Science & Technology Development Journal – Engineering and Technology 2024, 7(2):2248-2257

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Research Article

Fabrication and Characterization of Hydrophobic Aerogels Containing Bacterial Cellulose Coated with Copper Species via Mild Reduction

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ABSTRACT

In this study, bacterial cellulose (BC) was coated with copper species via a room-temperature hydrazine-mediated reduction reaction of copper(II) acetate in a suspension phase of *nata de coco*, which was treated by freeze-drying, yielding hydrophobic lightweight aerogels. Structural and textural characteristics of the prepared aerogels were discovered using several techniques including X-ray diffraction (XRD), thermogravimetry analysis (TGA), water contact angle measurement and isothermal nitrogen physisorption. In detail, XRD results indicated the formation of the metallic copper phase in the aerogel while the high cellulose crystallinity was remained unchanged. No oxidized copper phases were detected in the material. Via the TGA profiles, the Cu loading was determined to be in the range from 3.9 to 13.4 wt.%, depending on the copper(II) acetate amount used for the reduction reaction. These values were generally lower than the theoretical Cu contents probably due to the unexpected Cu losses during the preparation procedure. In addition, increasing the Cu content in the BC aerogel led to a significant decrease in the specific surface area with the presence of Cu in the porous structure. On the other hand, as expected, the hydrophobicity of the BC aerogel was significantly enhanced with the Cu content. Indeed, the Cu-coated BC aerogels with high copper contents (> 6.2 wt.%) were hydrophobic, showing a large water-contact angle of up to 138°. Therefore, the resulting hydrophobic aerogels well interacted with water-immiscible organic solvents including diesel oil and cyclohexane with adsorption capacities varied from 20 to 30 g/g. The successful fabrication of the hydrophobic aerogels upon the simple surface modification of abundant bacterial cellulose with Cu species can introduce novel and efficient biomass-based material for the treatments of oil-based liquids in the aqueous environment.

Key words: hydrophobic aerogel, bacterial cellulose, coating, copper, mild reduction

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History

- Received: 20-2-2024
- Accepted: 23-5-2024
- Published Online: 30-9-2024

DOI :

https://doi.org/10.32508/stdjet.v7i2.1339

INTRODUCTION

In the era of industrialization, inevitable water pollution caused by hydrophobic organic solvents and oil spills threatens both ecosystems and human health [1,2]. These incidents inflict substantial burdens due to lost resources, cleanup efforts, and potential disruptions to industries reliant on clean water [3]. As a result, the consequences prompted the necessity for effective and innovative remediation techniques to minimize their environmental impact and ensure public health. At present, the common strategies employed to cope with including adsorption, chemical treatment, incineration and biotreatment [1]. Among them, adsorption is deemed a promising approach to capture the contaminants since the process is simply, cost-effective and does not generate secondary pollution [3]. It is, therefore, essential to design an effective adsorbent. In particular, the ideal sorbents should exhibit high trapping efficiency, high uptake rate, commercial viability, environmental friendliness, and facile recyclability [4].

Aerogels are an outstanding class of porous materials, with an extremely low bulk density, a very high porosity, and a low thermal conductivity [5,6]. In particular, carbon nanotubes (CNTs), graphene, as well as biomass-derived materials [7–10] have been the outstanding precursors for aerogels fabrication towards their application in oil spill cleanup and water treatment. However, the limitations for the application of CNTs and graphene are high precursor cost, complex fabrication procedures or the need for specialized equipment [5]. In contrast, biomass-based materials can offer distinct advantages, including sustainability, biodegradability and inherent safety [11]. Recently, aerogels derived from bacterial cellulose have attracted attention of the scientists as a potential material for environmental treatments owing to their low cost, sustainability, low density, high porosity and biodegradability [12,13]. The oil

Cite this article : Le H V, Nguyen H H M, Nguyen T T P, Nguyen T T T, Dang K D, Le K A, Nguyen K D, Phan H T. **Fabrication and Characterization of Hydrophobic Aerogels Containing Bacterial Cellulose Coated with Copper Species via Mild Reduction**. *Sci. Tech. Dev. J. – Engineering and Technology* 2025; 7(2):2248-2257.

