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Preparation and Characterization of Chitosan–Durian Seed Composite Adsorbent for Tofu Wastewater Treatment

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A B S T R A C T

The tofu industry generates wastewater containing high concentrations of total dissolved solids (TDS) and total solids (TS), posing significant environmental concerns. This study aimed to develop a chitosan-modified durian seed (Durio zibethinus) composite bioadsorbent for the removal of TS and TDS from tofu wastewater. The composite was characterized using scanning electron microscopy (SEM), X-ray fluorescence (XRF), and particle size analysis (PSA), followed by batch adsorption experiments at different adsorbent dosages. The initial wastewater contained 7,528 mg/L TDS and 9,139 mg/L TS. The composite exhibited superior adsorption performance compared with pristine chitosan, achieving a maximum TDS removal efficiency of 65.84%, reducing the TDS concentration to 2,572 mg/L, while TS removal reached 47.70%, with the final TS concentration decreasing to 4,780 mg/L at an adsorbent dosage of 1 g. Material characterization demonstrated that chitosan modification improved the composite morphology and physicochemical properties, thereby enhancing its adsorption performance. These findings indicate that the chitosan-modified durian seed composite is an effective, low-cost, and environmentally friendly bioadsorbent for tofu wastewater treatment. Furthermore, the utilization of durian seed waste as a bioadsorbent contributes to agricultural waste valorization and supports circular economy practices.

Contribution to Sustainable Development Goals (SDGs):
SDG 6: Clean Water and Sanitation
SDG 9: Industry, Innovation, and Infrastructure
SDG 12: Responsible Consumption and Production
SDG 13: Climate Action
SDG 15: Life on Land

1. INTRODUCTION

1.1. Research Background

The rapid expansion of the tofu industry has significantly increased wastewater generation, particularly in developing countries where wastewater treatment facilities remain limited. Tofu production requires substantial quantities of water during washing, soaking, grinding, cooking, and soybean coagulation, resulting in wastewater rich in suspended and dissolved solids originating from soybean residues, proteins, carbohydrates, lipids, minerals, and other organic constituents [1], [2]. Among the various wastewater quality parameters, Total Solids (TS) and Total Dissolved Solids (TDS) are considered critical indicators because they represent the overall solid load discharged into receiving water bodies and directly influence aquatic ecosystem quality.

Previous studies have reported TS concentrations exceeding 700 mg/L and TDS concentrations above 8,400 mg/L in tofu wastewater, values that surpass the permissible discharge standards for industrial effluents [3], [4]. Excessive TS increases turbidity, promotes sediment accumulation, reduces light penetration, and inhibits photosynthetic activity in aquatic ecosystems. Meanwhile, elevated TDS increases electrical conductivity, alters osmotic balance, disrupts ionic equilibrium, and degrades aquatic habitats [5]. Moreover, dissolved organic constituents contributing to TDS can undergo microbial degradation, thereby increasing dissolved oxygen demand and accelerating water quality deterioration. These environmental impacts highlight the necessity of simultaneously removing TS and TDS to improve the sustainability of tofu wastewater management.

Numerous treatment technologies have been investigated for tofu wastewater, including coagulation–flocculation, biological treatment, anaerobic digestion, membrane filtration, and biofiltration [6]. However, these approaches still exhibit several limitations. Biological treatment is generally ineffective for dissolved solids removal, membrane filtration requires high investment and operational costs and is prone to membrane fouling, while coagulation and biofiltration generate substantial sludge and often suffer from declining performance due to media saturation and operational fluctuations [7]. Therefore, the development of an efficient, low-cost, and environmentally sustainable treatment technology remains highly desirable, particularly for small- and medium-scale tofu industries.

Adsorption has attracted considerable attention as an alternative wastewater treatment technology owing to its high removal efficiency, operational simplicity, low energy requirement, and compatibility with renewable biomass-based adsorbents [8]. The adsorption performance of biomass-derived materials is strongly influenced by their surface area, pore structure, and surface functional groups capable of interacting with pollutants through physical adsorption, electrostatic attraction, hydrogen bonding, and surface complexation [9]. Consequently, lignocellulosic biomass has become an increasingly attractive precursor for developing sustainable bioadsorbents.

