagasse is a fibrous material containing cellulose as its main component and therefore, it is a potential raw material for producing aerogels. These advanced gels are an outstanding class of porous materials, with an extremely low bulk density, and very high porosity, and a low thermal conductivity (Nguyen et al., 2021). Aerogels are being considered as the most potential material for water treatment and desalination. Cellulose based-waste materials including sugarcane bagasse are ideal sources for developing aerogels due to their renewable, and biodegradable properties incineration (Thai et al., 2020). Our study, therefore, aimed to transform sugarcane bagasse into cellulose nano fibers (CNF) and applied them for producing aerogels. Besides, various additives including polyvinyl alcohol (PVA), polyethylene glycol (PEG) and graphene oxide (GO) was fabricated with CNF to strengthen the structure of the aerogels.

2. Materials and Methods

2.1. Materials

Sugarcane bagasse was collected from sugarcane juice carts. Chemical substances including polyvinyl alcohol (PVA), polyethylene glycol (PEG), sulfuric acid (H_2SO_4), hydrochloric acid (HCl), hydrogen peroxide (H_2O_2), sodium hydroxide (NaOH), graphite powder (94%), sodium nitrate ($NaNO_3$) and potassium permanganate ($KMnO_4$) were provided by Xilong Scientific (China). All chemical subtances were in the analytical grade.

2.2. Preparation of cellulose nanofibers (CNF)

Cellulose nanofibers were prepared based on the combination of ultrasound homogenization with chemical pretreatments (Chen et al., 2011b; Xu et al., 2017). Briefly, raw materials were cut into 1 - 2 cm pieces and washed several times with water to remove impurities. Then, the sugarcane fibers were dried in a laboratory oven (Memmert, Germany) at 90^{o}C for 72 h to the moisture content of 65% and finely ground into powder. The size of dried powder was controlled using a laboratory shaking sieve (BK-TS200, Biobase, China). Sugarcane fiber powders with a size range of 0.25 mm were reacted with NaOH solution (3% wt) with a ratio of 1:5 at 90^{o}C for 3 h to break lignocellulosic linkages. Then, the sample was bleached by H_2O_2 (10% wt) with a ratio 1:5 at 60^{o}C for 10 h. The mixture was rinsed with distilled water to neutral pH and dried at 70^{o}C for 24 h to obtain cellulose fibers. These fibers, then, were grinded finely and soaked into distilled water at 80^{o}C for 1 h before ultrasound dispersing for 15 min to obtain the CNF suspension. CNF was refrigerated at 4^{o}C for use in later experiments.

2.3. Preparation of graphene oxide (GO)

Graphene oxide was prepared according to the modified Hummer's method (Li et al., 2019). Firstly, the mixture including graphite (2 g), $NaNO_3$ (1 g) and $KMnO_4$ (6 g) was slowly added into H_2SO_4 (98% wt). Reaction was conducted at 5°C for 90 min and hold at 40^{o}C for 30 following minutes. Then, 92 mL of deionized water was added dropwise to the solution, and the temperature of the reaction was maintained at 95^{o}C until the color of mixture turns from dark green to light brown. H_2O_2 (30% wt) was added to the reaction mixture until no more visible bubbles were produced, and the mixture was allowed to stand. The solution was filtered with HCl (2% wt) to completely remove the remaining Mn ions. After adjusting to pH of 5 - 6 (using distilled water), the solution was dried at 70^{o}C for 24 h to obtain GO.

2.4. Preparation of CNF/PVA & CNF/PEG & CNF/GO

The PVA and PEG solutions (1.5% wt) were prepared by dissolving these polymers in deionized water at 75°C for 12 h. Hydrogels were formed by mixing CNF suspension (2.5% wt) with PVA/PEG/GO solution (1.5% wt) and then ultrasound homogenizing the mixture for 15 min. Aerogels were formed by freeze-drying hydrogels for 72 h using a laboratory lyophilizer (BK-FD10S, Biobase, China).

