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Optimization of Sumatera Bentonite by Ammonium-impregnated as A Coagulant for Cassava Wastewater Treatment

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Abstract

The original Sumatera Bentonite (SB), which has been impregnated to be ammonium-bentonite (NH-B), was applied as a cassava wastewater coagulant. The modification was conducted using multi-step impregnation initiated by bentonite activation (sodium cation exchange), followed by ammonium impregnation. The optimization parameter focused on the coagulant dose that was used. The result of cassava wastewater coagulation by the ammonium-impregnated bentonite (with dose: 0.4 g/100 mL) shows high-efficiency reduction for turbidity up to 73.97% (from 1099 to 186 NTU), Total Suspended Solid up to 86.56% (from 588 to 79 mg/L), and Chemical Oxygen Demand up to 88.6% (from 559.7 to 63.3 mg/L). The existence of ammonium impregnated is characterized by X-Ray Diffraction analysis based on 2θ shifting at 10° and Fourier Transform Infra-Red analysis at 464, 521, and 1429 cm^{-1} as ammonium binding.

Keywords

Bentonite, Impregnation, Ammonium, Coagulation, Cassava Wastewater

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1. INTRODUCTION

Cassava (*Manihot esculenta*) is classified as the third largest carbo source and give a crucial role in the socio-economies of tropical zone countries such as Indonesia, Thailand, Brazil, Ghana, and Nigeria (Aryee et al., 2006; Franck et al., 2011; Léotard et al., 2009; Nwokocha et al., 2009; Sánchez et al., 2017; Zanetti et al., 2014; Zhang et al., 2016). The Cassava processing generates up to 2.75-6.3 m^3 of solid waste for every ton of raw materials (Cooke et al., 1988; Hasanudin et al., 2019; Kuczman et al., 2017; Ocieczek et al., 2022). According to Ratnadewi et al. (2016), cassava waste compiles 16% of the total root weight with 70% wastewater that is fractioned into hemicellulose of 27%, cellulose of 14%, lignin of 11%, the raw fiber of 10%, and protein of 3.5% with an insufficient of hydrogen cyanide (HCN) substance (Adebowale et al., 2008; Candrawati et al., 2017; de Souza Neto et al. 2018; Eke-emezie et al., 2022; Photharin et al., 2022). Direct cassava wastewater disposal might initiate the eutrophication of aquatic ecosystems and soil contamination due to the high composition of organic matter and toxic cyanide (Nnaji and Akanno, 2022; Acchar and da Silva, 2020; de Souza, 2021).

Recently, researchers focused on cassava wastewater treatment through several strategies, such as phytoremediation/bioremediation, electrocoagulation, anaerobic treatment, activated sludge, and coagulation (de Faria Ferreira Carraro et al., 2021; Fleck et al., 2017; Ibiene et al., 2021; Kaewkannetra et al., 2011; Nuraini and Felani, 2015; Paulo et al., 2013; Sudibyo et al., 2017). Coagulation is chosen as the more effective technique due to its efficiency, economic, and facile operation, and it also shows satisfying results (Aguilar et al., 2003; Ahmad et al., 2005; Adebowale et al., 2008). The coagulation process was affected by several parameters such as type and coagulant doses, timing and speed of mixture, pH condition, and temperature (Ramli and Aziz, 2020; Rosińska and Dąbrowska, 2021; Dayarathne et al., 2022). Fortunately, the utilization of bentonite as a coagulation aid has been researched and shows a high ability in organic pollutant removal when used with another main coagulant (Zhang et al., 2023).