Durian seed (Durio zibethinus), an abundant agricultural by-product in Southeast Asia, represents a promising lignocellulosic biomass for bioadsorbent production. Its cellulose, hemicellulose, and lignin components provide abundant hydroxyl and carboxyl functional groups, while its naturally porous structure facilitates pollutant adsorption [10]. Previous studies have demonstrated the effectiveness of durian seed-derived adsorbents for removing heavy metals and organic contaminants from aqueous solutions [11]. Nevertheless, the adsorption capacity, chemical stability, and mechanical strength of raw biomass remain limited, restricting its practical application.

To overcome these limitations, chitosan has been extensively employed as a modification material because of its abundant amino ($-NH_2$) and hydroxyl (–OH) functional groups that provide additional adsorption sites. Incorporating chitosan into lignocellulosic biomass improves structural integrity, enhances mechanical stability, increases the availability of active functional groups, and facilitates adsorbent recovery after treatment [12], [13]. Furthermore, fabricating the composite into bead form offers superior mechanical durability, lower particle loss, and improved applicability in continuous and repeated wastewater treatment systems compared with powdered adsorbents [14].

Despite these advances, previous studies on chitosan–durian seed composites have primarily focused on the removal of heavy metals, synthetic dyes, or individual contaminants using synthetic aqueous solutions [9], [10]. Their application for the simultaneous removal of TS and TDS from real tofu wastewater has received little attention. Furthermore, the relationship between the physicochemical characteristics of chitosan-modified durian seed composite beads and their adsorption performance toward TS and TDS has not been systematically investigated.

Therefore, this study aims to develop chitosan-modified durian seed (Durio zibethinus) composite bioadsorbent beads for the simultaneous removal of TS and TDS from real tofu wastewater. The novelty of this work lies in the fabrication of biomass-based composite beads specifically designed for treating actual tofu wastewater, accompanied by a systematic investigation of the relationship between the physicochemical properties of the adsorbent and its adsorption performance. The results are expected to advance the understanding of the structure–performance relationship of biomass-based composite adsorbents while providing an economical, environmentally sustainable, and circular economy-oriented solution for tofu wastewater treatment.

2. MATERIALS AND METHODS

2.1. *Materials and Equipment*

Chemicals used in this study comprised durian seeds as the biosorbent precursor, chitosan (degree of deacetylation ~70%), 2% (v/v) acetic acid (CH_3COOH), sodium tripolyphosphate (STPP) as the crosslinking agent, and distilled water. The wastewater sample was collected from a household-scale tofu industry. Morphological characterization of the adsorbent was conducted using scanning electron microscopy (SEM).

2.2. *Preparation of Durian Seeds*

Durian seeds were thoroughly washed to remove impurities, cut into small pieces, and oven-dried at approximately 100 °C for 24 h. The dried seeds were then ground into powder and sieved through a 100-mesh sieve to obtain a uniform particle size. The resulting durian seed powder was chemically activated with 0.1 M HNO_3 for 1 h, followed by washing with distilled water until the pH was neutral. The material was subsequently dried again at approximately 70 °C.

2.3. *Preparation of Chitosan–Durian Seed Beads (Ionic Gelation with STPP)*

A total of 2 g of chitosan was dissolved in 100 mL of 2% (v/v) acetic acid and stirred for approximately 2 h until a homogeneous solution was obtained. Durian seed powder was then added at a 1:1 (w/w) ratio and mixed to form a uniform suspension. The suspension was dropped into 2% (w/v) STPP solution using a syringe, producing beads via ionic gelation between protonated

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amine groups of chitosan (–NH_3^+) and polyphosphate ions. Beads were left for approximately 2 h to complete crosslinking, washed with distilled water to neutral pH, and oven-dried at approximately 60 °C for 24 h.

2.4. Adsorption Procedure

A total of 250 mL of tofu wastewater was transferred into an Erlenmeyer flask, followed by the addition of bead adsorbent at dosages of 0.25, 0.5, 0.75, and 1 g. The mixture was agitated at approximately 150 rpm for 120 min under constant contact time. After adsorption, the suspension was filtered to separate the adsorbent. The resulting filtrate was subsequently analyzed for TS and TDS.