2.5. Physicochemical properties characterization of sugarcane bagasse aerogels

The density (ρ) and porosity (P%) of aerogels were determined based on their dimensions (20 x 40 mm) and weight. The specific surface area of the aerogel was determined through the N_2 adsorption–desorption isotherm using the Brunauer–Emmett–Teller (BET) method. Besides, mechanical properties of products were determined based on texture profile analysis (TPA) and stress relaxation (SR) methods using a texture analyzer (TA.XT Plus, Stablemicrosystem, UK) equipped with a cylinder geometry (diameter of 40 mm). The strain deformations of TPA and SR methods were 50 and 10%, respectively while the relaxation time was 300 sec.

The morphology of aerogels was observed using a polarized light microscope (DM2500P, Leica, Germany) and a scanning electron microscope (JSM-IT500, Japan). Fourier Transform Infrared (FTIR) spectroscopy of samples was performed in the range of 400 - 4000 cm, with a 4 cm resolution at room temperature (Tenser 27, Bruker, Germany).

3. Results and Discussion

3.1. Morphology and microstructure of sugarcane bagasse aerogels

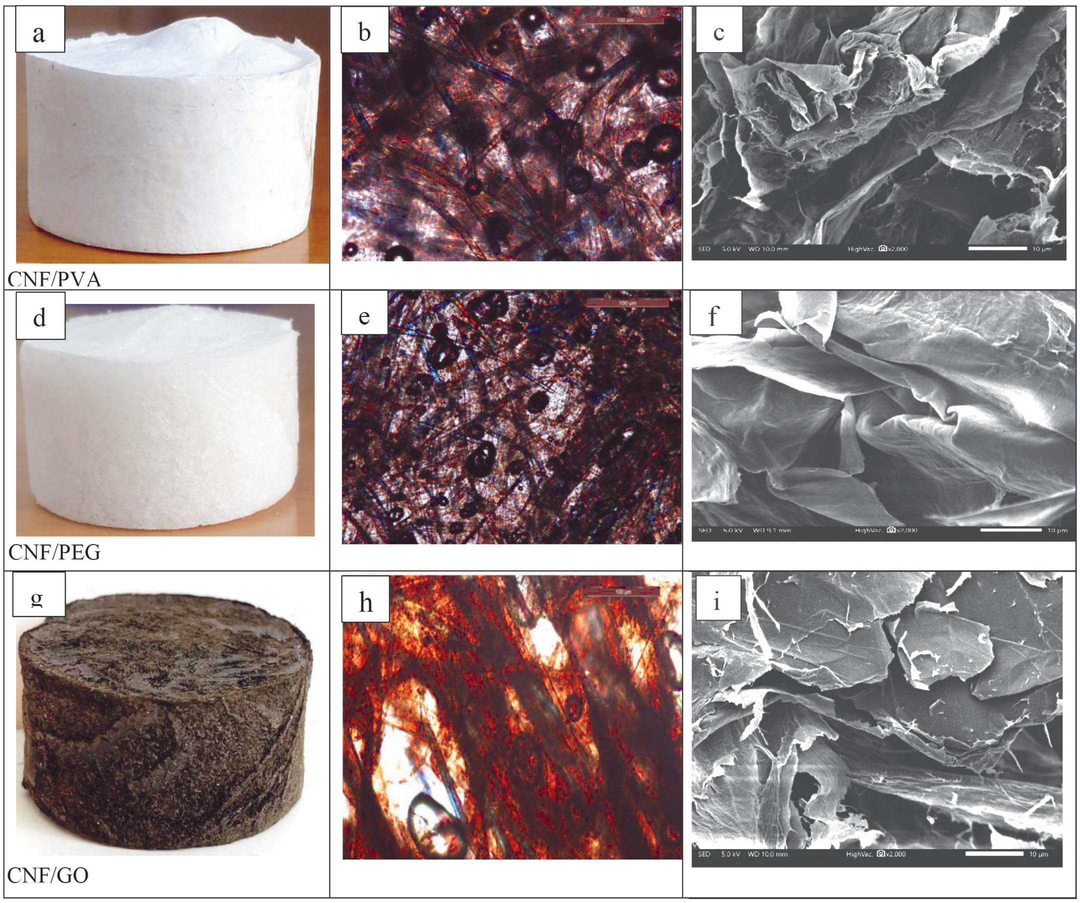

Figure 1. Morphology and microstructure of aerogels observed by naked eye (a, d, and g), polarized light microscopy (b, e, and h) and scanning electron microscope (c, f and i). CNF: cellulose nano fibers; PVA: polyvinyl alcohol; PEG: polyethylene glycol; GO: graphene oxide.

The effect of additives on the morphologies and microstructure of aerogels was displayed in Figure 1 while their physical properties were summarized in Table 1. As expected, polarized light microscope (PLM) and scanning electron microscope (SEM) observations revealed that polymer fibers from sugarcane baggage were CNF with a diameter in a range of 50 - 500 nm. Similar