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Synthesis of waste pineapple peel cellulose based hydrogels and aerogels

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Abstract: Aerogels were one of the groups of nanoporous materials with superior physicochemical properties. Their unique physical, chemical, and mechanical properties make aerogels as promising candidates for different applications including drug delivery, tissue engineering, medical implantable devices, biotechnology and wastewater treatments. The organic (silica) and inorganic (biopolymers) compounds can be used to synthesize aerogels. Cellulose found the most abundant in world was one of these biopolymers. Cellulose has properties such as biocompatibility, recyclability, excellent mechanical strength, adjustable optical appearance, thermostabilizing, non-toxicity make to prefer in aerogel studies. In this study, pineapple peel waste cellulose was used to synthesize aerogel. To obtain cellulose hydrogels cellulose and carboxymethyl cellulose (monomers) were mixed with epichlorohydrin (cross-linker). Alcogels (by solvent exchange) and aerogels (by freeze-thaw) was synthesized from obtained hydrogels. The characterization studies water adsorption capacity and transparency tests were performed waste based hydrogel and aerogels.

Keywords: Bioaerogel, waste-based cellulose, pineapple peel, adsorption capacity

Atık ananas kabuğu selülozu temelli hidrojellerin ve aerojellerin sentezlenmesi

Özet: Aerojeller, eşsiz fizikokimyasal özelliklere sahip nanoporöz malzeme gruplarından birisidir. Eşsiz fiziksel, kimyasal ve mekanik özellikleri aerojelleri; ilaç taşınımı, doku mühendisliği, implant medikal cihazlar, biyoteknoloji ve atık su arıtımları gibi farklı uygulamalar için umut verici adaylar haline getirir. Aerojel sentezinde inorganik (silika) ve organik bileşikler (biyopolimerler) kullanılabilir. Dünyada en çok bulunan selüloz bu biyopolimerlerden birisidir. Selüloz, biyouyumluluk, geri dönüştürülebilirlik, üstün mekanik kuvvet, ayarlanabilir optik görünüm, termostabilizasyon, toksik olmaması gibi özelliklerinden dolayı aerojel çalışmalarında tercih edilmektedir. Bu çalışmada ananas kabuğu atığı selülozu aerojel sentezinde kullanılmıştır. Selüloz hidrojellerini elde etmek için selüloz ve karboksimetil selüloz (monomerler) epiklorohidrin (çapraz bağlayıcı) ile karıştırılmıştır Elde edilen hidrojellerden, alkojeller (solvent değişimi ile) ve aerojeller (dondurma-çözme ile) sentezlenmiştir. Atık selülozdan hazırlanan hidrojel ve aerojellerin karakterizasyonu için su adsorpsiyon kapasitesi ve saydamlık testi yapılmıştır.

Anahtar Kelimeler: Biyoaerogel, atık bazlı selüloz, ananas kabuğu, adsorpsiyon kapasitesi

1. Introduction

Aerogels were porous solid materials have unique properties such as bulk density, high porosity, large surface area. They had many different application areas; food industry, pharmaceutical, air filtration, wastewater treatment, dye adsorption, thermal insulation. (Azimi et al. 2024; Chen et al. 2022; Do et al. 2020; Ganesamoorthy et al. 2021; Groult et al.2022; Gu et al. 2022; Guastaferro et al. 2021; Ihsanullah et al. 2022; McNeil et al. 2022; Sanchez et al. 2023; Sozcu et al. 2024; Sun et al. 2022). Depending on the nature of the initial precursors, aerogels were classified as inorganic, organic and hybrid. Inorganic aerogels were based on metals, oxides and silica. The organic aerogels were based on synthetic polymers (polystyrenes, polyurethanes, poly alcohols, polyacrylates) or natural biopolymers (protein, carbohydrates, pectins, mucilage) (He at al. 2023; Meti et al. 2022; Tafreshi et al. 2022; Wei et al. 2022). The waste based aerogels, generally had natural biopolymer precursor also known as bioaerogels. The biowaste that constitute the lignocellulosic mass were excellent bioaerogel precursors (renewable, cheap). The lignocellulosic mass could be agricultural, food or fruit wastes (He at al. 2023; Joshi et al. 2023; Abdul Khalil et al. 2020). The components of lignocellulosic biomass were cellulose, hemicellulose and lignin. Cellulose had increasing interest in hydrogel/ aerogel areas due to its biocompatibility, thermal stability, renewability, biodegradability and non-toxicity (Joshi et al. 2022;Abdul Khalil et al. 2020; Al Abdallah et al. 2024; Chang et al. 2010a; Budtova 2019; Jianan et al. 1996; Li et al. 2024; Long et al. 2018)

