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ORIGINAL ARTICLE

Fused nanogranular polyaniline-sawdust (Cocos nucifera) composite for Lead adsorption application

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Abstract

The problem of heavy metal contamination in industrial effluents requires new and environment-friendly agents for wastewater treatment. In this study, we developed fused nanogranular polyaniline-sawdust (PANI/SD) composites for lead ion removal through the adsorption process. To prevent the aggregation of polyaniline (PANI), coconut (*Cocos nucifera*) sawdust (SD), an agricultural waste, was used as a substrate via *in situ* chemical polymerization in varied sawdust-to-aniline ratios. The scanning electron micrographs (SEM) of the obtained PANI/SD composites revealed a nanogranular structure that indicated the complete coating of polyaniline on sawdust. Furthermore, the PANI/SD composites were verified to be in the emeraldine oxidation state through Fourier-Transform Infrared (FT-IR) spectroscopy. Among the formulations studied, the PANI/SD composite with a sawdust-to-aniline ratio of 1.2 g/mL was found to have the highest adsorption capacity of 738.9 mg/g. This study presents the promising potential of PANI/SD as a novel and cost-effective adsorbent material to remove lead from contaminated water.

Keywords: *Adsorption; Lead; Polyaniline; Sawdust; Wastewater.*

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INTRODUCTION

Lead is a naturally occurring metal that is used in various industrial applications because of its malleability, high density, and low melting temperature [1]. The advancement of technology and the increase in population demand more usage of this metal, leading to an increase in the production of materials containing lead. Hence, the probability of the dispersion of this toxic metal and its contact with the environment also rises [2]. Predominantly, pollution-causing metals in natural waters come from the emission of domestic and industrial effluents and the dumping of sewage sludge. The increase in the amounts of lead in soil, air, and water leads to the build-up of this toxic chemical in the food chain, affecting humans and other living organisms. Exposure to this toxic metal can lead to psychological and neurobehavioral dysfunctions and can detriment almost all organ systems in the body especially the central nervous system, kidneys, and blood, and can lead to death at extreme exposures [3].

Conventional means of treating lead-containing wastewater include chemical precipitation, ion exchange, electrolytic recovery, photocatalysis, coagulation and flocculation, reverse osmosis, and ultrafiltration. Despite their advantages in removing lead from wastewater, these processes entail high-operational costs, high-energy consumption, and handling costs for sludge disposal [4]. Hence, low-cost alternatives

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have been developed to address this problem.

Adsorption, a process that involves the accumulation of a solute on the surface of a material, offers a comparatively simpler process for treating wastewater and effluents. It is an attractive option for cleaning wastewater, as it does not require high-energy-operating equipment or incur high-operational costs. Activated carbons, phosphatic clay, and biosorbents have been widely used as adsorbents to remove heavy metals from wastewater. Additionally, various studies on heavy metal removal have utilized peanut and almond shells, acacia gum, waste tea leaves, eggshells, and polymers [5-9].

Polymers exhibit superior properties as synthetic and natural adsorbents because they are easy to handle, selective, and cost-effective [10]. One of those polymers is polyaniline (PANI), a conducting polymer that has been the subject of many recent researches due to its environmental stability, affordability, and ease of synthesis. PANI has been applied to a variety of applications, including its use as a fluorescence sensor, ion exchange material, and for the removal of toxic metals [5, 11, 12]. However, PANI particles tend to aggregate in solution, resulting in low adsorption capacity due to reduced surface exposure and slow kinetics [13]. Researchers have utilized templates to provide surfaces where PANI chains anchor, thereby preventing the formation of aggregates. Hence, polyaniline composites were developed to enhance the adsorption efficiency of PANI-based materials. Furthermore, deprotonating the composite can further enhance its capability to remove heavy metals [14].

