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Coagulation-Flocculation of Tofu Wastewater using Natural Coagulant of Chempedak (Artocarpus integer) Seed

Koagulasi-Flokulasi Limbah Cair Tahu dengan Koagulan Alami Biji Cempedak (Artocarpus integer)

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ABSTRAK

Limbah cair tahu mencemari badan air dan menyebabkan bau tidak sedap di lingkungan. Koagulasi-flokulasi adalah metode efektif untuk menyisihkan kontaminan di limbah cair tahu karena metode ini memiliki efisiensi tinggi dan kebutuhan energi rendah. Namun, penggunaan koagulan kimia menghasilkan lumpur yang berbahaya, sehingga koagulan dari bahan alami lebih disukai karena aman untuk lingkungan dan manusia. Penelitian ini bertujuan menganalisis potensi ekstrak biji cempedak untuk menyisihkan COD, TSS, dan kekeruhan pada limbah cair tahu. Biji cempedak diekstrak menggunakan n-heksana dan NaCl untuk meningkatkan karakteristik koagulan. Eksperimen dilakukan secara *batch* menggunakan jar-tes. Dosis yang digunakan adalah 1,5–4 mL/L dengan pengadukan cepat 120 rpm selama 3 menit, pengadukan lambat 60 rpm selama 15 menit, dan sedimentasi 60 menit. Hasil penelitian menunjukkan bahwa koagulan biji cempedak dapat menurunkan kekeruhan dan TSS, tetapi meningkatkan konsentrasi COD. Gugus fungsi hidroksil dan karboksil memegang peranan penting dalam proses koagulasi. Kenaikan dosis koagulan dapat meningkatkan efisiensi penyisihan kekeruhan dan TSS. Dosis berlebih dapat menyebabkan resuspensi yang meningkatkan kekeruhan air. Dosis optimum untuk menyisihkan kekeruhan dan TSS di limbah cair tahu adalah 2,5 mL/L pada pH 9 dengan efisiensi penyisihan 78%. Efisiensi penyisihan TSS sebesar 77% didapatkan pada dosis 3,5 mL/L.

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ABSTRACT

The tofu wastewater contaminates water bodies and causes unpleasant odors to the environment. Coagulation-flocculation is an effective method to remove contaminants because of its high removal efficiency and low energy requirement. However, the use of chemical coagulants generates a significant amount of toxic sludge. Thus, a natural coagulant is more favorable because it is biodegradable and safe for human health. The objective of this study is to analyze the performance of chempedak seed extract to remove chemical oxygen demand (COD), total suspended solids (TSS), and turbidity in tofu wastewater. Chempedak seed was extracted using n-hexane and NaCl to improve the coagulant characteristics. The experiment was conducted in the batch system using a jar test apparatus. The experiment was carried out on a dosage range of 1.5–4 mL/L with a flash mixing rate of 120 rpm for 3 minutes, slow mixing of 60 rpm for 15 minutes, and sedimentation for 60 minutes. The result proved that the chempedak seed coagulant could reduce turbidity and TSS but increase COD concentration. The hydroxy and carboxy functional groups play a crucial role in coagulation activity. The increasing coagulant dosage increased the removal efficiency of turbidity and TSS. Overdosing can influence resuspension conditions that increase final turbidity. The optimum dosage to remove turbidity and TSS in tofu wastewater was 2.5 mL/L at a pH of 9 with a removal efficiency of 78%. The TSS removal efficiency of 77% was achieved at a dosage of 3.5 mL/L.

1. INTRODUCTION

1.1 Background

Tofu is the most popular food to fulfill protein nutrient needs in Indonesia because of its affordable cost and nutritional content. Small-scale home industries are the most common tofu producers in Indonesia. The tofu production process generates waste by-products, including solid and liquid waste. Due to the protein content in the solid waste of tofu processing waste, other food industries utilize it as the raw material. Around 9–10 tons of liquid waste are discharged per 1 ton of processed soybeans (Purnawan et al., 2021). The home industry of tofu cannot afford wastewater treatment costs, so the wastewater is directly discharged to water bodies without appropriate treatment. Total suspended solids (TSS), chemical oxygen demand (COD), and biological oxygen demand (BOD) contaminants in tofu wastewater ranged from 6000–8000 mg/L, 7000–2600 mg/L, and 5000–1000 mg/L, respectively (Prawati et al., 2019). Carbohydrates, protein, fat, and amino acids in tofu wastewater will physically, chemically, and biologically react with other substances to produce toxic substances that pollute water bodies (Murwanto et al., 2021). Tofu wastewater can depress dissolved oxygen concentration and produce a pungent odor in the environment because of the organic matter decomposition product (Putro et al., 2021; Seroja et al., 2018).