The coagulation aid ability of bentonite is generated from the structural composition layers of alumina octahedral between two silica tetrahedral which provide a negative charge area and the basal spacing between layers that are filled up with cations like Ca+, K$^+$, Mg$^+$, and Na$^+$ (Gandhi et al., 2022). The surface charge of bentonite will help in floc formation (Brandt et al., 2017). As the coagulation aid, there is prior research about bentonite utilization such as Ahari et al. (2019) reported the achievement of a combination of alum (as the coagulant) and bentonite (as the coagulant aid) to eliminate turbidity at 96.72% from the Ait Baha Water Station, Morocco. Research conducted by Marey (2019) applied chitosan (as the coagulant) assisted by bentonite (as the coagulant aid) for removing the wastewater from the Ismaili Canal of the Nile River in Egypt and showing turbidity removal at 97.7%. A research report from Zhang et al. (2023) explained that the combination between chitosan and polyglutamic acid (as a coagulant system) and bentonite-based material (as the coagulant aid) successfully reach a removal rate of COD and turbidity up to 86.2% and 98.2%, respectively from the starch wastewater.

Since bentonite has a coagulation aid ability, there is a need to improve bentonite as the primary natural coagulant in organic wastewater, especially cassava wastewater. Furthermore, previous plant-based coagulants effectively removed cassava wastewater (Ang and Mohammad, 2020). Trevisan et al. (2019) started to observe the coagulation ability of *Moringa oleifera* seeds extract and reported the removal of turbidity at 89.16% and COD at 66.39% from the cassava wastewater. Ibiene et al. (2021) reported that coagulants from Moringa oleifera powdered seeds successfully reduced the COD by up to 74.1% and turbidity by up to 63.2% from the cassava wastewater. Differently, dos Santos et al. (2018) applied different commercial coagulants (Acquapol WW, Acquapol S5T, Tanfloc SL, and Tanfloc SG) to observe the effectivity on cassava wastewater removal. As the best coagulant, Acquapol S5T shows turbidity removal at 88.5%. Unfortunately, there is a need update on the literature about cassava wastewater management using bentonite-based coagulant. Here, we propose the modification of Sumatera Bentonite by impregnation through cation exchange, expected to transform the bentonite into the primary natural coagulant.

This work conducts the Sumatera Bentonite (SB) modification by multi-step impregnation, which means there are two processes of activation bentonite (sodium impregnation) and ammonium impregnation. Ammonium groups are proven to effectively change the bentonite interaction feature to remove the organic contaminant hydrophobically (Gönülşen et al., 2003). Several characterizations, including XRD, FTIR, and SEM-EDX analyses, investigated the modified materials. The bentonite-modified will be examined to coagulate the cassava wastewater imported from the tapioca industry in Palembang, South Sumatera. Optimum dosages as parameters focus examined removing the turbidity, suspended solid (TSS), and chemically dissolved (COD).

2. EXPERIMENTAL SECTION

2.1 Chemicals and Instrumentation

This work modified the Sumatera Bentonite as a precursor (SB). The chemicals used, including NaCl, HCl, NH_4Cl, NaOH, and ethanol, were purchased from Sigma Aldrich, H_2SO_4 was purchased from Mallinckrodt Ar Laboratory Chemical, then $K_2Cr_2O_7$ and $Fe(NH_4)_2(SO_4)_2 \cdot {}_6H_2O$ (ferro ammonium sulfate/FAS) were purchased from Merck. All the chemicals are pure-grade items. The distilled water (DW) was purchased from Bratachem. The cassava wastewater was imported from the tapioca industry in Palembang, South Sumatra.

The modified bentonite was characterized using JSM 6510-LA SEM-EDX using 10,000 magnifications, Rigaku Miniflex600 X-Ray Diffractometer on the scanning scope from 2θ of 10-90°, and Perkin-Elmer UATR Spectrum two Fourier Transfer Infra-Red using 400-4000 cm^{-1} scan ranges. An additional instrument to measure the turbidity level of the treated waste is Eutech TN-100 Turbidimeter.