and oleophilic liquid adsorption performance of sorbents are not only determined by their density and porosity, but is also significantly influenced by surface properties[14]. Common strategies for cellulose fibers comprising of chemical vapor deposition, cold plasma treatment and atomic layer deposition are applied with the low-surface-energy alkyl or fluorine functional groups to obtain the hydrophobicity[15–17]. However, these methods involve the expensive and toxic organic modifiers. Therefore, the development of a facile and cost-effective approach for the fabrication of hydrophobic cellulose aerogels is of importance. As ideal sorbents for water-immiscible solvents and oil, cellulose-based aerogels need tailoring to tune the aerogel structure toward improved hydrophobicity[4]. Recently, we have demonstrated promising hydrophobic organic solvent adsorption by copper-modified bacterial cellulose aerogels, which was attributed to the copper particles covering polar hydroxyl groups via mild reduction reaction[5]. However, the hydrophobicity of the obtained aerogels was not fully investigated.

Herein, the present study focused on a further investigation of the hydrophobic characteristics of copper-coated BC-based aerogels, thereby achieving a complete hydrophobicity and enhancing the selectivity to the oil phase for practical application.

MATERIALS AND METHOD

Materials

Nata de coco pieces with an average BC content of 0.8 wt.% were purchased from the Bich Lien Duong supplier (Ben Tre, Vietnam). A Philips HR2531 handblender (650 W) was employed to grind the mixture of *nata de coco* pieces (125 g, containing approximately 1 g of BC) and water (125 g) for 2 min, yielding a suspension phase of *nata de coco*.

In a typical procedure for coating BC with Cu which was based on the previous study with minor modifications, the obtained suspension phase of *nata de coco* was added with $Cu(CH_3COO)_2.H_2O$ (1 mmol) in a 500-mL Erlenmeyer flask under vigorous stirring for 3 h. Subsequently, 50 equivalents of hydrazine hydrate ($N_2H_4.H_2O$) was added dropwise under vigorous stirring, followed by the Cu^{2+} reduction reaction for 15 h. After reaction completion, the Cu-modified BC was collected by gravity filtration and washed repeatedly with water until a neutral pH value was obtained. The resulting mixture was remained on the filter paper for 30 min for further water release and then transferred to propylene boxes, which were frozen at −20 °C for 24 hours. The bacterial cellulose aerogels were obtained via freeze-drying, yielding cylinder-shaped aerogels, which were denoted as "Cell–Cu=1:1" expressing 1 g of BC:1 mmol of $Cu(CH_3COO)_2$. Further samples including Cell–Cu=1:2, Cell–Cu=1:3, and Cell–Cu=1:4 were fabricated under similar conditions using 1 g of BC and the varied $Cu(CH_3COO)_2$ amount, namely, 2, 3, and 4 mmol, respectively. An aerogel sample named "Cell" was prepared without the modification of Cu for the comparison purpose[5].

Characterization of the obtained materials

Crystallinity of the materials was discovered by X-ray diffraction measurements on a diffractometer device using Cu radiation (D8 Advance, Bruker, Germany). Morphological photographs of the aerogel samples were achieved on an electron scanning microscope (S-4000, Hitachi, Japan). Thermal behavior of the aerogels was investigated on a thermal gravimetric analyzer (SDT Q600, TA Instruments, USA). Their textural properties were determined by 77 K-isothermal nitrogen adsorption/desorption using a high-performance sorption analyzer (ASAP 2020, Micromeritics, USA).

Adsorption study

The adsorption capacity of the copper-coated aerogels for cyclohexane and diesel oil was discovered. 0.02 g of the aerogel sample was dipped into a glass vial containing 10 mL of the corresponding solvent. The solvent-trapping sample was taken out from the liquid phase but still remained in the vial. Until there were no more solvent drops back to the liquid phase, the sample was completely removed. The cyclohexane solvent adsorption efficiency of the copper-coated aerogels was calculated according to the formula: $Q = (m_1 - m_2)/m_{aerogel}$ (g/g), where m_1 and m_2 are the total weight of the glass vial containing the tested solvent before and after the adsorption, respectively.