Several methods have been investigated to remove contaminants in tofu wastewater. Biological treatments, namely anaerobic and phytoremediation, can treat high-strength tofu wastewater (Adisasmito et al., 2018; Santosa et al., 2023). However, the anaerobic biological process requires a longer hydraulic retention time, so it will take a long time to treat large volumes of wastewater. Another method is the aerobic biological process, which provides continuous aeration but requires an extensive energy supply (Rinaldi et al., 2018). The physical method of membrane filtration has disadvantages of membrane fouling and pre-treatment of the precipitation process (Putri & Kartohardjono, 2018). Ozonation is a rapid chemical process to remove impurities, but the process depends on higher pH, accommodating OH radical formation (Hadiyanto, 2018; Karamah et al., 2019). Coagulation-flocculation is a favorable chemical process because of its high removal efficiency, low cost, and low energy requirement (Ayangunna et al., 2016; Iwuozor, 2019).

Chemical substances, for instance, polyaluminum chloride (PAC), alum, and ferric salt, are commonly used as coagulants to remove pollutants in the water. Although chemical coagulant offers good removal efficiency, it has some drawbacks, notably generating a significant amount of toxic sludge and residue in drinking water that may impact health deterioration (Bahrodin et al., 2021). Natural coagulants from plants or animals are more favorable than chemical coagulants because they are biodegradable, environmentally friendly, and safe for human health. Several studies have investigated using plant-based coagulants to treat tofu wastewater. A previous study reported that Moringa oleifera seed has a removal efficiency of around 50% to remove contaminants in tofu wastewater (Setyawati & Sari, 2019). Trembesi seed extract could remove pollutants in tofu wastewater by 83.79% of SS, 79.55% of COD, and 87.5% of BOD (Putri et al., 2020). Corn flour extract has good turbidity removal, around 71%, but only 31.71% for COD (Prihatinningtyas & Effendi, 2022). Animal-based coagulant of chitosan, which is extracted from milkfish scales and crab shells, has the capability of approximately 25% to remove COD in tofu wastewater (Bija et al., 2020; Soviana et al., 2020).

Chempedak (Artocarpus integer) is a plant from Kalimantan Island with a high potency as a local commodity. The chempedak fruit is consumed, but the seeds, rags, and peels are discharged. A past study shows that chempedak has a protein content of around 4% (Irvan et al., 2016). The protein in natural coagulants is an active component that destabilizes colloids in the wastewater during the coagulation-flocculation process (Adesina et al., 2019). Extraction of protein content using the salt solution enhances coagulation activity (Birima et al., 2013).

The utilization of natural plants as coagulants to remove contaminants in tofu wastewater has been studied over forty years ago. However, a systematic understanding of how chempedak as a natural coagulant contributes to treating tofu wastewater still needs to be improved. Thus, the specific objective of this study was to investigate the performance of extracted chempedak seed as a coagulant to remove turbidity, TSS, and COD in tofu wastewater. Furthermore, a coagulant evaluation using Fourier transform infrared (FTIR) was carried out to identify the protein content in chempedak seed coagulants after extraction. The experimental work presented provides the first investigations into how to treat tofu wastewater using an extract of chempedak seed.

1.2 Objectives

This study aims to investigate the performance of extracted chempedak seed as a coagulant to remove turbidity, TSS, and COD in tofu wastewater.