2.2 Bentonite Impregnation

The multi-step impregnation is initiated by bentonite activation using the sodium cation exchange. First, the saturated NaCl solution was mixed for 120 minutes with 100 g of SB, then diluted by DW (the ratio is 1:2 for the mixture to DW) for 10 minutes mixing. The precipitate was separated and continued re-dissolved saturated NaCl solution for 120 minutes mixing (Laipan et al., 2020; Lubis, 2007). The precipitate was washed with boiled DW and calcined for 12 hours at 200°C. Then this activated bentonite is used for the next step of impregnation.

The next impregnation step is ammonium impregnation which is functionalized to adapt the bentonite feature in organic cassava wastewater. The process was carried out by preparing 50 g of the purified bentonite in saturated NH_4Cl solution for 2 hours of mixing, then diluted for 10 minutes in DW (the ratio of DW to the mixture is 2:1). The precipitate was separated followed by re-dissolved for 120 minutes in 165 mL of saturated NH_4Cl solution. The final precipitate was washed with boiled DW and calcined for 12 at 200°C. Then, this material is used as a coagulant and denoted as NH-B.

2.3 Coagulation Work

The coagulation process was initiated by pH measuring to stability checking for seven days every 24 hours. In addition, the effect of coagulant doses was investigated in steps as follows: 100 mL of cassava wastewater was added by bentonite-modified coagulant at 0.1, 0.2, 0.3, 0.4, and 0.5 g, then agitated at 750 rpm for 3 minutes followed by settling down for 25 minutes. Finally, the turbidity and pH of the final condition of coagulation were measured.

The total suspended solids (TSS) and chemical oxygen demand (COD) were analyzed according to the National Standard of Indonesia (SNI) 6989.3:2019 method (based on the gravimetry method) and the National Standard of Indonesia (SNI) 6989.73:2019 (based on the volumetric method), respectively. The analysis of TSS measured the precipitation of the suspended solid sample using a coagulant of NH-B (0.4 g of optimum NH-B coagulant dose/100 mL of cassava wastewater) and without a coagulant (as a control). Then, the separated precipitate was washed using DW and ethanol, and the dried sample mass was compared.

Furthermore, the COD was analyzed in the closed reflux by volumetric technique as follows: 2.5 mL of solution from the coagulation system (without the addition of coagulant and after added by 0.4 g NH-B coagulant) mixed with 1.5 mL of 0.1 N $K_2Cr_2O_7$ and 3.5 mL of H_2SO_4 in the COD pump reactor, then the mixture is homogenized followed by heating at 150°C for 2 hours. The mixture was settled down, followed by adding indicator ferroin then the mixture was titrated by ferroammonium sulfate solution (FAS) 0.25 N. The volume of titrant was noted to calculate the COD value.

2.4 Analytical Methods

The TSS of the sample in the coagulation process was calculated using Equation (1):

$$C_{tss} = \frac{(W_1 - W_0) \times 1000}{V_{\text{sample}}} \quad (1)$$

Where the W_1 and W_0 refer to the total mass (g) after and before coagulation.

The COD calculation was determined by the normality of FAS at Equation (2), followed by the calculation of the total COD value at Equation (3) as follows:

$$N_{FAS} = \frac{V_{K_2Cr_2O_7}}{V_{FAS}} \times N_{K_2Cr_2O_7} \quad (2)$$

$$C_{COD} = \frac{(V_{FAS0} - V_{FAS1}) \times N_{FAS} \times 8000}{V_{\text{sampel}}} \quad (3)$$

Where the V_FAS0 and $_VFAS1$ (mL) refer to the volume of FAS without a coagulant and with a coagulant.

The efficiency of coagulation to remove the turbidity, TSS, and COD was measured by the Equation (4):

$$\%_{efficiency} = \frac{(C_0 - C_1)}{C_0} \times 100 \quad (4)$$

Where the C_0 and C_1 (mg/L) refer to the concentration of cassava wastewater before and after the coagulant is added.