Nanomaterials were integrated into the polyaniline polymer matrix for the recovery of heavy metals, including metal oxides or hydroxides, carbon- and silica-based materials, and magnetic materials [15, 16]. However, these nanomaterials are associated with high costs and potential nanotoxicity risks [17]. Agricultural waste has been widely utilized as a substrate for polyaniline. For instance, PANI/rice husk, PANI-sawdust composites, such as pine sawdust, and polyaniline-coconut fiber composites exhibit increased removal of heavy metal ions from aqueous solutions. [10, 18-23]. Synthesizing PANI on low-cost templates such as agricultural wastes, is a viable method to enhance its adsorption capacity. While several templates have been employed for the synthesis of PANI-based composites, there is a notable lack of studies focusing on PANI/*Cocos Nucifera* (coconut) sawdust composites and their adsorption capacity. This research gap presents a unique opportunity to explore the potential of PANI/*Cocos Nucifera* sawdust composites as an alternative and efficient adsorbent for heavy metal removal. It is highly available in the country with minimal or no production cost. By capitalizing on this agricultural waste, we can harness its potential and contribute to a more eco-friendly and cost-effective approach to heavy metal recovery.

In this research, we present the potential of polyaniline-coconut sawdust (PANI/SD) composites as an adsorbent material to remove lead in aqueous solutions. PANI/SD composites were chemically synthesized using potassium dichromate as an oxidant and hydrochloric acid as a dopant. The *in situ* polymerization of aniline on coconut (*Cocos nucifera*) sawdust was done with varying sawdust-to-aniline (SD-An) ratios. The lead adsorption capacity and percentage of PANI/SD were determined through batch adsorption experiments.

EXPERIMENTAL

Preparation of sawdust

The coconut (*Cocos nucifera*) sawdust (SD), obtained from a local sawmill, was sieved, and washed with tap water until the water became colorless. The sawdust was then subjected to acid treatment, following a referenced method with some modifications [24]. It was washed successively with 2 M hydrochloric acid (HCl) and distilled water using a magnetic stirrer rotating at 890 rpm. The sawdust was dried at room temperature for two (2) days and then oven-dried at 110°C for 3 hours and kept in a sterilized jar.

Synthesis of polyaniline and polyaniline-sawdust composites

Polyaniline (PANI) composite was synthesized with an aniline-dichromate ratio of 3.0, which was previously used to achieve 84% polymer yield, resulting in PANI in the emeraldine oxidation state [25, 26]. It was carried out by preparing 100-mL solutions of 6.02 mL of aniline and 4.816 g of sawdust, with an 0.8 g/mL sawdust-to-aniline (SD-An) Ratio. Additionally, 6.48 g of potassium dichromate ($K_2Cr_2O_7$) was dissolved separately in 3.5 M HCl. These solutions were mixed and stirred for 15 minutes to form a homogenous solution with an aniline-dichromate ratio of 3.0. During the polymerization process, temperature changes in the mixture were recorded every 10 seconds for the initial 15 minutes of the reaction to obtain its temperature profile. The mixture was then left to undergo complete polymerization for sixteen (16) hours. The resulting polymer was washed with 600 mL of distilled water and filtered to remove impurities. The product was dried at room temperature for 24 hours and further oven-dried at 70ºC for 3 hours. The obtained PANI composite was pulverized. The sawdust-to-aniline (SD-An) ratios were varied from 0.8 g/mL to 2.0 g/mL with a 0.4 interval. Control set-ups were also prepared, containing pure aniline and pure sawdust.

To deprotonate the composites, 25 mL of 0.5 M of ammonia solution was added for every gram of polyaniline-sawdust (PANI/SD) powder. The mixture was stirred and left for sixteen (16) hours. Then, the solution was filtered and dried in an oven at 60-70ºC for two hours to completely remove any excess ammonia [27].

Characterization

The Fourier Transform Infrared (FT-IR) spectrum was recorded by using Spectrum 100 FT-IR spectrometer (Perkin Elmer, UK) in the range of 4000-450 cm^{-1}. The oxidation state of PANI was determined by calculating the ratio of quinoid to benzenoid band intensities (Q/B ratio). A Q/B ratio equal to 1 indicates that the polymer is in its emeraldine oxidation state [28]. The morphology of PANI/SD composites was examined by using a scanning electron microscope (SEM, equipment model: JEOL) with a magnification range of 500-20,000×.

