Nanocellulose-Based Adsorbent for Cu(II) Adsorption

Haziqatulhanis Ibrahim
Faculty of Mechanical and Automotive Engineering Technology, Universiti Malaysia Pahang, Malaysia
haziqahanis.ibrahim@gmail.com

Norazlianie Sazali
Faculty of Manufacturing and Mechatronic Engineering Technology, Universiti Malaysia Pahang, Malaysia
azlianie@umpsa.edu.my (corresponding author)

Kumaran Kadirgama
Faculty of Mechanical and Automotive Engineering Technology, Universiti Malaysia Pahang, Malaysia
kumaran@umpsa.edu.my

Wan Norharyati Wan Salleh
Advanced Membrane Technology Research Centre (AMTEC), Universiti Technology Malaysia, Malaysia
hayati@petroleum.utm.my

Triyanda Gunawan
Department of Chemistry, Faculty of Science and Data Analytics, Institut Teknologi Sepuluh Nopember, Sukolilo, Indonesia
triyanda@its.ac.id

Nurul Widiastuti
Department of Chemistry, Faculty of Science and Data Analytics, Institut Teknologi Sepuluh Nopember, Sukolilo, Indonesia
nurul_widiastuti@chem.its.ac.id

Afdhal Junaidi
Department of Chemistry, Faculty of Science and Data Analytics, Institut Teknologi Sepuluh Nopember, Sukolilo, Indonesia
aafdhaljunaidi@gmail.com

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ABSTRACT

This study addresses the critical issue of copper removal from wastewater due to environmental and health concerns. Choosing pandan leaves as a source of cellulose was a deliberate decision due to their abundant availability in nature and minimal ecological footprint. Through the utilization of these properties, this study synthesized nanocellulose with enhanced adsorption capabilities by employing chemical pretreatments, sulfuric acid hydrolysis, and acrylamide grafting with the aid of ceric ammonium nitrate (CAN) as an initiator. In order to thoroughly evaluate the synthesized material, X-Ray diffractometer (XRD) and Fourier transform infrared (FTIR) spectroscopy were used. These characterization methods revealed insights into the morphology, functionality, and crystallinity of nanocellulose. The removal of copper(II) ions is investigated by employing an atomic absorption spectrometer (AAS), focusing on three important factors: pH variation, initial concentration, and adsorbent dosage, which are carefully

examined. Grafted nanocellulose demonstrates superior performance, achieving over 85% grafting efficiency. Optimal Cu(II) removal conditions are identified at pH 6, with an initial metal ion concentration of 30 ppm and an adsorbent dose of 2.2 g/L. This study not only addresses a critical concern in wastewater treatment, but also explores the potential of pandan leaf-derived nanocellulose as a sustainable solution for heavy metal removal.

Keywords- nanocellulose; copper removal; pandan leaves; wastewater treatment

I. INTRODUCTION

Clean water sources are increasingly limited as industrialization and globalization continue to progress. Water is an essential resource for all living organisms, but ensuring its purity in today's world is a significant challenge [1]. On a daily basis, a significant amount of industrial wastewater is released into the water system [2]. This detrimental practice impacts both oceanic and river ecosystems. The water discharged from industries is contaminated with organic pollutants and toxic heavy metals like Ni^{2+}, Cu^{2+}, and Zn^{2+}, posing a threat to the environment [3-5]. Marine life is likely to be subjected to significant exposure to toxic contaminants, while other beings such as humans and land animals are also at high risk of being impacted through the food chain [6]. Copper, specifically in its Cu(II) form, has become a noteworthy pollutant, imposing serious health risks, including hypertension, fetal malformation, and kidney damage [7, 8].

Various methods have been employed to eliminate heavy metals from wastewater, including precipitation, ion exchange, membrane filtration, reverse osmosis, and electroplating [9]. Existing technologies are reaching their limits, necessitating the introduction of innovative approaches to tackle the issue. The most efficient and cost-effective solution is the removal of heavy metals in wastewater through adsorption [10]. Many sorbents, such as activated biomass, clay minerals, and natural zeolites, have been deployed for the purpose of heavy metal removal [11]. Traditional sorbents have limited effectiveness and poor results when it comes to extracting heavy metal ions [12].