3. RESULTS AND DISCUSSION

3.1 Characterization of Material

Figure 1 shows the difference XRD pattern of SB (a) and NH-B (b) to analyze the structure changes. According to Figure 1, the peak increase at 22.1°, 36°, and 62° indicates the high crystallinity of bentonite according to the montmorillonite mineral quality. Ammonium impregnation improves crystallinity reformation (Hidayat and Nugraha, 2018). There observed that quartz as impurities have decreased the peak due to the impregnation process (Elkhalifah et al., 2013). A unique peak was increased at 10° to define the ammonium (NH_4^+) intercalant inside the interlayer of the bentonite structure (Pérez-Maqueda et al., 2003). Overall, the modification of bentonite by ammonium impregnation changed the crystallinity level of the bentonite structure and lifted the lamellar structure (Lindgreen, 1994).

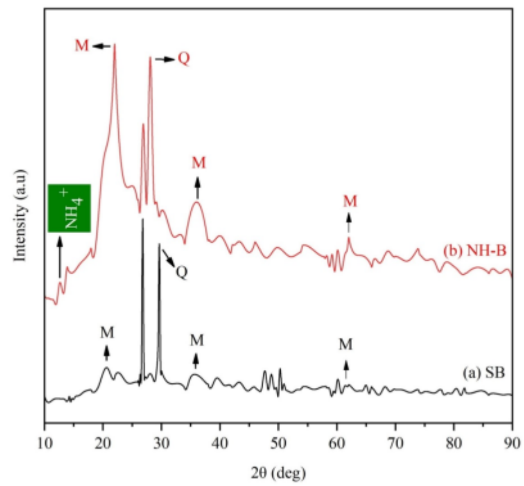

Figure 1. X-Ray Diffraction Spectrums Formation of SB (a) and NH-B (b) Coagulant

Table 1. EDX Analysis of Bentonite-impregnated Composition

Element	SB (%w)	NH-B (%w)
C	17.69	15.1
O	38.23	40.3
Ti	0.89	-
Fe	3.70	1.63
Mg	1.35	0.69
Na	0.17	1.9
Al	7.04	9.1
Si	23.2	25.3
K	0.76	3.3
Ca	6.99	2.8

The impregnation impacts the transformation of functional group changes from the bentonite, as shown by FTIR spectra in Figure 2. The multi-layer structure of bentonite established numerous octahedra hydroxyl groups indicated by stretching vibration peak at 3396 cm^{-1} (Li et al., 2022). The broadening spectrum at 3380 cm^{-1} of vibration H-O-Si on NH-B was possibly due to the breaking of hydroxyl groups and water molecules in the surface coagulant (Langeroudi and Binaeian, 2018). This phenomenon triggers destabilizing scheme in cassava wastewater, which is dissolved in water through hydration breaking. As a result, the water molecule of cassava wastewater will attract to interlayer ammonium-based bentonite coagu-