2. METHODS

2.1 Materials Preparation

Chempedak seeds were collected from a local food store in Balikpapan, East Kalimantan. The seed was cut and dried in the oven at a temperature of 105 °C for 24 hours. The dried seeds were crushed using mortar and sieved with a sieve size of 60 mesh. The seed powder was extracted using n-hexane to remove oil contents. The ratio of seed and n-hexane was 1:5 (w/v). The mixture solution was initially soaked for six hours, agitated at 100 rpm for 10 minutes, and finally immersed again for 18 hours. After being immersed, the mixture was filtered and dried at room temperature. The dried powder was extracted using 100 mL NaCl 1.5 M, then stirred for 15 minutes. The final solution was used as a coagulant.

Tofu wastewater was taken from the effluent of the wastewater treatment plant of the tofu industry in East Kalimantan. The initial tofu wastewater quality, including pH, COD, TSS, and turbidity, was analyzed using standard methods for examining water and wastewater (APHA, 2005).

2.2 Experimental Methods

This study was carried out in a batch reactor using a jar test apparatus. The preliminary experiment was conducted to determine the optimum flash mixing rate and initial pH. The flash mixing rate was 120 rpm, 150 rpm, and 180 rpm. The range of initial pH from 4–9 was used with sodium hydroxide (NaOH) 3 M and sulfuric acid (H_2SO_4) 98% as pH adjustment solution. The dosage of 1.5 mL/L and coagulant size of 60 mesh was applied for the preliminary study. Tofu wastewater of 500 mL was loaded into a beaker glass and injected with a coagulant. The experiment was performed using a jar test apparatus with a flash mixing rate for 3 minutes, slow mixing of 60 rpm for 15 minutes, and sedimentation for 60 minutes. The optimum flash mixing rate and initial pH were used to examine the effect of coagulant dosage in the coagulation-flocculation process. The dosage ranges from 1.5–4 mL/L were used in the experiment. After sedimentation, the supernatant was collected to determine the final turbidity, TSS, and COD concentration using standard water and wastewater examination methods. The removal efficiency was calculated using equation (1).

$$\text{Removal efficiency}(\%)=\frac{C_o - C_e}{C_o}\times 100\%\text{.......................................}(1)$$

where C_o resembles the initial concentration of COD, TSS, and turbidity. C_e describes the final COD, TSS, and turbidity concentration after the coagulation-flocculation process.

2.3 Analysis Methods

Fourier transform infrared (FTIR) analysis was conducted to identify the functional group of coagulant and flocs after the coagulation process. Wavenumber of 600–4000 cm^{-1} was applied in Fourier transform infrared (FTIR) analysis (Bruker, ALPHA II). All spectra were plotted as a function of the percentage transmittance (%T) and wavenumber (cm^{-1}).

3. RESULTS AND DISCUSSION

3.1 Characterization of Tofu Wastewater

Table 1 shows the effluent of the tofu wastewater treatment plant. The plant consists of equalization, sedimentation, and wetland units. The results show that although the wastewater has been treated, the effluent quality still did not meet the quality standard of tofu wastewater. Tofu industries use soybeans as the primary raw material in tofu production. Hence, tofu wastewater has high COD concentrations of 731 mg/L due to protein (40–65%), carbohydrate (25–50%), and fat (10%) content (Nurjuwita et al., 2020). Tofu wastewater generally has low pH because of acetate acid utilization in the tofu-making process (Pangestika et al., 2018). However, the effluent has a pH ranging from 6.2 to 6.7 because of assimilation decay, microorganism activity, and the photosynthesis process in the wetland (Yin et al., 2016). The high turbidity of 238–294 NTU in tofu wastewater was caused by the release of colloid and organic particles during tofu production. The tofu wastewater has a TSS of 294 mg/L. The majority of TSS was released during the soybean boiling process in tofu and tempeh production (Pramaningsih et al., 2022).

3.2 Process Optimization

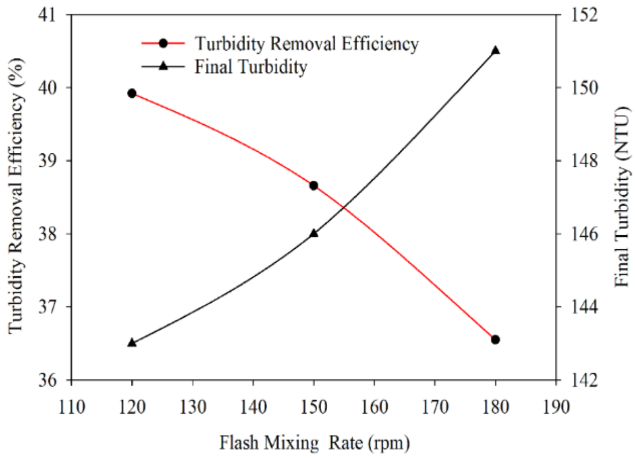

Figure 1. Effect of flash mixing rate on turbidity removal efficiency with initial turbidity of 238 NTU and initial pH of 6.4