2.5. Total Solids (TS) – Gravimetric Method

Porcelain crucibles were dried at 103–105 °C, cooled in a desiccator, and weighed (W_0). The sample was then added and dried to constant weight, then reweighed (W_1). Total solids were calculated using:

$$TS\,(mg/L) = \frac{(W_1 - W_0) \times 1000}{V}$$

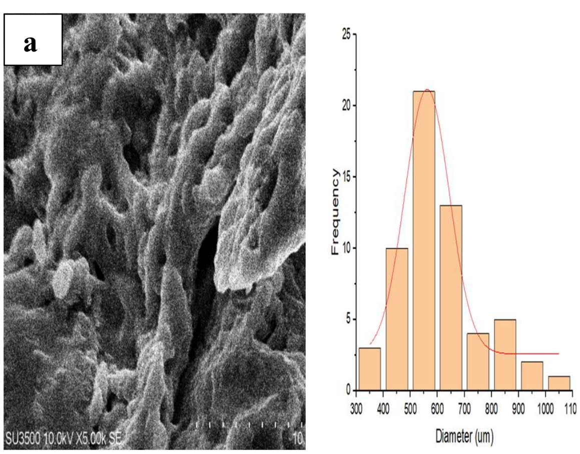
2.6. Total Dissolved Solids (TDS) – Gravimetric Method

The sample was first filtered, and the filtrate was transferred into a crucible. It was then dried at 180 °C to constant weight and weighed. Total dissolved solids were calculated as:

$$TDS\,(mg/L) = \frac{(W_1 - W_0) \times 1000}{V}$$

3. RESULT AND DISCUSSION

3.1. Characterization of SEM and PSA Before Adsorption



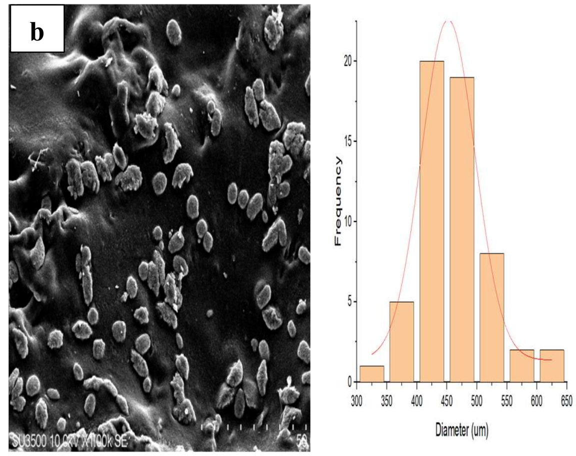


Figure 1. (a) SEM and PSA of chitosan; (b) SEM and PSA of chitosan–durian seed composite.

Figure 1 presents the surface morphology and particle size distribution of chitosan and the chitosan–durian seed composite as characterized by Scanning Electron Microscopy (SEM) and Particle Size Analysis (PSA). As shown in Figure 1a, chitosan exhibits a porous surface morphology with a relatively uniform pore distribution. This porous structure provides a large specific surface area and facilitates mass transfer by offering accessible diffusion pathways during the adsorption process [15]. Moreover, the amino (–NH_2) and hydroxyl (–OH) functional groups on the chitosan surface provide abundant active sites for adsorption through electrostatic attraction, hydrogen bonding, and complexation with contaminants [16].

The PSA results indicate that chitosan has an average particle size (Z-average) of 2,144.5 nm, a polydispersity index (PDI) of 0.882, and a dominant particle size (mode) of 1,137.6 nm. The relatively high PDI value indicates a broad particle size distribution, suggesting considerable heterogeneity in particle size generated during material preparation.

Following modification with durian seed biomass, the composite exhibited substantial morphological changes, as illustrated in Figure 1b. The composite surface became denser, more compact, and more integrated than that of pristine chitosan, indicating the formation of strong interactions between the chitosan matrix and the lignocellulosic constituents of the durian seed. These observations confirm that chitosan acts as a crosslinking matrix, enhancing the structural cohesion and mechanical stability of the composite [17]. Furthermore, the cellulose, hemicellulose, and lignin present in the durian seed contribute additional oxygen-containing functional groups, thereby increasing the density of active adsorption sites. Similar structural reinforcement has been widely reported to improve both the stability and adsorption performance of chitosan-based composites [18].