The pineapple or *Ananas comosus*, a member of *Bromeliaceae* family and was grown in many parts of world. It is eaten as preserved or fresh fruit. Also, it was used jams, concentrates and juices. Since 60% of pineapple fruits can be eaten, the 40 % of parts were as wastes. The pineapple wastes were leaves, peels, roots and farm stems (Nath et al. 2023). Conversion of biomass wastes as a sustainable resource into bioaerogels was a new and innovative process. By this way utilizing biomass and wastes reduced environmental problems (Asim et al. 2019; Partow et al. 2022). Also, the properties of biomass waste (biodegradability, non-toxicity), make bioaerogel as an eco-friendlier material compared with silica based inorganic aerogels (Mazrouei-Sebdani et al. 2021) . For this aim, we used free of charge pineapple peel waste as a cellulose was a source of bioaerogel

While synthesizing cellulose aerogel, the biggest problem was the solubility of cellulose. The chemical structure (high crystallized form, robust inter and intra molecular hydrogen bonds-high number hydroxyl groups) make cellulose insoluble in water. There were many solutions of this problem, but most of them included specific and toxic chemicals (Chang et al. 2010b). The eco-friendly choice for this dissolution problem was blending cellulose with cellulose derivatives such as carboxymethyl cellulose (CMC), ethyl cellulose (EHC), hydroxyethyl cellulose (HEC), hydroxypropyl cellulose (HPC). The carboxymethyl cellulose exhibited great potential for using this aim. Because, CMC had good water solubility, biocompability, biodegradability and sensitivity to pH or ionic strength. (Chang and Zhang 2011). After blending with carboxymethyl cellulose, cellulose became more soluble.

In this study, we converted pineapple peel wastes into cellulose based aerogels. For modifying and to solve the dissolution problem of cellulose we blended waste cellulose with CMC in sodium hydroxide/ urea. The cellulose hydrogels crosslinked with epichlorohydrin (ECH). To convert hydrogel to alcogel and then aerogel, we studied a multistage solvent exchange (ethanol) and freeze-drying process. The water adsorption studies and swelling degree, transparency of hydrogels and aerogels were studied for characterization.

2. Materials and Method

The pineapple peels were taken from local market free of charge (waste). The sodium hydroxide, urea, carboxymehyl cellulose, epichlorohydrin (ECH), hydrogen peroxide, potassium hydroxide, hydrochlororic acid 37 %, ethanol 99.9 % were purchased from Sigma-Aldrich Chemical Co. Ltd. All other chemicals were analytical grade.

2.1. The isolation of cellulose from pinapple peel waste

The cellulose could be isolated from biomass via mechanical, disintegration, chemical treatment with alkaline solutions and biological treatments. In this study; the cellulose extraction from pineapple peel was carried out with potassium hydroxide (Dai et al. 2016). The pineapple peels were taken from local markets as waste (free of charge). Pineapple peels were cut into small pieces and dried at 50°C. Dried pineapple peels were ground in mortar (Retsch RH 100). 50 grams of dry pineapple peel was boiled for 2 hours at 80 ° C in 1L distilled water and filtered through filter paper. This process was repeated 3 times. Soluble fiber tissues were removed and filtered again. The residue was mixed with 5% hydrogen peroxide and stayed at 75 ° C for 4 hours (to remove the lignin) and filtered. The residue was washed until neutralized with 95% ethanol and distilled water. The sample was dried at 50 ° C for 16 hours. To remove hemicellulose, the pellet as mixed with 10% potassium hydroxide for 24 hours and filtered. The residue (cellulose) was washed until neutralized with 95% ethanol and water and dried.

2.2. Preparation of waste cellulose based hydrogel and aerogel

To obtain 3 % cellulose solution; the waste cellulose was dissolved in 6 % NaOH/ 4 % urea / 90 % distilled water by stirring 5 minutes and then stored at -20° C for two days. After two days, the solid was thawed and stirred at room temperature till a transparent cellulose solution was taken. 3 wt% CMC was dissolved in same solution (6 % NaOH/ 4 % urea / 90 % distilled water). The cellulose and CMC solutions were mixed different ratios (5:5: 6:4;8:2; 9:1) respectively. ECH was added to the cellulose/ CMC mixtures as cross-linker (0.1 % v/v) stirred at room temperature for 2 days to obtain a homogeneous solution, and then kept at room temperature for four days to get hydrogel forms. Hydrogels were washed with distilled water before next step.