Various nanostructured materials have been developed to address the problem at hand, demonstrating superior sorption efficiency compared to traditional adsorbents [13]. Recent technological advancements have led to a growing body of research focused on the use of nano-sized particles for the removal of heavy metal pollutants. The surface modifiability of nanomaterials and the high surface-to-volume ratio make them a preferred choice among researchers [14, 15]. Numerous studies have been conducted over a decade on the properties of nanomaterials, leading to the agreement that they hold great promise in addressing environmental concerns. Currently, researchers are primarily focused on nanocellulose as an environmentally friendly and easily obtainable substance [16, 17].

Cellulose can be typically derived from many sources, mainly cellulose-containing plants, such as lemon peels, pea peels, watermelon rinds, and more [18, 19]. However, in the present study, cellulose was extracted from the leaves of Pandanus amaryllifolius. These leaves are abundantly encountered in Malaysia and were chemically treated to break down the structure into nanocellulose. Nanomaterials are known to have the promising characteristic of increased sorption efficiency due to their large surface-to-volume ratio. Therefore, the modification of the cellulose backbone through graft copolymerization allows more functional groups, increases the sites for adsorption, and improves the metal-binding ability [20]. To date, many vinyl monomers have been reported for the purpose of grafting with natural adsorbents [21]. However, works highlighting the heavy metal adsorption performance of nanocrystal cellulose derived from pandan leaves and grafted with polyacrylamide (PAM) remain scarce. Thus, this paper presents the removal of Cu(II) from wastewater by using a nanocellulose adsorbent extracted from pandan leaves.

II. MATERIALS AND METHODS

A. Materials

Cellulose was extracted from fresh raw pandan leaves, which were collected from a residential area in Johor, Malaysia. The chemicals utilized to extract cellulose consisted of nitric acid (HNO_3) 65%, sodium chlorite ($NaClO_2$) 80%, and sulfuric acid (H_2SO_4) 97%, obtained from Sigma-Aldrich (Merck). Sodium hydroxide pellets (NaOH) were purchased from QReC. For graft copolymerization, ceric ammonium nitrate ($(NH_4)_2Ce(NO_3)_6$), acrylamide ($CH_2CHCONH_2$), and dimethylformamide (DMF) from Sigma-Aldrich were implemented. A 1000 ppm stock solution of Cu(II) nitrate trihydrate $Cu(NO_3)2.3H_2O$ was prepared for the adsorption studies and distilled water was used throughout the experiment.

B. Methods

1) Adsorbent Preparations

The adsorbents deployed in this experiment were nanocellulose (ungrafted) and nanocellulose-g-polyacrylamide (grafted), synthesized in the laboratory. First, fresh pandan leaves were washed with running water to remove dirt before being cut into smaller pieces and dried in an oven. The dried leaves were then ground into powder form and treated under an alkali treatment and bleaching treatment by using a 5 wt% NaOH solution and $NaClO_2$ at 125°C for 2 hours. The samples were neutralized after each step until they reached pH 7. In the bleaching treatment, a few drops of HNO_3 were added to the solution, and at the end a white sample was obtained. The acid hydrolysis of the cellulose was contacted according to the method described in [22]: The white sample was supplemented with 60 wt% of H_2SO_4, followed by a cellulose-to-liquor ratio of 5:100 (wt%) at 125°C for 1 hour. Next, cold distilled water was added to the mixture, and the white suspensions were dialyzed until the pH turned neutral.

2) Graft Copolymerization

Grafted cellulose was prepared by adding 10 g/L nanocellulose to a Ceric Ammonium Nitrate (CAN) initiator for 10 min. Then, dissolved acrylamide was added to the cellulose mixture and subjected to a reaction for 1 hour at 60 °C. The crude grafted cellulose was filtered and washed with deionized water before being subjected to drying at 40 °C until a constant weight was obtained. To remove traces of homopolymer, it was washed with a DMF solution and dried for 48 hours.