RESULTS AND DISCUSSION

Coating copper particles on the surface of bacterial cellulose fibers and bundles was employed via the Cu(II) to Cu(0) reduction stage by hydrazine in an aqueous phase, which was considered as an effective reducing agent thanks to numerous advantages of high efficiency, fast reaction speed, and room-temperature operation[5,18]. Due to the addition of hydrazine, the solution color changed from blue of the Cu^{2+} cation to red-brown of the Cu^0 clusters (Figure 1), proving the successful reduction of Cu^{2+} to Cu^0 by hydrazine under ambient conditions. The

presence of the BC fibers in the same reaction environment led to the development of the Cu crystal on the fiber surface, affording successful Cu coating.

Pure cellulose aerogels exhibited non-selective affinity for both water and oil. This limitation can be solved by introducing copper species to the aerogel, which modified the surface properties of the aerogels. Copper particles effectively shielded the hydroxyl groups (-OH) on BC fibers, decreasing hydrophilicity of the BC aerogels. As described in our previous study, upon contact with the copper-coated aerogels, the water droplet was remained on the surface with a water-contact angle of 133°, preventing water penetration into the porous structure. In contrast, cyclohexane was rapidly trapped into the cellulose matrix[5]. The present work focuses on further investigating the influence of the Cu content on the material hydrophobicity.

XRD analysis was employed to confirm the successful deposition of copper onto the bacterial cellulose (BC) surface and evaluate its impact on the crystalline structure (Figure 2). The XRD pattern exhibits two characteristic diffraction peaks at 2θ = 14.6 °, 16.7 ° and 22.7 °, corresponding to the ($1\bar{1}0$), (110) and (020) lattice planes of crystalline cellulose, respectively[19]. The result indicated that the copper coating process had negligible impact on the inherent crystallinity of the BC. Notably, no further required toxic chemicals-involving treatments as compared to plant cellulose[20,21]. In addition, the XRD pattern indicated the successful incorporation of copper particles on cellulose fibers, as confirmed by the presence of characteristic peaks of 2θ = 43.5°, 50.5° and 74.2°, corresponding to the lattice planes of (111), (200), (220) of pure metallic copper phase (JCPDS No. 003–1018), thus proving the effectiveness of the reduction reaction of Cu2+ to Cu0 at room temperature[5,22]. These results are consistent with previous research by Li and coworkers on the modification of plant-derived cellulose with copper nanoparticles[18].

Further analysis using TGA revealed the thermal stability and discovered the copper content of the aerogels (Figure 3). The obtained aerogels exhibited the thermal stability were up to 220 °C with a minor mass loss of approximately 6% due to the elimination of adsorbed water from the aerogel matrix, which was consistent with the previous study of Mohite and co-workers[23]. Above 220 °C, the rapid decomposition occurred, generating carbon oxides, water and other gaseous compounds[24]. As reported in our previous study, upon the completion of the combustion, a negligible residual mass of approximately 0.7% for

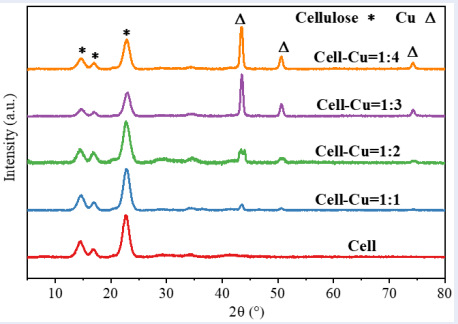

Figure 2: XRD patterns of pristine and Cu-coated bacterial cellulose aerogels.

the pristine BC aerogel was observed, which was attributed to unwashed inorganic additives used in the preparation of *nata de coco*[5]. For the Cu-containing aerogel samples, assuming that the residue was copper(II) oxide after the combustion in air, the Cu content was determined to be 3.9, 6.2, 10.2, and 13.4 for Cell:Cu=1:1, Cell:Cu=1:2, Cell:Cu=1:3, Cell:Cu=1:4, respectively (Table 1).

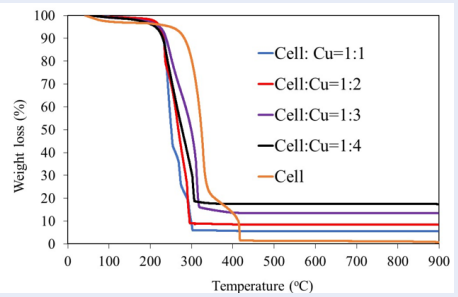

Figure 3: TGA profiles of the Cu-free and Cu-modified BC aerogels[5].