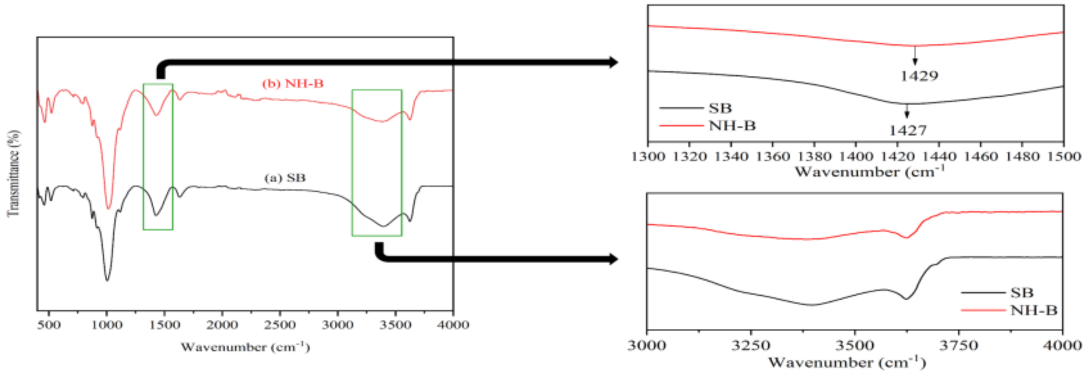

Figure 2. FTIR Spectrums Analysis of SB (a) and NH-B (b) Coagulant

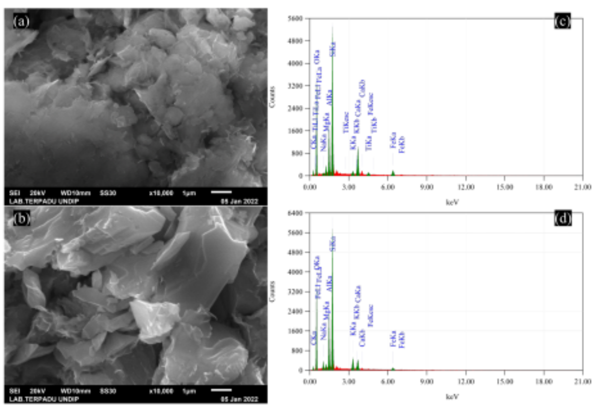

Figure 3. SEM Structure Analysis for SB (a), NH-B (b), and Graph of EDX Analysis for SB (c), NH-B (d) Coagulant

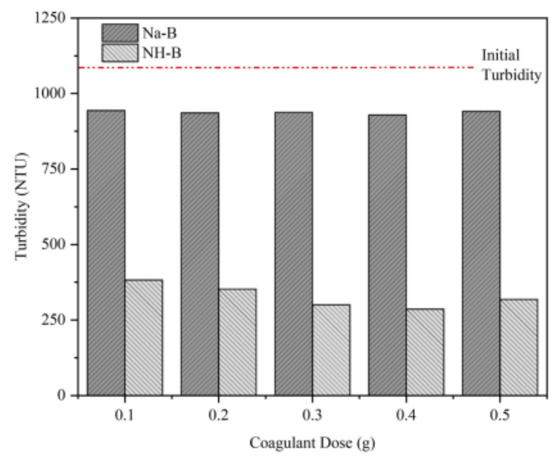

Figure 4. Turbidity Level on Different Coagulant Levels Used According to the literature, the coagulation will affect the pH final of the wastewater treated

lant. Other H-O-H vibration comes from the water molecules confirmed by a peak at 1630 cm^{-1}.

The fundamental structure of bentonite was confirmed by strong vibration stretching of Si-O-Si at 1013 cm^{-1} and Al-O-Al's vibration at 795 cm^{-1}. A slight difference in sharpness at 521 and 464 cm^{-1} is associated with the effect of the impregnation process; it is detected as the oscillation from the ammonium intercalant (Cheddie, 2012). Overall interpretation of FTIR analysis shows a slight transformation of clay's functional group. Surprisingly, ammonium impregnation significantly improves coagulant ability on bentonite-based coagulants.

The SEM analysis with ten-thousand zoom-in figured out the surface morphology transformation from the natural SB (to the impregnated one with increasing smoothness on the layer surface as a consequence of impurities reduction (see Figure 3a-b) (Lian et al., 2019). Furthermore, the expanded layer on NH-B provides numerous active sites in the bentonite layer and supports the neutralization and the bridging step in floc formation (see Figure 3b) (Sarkar and Dana, 2022; Yaghmaeiyan et al., 2022; Zhang et al., 2022). The EDX graph compared element amount in SB and NH-B as the peak height (see Figure 3c-d). The disappearance of the Ti+ element, according to the EDX graph, indicated that it is the impurities in the bentonite structure (Sirait et al., 2017). Table 1 describes the detailed composition of bentonite elements and shows the different conditions of Ti^{+}.

