

DISSERTATION INFORMATION

Title: Synthesis of cellulose aerogels from coconut fiber for adsorption of organic dyes and lubricating oil

Major: Chemical Engineering

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Major contributions of this dissertation:

Cellulose aerogel, emerging as a novel absorbent in the 21st century, offers unique and impressive features including low density, abundant pore structure, a seamless 3D network, and large specific surface area. Cellulose aerogel holds promise for adsorption dye from water and oil by combining aerogel characteristics with the advantages of abundant supply, renewability, bio-renewability, and facile surface modification. The synthesis method and the linker of cellulose aerogel can significantly impact its adsorption capacity. Therefore, the synthesis process of cellulose aerogel to prepare materials for adsorbing organic dyes and oil holds substantial scientific and practical significance.

In this dissertation, two cellulose aerogels were synthesized from coconut fiber and the linkers for adsorbents for organic dyes and lubricating oil. Firstly, cellulose aerogel was fabricated from alkalinized coconut fiber and physical cross-linkers (polyvinyl alcohol (PVA) and xanthan gum (XTG), symbol of PXA). Secondly, cellulose aerogel was synthesized from bleached coir and chemical cross-linker tetraethylenepentamine (TEPA) via the sol-gel process (symbol of NUTA). The physicochemical properties of cellulose aerogels were analyzed by X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FT-IR), compressive Young's modulus, and thermogravimetry (TGA). The PXA demonstrated remarkably low density (27.59–47.78 g/cm³), high porosity (96.74–98.03%), and compressive Young's modulus (3.82–12.66 kPa). The resulting aerogel demonstrated a highly porous structure (>90%), low density (91.89–114.68 mg/cm³), and abundant surface amine groups advantageous for removing organic dyestuffs. PVA and XTG form cross-links with the hydroxyl group of cellulose, enhancing the bonding between fibrils and strengthening the 3D

structure of aerogel. In contrast, the functionalized materials exhibit an intricately interconnected three-dimensional porous structure achieved through covalent crosslinking with TEPA.

These aerogels were tested for methylene blue (MB), crystal violet (CV), and methyl orange (MO) adsorption to evaluate their effectiveness in treating dye-polluted water via various conditions such as contact time, pH values, initial concentrations, temperature, and reusability. The mechanism of the adsorption process is studied through three kinetic models, four isotherm models, and differences in characteristics before/after adsorption (including FT-IR spectrum and energy dispersive X-ray spectroscopy). PXA3 (3 wt% cellulose, 0.6 wt% PVA và 0.3 wt% XTG) had removal capacities for MB, CV, and MO 50 mg/L of 80.57, 82.04, and 16.67% after 90 minutes at pH 7–11. Adsorption followed the second-order kinetics and the Redlich-Peterson isotherm model. NUTA2 (2 wt% cellulose, ratio of solution 2TEPA:5EtOH:3H₂O) demonstrated the percentage removal for MB and CV 50 mg/L of 98.65 and 89.92% mg/g after 120 minutes. Adsorption followed the second-order kinetics and the Freundlich isotherm model. The fabricated aerogels exhibited a three-stage adsorption process across a wide pH range (pH 5–11), with the adsorption mechanism attributed to electrostatic interaction, Vander Waals forces, and hydrogen bonding between cellulose aerogel and organic dye molecules. As a result, NUTA2 is potential for organic dye removal because of its high adsorption capacity, reusability, and retention of 3D structure in solution.

Cellulose aerogel contains numerous hydrophilic hydroxyl groups on its surface, which undergoes modification with methyltrimethoxysilane (MTMS) to improve its lubricating oil adsorption efficiency. The introduction of silane functional groups and the distribution of silicon on the surface confirm that chemical vapor deposition is an effective method for hydrophobically modifying the cellulose aerogel. The study examined the impact of viscosity on adsorption capacity, including paraffin oil, 5w30 oil, and vacuum pump oil. The results revealed that viscosity increased and adsorption capacity decreased. Moreover, while PXSA1 adsorbed oil up to 20.32 g/g, NUSA1 reached only 17.10 g/g. Additionally, PXSA1 demonstrated the ability to be reused after three consecutive adsorption-desorption cycles, indicating its potential for lubricating oil adsorption.

In conclusion, the research outcomes of the thesis contribute to the development of two new materials capable of absorbing organic dyes and lubricating oil. Specifically, materials containing cellulose and physical cross-linkers are suitable for oil adsorption applications, while materials containing cellulose and chemical cross-linkers are proposed for organic pigment

adsorption. Both cellulose aerogel synthesis processes are designed to be straightforward, involve freezing in a freezer, and use freeze-drying to preserve the 3D structure of the material. The roles of cross-linking agents, material synthesis, and adsorption mechanisms are elaborated upon, providing a foundation for further research and development of adsorbent materials.

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