As can be seen in Table 1, the theoretical copper content was higher than the TGA-based result, suggesting copper losses probably during the sample preparation. Under the applied preparation conditions, the loss reason might be explained based on the two facts that excess, non-adherent copper species on cellulose fibers could be leached into water and nano-sized copper particles could pass through the filter paper.

Nitrogen sorption analysis revealed the textural characteristics of the fabricated aerogels (Figure 4). The isotherms exhibited similar adsorption tendencies at P/P_o values between 0 and 0.8, indicating negligible presence of micro- and mesopores. A sharp rise in adsorption capacity above P/P_o of 0.8 confirmed that macropore was dominant in the aerogel structures.

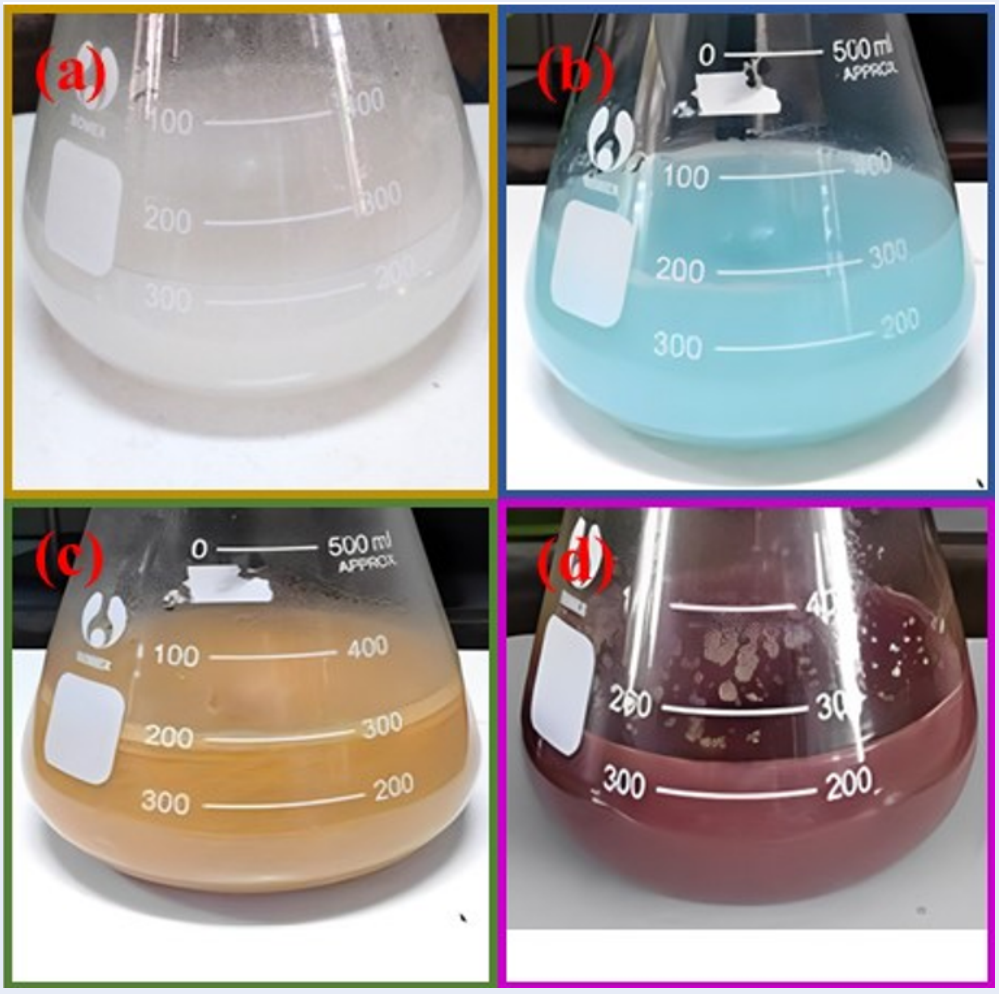

Figure 1: The BC suspension phase in water (a); after the addition of copper(II) acetate (b); after the addition of hydrazine for 1 min (c); after the addition of hydrazine for 14 h (d)

Table 1: Copper content in Cu-modified bacterial cellulose aerogels.