3.2 Coagulation in Cassava Wastewater

The characterization of impregnated bentonite was used as the fundamental hypothesis that the bentonite provided a coagulation ability. The application of bentonite coagulant was completed to reduce the turbidity, total suspended solids (TSS), and chemical oxygen demand (COD). Moreover, the result of coagulation was matched to the Law of number 5/2014 from

Table 2. The Quality Standard of Cassava Wastewater According to the Law of the Ministry of Environmental and Forestry of Indonesia Numb 5/2014

Parameters	Max. Quantity (mg/L)	Highest Pollution Load (Kg/tons)
COD	300	9
BOD	100	1.5
TSS	100	3
Cyanide (CN)	0.3	0.009
pH	6.0-9.0	
Waste Discharge	30 m3/tons of cassava product	

Table 3. Several Coagulants to Coagulation of Cassava Wastewater

Coagulant	Reduction Value (%)			Ref.
	Turbidity	TSS	COD	
Acquapol WW	88.5			
Acquapol S5T	88			
Tanfloc SL	90	Not examined		(dos Santos et al., 2018)
Tanfloc SG	92.5			
$Al_2(SO_4)_3$	55			
Extracts of Moringa oleifera seeds	89.19	Not examined	70.98	(Trevisan et al., 2019)
Powdered Seeds of Moringa oleifera	63.2	Not examined	74.1	(Ibiene et al., 2021)
NH-B	73.97	86.56	88.6	This work

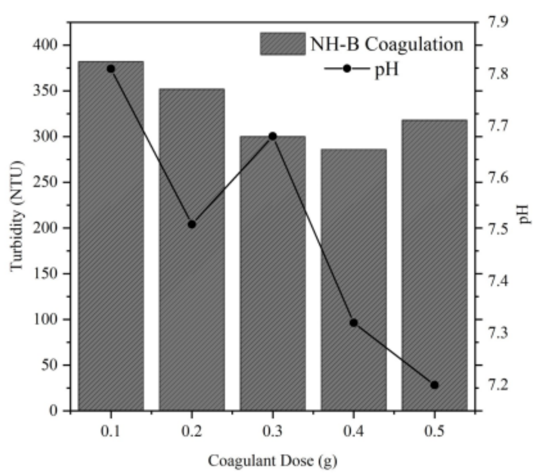

Figure 5. The Effect of pH on Turbidity Reduction

The Ministry of Environment and Forestry of Indonesia, which organized the quality standard of cassava wastewater allowed to dispose of in the water bodies. The details of data obligation are shown in Table 2.

The coagulation was initiated by determining the optimum coagulant doses to reduce the cassava wastewater turbidity. The testing condition was prepared with the variation of coagulant doses from 0.1 – 0.5 g of bentonite coagulant, and the comparison removal capability was shown in Figure 4. According to Figure 4, the optimum doses reached at 0.4 g coagulant indicated by the significant turbidity decreases from 1099 NTU to 186 NTU (up to 73.97% cassava wastewater removal). The comparison of the coagulation capability of SB decreased the turbidity from 1099 NTU to 929 NTU (only 15.47% cassava wastewater removal).

According to the literature, the coagulation will affect the pH final of the wastewater treated. Thus, the study was completed to expect the effect of bentonite coagulant utilization on the pH changes of wastewater (Naceradska et al., 2019). Figure 5 shows the final pH of cassava wastewater after coagulant in variation doses. The highest coagulation activity using 0.4 g of coagulant changed the pH of cassava wastewater to 7.32, which is acceptable in the environment according to the quality standard of cassava wastewater in the law of the Ministry of Environmental and Forestry of Indonesia Numb 5/2014.

The coagulation parameter of the suspended solid was measured by reducing the total suspended solid (TSS) using the gravimetry methods. This method was based on determining the floc formed, precipitated, and filtrated; then, the dried floc was weighed. The TSS level reduction was determined by calculating the different weights of the suspended from coagulation using NH-B and without coagulation; the results are shown in Figure 6a. Under the optimum dose of 0.4 g NH-B, the maximum TSS removal was reached up to 85.56% (the initial TSS concentration is 588 mg/L decreased to 79 mg/L).