These observations are further supported by the PSA results. The chitosan–durian seed composite exhibited an average particle size of 1,159.2 nm, a PDI of 0.531, and a dominant particle size of 111.8 nm. Compared with pristine chitosan, the composite showed a lower average particle size and a narrower particle size distribution, as reflected by its lower PDI value. The more uniform particle size distribution suggests that the composite formation process was successful, producing a more stable granular structure. Such characteristics are advantageous for adsorption applications because they increase the effective surface area, facilitate mass transfer, and improve the separation of the adsorbent from the treated solution after the adsorption process [19].

The improved morphological characteristics and particle size distribution are expected to contribute to a more efficient adsorption mechanism. The amino (–NH_2) and hydroxyl (–OH) groups of chitosan provide a high affinity toward contaminants, while the lignocellulosic components of durian seed introduce additional hydroxyl, carboxyl, and other oxygen-containing functional groups, thereby increasing both the number and diversity of active adsorption sites [12], [20]. The synergistic combination of these two materials enables contaminant removal through multiple simultaneous mechanisms, including electrostatic attraction, hydrogen bonding, flocculation, and complexation. Electrostatic attraction occurs through interactions between protonated amino groups on chitosan and negatively charged particles, whereas hydrogen bonding involves interactions between hydroxyl and carboxyl groups and organic molecules. In addition, chitosan acts as an effective flocculating agent that promotes the aggregation and settling of suspended particles, while complexation occurs through coordination interactions between active functional groups and various contaminant species [21].

The more compact yet porous composite structure also enhances pore accessibility and facilitates molecular diffusion into the internal active sites, allowing adsorption to occur not only on the external surface but also within the interior of the adsorbent through intraparticle diffusion. The synergistic contribution of these adsorption mechanisms results in superior adsorption capacity and structural stability compared with pristine chitosan, highlighting the potential of the chitosan–durian seed composite as an effective bioadsorbent for the removal of total solids (TS) and total dissolved solids (TDS) from tofu wastewater [1], [22].

3.2. SEM and AFM Characterization of Adsorbent After Adsorption

The surface morphology using Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM), as illustrated in Figure 2. The SEM micrograph presented in Figure 2(a) reveals substantial morphological alterations after the adsorption process. The adsorbent surface became rougher, denser, and more aggregated, while several pores appeared partially blocked by adsorbed particles [39]. These observations indicate that contaminants in tofu wastewater were successfully adsorbed onto the adsorbent surface. The reduction in visible pore structures further suggests that adsorption occurred not only on the external surface but also within the adsorbent's porous matrix.[40]

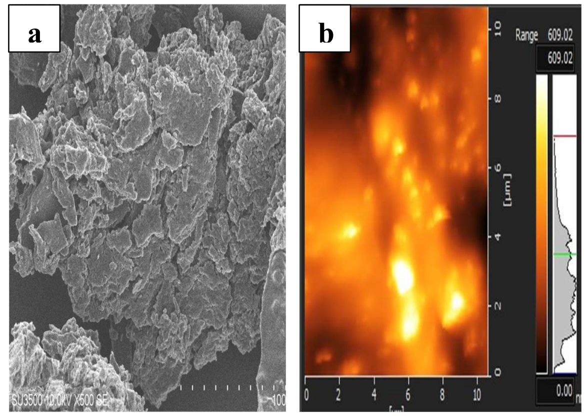


Figure 2. (a) SEM image of the adsorbent after adsorption; (b) AFM topography of the adsorbent after adsorption.

The observed morphological transformation can be attributed to the interaction between pollutant molecules and the functional groups of the cellulose–chitosan biocomposite. Cellulose and chitosan contain abundant hydroxyl (–OH) and amino (–NH_2) groups that serve as active adsorption sites and facilitate pollutant binding via hydrogen bonding, electrostatic attraction, and surface complexation [23]. The synergistic combination of cellulose and chitosan also enhances adsorption performance by improving structural stability, hydrophilicity, and surface reactivity [24].