Sample	Cu content (wt.%)	
	Theoretical calculation	TGA calculation
Cu:Cell=1:1	6.02	3.90
Cell:Cu=1:2	11.40	6.16
Cell:Cu=1:3	16.10	10.20
Cell:Cu=1:4	20.40	13.39

It should be noted that increasing the copper content deposited on the BC fibers resulted in a significant reduction in the surface area compared with the pristine BC aerogel whose surface area was previously reported to be 45 m^2/g[5]. In detail, for the samples Cell:Cu=1:1 and Cell:Cu=1:2, the surface area was remained stable at approximately 13 m^2/g. However, further increasing the copper ratio to 10.2 wt.% led to a drastic drop in surface area to approximately 4 m^2/g (sample Cell:Cu=1:3), similar to the value observed at the sample Cell:Cu=1:4 (Table 2). This observed trend suggested that copper particles preferentially occupied the pores available in the pristine aerogel, thus reducing its surface area and pore volume[18].

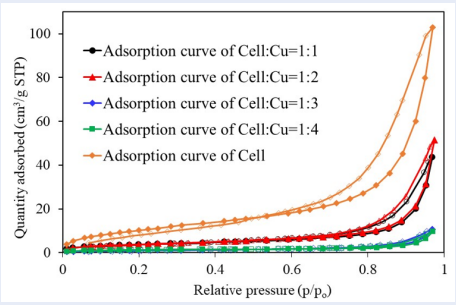

Figure 4: N_2 sorption isotherms of the pristine and Cu-modified BC aerogels[5].

Water contact angle analysis revealed the surface characteristics of copper-loaded aerogels (Figure 5). The Cell:Cu=1:1 aerogel completely interacted with water, indicating insufficient copper particles to encapsulate the highly polar hydroxyl groups of bacterial cellulose. In contrast, the samples Cell:Cu=1:2, Cell:Cu=1:3, and Cell:Cu=1:4 exhibited increasing contact angles, particularly 115.6 °, 131.4 °, and 137.6 °, respectively, implying the inconsiderable interaction between water and copper-coated cellulose fibers. Water droplets could be remained on the aerogel surface. As a result, the aerogels Cell:Cu=1:3 and Cell:Cu=1:4 could float on water. The observed behavior demonstrated the good hydrophobicity of Cell:Cu=1:3 and Cell:Cu=1:4 aerogels. This study suggested a critical copper loading threshold for accomplishing hydrophobicity, highlighting the potential for tuning the surface properties via controlling copper content incorporation.

The oleophilic-liquid adsorption capacity of the BC aerogels was discovered via using cyclohexane solvent as a typical hydrophobic phase (Figure 6). The obtained aerogels exhibited good adsorption due to the low surface energy of cyclohexane, enabling facile penetration into the web-like skeleton. Notably, the copper-free BC-aerogels demonstrated the highest adsorption capacity, reaching 37.7 g/g. However, introducing copper particles led to a decrease in adsorption capacity. In particular, at a 6.02% copper loading (Cell:Cu=1:1), the adsorption capacity decreased by 21%. A further increase in copper content (Cell:Cu=1:3) resulted in a 17.2% lower adsorption efficiency compared to Cell:Cu=1:1, but only 4% lower than Cell:Cu=1:2. The lowest adsorption capacity was observed for the Cell:Cu=1:4, which was a significant reduction compared to the copper-free and lower copper-loading samples. In fact, pristine cellulose aerogels possess the ability to adsorb different liquids but with an unclear selectivity in terms of polarity, which could inhibit the removal efficiency of immiscible liquid from aqueous medium[18,25].

Extended investigation into the adsorption capacity of BC - based aerogels for diesel oil revealed a similar trend observed for cyclohexane, namely a decrease in adsorption efficiency with increasing copper content. This might be owing to the reduction in pore size upon copper incorporation, consequently constraining the available volume within the porous structure. As expected, the Cell:Cu=1:1 sample exhibited the highest adsorption capacity, followed by relatively similar values for Cell:Cu=1:2 and Cell:Cu=1:3, with an obvious drop observed for Cell:Cu=1:4. This suggested that factors beyond pore size, namely hydrophobicity and hydrophilicity balance also determined adsorption behavior. It should be noted that the Cell:Cu=1:1 sample remained hydrophilic, promoting oil adsorption, while the Cell:Cu=1:4 sample turned hydrophobic, potentially promoting oil interaction. However, its significantly lower surface area inhibited its overall adsorption capacity. These mutual influences might be responsible for the decrease in adsorption tendencies observed, aligning with the proposed explanation regarding pore size limitations.