3) Characterization

The samples were characterized by a Fourier transform infrared spectrophotometer (FTIR, Perkin Elmer). The crystallinity of the samples was analyzed putting into service an X-ray diffractometer (XRD, D5000 Siemens). The concentration of Cu(II) ions was analyzed by atomic adsorption spectroscopy (AAS, AA-7000 Shimadzu).

4) Adsorption Studies

Batch-mode experiments were conducted to study the equilibrium conditions for Cu(II) removal from wastewater using nanocellulose and grafted nanocellulose as adsorbents. Three parameters were studied: solution pH (2–6), initial metal ion concentrations (15–75 ppm), and adsorbent dosage (0.6–2.2 g/L) to optimize adsorption efficiency. These ranges consider factors such as the predominant ionization states of heavy metals, maintaining a suitable concentration gradient for adsorption, and achieving optimal utilization of adsorbent material without wastage. These ranges optimize conditions for efficient heavy metal removal while reducing operational constraints and resource usage. The pH values were adjusted by utilizing 0.1M HCl or 0.1M NaOH. All experiments were carried out at room temperature. The equilibrium adsorption capacity for the adsorbent was calculated via the following equation:

Adsorption capacity, q_e (mg/g)

$$q_e = \frac{(C_0 - C_e)V}{m} \tag{1}$$

where C_0 (ppm) and C_e (ppm) are the concentrations at initial and equilibrium, respectively and V (L) is the solution volume and m (g) is mass of adsorbent used.

III. RESULTS AND DISCUSSION

A. FTIR Analysis

The FTIR spectra of raw (dried pandan), treated (after bleaching), and grafted samples are shown in Figure 1. These spectra indicate that the samples have a similar chemical composition. In particular, the spectra a and b depict the typical cellulose composition with characteristic bands at 3000–3700 cm^{-1} (-OH stretching), 2915 cm^{-1} (C=C-H stretching), 1425 cm^{-1} (CH2 symmetric bending), 1150 cm^{-1} (C-O of primary -OH stretching), and 1040 cm^{-1} (C-O-C anti-symmetric bridge stretching [23]. On the contrary, the peak disappearance and reduced transmittance encountered between a and b denote the removal of lignin, hemicellulose, and other extractives as a result of the chemical pretreatment process. The properties of amide are further noted in c, where a small peak at 3340 cm^{-1} is attributed to the N-H stretching and the peak at 1640 cm^{-1} to the C=O stretching from the amide group, respectively [24].

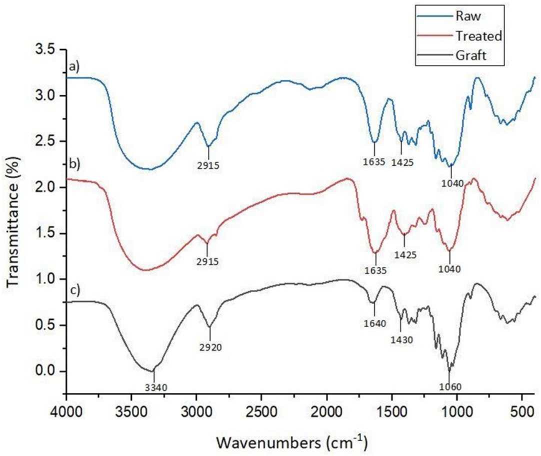

Fig. 1. FTIR spectra for a) raw pandan leaves, b) treated cellulose, and c) grafted nanocellulose.

B. XRD Analysis

The XRD graphs of raw (i.e., dried pandan), treated (i.e., post-bleaching), and grafted samples are portrayed in Figure 2, thus explaining the changes observed after the pre-treatment and grafting processes. Naturally, the raw sample revealed a highly amorphous percentage due to the presence of components other than cellulose on the molecular chain. Furthermore, the diffraction peaks that denote the crystallinity and amorphous regions of cellulose could be observed at 2θ = 16° and 22.5° for the three patterns [25]. A comparison of the samples revealed greatly increased crystallinity for the treated and grafted samples as opposed to that of the raw sample, proving the enhancement in structural stability. However, the XRD pattern for the grafted sample depicted lower crystallinity compared to the treated sample because of the grafting process, which involved nanocellulose dispersion in water. This increases its amorphous regions [26].