Moreover, the incorporation of chitosan into cellulose-based composites has been reported to increase the density of active binding sites owing to the presence of reactive amino groups. These functional groups exhibit a strong affinity for dissolved contaminants, thereby promoting the accumulation of pollutants on the adsorbent surface and within the pore structure [25]. As a consequence, the adsorbent surface after adsorption appeared more compact and agglomerated. Similar morphological changes have been reported in cellulose–chitosan composite adsorbents, where adsorption processes resulted in surface coverage and pore blockage due to contaminant deposition [26].

The AFM image shown in Figure 2(b) further confirms the modification of surface topography following adsorption. The AFM topography shows heterogeneous surface features, with irregular protrusions and valleys of varying heights. Bright regions in the AFM image represent elevated surface domains, whereas darker regions correspond to lower surface areas. The increased surface irregularity and roughness after adsorption indicate the successful attachment of contaminant particles onto the adsorbent surface. These topographical changes demonstrate that the adsorption process significantly altered the material's surface texture.

The increase in surface roughness observed from AFM analysis is closely associated with the accumulation of dissolved solids and organic compounds from tofu wastewater onto the cellulose–chitosan matrix. A heterogeneous surface structure is advantageous for adsorption because it provides a larger number of accessible active sites and enhances interaction between the adsorbent and contaminants. Previous studies on biopolymer-based adsorbents similarly reported that adsorption processes increased surface heterogeneity and roughness by depositing pollutants within the adsorbent matrix [16], [17]. Overall, the SEM and AFM analyses clearly demonstrate that the adsorption process induced significant changes in the morphology and

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topography of the adsorbent surface. The formation of a rough, compact, and heterogeneous structure after adsorption confirms the effective interaction between the cellulose–chitosan biocomposite and contaminants in tofu wastewater. These findings highlight the potential of cellulose–chitosan-based adsorbents as sustainable, environmentally friendly materials for wastewater treatment.

3.3. XRF Characterization of Adsorbent and Its Role in TDS and TS Removal

Table 1. XRF Analysis of Chitosan–Durian Seed Composite Adsorbent

No.	Oxide Component	Composition (wt.%)
1	Na_2O	38.20
2	CaO	27.90
3	MgO	9.46
4	MnO	5.87

The X-ray Fluorescence (XRF) analysis presented in Table 1 shows that the chitosan–durian seed composite is mainly composed of Na_2O (38.20 wt.%), CaO (27.90 wt.%), MgO (9.46 wt.%), and MnO (5.87 wt.%). The detected oxide composition indicates that the synthesized composite contains inorganic constituents originating from both chitosan and durian seed biomass, providing evidence for the successful incorporation of the biomass into the composite structure.

The predominance of Na_2O is likely associated with residual sodium species derived from the NaOH treatment and sodium tripolyphosphate (STPP), which were employed during the composite preparation process. Meanwhile, the presence of CaO, MgO, and MnO reflects the naturally occurring mineral constituents of durian seed biomass that remained incorporated within the composite matrix after chemical modification. These findings suggest that the synthesis procedure preserved part of the intrinsic mineral composition of the biomass while producing a chemically stable composite material [27].

It should be noted that the XRF analysis provides information on the elemental and oxide composition of the composite rather than identifying the active adsorption sites. Therefore, the detected mineral oxides should be interpreted as compositional evidence supporting the successful formation of the composite instead of direct contributors to the adsorption mechanism. The adsorption behavior is more reasonably attributed to the synergistic combination of the chitosan matrix and the lignocellulosic biomass, which provides abundant amino (–NH_2), hydroxyl (–OH), and other oxygen-containing functional groups available for interaction with contaminants [28].

The XRF results are consistent with the structural characterization obtained from SEM, PSA, and AFM analyses. SEM observations revealed notable morphological changes following composite formation, PSA demonstrated a more homogeneous particle-size distribution, and AFM confirmed changes in surface topography. Collectively, these complementary characterization results verify the successful synthesis of the chitosan–durian seed composite and demonstrate that the incorporation of durian seed biomass altered both the chemical composition and the structural characteristics of the adsorbent, which are expected to be beneficial for wastewater treatment applications.