Nanocellulose aerogel modified with hexadecyltrimethoxysilane exhibited an excellent adsorption performance for cyclohexane, reaching approximately 100 g/g while multifunctional polyimide aerogels revealed the similar cyclohexane adsorption capacity of 33 g/g[26,27]. In contrast, the sodium alginate/graphene oxide/silicon oxide aerogel modified with methyltrimethoxysilane possessed lower uptakes for the removal of organic solvent and oils, namely 22 and 23 g/g for cyclohexane and diesel oil, respectively[28]. It should be noted that manufacturing these materials required costly precursors and complicated processes which could inhibit the application cope in capturing oil and

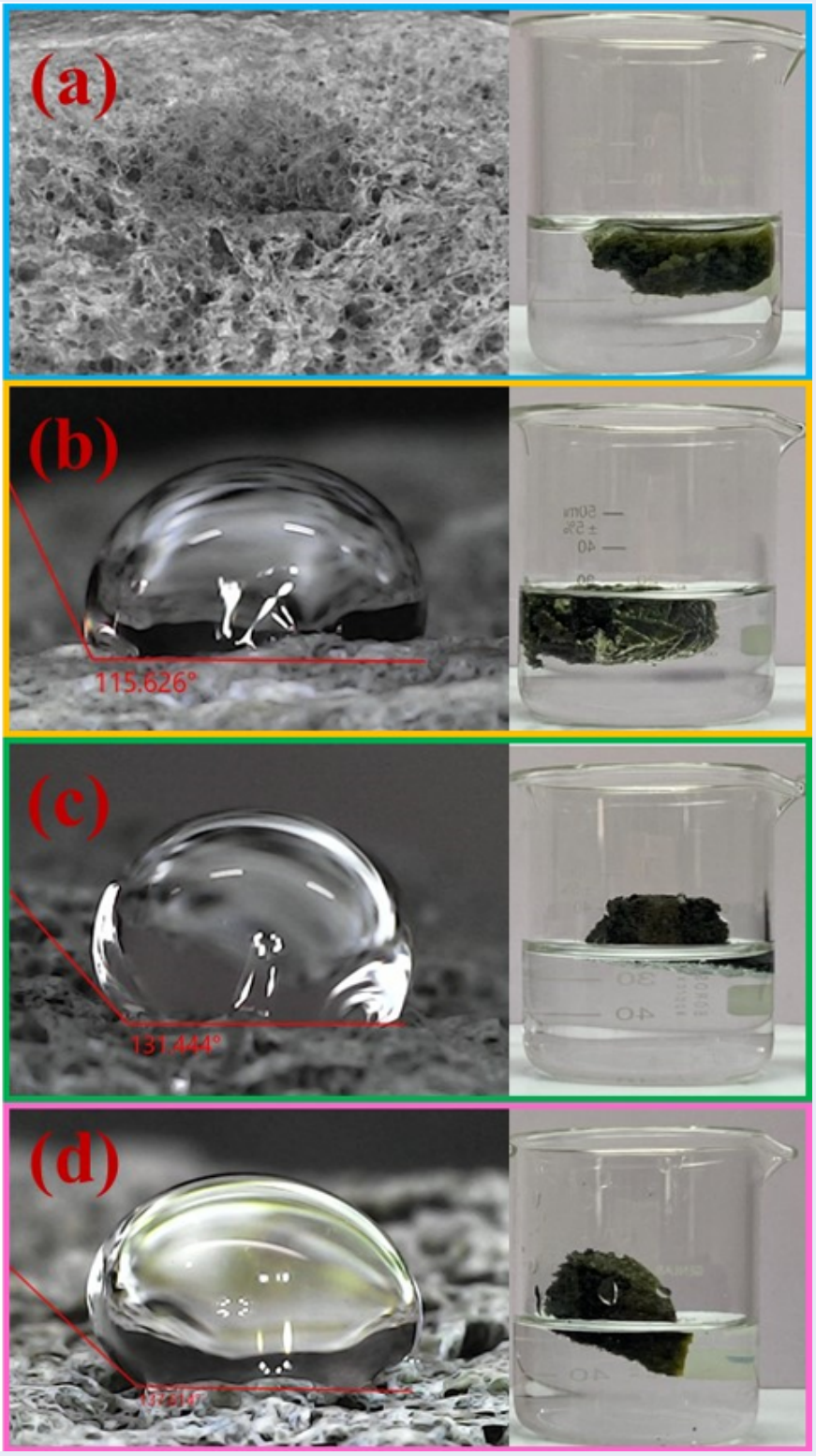

Figure 5: Photographs of water contact angle and water interaction for the Cu-containing BC aerogel samples Cell:Cu=1:1 (**a**); Cell:Cu=1:2 (**b**); Cell:Cu=1:3 (**c**); Cell:Cu=1:4 (**d**).

Table 2: BET specific surface area of pure and Cu-modified BC aerogels[5].

Sample	Specific surface area (m^2/g)
Cell:Cu=1:1	13.4
Cell:Cu=1:2	13.9
Cell:Cu=1:3	4.05
Cell:Cu=1:4	4.59
Cell	45.0

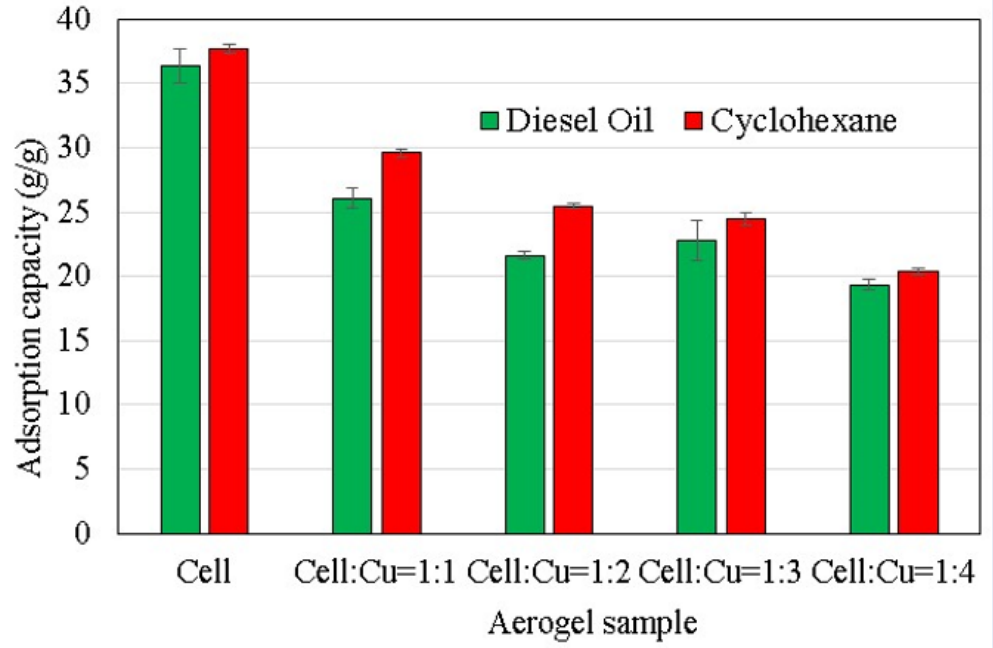

Figure 6: Effect of Cu content and BC ratio on the cyclohexane and diesel oil adsorption capacity.

organic solvents. Therefore, the present study could offer a cost- and step-efficient procedure to yield hydrophobic aerogels for the environmental treatments.

CONCLUSIONS

In conclusion, this study successfully accomplished surface-modified BC aerogels via a feasible liquid-phase reaction and subsequent freeze-drying. The hydrophobic property of the aerogel can be obtained due to the Cu coating. Notably, increasing the Cu content can significantly improve the hydrophobicity of the aerogel while its surface area was declined. On the other hand, the adsorption capacity for cyclohexane and diesel oil in the range of 20-30 g/g demonstrated the high potential of this composite aerogel toward cleaning-up oil spills in the aqueous environment. Focusing on this objective, the selective adsorption of the oil phase in the presence of water or in an emulsion phase would be intensively investigated.

ACKNOWLEDGMENT

We acknowledge Ho Chi Minh City University of Technology (HCMUT), VNU-HCM for supporting this study.