The cellulose alcogels were prepared from hydrogels first replacing the water in the hydrogel with ethanol (EtOH) by multistage solvent exchange process. During the solvent exchange process, the hydrogels were soaked in 30; 50; 70; 100 (%) EtOH and 100 % EtOH for 5 days, where the ethanol was centrifuged and replaced fresh EtOH everyday to obtain alcogels. The alcogels were then soaked with 100 (%) EtOH for 2 hours and centrifuged. The alcogels were freze-dried at -80° C for 4 days to obtain aerogel. The final product cellulose aerogels were stored at dry place (room temperature) for further experiments (Gan et al. 2017; Paksung et al. 2020; Paulauskiene et al. 2022; Wang et al.2016).

2.3. Characterization Studies
2.3.1.Water adsorption studies

To calculate water adsorbing capacity of hydrogels and aerogels, the samples were immersed into distilled water to acquire different conditions such as pH, temperature and time. We studied different time effect on water adsorption of hydrogels and aerogels in this study.

The hydrogels and aerogels (constant weight) were immersed in distilled water for 15-30-45 min- 1h -2 h-4 h-6 h. The weight of each sample was measured (before and after immersed) and the percentage of water adsorption capacity (WAC %) was calculated according to Equation 1.

$$WAC(\%) = \frac{Ws-Wd}{Wd} * 100 \text{ (Eq. 1)}$$

Where Wd and Ws were the weights of the hydrogel and aerogel before and after immersion in respectively.

2.3.2. The transparency of the hydrogels and aerogels

The transparency of aerogels were determined by UV-Vis microplate spectrophotometer at a wavelength ranging from 200 to 1000 nm

3. Results and Discussion

3.1. The preparation of waste based cellulose aerogels

The cellulose aerogels had the advantages renewability, biocompability, biodegradability as cellulose. Also; the cellulose aerogels could be ecofriendly because their natural decomposition and also they can be studied at many different areas because of their stability in harsh conditions. The cellulose aerogels was studied by using waste cellulose plant or plant based materials (cotton, hemp, coconut, pineapple waste). Depending on source the extraction of cellulose involves pretreatment, post treatment and dissolution stages. The extraction methods can change the characteristics of cellulose (size, degree of polymerization, thermal stability, degree of hydroxylation) The quality of extracted cellulose effect also the performance of the cellulose aerogels (Nguyen et al. 2022; Liu et al. 2024; Long et al. 2018; Asim et al. 2019).

The plant cellulose fibers fills with lignin and hemicellulose penetrates the cellulose skeleton to form 3D structure via covalent and hydrogen bonds by using alkaline solutions (sodium or calcium hydroxide) (Mujtaba et al. 2023). To unform this stable compact structure, many deconstruction techniques can be used such as ball-milling, acidic fraction, alkaline fraction, etc. All techniques resulted the different form of cellulose (fibers, nanowhisker, particle). In this study we used alkaline process. The alkaline fraction process is done to deconstruct lignocellulosic form at a certain tempertaure and residence time. The advantages of this process removes lignin and breaking bonds between hemicellulose, retaining cellulose in solid fraction as cellulose fibers and can be ingrated other techniques which were related the solubility of cellulose (Liyanage et al. 2021; Gan et al. 2017).

The extraction quality of waste cellulose effected the formation of hydrogel and the initial hydrogel characteristics (porosity, pore volume) were the results of cellulose extraction. The aerogel properties mostly depend on the hydrogel 3D form. However the surface area of aerogels only depends on drying method of aerogel while preparing. The different methods were existed for this aim; freeze-drying, supercritical drying (CO_2, acetone, methanol or ethanol), ambient pressure drying, vacuum drying, and microwave drying. These methods were used to remove the existed solvent inside the pores of the hydrogel maintaining the porous structure. Because of its simplicity, environmentally friendly and cheap cost, freeze-drying technique was used for waste based cellulose aerogel in this study. Freeze- drying of cellulose hydrogels involved three stages. The first stage consisted of freezing the hydrogels into ice crystals leading the ordered structure. The critical point of freezing was the temperature, we optimized the temperature at -80°C. This temperature affected the structure of the ice formed in aerogel and the quality of the aerogel. Primary drying was performed to remove 95 % of water and secondary drying was performed to eliminate unfrozen water (Simon-Herrero et al. 2016).

According to our results (Fig 1.) the hydrogels lost almost 95 % of their weight after freeze- drying and ultra weight cellulose aerogels were got.

Fig. 1 The different ratio of waste cellulose: CMC based hydrogels (a), alcogels (b) and aerogel (c) after freeze-drying

3.2. The results of characterization studies
3.2.1.Water adsorption capacities of hydrogels and aerogels

To confirm environmental adaptation of hydrogel and aerogel the adsorption capacity was studied. The water adsorption capacity of waste cellulose: CMC (9:1; 8.2; 6:4; 5:5) hydrogels and aerogels were studied according to weight changing with time.