The performance of coagulation-flocculation is strongly reliant on the flash mixing rate. In addition to causing particle and coagulation collision in water through gradient velocity, mixing also facilitates the ability for the suspension to be distributed uniformly (Sun et al., 2019). Figure 1 presents the influence of the flash mixing rate on turbidity removal. The coagulation-flocculation process removed initial turbidity from 238 NTU to 151–143 NTU. The result shows that increasing the flash mixing speed from 120 to 180 rpm increased the final turbidity and declined turbidity removal efficiency slightly from 40% to 36%. The lowest turbidity removal efficiency was obtained at the mixing rate of 180 rpm. Pearson correlation test showed that there was no significant correlation between mixing rate and turbidity removal efficiency ($p>0.05$). The mixing rate of 120 rpm was chosen in coagulation-flocculation experiments because of the lowest mixing rate. At the high mixing velocity, floc splintering occurs due to surface erosion and turbulent drag, so final turbidity is increased (Cheng et al., 2021; Jun et al., 2019).

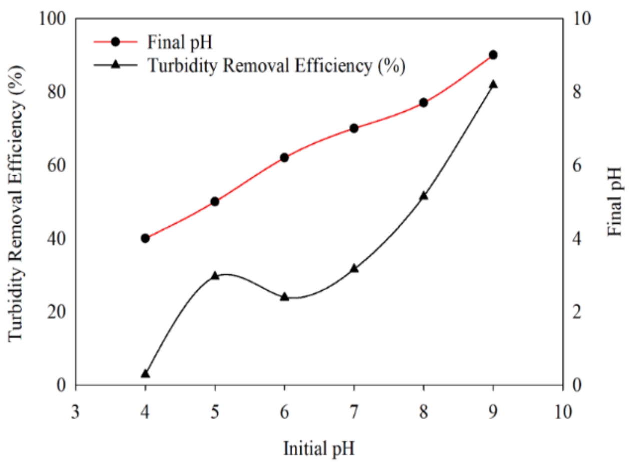

Figure 2. Effect of initial pH on turbidity removal efficiency with initial turbidity of 247 NTU

Table 1. Effluent quality of tofu wastewater treatment plant

Parameter	Value	Quality Standard of Indonesia Regulation*	Unit
COD	731,18	300	mg/L
TSS	294	100–200	mg/L
Turbidity	238–294	-	NTU
pH	6.2–6.7	6–9	-

*(Indonesian Ministry of Environment and Forestry, 2014)

The pH is a crucial parameter in the coagulation-flocculation process because it influences the surface charge of natural coagulants and the stabilization of suspension (Naceradska et al., 2019; Priyatharishini & Mokhtar, 2020). The coagulation process is enhanced at the range of a certain pH. Adjusting the pH of the solution from 4 to 9 was conducted to optimize coagulation performance. The influence of initial pH on turbidity removal efficiency and final pH is shown in Figure 2. The finding demonstrates that increasing the pH value promoted better turbidity removal efficiency. The coagulant had a removal efficiency of less than 20% at a pH of 4 while increasing the initial pH improved the removal efficiency to above 60%. At the pH of 9, the highest turbidity removal efficiency of around 80% was obtained. Based on the Pearson correlation test, the initial pH and turbidity removal efficiency had a statistically significant linear relationship ($p<0.05$). Initial pH and turbidity removal efficiency are positively correlated (R=0.928), which means increasing pH enhances turbidity removal efficiency. The initial pH of 9 was used in coagulation-flocculation experiments with varying dosages. At lower pH, the abundance of hydrogen ions competes with the contaminant on the coagulant surface, whereas the presence of hydroxyl ions suppresses contaminant adsorption at higher pH (Desta & Bote, 2021). The finding indicates that the chempedak seed extracts contain cationic protein, an effective active coagulant agent at higher pHs (Hussain et al., 2019; Noor et al., 2021).