to the findings from previous studies (Chen et al., 2011a, 2011b & 2011c), sugarcane bagasse-based aerogels also had a characteristic porous structure (with a pore size diameter of 10 - 50 μm) and a high porosity degree (97 - 98%). In comparison between PVA and PEG (Figure 1c & 1f), the latter showed a better compatibility with cellulose particles resulting in a better homogenous crystal network for aerogel as well as a lower surface area (12.97 and 2.70 m^2/g for CNF/PVA and CNF/PEG, respectively). However, the difference of porosity degree between these samples was not significant. For sample fabricated with GO (Figure 1i), a more highly ordered structure was observed. Instead of the heterogenous aggregation of CNF, GO nanosheets tended to parallel arrange during the drying and therefore helped to orientate the CNF in the gel structure (Nguyen et al., 2021). Besides, the fabrication of GO on sugarcane bagasse CNF also significantly increased the density of aerogel ($P < 0.05$) in compared with samples adding PVA or PEG (Table 1).

Table 1. The density and porosity of sugarcane bagasse aerogels with different additives[1]

Sample	Density (g/cm^3)	Porosity (%)
CNF/PVA	0.032 ± 0.003^a	96.89 ± 0.002^a
CNF/PEG	0.035 ± 0.005^a	97.06 ± 0.001^a
CNF/GO	0.041 ± 0.002^b	98.61 ± 0.001^a

[1]*Data expressed as mean ± standard deviation (n = 3).*

[a,b]*Values within the same column having different letters are significantly different (P < 0.05) between means of samplesd.*

CNF: cellulose nano fibers; PVA: polyvinyl alcohol; PEG: polyethylene glycol; GO: graphene oxide.

3.2. FTIR

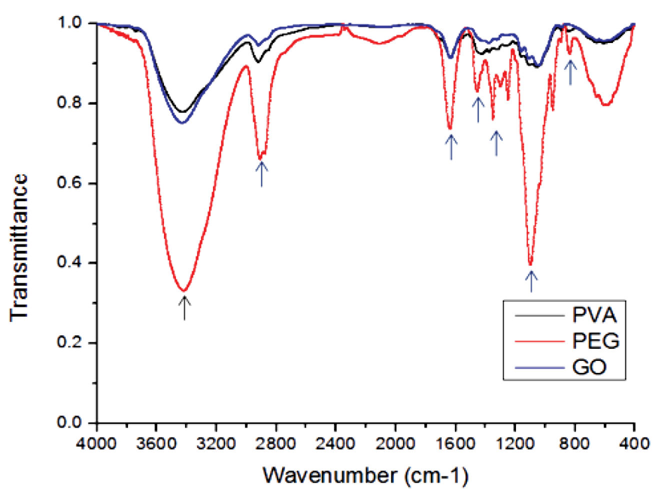

Figure 2. Fourier transform infrared spectroscopy spectra of sugarcane bagasse aerogels with different additives. PVA: polyvinyl alcohol; PEG: polyethylene glycol; GO: graphene oxide.