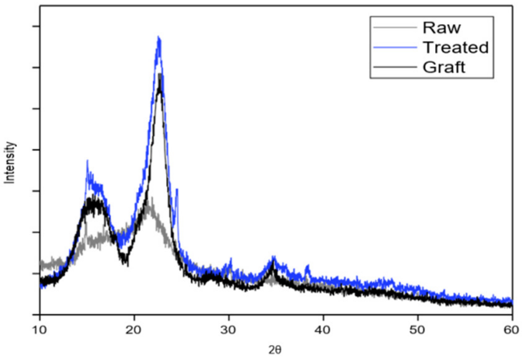

Fig. 2. XRD graph for raw pandan leaves, treated cellulose and grafted cellulose.

C. Solid Accumulation

The influence of the Vinyl Monomer to Cellulose (VM:C) ratio and the amount of initiators on the Grafting Efficiency (GE) is presented in Table I. A decreasing trend was observed related to the influence of the VM:C ratio used, which was contradictory to the pattern reported in [20]. Therefore, this could be attributed to the decreasing sites found on the cellulose backbone that are available for the monomer to attach itself. In particular, the optimum condition for graft copolymerization between the VM:C ratio was obtained when 1g of cellulose and 1g of acrylamide were utilized. On the contrary, the employment of 0.7g of redox initiator yielded the highest percentage of grafting efficiency, 87.40%. Beyond the optimum condition (0.7g of CAN), the grafting efficiency exhibited a significant decrease [27]. According to [28], CAN ions prefer the formation of a complex with acrylamide to cellulose, while homopolymer formation reduces the efficiency of the process.

TABLE I. GRAFTING EFFICIENCY FOR VARIOUS MONOMER-TO-CELLULOSE RATIOS AND INITIATOR AMOUNTS

VM:C	GE (%)	CAN (g)	GE (%)
1:1	86.68	0.1	75.48
3:2	83.82	0.3	79.85
2:1	81.53	0.5	86.68
5:2	79.29	0.7	87.40
		0.9	72.44

D. Adsorption Study

1) Effect of pH

As evidenced in Figure 3, the trendline for the adsorption capacity increases with pH. This could be explained by the gradual decrease in the concentrations of hydronium and H_3O^+ ions along with the pH increase, while a positively charged Cu ion could bind to the active sites on the adsorbent surface. Beyond pH 7 and approaching alkaline conditions, the adsorption capacity could not be measured as the Cu started to precipitate and yield $Cu(OH)^{2+}$ [29]. In particular, most researchers have reported an optimum removal of Cu ions at pH values between 5 and 6 [24], [30, 31]. Thus, in this work the pH was limited to 6 accordingly. For the grafted samples, the acidic condition caused the amino groups to undergo protonation due to the high H^+ concentration. Therefore, the adsorption capacity of the grafted adsorbent was 27.37 mg/g at the optimal pH, which denotes Cu removal around 91% of the initial concentration.

2) Effect of Initial Concentration

The initial concentration of the metal ions is another important factor that affects their uptake. For example, if there are more heavy metals in a solution at the beginning, the adsorption capacity will be higher because of the concentration gradient. However, when the initial concentration exceeds the optimal value, the adsorption performance declines due to insufficient binding sites available on the adsorbent surface. Figure 4 manifests satisfactory removal performance for both grafted and non-grafted samples at the initial metal ion concentration of 30 ppm. A possible limitation may be due to the low amount of the adsorbent dosage used in the experiment. For the initial concentration study, the pH employed was 6, which was found to be the optimum concentration for Cu removal according to Figure 3. Previous studies have noted that the initial concentration of heavy metals typically reaches the optimum adsorption capacity at a concentration ranging from 10 to 100 ppm [32].