Batch adsorption experiments

To determine the adsorption capacities of pure PANI, PANI/SD composites, and pure sawdust, batch adsorption experiments were conducted. First, 45 mL of 0.1 M lead nitrate solution was added to each beaker containing the adsorbents. Upon establishing the adsorption equilibrium, the mixtures were filtered, and the filtrates were analyzed using Atomic Absorption Spectrophotometer (AAS) at the Department of Science and Technology IX-Regional Standards and Testing Laboratories, Zamboanga City, Philippines. The analysis was conducted using the official AOAC 999.10 protocol as provided by the Association of Official Analytical Chemists International [29]. The experimental runs were conducted and the concentrations of adsorbed lead (II) ions were measured and recorded. The initial concentration of the adsorbate solution (0.1 M), contact time between PANI/SD and lead nitrate solution (15 minutes), and the dosage of PANI/SD (1g per 45 mL lead nitrate solution), were held constant in this study. The adsorption capacity (q) and adsorption percentage (φ) were obtained using the following formula [19-21]:

$$q = (C_0 - C_e)V/m \tag{1}$$

$$\varphi = 100(C_0 - C_e)/C_0 \tag{2}$$

In these equations, C_0 and C_e are the initial and equilibrium concentrations (mg/L) of the adsorbate, respectively. V denotes the volume of the test solution used in the adsorption experiment, and m is the mass of the adsorbent.

RESULTS AND DISCUSSION

Scanning Electron Microscopy (SEM) Analysis

Fig. 1 shows the SEM micrographs of pure sawdust (SD), polyaniline (PANI), and polyaniline-sawdust (PANI/SD) composites. The micrograph of pure sawdust (Fig. 1a) reveals a structure composed of bundled nanofibers. On the other hand, the PANI/SD composites (Fig. 1c-1f) showed fused nanogranular structures similar to those observed in pure PANI (Fig. 1b). The diameters of the particles of PANI/SD composites were found to range from 282 to 380 nanometers. The sawdust nanofibers were no longer observed after the fabrication of the PANI/SD composites, indicating complete coating of the sawdust particles with PANI.

Stejskal et. al. [30] reported that granular structure is a common morphology observed in PANI obtained through the precipitation polymerization of aniline in highly acidic media. The individual PANI/SD nanostructures formed during synthesis can be described by the following mechanism. During the induction period, short aniline oligomers, known as nucleates, are formed. Due to their hydrophobic nature, these nucleates adsorb onto the surfaces of the sawdust. The nucleates then undergo random and continuous aggregation or “stacking.” The hydroxyl groups of the cellulose in sawdust form hydrogen bonds with the free nitrogen groups in PANI. This starburst growth of PANI leads to the formation of the first granule of PANI/SD. Subsequently, the nucleates accumulate as droplets on the surfaces of the PANI/SD granules and initiate the growth of new