To elaborate further the effect of additive on sugarcane bagasse-based aerogels, FTIR spectroscopy was used to analyze functional groups of samples and the results was depicted on Figure 2. As hypothesized, lignin lignocellulose fibers have been significantly removed after alkaline treatment and bleaching step. Therefore, their characterized peak at 1730 cm was not recognized in the FTIR spectrum of investigated aerogels (Thai et al., 2020). For CNF, strong adsorption peaks were observed at 1606 cm and 1010 cm characterizing for the presence of C=O and C-O-H bonds of their backbone (Dao et al., 2022) while other peaks at 3300, 2900 and 1000 cm were contributed by O-H, C-H and C-H_2 linkages of glucose units. Besides, the addition of PVA and GO seem not to affect the chemical properties of CNF. For sample added PEG, some extra adsorption peaks were recognized at 1452 cm and 1245 cm. They should be associated with the bending and stretching vibrations of C-H and C-O groups, respectively (Sahu et al., 2022).

3.3. Mechanical properties of aerogels

Table 2. Mechanical properties of sugarcane bagasse aerogels with different additives[1]

	Texture profile analysis		Stress relaxation (SR)	
Sample	Hardness (N)	Cohesiveness	Firmness (N)	SR (%)
CNF/GO	55,593 ± 1,15[a]	0,942 ± 0,001[a]	31,096 ± 2,93[a]	40,08[a]
CNF/PVA	48,86 ± 1,296[a]	0,877 ± 0,012[b]	28,75 ± 0,8[a]	45,51[a]
CNF/PEG	27,261 ± 1,098[c]	0,775 ± 0,015[c]	18,22 ± 0.11[b]	65,65[b]

[1]Data expressed as mean ± standard deviation (n = 3).

[a-c]Values within the same column having different letters are significantly different (P < 0.05) between means of samples.

CNF: cellulose nano fibers; PVA: polyvinyl alcohol; PEG: polyethylene glycol; GO: graphene oxide.

The mechanical properties of aerogels play an important role for their applications as well as their shelf-life. Two different testing methods including TPA and SR were applied to compare the texture properties between samples and the results were summarized in Figure 3 and Table 2. As expected, aerogels adding GO had a highest hardness and mechanical resistance. The presence of GO particles contributed to improving the strength for the crystal network of sugarcane bagasse-based aerogel (Figure 1i). In line with the findings from FTIR analysis, mechanical parameters between CNF/PVA and CNF/GO aerogels had a high similarity, and the difference was mainly recognized for CNF/PEG sample. The hardness and firmness of the latter was approximately 40% of CNF/GO aerogel. All samples showed a well recovery property with a high cohesiveness (0.8 - 0.9) but their SR degrees showed a significant difference. While CNF/PVA and CNF/GO aerogels showed a well balance between the elastic and viscous components (SR in a range of 40 - 50), the addition of PEG to aerogels significantly increased the loss modulus of sample. It could be assumed that the presence of hydrophilic groups in PEG affected the linkages between hydrophobic groups of polymers in CNF aerogels.