ABBREVIATION

BC: Bacterial cellulose

SEM: Scanning electron microscope

TGA: Thermogravimetry analysis

XRD: X-ray diffraction

CONFLICTS OF INTERESTS

The authors declare that they have no competing interests.

AUTHORS' CONTRIBUTIONS

Ha V. Le: Conceptualization, Methodology, Data curation, Writing – review & editing. **Hanh H. M. Nguyen**: Investigation, Methodology, Writing – original draft. **Trang T. P. Nguyen**: Investigation, Formal analysis, Methodology. **Truc T. T. Nguyen**: Investigation, Formal analysis, Writing – original draft. **Khoi D. Dang**: Methodology, Data curation. **Kien A. Le**: Methodology. **Khoa D. Nguyen**: Writing – review & editing. **Hoan T. Phan**: Funding acquisition, Project administration, Writing – original draft.

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Tạp chí Phát triển Khoa học và Công nghệ – Engineering and Technology 2024, 7(2):2248-2257

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Bài nghiên cứu

Tổng hợp và phân tích vật liệu aerogel kỵ nước chứa cellulose vi khuẩn được phủ đồng thông qua phản ứng khử êm dịu

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TÓM TẮT

Trong nghiên cứu này, cellulose vi khuẩn (bacterial cellulose, BC) đã được bao phủ với đồng thông qua phản ứng khử ở nhiệt độ phòng giữa đồng(II) acetate và hydrazine trong hệ phân tán của thạch dừa trong nước, sau đó được sấy đông khô để tạo thành các aerogel siêu nhẹ kỵ nước. Các đặc trưng cấu trúc của aerogel đã được xác định bằng các kỹ thuật khác nhau bao gồm phương pháp nhiễu xạ tia X (XRD), phân tích nhiệt trọng lượng (TGA), đo góc thấm ướt và hấp phụ nitrogen đẳng nhiệt. Kết quả XRD đã chứng minh sự hình thành của pha đồng kim loại trong aerogel trong khi độ tinh thể cao của cellulose vẫn được duy trì. Hơn nữa, các pha đồng khác không xuất hiện trong vật liệu. Bằng phân tích TGA, hàm lượng đồng có mặt trong vật liệu đã được xác định trong khoảng 3.9 đến 13.4% tùy thuộc vào hàm lượng đồng(II) acetate được sử dụng trong phản ứng khử. Các giá trị này nhìn chung thấp hơn so với hàm lượng đồng theo lý thuyết do sự thất thoát trong quá trình tổng hợp vật liệu. Việc tăng hàm lượng đồng trong aerogel đã dẫn đến diện tích bề mặt của vật liệu giảm đáng kể với sự có mặt của đồng trong cấu trúc mao quản của vật liệu. Tuy nhiên, như mong đợi, tính kỵ nước của vật liệu đã được cải thiện đáng kể khi tăng hàm lượng đồng. Các mẫu aerogel chứa hàm lượng đồng cao (> 6.2 wt.%) kỵ nước với góc thấm ướt lên đến 138°. Do đó, các aerogel kỵ nước tương tác tốt với các dung môi hữu cơ không tan trong nước như diesel oil và cyclohexane với hiệu quả hấp phụ từ 20 đến 30 g/g. Việc tổng hợp thành công các aerogel kỵ nước dựa trên sự biến tính bề mặt BC với đồng có thể cung cấp những vật liệu hấp phụ mới và hiệu quả trên cơ sở sinh khối hướng đến việc xử lý các chất lỏng dạng dầu trong môi trường nước.

Từ khoá: aerogel kỵ nước, cellulose vi khuẩn, bao phủ, đồng, phản ứng khử êm dịu

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Lịch sử

- Ngày nhận: 20-2-2024
- Ngày chấp nhận: 23-5-2024
- Ngày đăng: 30-9-2024

DOI : https://doi.org/10.32508/stdjet.v7i2.1339

Trích dẫn bài báo này: Hà L V, Hạnh N H M, Trang N T P, Trúc N T T, Khôi D D, Kiên L A, Khoa N D, Hoàn P T. **Tổng hợp và phân tích vật liệu aerogel kỵ nước chứa cellulose vi khuẩn được phủ đồng thông qua phản ứng khử êm dịu**. *Sci. Tech. Dev. J. - Eng. Tech.* 2024, 7(2):2248-2257.