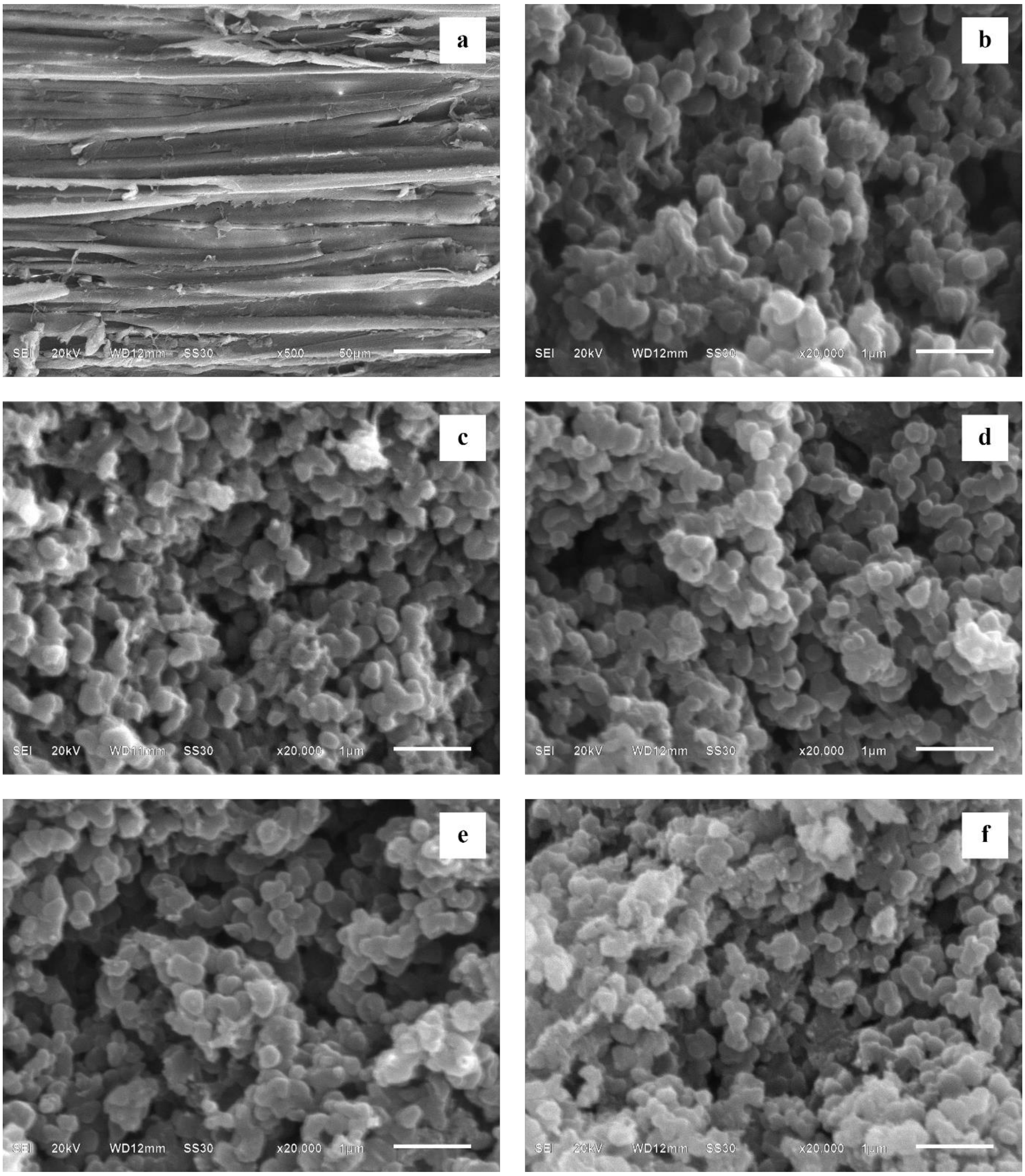

Fig. 1. Scanning electron microscopy (SEM) images of (a) Pure sawdust at 500× magnification, (b) Pure PANI, (c) 0.8 SD-An Ratio PANI/SD composite, (d) 1.2 SD-An Ratio PANI/SD composite, (e) 1.6 SD-An Ratio PANI/SD composite, and (f) 2.0 SD-An Ratio PANI/SD composite, all at 20,000× magnification.

granules at the surface of the completed ones. As a result, a fused nanogranular morphology is formed.