3.3 Influence of the Dosage of Chempedak Seed Coagulant

Coagulation-flocculation aims to remove turbidity due to colloid particles in tofu wastewater. Coagulant destabilized negative charge of colloid particles form floc that must be settled in sedimentation (El-taweel et al., 2023). Figure 3 presents the influence of the dosage of chempedak seed coagulant on turbidity removal in tofu wastewater. The 1.5–2.5 mL/L dosage decreased final turbidity from 283 NTU to 70–60 NTU. The finding shows that increasing the coagulant dosage increased the turbidity removal efficiency. For instance, a dosage of 1.5 mL/L had a turbidity removal efficiency of 75%. The increasing dosage to 2.5 mL/L caused a slight increase in removal efficiency to 78% with a final turbidity of 61.4 NTU. Because the ionic strength of the solution and the solubility of the active compounds are improved, the extracted chempedak coagulant can reduce turbidity in wastewater (Dhivya et al., 2017). The coagulant was improved by dissolving the protein component using NaCl.

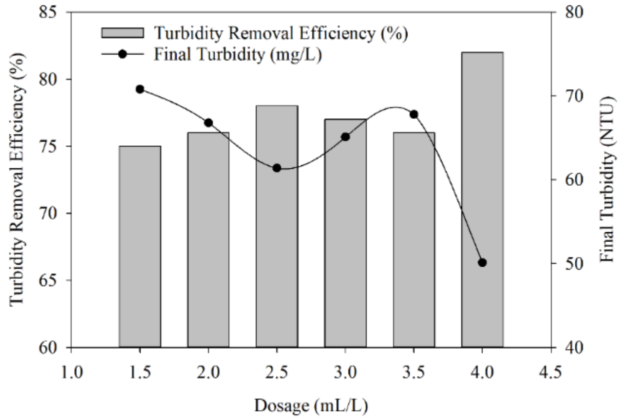

Figure 3. Effect of dosage on turbidity removal efficiency with initial turbidity of 283 NTU

At further increasing dosage to 3.5 mL/L, the removal efficiency decreased, which caused final turbidity to rise again to 70 NTU. Overdosing on the coagulant interrupts sedimentation and causes resuspension of flocs (Zedan et al., 2022). Moreover, the resuspension is also called the flocculation window phenomenon. A flocculation window is a range of effective dosages to remove colloid particles in a specific turbidity range (Kazuhiro, 2018). The flocculation window occurred in the range of dosage of 2–3.5 mL/L. Electrostatic repulsion sustains suspended flocs in stable conditions before and after flocculation windows (Borchert et al., 2021). So, the final turbidity increases outside the flocculation window range.

Total suspended solids and turbidity in wastewater have a linear relationship (Maurya & Daverey, 2018). Therefore, the coagulant not only can remove turbidity but also total suspended solids. The influence of dosage on total suspended solid removal is presented in Figure 4. The removal efficiency was around 29.7% in the 1.5 mL/L coagulant dosage. When the dosage was increased to 3.5 mL/L, the removal efficiency increased considerably to below 80%. From this point onwards, the removal efficiency gradually declined to around 54%. The removal efficiency increased with the increase of coagulant dosage until it reached the optimum dosage. The excessive dosage has a negative effect on coagulant activity, which causes an increase in the final total suspended solids in the solution. Due to the opposite net charge on the suspended particles in wastewater, this excessive dosage decreased the coagulation performance (Sibiya et al., 2021). Additionally, re-stabilization has appeared because polymer molecules in the coagulants have difficulty finding available vacant sites for adsorption (Abreu et al., 2020).