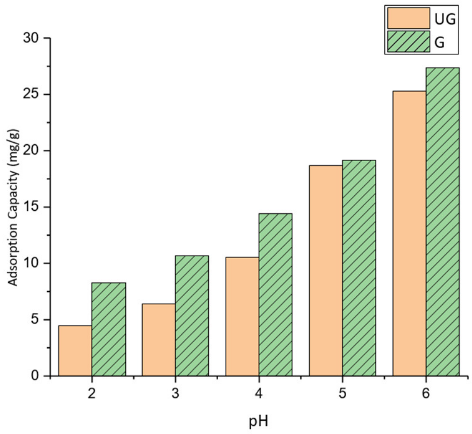

Fig. 3. Effect of solution pH on the adsorption capacity of the adsorbent to remove Cu(II) ions (adsorbent dosage = 1 g/L; initial concentration of metal ions = 30 ppm; reaction time = 180 mins).

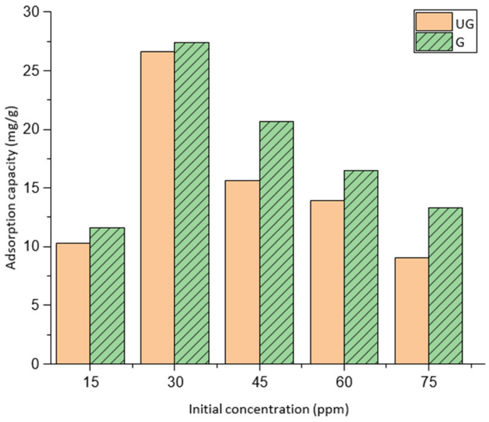

Fig. 4. Effect of the initial concentration of metal ions on the adsorption capacity of the adsorbent to remove Cu(II) ions (adsorbent dosage = 1 g/L; solution pH = 6; reaction time = 180 mins).

3) Effect of Adsorbent Dosage

The significance of adsorbent dosage cannot be denied in the adsorption process, as it determines the percentage of removal and the process economy. Therefore, increasing the dose of the absorbent to a certain limit offers more adsorbing sites for the binding of metal contaminants, thus amplifying the effectiveness of ion removal from a solution [33]. Contrarily, the adsorption capacity shows a decrease when the adsorbent dosage is increased due to the unsaturation of metal ions on the adsorbent surface. This is observed in Figure 5, following the pattern depicting a decreasing value of the adsorption capacity. The grafted sample yielded better performance compared to the non-grafted sample at the same amount of adsorbent dosage, this may be possibly attributable to the additional functional groups obtained from the acrylamide monomer. After reaching the optimal dose, no significant increase in the removal percentage was reported, while a further increase in the amount of dosage led to a decrease in removal efficiency [34].

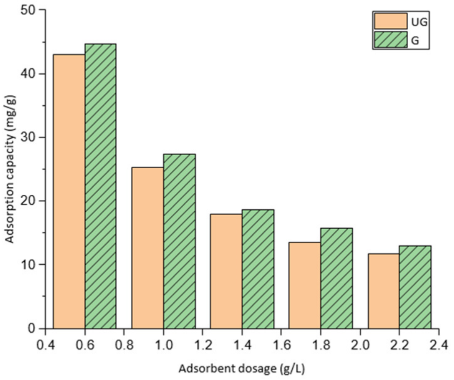

Fig. 5. Effect of the dosage of the adsorbent on the adsorption capacity of the adsorbent to remove Cu(II) ions (initial concentration of metal ions = 30 ppm; solution pH = 6; reaction time = 180 mins).

IV. CONCLUSIONS

In this study, nanocellulose was successfully extracted from pandan leaves and underwent graft copolymerization with acrylamide to modify its surface. The maximum grafting efficiency reached 87.40% when the ratio of vinyl monomer to cellulose was 1:1 and 0.7g of the initiator amount was implemented. The FTIR analysis confirmed the presence of acrylamide functional groups on the cellulose backbone. Batch adsorption tests done under different conditions on the two types of adsorbents discloded that grafted samples have slightly higher performance on copper ions removal compared to ungrafted nanocellulose. Despite the laboratory-scale experiment carried out, further optimization is necessary to ensure its cost-effectiveness in industrial-scale wastewater, as industrial wastewater is much more complex and consists of multiple pollutants and heavy metals. However, this work could be the first step in the commercialization of a natural cellulose-based adsorbent in water treatment technology.

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