Fourier Transform Infrared (FT-IR) Spectroscopy

Fig. 2 shows the FT-IR spectra of sawdust, PANI and PANI/SD composites synthesized in various sawdust-aniline ratios. Characteristic peaks of PANI are observed in the absorbance spectra of PANI/SD composites. The bands within the region of 1499-1592 cm^{-1} correspond to the nitrogen-bonded benzenoid (N=B=N) and quinoid (N=Q=N) stretching. The bands at 1304-1307 cm^{-1} are attributed to the C-N stretching of a secondary

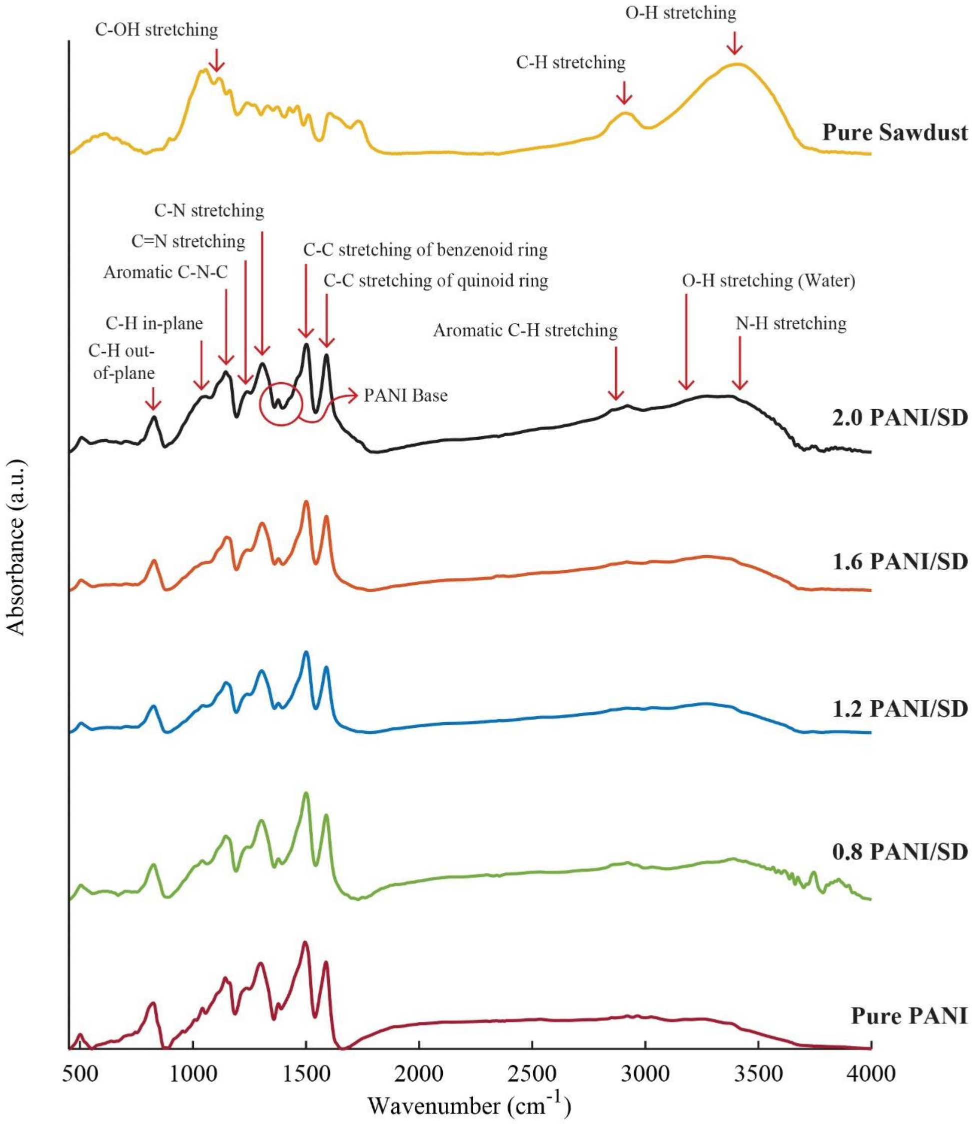

Fig. 2. FT-IR spectra of pure sawdust, PANI and PANI/SD composites along with their corresponding vibrational assignments.

aromatic amine. Furthermore, peaks are observed at 2919-2922 cm^{-1} (aromatic C-H stretching) and at 829-833 cm^{-1} (C-H out-of-plane deformation). Characteristic peaks of water are also present at 3100-3700cm^{-1}, indicating that the samples were not completely dry during the characterization process.