3.4. Analysis (TDS) and (TS)

Total dissolved solids (TDS) and total solids (TS) are important indicators for evaluating wastewater quality because they represent the concentrations of dissolved and total solid materials present in water. The tofu wastewater used in this study exhibited initial TDS and TS concentrations of 7,528 mg/L and 9,139 mg/L, respectively, indicating a high load of organic and inorganic constituents generated during soybean processing. These solids mainly originate from proteins, carbohydrates, lipids, minerals, and suspended particles released throughout the tofu production process, all of which contribute to the high pollution load of the wastewater [7]. Therefore, the removal of TDS and TS was selected as the primary performance indicator for evaluating the effectiveness of the developed chitosan–durian seed composite bioadsorbent.

Table 2. Effect of bioadsorbent dosage on TDS and TS removal efficiency.

Adsorbent	Dose (g)	TDS (mg/L)	Removal (%)	TS (mg/L)	Removal (%)
Initial wastewater	-	7528	-	9139	-
Chitosan	0.25	5608	25.51	7890	13.67
Chitosan	0.50	3252	56.80	6540	28.44
Chitosan	0.75	5673	24.64	6745	26.20
Chitosan	1.00	6316	16.10	6134	32.88
Composite	0.25	5432	27.84	6780	25.81
Composite	0.50	4588	39.05	7038	22.99
Composite	0.75	4470	40.62	7636	16.44
Composite	1.00	2572	65.84	4780	47.70

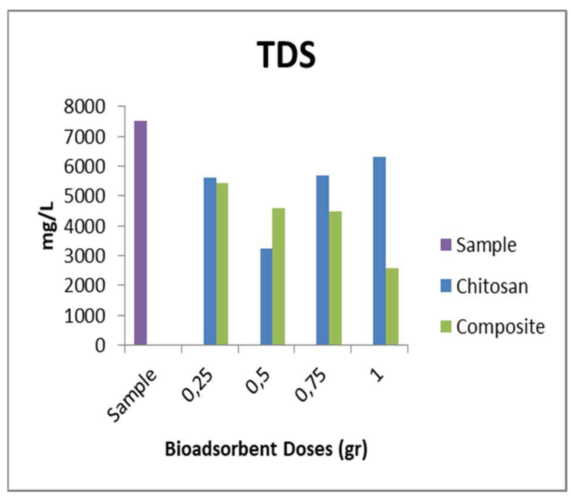

Figure 3. Effect of bioadsorbent dosage on TDS reduction using chitosan and composite adsorbents.

As presented in Table 2 and Figure 3, increasing the adsorbent dosage generally improved the removal efficiency of total dissolved solids (TDS), particularly for the chitosan–durian seed composite. The TDS concentration decreased from 7,528 mg/L to 2,572 mg/L when 1 g of the composite adsorbent was applied, corresponding to a removal efficiency of 65.84%. In contrast, pristine chitosan reduced the TDS concentration only to 6,316 mg/L, resulting in a removal efficiency of 16.10% at the same adsorbent dosage.

The improvement in TDS removal with increasing adsorbent dosage can be attributed to the greater availability of adsorption sites and the larger effective surface area for contaminant uptake. A higher adsorbent dosage provides more accessible active sites for the adsorption of dissolved species, thereby increasing the probability of adsorbent–adsorbate interactions and enhancing mass transfer during the adsorption process. In addition, the greater number of available pores facilitates the diffusion and retention of dissolved organic and inorganic constituents within the porous structure of the adsorbent[29], [30].

The superior performance of the composite compared with pristine chitosan indicates a synergistic effect between the chitosan matrix and durian seed biomass. Chitosan contributes amino (–NH_2) and hydroxyl (–OH) functional groups, while the lignocellulosic components of durian seed provide additional hydroxyl, carboxyl, and other oxygen-containing functional groups, thereby increasing both the abundance and diversity of potential adsorption sites [11], [31]. Furthermore, SEM characterization revealed that the composite possessed a more compact surface with interconnected pores, whereas PSA analysis demonstrated a more homogeneous particle-size distribution than that of pristine chitosan. These structural characteristics are expected to increase the effective surface area and facilitate contaminant diffusion into the internal pores of the adsorbent, thereby contributing to the enhanced TDS removal efficiency observed for the composite.