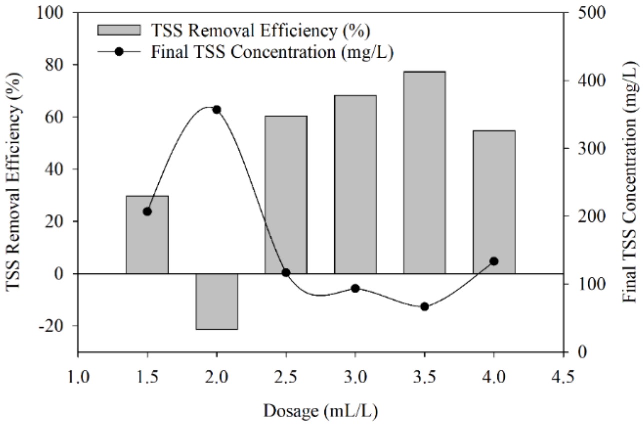

Figure 4. Effect of dosage on total suspended solid with initial TSS 294 of mg/L

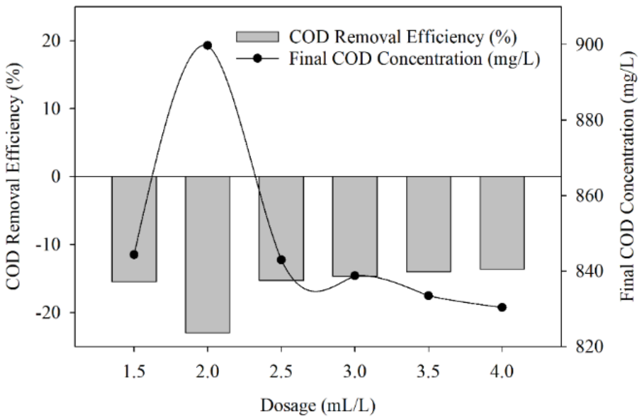

Figure 5. Effect of dosage on COD removal efficiency with the initial COD concentration of 731 mg/L

Figure 5 illustrates COD concentration after the coagulation-flocculation process using an extract of chempedak seeds. Tofu wastewater had an initial COD value of 731.18 mg/L. However, the results of this study show that the COD concentration increased significantly to 820–900 mg/L after adding the dosage of chempedak seed, ranging from 1.4 to 2.0 mL/L. Despite performing similarly to chemical coagulants in removing turbidity and TSS, some natural coagulants may raise the concentration of COD. The past study reported that increasing COD concentration after coagulation using natural coagulants. The previous study found that Moringa oleifera seed increased COD concentration from 132 to 164 mg/L after coagulation (Shan et al., 2017). Adding tamarind and winged bean seed has increased the COD concentration from 6600 mg/L to 7000–9000 mg/L (Elpani et al., 2020). The COD level rise occurred due to natural coagulants containing organic matter, such as carbohydrates and lipids, that remained dissolved in the solution after coagulation-flocculation (Nimesha et al., 2022; Vega et al., 2021). The n-hexane was used in the extraction of chempedak seed. The solvent used also increased the COD level in wastewater after coagulation-flocculation (Shan et al., 2017).

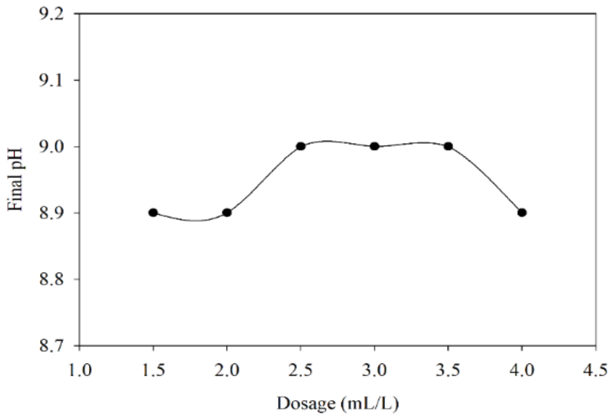

Figure 6. Effect of dosage on final pH with initial pH of 9

The final pH was determined to analyze the pH of wastewater after the coagulation-flocculation process. The final pH after coagulation-flocculation using chempedak extract coagulant did not alter considerably, as observed in Figure 6. At the dosage of 1.5–2.0 mL/L, the final pH decreased slightly from 9 to 8.9. The increasing dosage of 2.5–3.5 mL/L did not change the final pH, which was maintained at a pH of 9. Several plant-based coagulants, namely dragon fruit foliage and cactus powder, are effective at maintaining pH levels before and after coagulation (Beyene et al., 2016; Rayudu et al., 2022). The hydroxide ions in natural coagulants and wastewater hydroxide ions may have been balanced, which resulted in a little shift in the final pH (Ng & Elshikh, 2021). Natural coagulants are more effective than chemical coagulants at maintaining the pH of the suspension. Moreover, the chemical coagulants commonly used, such as alum and ferric salt, suppress the pH after coagulation-flocculation because of alkalinity consumption (Devlin et al., 2019).