The ratio of the absorption intensities of the quinoid to benzenoid peaks of PANI and PANI/SD composites were computed and were found to range from 0.77 to 0.89. These values are close to the ideal Q/B ratio of 1 for emeraldine. This finding confirms that both PANI and PANI/SD composites are in emeraldine oxidation state. Additionally, the FT-IR spectra of PANI and PANI/SD composites exhibit a weak absorbance peak at 1375 cm^{-1} (indicated by the red circle in Fig. 2) indicating successful deprotonation of PANI and PANI/SD composites.

The bands in the region of 1713-1747 cm^{-1} suggest the presence of C-NH stretching. Vibrational frequencies within this region for the PANI/SD composites are observed to shift to higher wavenumbers, compared to PANI. This can be attributed to the physiochemical interaction between PANI and the sawdust, further supporting our claim that the hydroxyl groups of sawdust interacted with the NH groups in PANI.

Table 1. Adsorption capacities and percentages of the adsorbents.

Adsorbents	Adsorption Capacity (mg/g)	Adsorption Percentage (%)
Pure Sawdust	239.4	25.7
Pure PANI	437.4	46.9
PANI/SD (SD-An Ratio)		
0.8	257.4	27.6
1.2	738.9	79.2
1.6	266.4	28.6
2.0	86.4	9.27

Sawdust is mainly composed of cellulose $(C_6H_{10}O_5)_n$ and shows a fibrous structure, as seen in its SEM image (Fig. 1a). Its FT-IR spectrum shows peaks at 3463, 2911, and 1118 cm^{-1}, which correspond to O-H deformations, C-H stretching and C-OH stretching, respectively. However, these prominent peaks are not observed in the FT-IR spectra of the PANI/SD composites. This supports the SEM observation that PANI completely coated the surface of the sawdust particles.

Atomic Absorption Spectroscopy (AAS)

After conducting the batch adsorption experiments, the filtrates were analyzed via AAS. This instrument measures the remaining amount of lead (II) in the filtrates after the experiments. Using these data, the adsorption capacities and percentages were computed. Table 1 shows the computed adsorption capacities and percentages of pure sawdust, PANI and PANI/SD composites.

Compared to other adsorbents in this study, PANI/SD with sawdust-to-aniline ratio of 1.2 had the highest adsorption capacity of 738.9 mg/g. This is greater than the adsorption capacity of pure PANI which was 437.4 mg/g. The enhanced adsorption capacity of PANI/SD can be attributed to the increase in the surface area due to the presence of sawdust. This suggests that the PANI coating on the surface of the sawdust was maximized.

However, the PANI/SD composite with a sawdust-aniline ratio of 0.8 exhibited a low adsorption capacity equal to 257.4 mg/g, which is attributed to the excessive amount of PANI. The excess PANI in the composite caused aggregation, resulting in a reduced adsorption capacity. Similarly, PANI/SD composites with sawdust-aniline ratios of 1.6 and 2.0 also demonstrated lower adsorption capacities, indicating that there were relatively fewer PANI coatings on the sawdust particles in these composites.

CONCLUSION

The polyaniline-sawdust (PANI/SD) composites were successfully synthesized in different sawdust-to-aniline ratios. Characterizations through the FT-IR and SEM analyses indicated that polyaniline (PANI) was effectively coated on the surface of the sawdust, resulting in composites in the emeraldine base form. Among the PANI/SD composites, the adsorbent with sawdust-aniline ratio equal to 1.2 had the greatest adsorption capacity of 738.9 mg/g. This study demonstrates that PANI/SD composites can be potentially used as a novel, cost-effective adsorbent to remove lead from contaminated water. We recommend conducting additional characterization techniques such as X-ray Diffraction (XRD), N2-adsorption desorption, and thermal behavior analysis. Furthermore, it is recommended to study the effects of pH, temperature, and dopant and oxidant concentration on the composites to optimize the formulation of PANI/SD adsorbents.

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CONFLICTS OF INTEREST

The authors do not have any conflicts of interest.

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