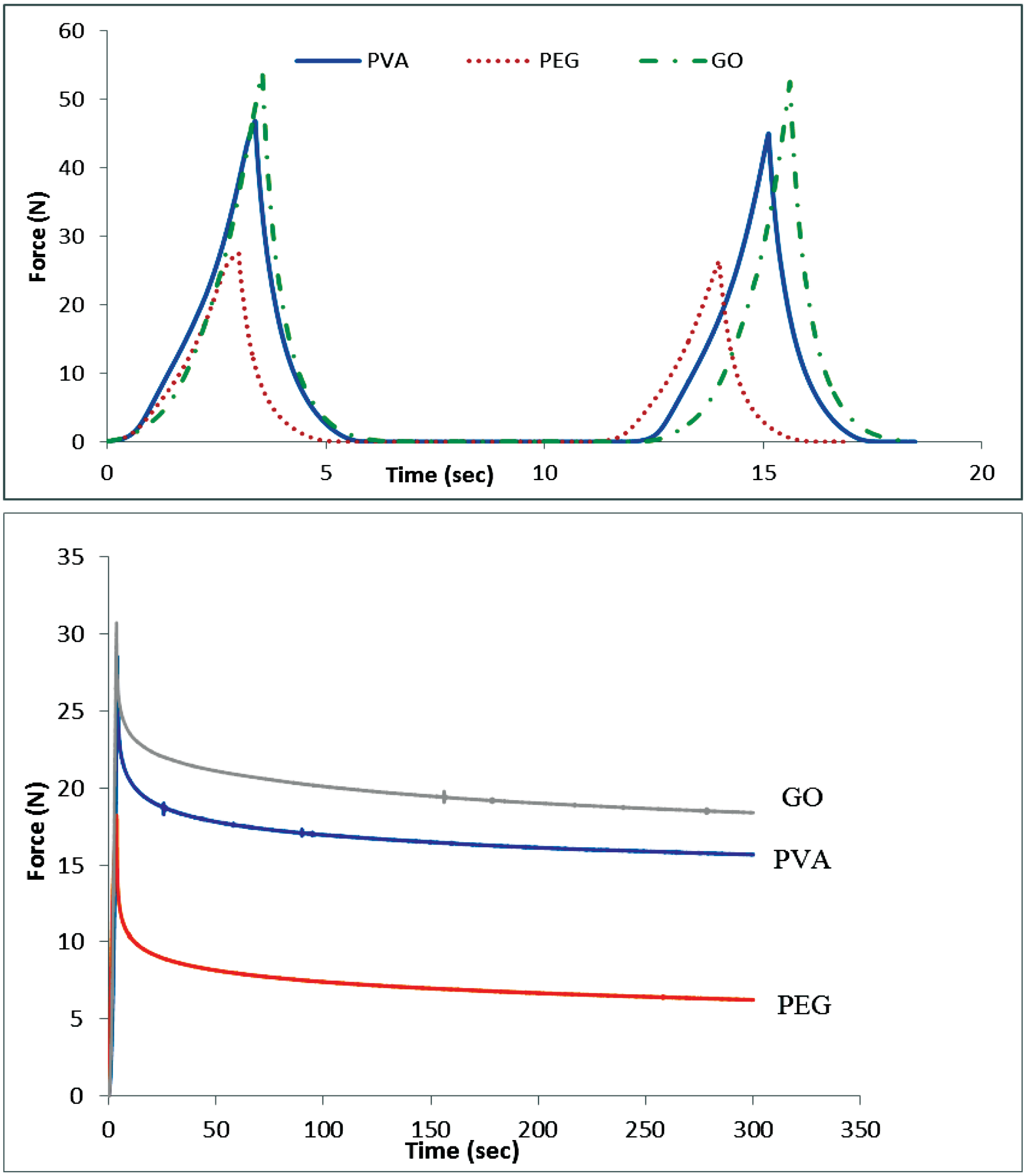

Figure 3. Texture profile analysis and stress relaxation of sugarcane bagasse aerogels. PVA: polyvinyl alcohol; PEG: polyethylene glycol; GO: graphene oxide.

4. Conclusions

This study successfully formulated aerogels from cellulose nano fiber of sugarcane bagasse. As expected, the microstructure of these aerogels was modified when using different additives (PEG/PVA/GO). In general, GO and PVA aerogels had a high similarity of physicochemical and mechanical properties while PEG aerogels had a weak crystal network. Further studies could apply these aerogels for filtration technology or water treatment.

Conflict of interest

The authors have no conflicts of interest to declare.

Acknowledgements

This research was financially supported by the Nong Lam University, Ho Chi Minh City research funding (the research code: CS-CB23-HHTP-03).

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