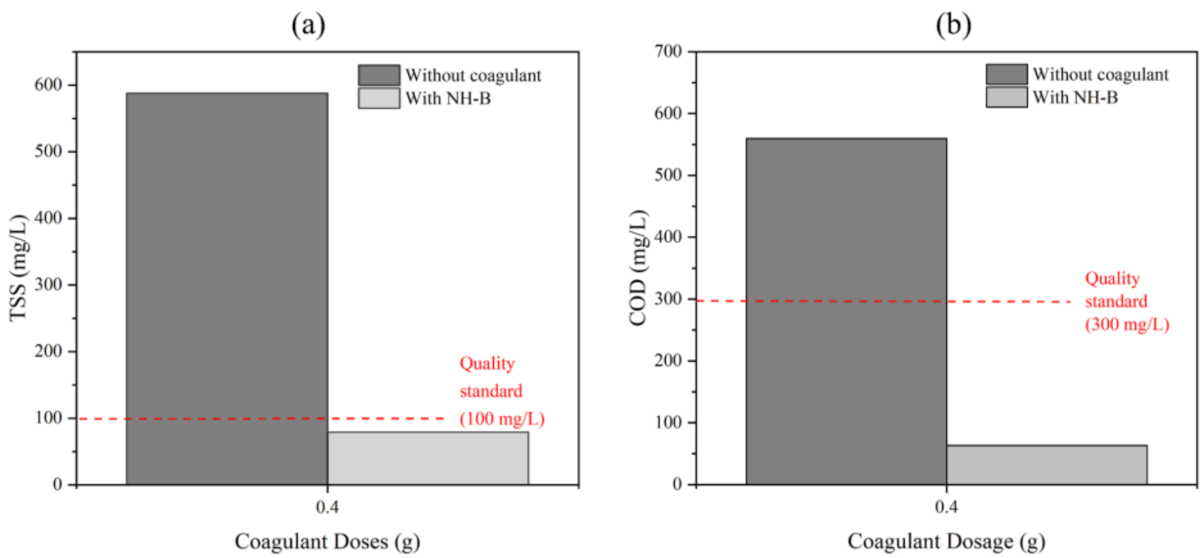

Figure 6. The TSS and COD Reduction Level After Coagulation Using NH-B

The chemical oxygen demand (COD) becomes a lethal parameter that determines the quality of the water ecosystem. Thus, the wastewater disposed of requires a prior treatment to keep the content of COD (Omer, 2019). Figure 6b shows the COD reduction level of cassava wastewater due to coagulation using NH-B coagulant; the reduction reached 88.6% (the initial COD concentration is 559.7 mg/L decreased to 63.3 mg/L). The proposed mechanism of flocs formation in suspended solid and reduction of COD level in the cassava wastewater was completed by neutralization of the negative charges and reactive particles in cassava wastewater and bridging floc formation due to the high mass of bentonite particles (Czemierska et al., 2015; Mcyotto et al., 2020; Precious Sibiya et al., 2021). We summarize the comparison of other cassava wastewater coagulants with our works in Table 3.

4. CONCLUSION

The Sumatera Bentonite was modified by ammonium impregnation, and then it was examined to remove cassava wastewater by coagulation process. The structure transformation and improvement were proved using XRD, FTIR, and SEM-EDX characterization. Several parameters from the cassava wastewater coagulation using NH-B coagulant, including the turbidity reduction of up to 73.97%, TSS reduction of up to 86.56%, and COD reduction of up to 88.6%, which decrease the waste content under the quality standard based on the law of Ministry of Environment and Forestry of Indonesia number 5/2014. Thus, this bentonite-based coagulant is effective in the coagulation of cassava wastewater.

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