3.4 Evaluation of Coagulants

Fourier transform infrared (FTIR) analysis was carried out to determine the functional group that presents and plays a vital role in natural coagulants. Figure 7 (a) shows the FTIR spectra of the coagulant before and after extraction using n-hexane and NaCl. After extraction, the chempedak seed had a strong peak at 3349 cm^{-1}, indicating the presence of O-H and N-H stretching. The hydroxyl group also was found in other Artocarpus species (Ndung'u et al., 2020). The hydroxyl group (O-H) is present because the chempedak seed contains amylum and amylopectin (Aliyatunnaim et al., 2022). The peak of 1635 cm^{-1} shows the presence of a carboxyl group (C=O) located in the wavenumber range 1630–1850 cm^{-1}. The peak of 1635 cm^{-1} represents the amide group, the secondary structural component of protein (Magalhães et al., 2021). This result suggests protein existence in chempedak seeds after extraction. The hydroxy and carboxyl functional groups are released into suspension and generate a positive charge bio-coagulant (Kurniawan et al., 2022). None of the significant alterations were found in FTIR spectra before and after coagulation, as seen in Figure 7 (b).

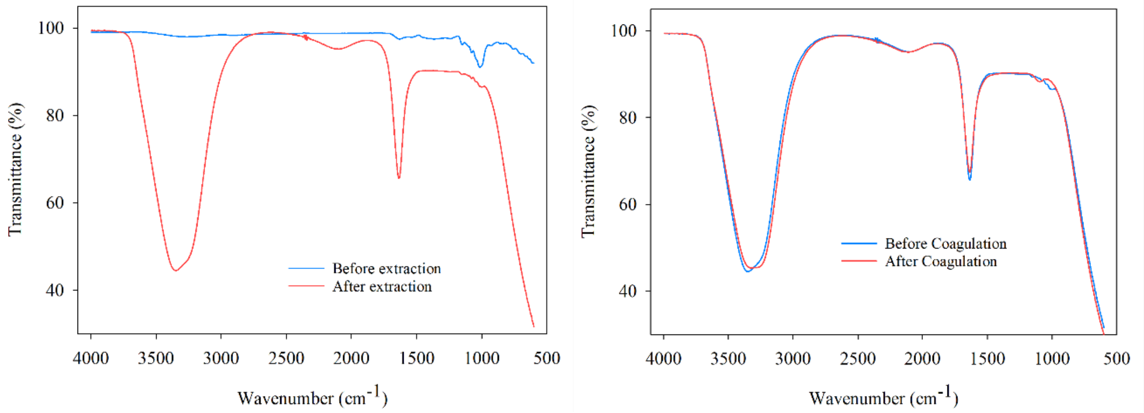

Figure 6. Spectra of FTIR before and after (a) coagulant extraction (b) coagulation-flocculation

4. CONCLUSION

The salt-extracted chempedak seed can potentially remove impurities in tofu wastewater. The optimum dosage to remove turbidity in tofu wastewater was 2.5 mL/L at a pH of 9 with a removal efficiency of 78%. The TSS removal efficiency of 77.32% was accomplished at the optimum dosage of 3.5 mL/L. The extract of chempedak seed increased the final COD concentration in the wastewater because of the organic content of the coagulant. The FTIR analysis confirmed the hydroxyl, carboxyl, and amide functional groups in the chempedak seed. These functional groups are active compounds in the coagulation-flocculation process. In addition, the extraction method using n-hexane and NaCl was conducted to improve the coagulant activity by dissolving protein into solution. These findings have significant implications for the understanding of how chempedak seed extract was used as a coagulant to remove turbidity, TSS, and COD in tofu wastewater. Further research on the extraction methods and coagulation-flocculation conditions can be conducted to optimize the coagulation-flocculation performance.

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