According to Figure 2; the water adsorption capacity of waste cellulose: CMC based hydrogels were reached adsorption equilibrium within 60 min and aerogels reached at 30 min. The different ratio of cellulose/ CMC did not affect the water adsorption capacity of hydrogels The adsorption capacity increased time by time. The reason of this could be surface and high pore size of aerogels. The adsorption capacity of aerogels increased significantly at first and then water filled the aerogels pores and adsorption reached saturation quickly. The strong hydrophilicity of aerogel surface absorbed the water completely. The percentage of changing weight of aerogels after water adsorption were 300-800 % of its weight whereas the percentage changing of weight for hydrogels were 20-100 % of its weight. The cellulose aerogel reached average 500% of water adsorption within 30 min. This suggested us the cellulose aerogel had a fast water adsorption ability due to the high porosity and abundant hydrophilic groups (hydroxyl, carboxyl).

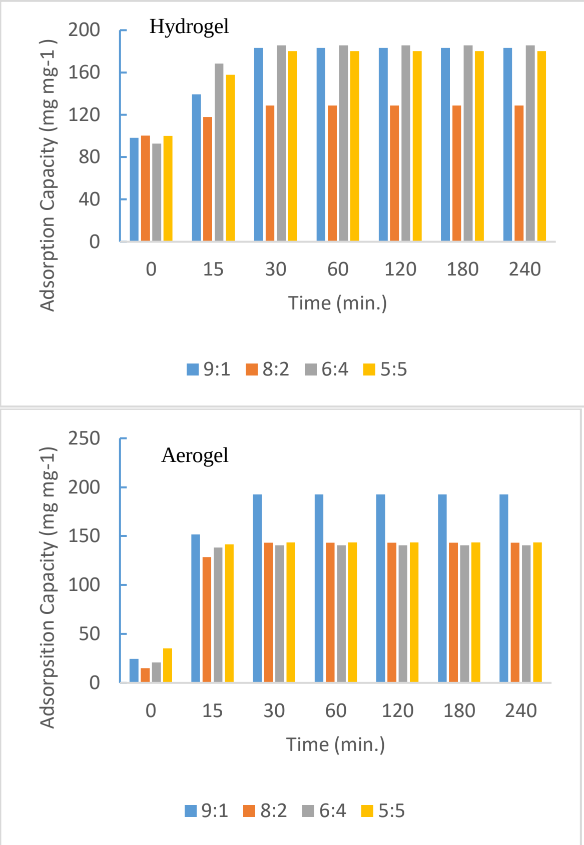

Fig. 2 The adsorption capacity (mg mg-[1]) of waste cellulose: CMC (9:1; 8:2; 6:4; 5:5) based hydrogel and aerogel

3.2.2. The transparency of the cellulose hydrogels and aerogels

The UV-Vis transmission spectra of cellulose aerogels were given at Fig 2. According to the spectra results, our cellulose aerogels do not transmit light after 200-400nm, while cellulose hydrogel do not transmit light after 400-600 nm (Gan et al. 2017). The results shown that cellulose aerogels were more transparency than cellulose hydrogels.

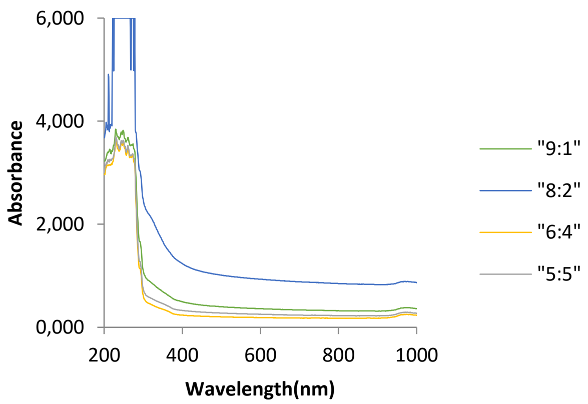

Fig. 3 The UV-Vis spectras of different ratio of waste cellulose: CMC based aerogels

4. Conclusion

The waste pineapple cellulose require no harsh chemicals to make bioaerogels owing to their structural formability. The results were shown that bioaerogels kept several weeks at room temperature and did not seen any visible degradation. The important advance of this study was the simplicity of the bioaerogel preparation process. The prepared bioaerogel also distinguished itself through water adsorption capacities which were crucial for the potential applications such as the treatment and removal of metals/dyes/toxic compounds from wastewaters. The aerogel production in large scale faced problems involved toxic precursor compound and the sustainability of initial compounds. Bioarogels overcome these problems by using raw materials (biomass-derived). So that using waste pineapple as the resource of bioaerogels is an alternative way of solving environmental pollution.

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Authors' contributions:

OS: performed the analysis, obtaining data, editing; BO: editing, writing

Conflict of interest disclosure:

The authors declare that there were no conflicts of interest in the realization of this research.

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