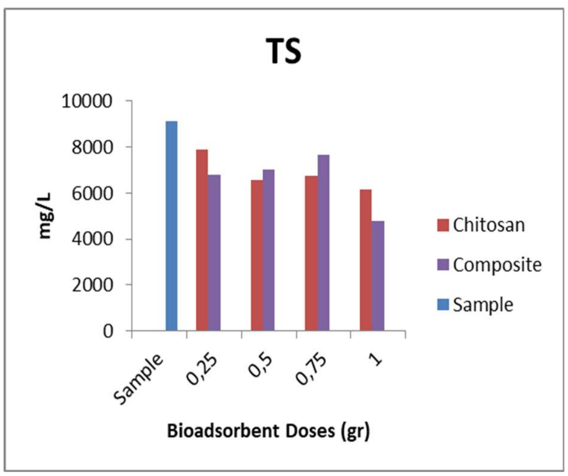

Figure 4. Effect of bioadsorbent dosage on TS reduction using chitosan and composite adsorbents.

A similar trend was observed for total solids (TS) removal, as presented in Table 2 and Figure 4. The initial TS concentration of 9,139 mg/L decreased to 4,780 mg/L following treatment with 1 g of the chitosan–durian seed composite, corresponding to a removal efficiency of 47.70%. This performance was superior to that of pristine chitosan, which reduced the TS concentration to 6,134 mg/L, achieving a removal efficiency of only 32.88%.

The reduction in TS indicates that the composite is effective in removing not only dissolved constituents but also suspended solids from tofu wastewater. This performance is likely attributed to the combined effects of adsorption and flocculation. Protonated amino groups in chitosan can promote charge neutralization of colloidal particles, facilitating particle aggregation, while the porous lignocellulosic matrix of the durian seed provides physical retention sites for suspended particles within the composite structure. In addition, the interconnected pore network observed by SEM may facilitate the entrapment of particulate matter, thereby enhancing the overall removal of total solids. Consequently, suspended particles become more readily separated from the wastewater through aggregation and subsequent retention within the adsorbent matrix [32].

The enhanced adsorption performance of the composite is supported by the material characterization results. SEM analysis revealed that the chitosan–durian seed composite exhibited a denser and more integrated morphology than pristine chitosan, while PSA analysis demonstrated a more homogeneous particle-size distribution. After the adsorption process, SEM images showed that several pores were partially occupied by deposited contaminants, whereas AFM analysis revealed an increase in surface roughness, indicating the accumulation of particulate matter on the adsorbent surface. These findings suggest that the incorporation of durian seed biomass successfully modified the structural characteristics of chitosan, resulting in a more stable porous framework and improved contaminant retention capacity.

Overall, the chitosan–durian seed composite exhibited superior adsorption performance compared with pristine chitosan for the removal of total dissolved solids (TDS) and total solids (TS) from tofu wastewater. This improvement can be attributed to the synergistic combination of the functional groups provided by chitosan and the lignocellulosic components of durian seed, together with the favorable structural characteristics of the composite that facilitate contaminant diffusion and retention. Under the optimum operating conditions, the application of 1 g of the composite achieved removal efficiencies of 65.84% for TDS and 47.70% for TS, demonstrating its potential as an environmentally friendly bioadsorbent for tofu wastewater treatment.

4. CONCLUSION

This study demonstrated that the chitosan-modified durian seed (Durio zibethinus) composite exhibited superior performance to pristine chitosan in reducing total dissolved solids (TDS) and total solids (TS) from tofu wastewater. The optimum adsorption performance was achieved at an adsorbent dosage of 1 g, resulting in a TDS removal efficiency of 65.84%, with the TDS concentration decreasing from 7,528 mg/L to 2,572 mg/L. The TS removal efficiency reached 47.70%, reducing the TS concentration from 9,139 mg/L to 4,780 mg/L. Characterization by SEM, XRF, and PSA confirmed that chitosan modification produced a composite with a more porous morphology, a more uniform particle size distribution, and elemental characteristics that contributed to enhanced adsorption performance. These findings suggest that the chitosan-modified durian seed composite is a promising, low-cost, and environmentally friendly bioadsorbent for tofu wastewater treatment. Moreover, the utilization of durian seed waste as a bioadsorbent provides added value to agricultural waste and supports the implementation of circular economy principles. Future studies should evaluate the regeneration and reusability of the composite and investigate its performance in continuous adsorption systems and at a larger scale to facilitate practical industrial wastewater treatment applications.

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