

## Research Article

## The Effectiveness of Green Scallop Shell Chitosan as Coagulant in Treatment of Tofu Industrial Liquid Waste

*Efektivitas Limbah Cangkang Kerang Hijau sebagai Koagulan Dalam Pengolahan Limbah Cair Industri Tahu*

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### Abstract

Tofu waste is gotten after processing soybean, and this waste contains a lot of polluting substances, hence it can pollute rivers and cause health problems. Meanwhile, one method used for treating wastewater into clean water is called the jarrest method and its processes include coagulation-flocculation and deposition. This study aims to determine the effectiveness of green mussel shells as coagulants in the treatment of tofu industrial liquid waste. The variables used were 100 mesh green mussel shell powder, 1000 mL of liquid tofu dregs, 150 rpm fast stirring speed for 2 minutes, and 60 rpm slow stirring speed for 15 minutes. Furthermore, chitosan was used with different weight variations in grams (0.5, 0.7, 0.9, 1.1, and 1.3) as well as precipitation time with variations in minutes (20, 30, 40, 50, and 60). The content of Chitosan water was 1.29% and its degree of deacetylation was 65.04%. The result of the preliminary analysis of tofu liquid waste with a coagulant showed BOD, COD, and TSS levels of 965.25mg/L, 435mg/L, and 395mg/L with pH 4 respectively. However, these levels were changed to 195.56mg / L; 299mg/L; and 195.32 mg/L with pH 6 after the final analysis of the liquid waste was conducted.

**Keywords:** coagulant; green mussel shells; jarrest method; tofu industry

### Abstrak

Limbah tahu merupakan sisa dari pengolahan kedelai yang terbuang karena tidak terbentuk menjadi tahu. Limbah tahu banyak mengandung pencemar yang dapat mencemari sungai dan menimbulkan gangguan kesehatan. Metode jarrest adalah suatu metode pengolahan air limbah menjadi air bersih yang prosesnya meliputi proses koagulasi-flokulasi dan pengendapan. Penelitian ini bertujuan untuk mengetahui efektivitas cangkang kerang hijau sebagai koagulan dalam pengolahan limbah cair industri tahu. Variabel yang digunakan yakni serbuk cangkang kerang hijau 100 mesh, 1000 mL ampas tahu cair, pengadukan cepat 150 rpm selama 2 menit, dan pengadukan lambat 60 rpm selama 15 menit. Variasi berat kitosan yang digunakan dalam gram antara lain 0,5; 0,7; 0,9; 1,1; 1,3; dan waktu pengendapan dalam menit 20, 30, 40, 50, 60. Kadar air kitosan 1,29% dan persentase deasetilasi 65,04%. Analisis awal limbah cair industri tahu dengan koagulan diperoleh kadar BOD, COD, TSS berturut-turut 965,25mg/L; 435mg/L; 395mg/L; dan pH 4. Pada analisis akhir limbah cair industri tahu, diperoleh perubahan kadar BOD, COD, TSS menjadi 195,56mg/L; 299mg/L; 195,32 mg/L dan pH 6.

**Kata kunci:** cangkang kerang hijau; industri tahu; koagulan; metode jarrest

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### 1. Introduction

Kenjeran, in Surabaya is one area that has at most 3 tons of shells discarded on the Kenjeran seaside [1]. These discarded shells are partly made up of the green mussel shell, which is a source of minerals with high carbonates. The shells are widely used by local people as decoration while the rest are left on the Kenjeran. Thus, the remaining shells can be converted into chitosan, which can be used as a coagulant for the process of purifying river water and waste. The content of green mussel shells that can produce chitosan is called chitin [2]. Furthermore, these shells contain calcium oxide (CaO), silicon dioxide (SiO<sub>2</sub>), iron (III) oxide (Fe<sub>2</sub>O<sub>3</sub>), magnesium (MgO), and aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) [3].

One known water contaminant is the tofu waste and it is produced by tofu industries. These industries are fast-growing in big cities and liquid waste is the dominant type of waste from the tofu production process [4]. These wastes contain high protein and thus pollute the environment with a bad smell as soon as they are disposed of without prior processing and this will automatically reduce the quality of the water bodies in which the waste is disposed of [5]. Furthermore, the waste contains both suspended and dissolved solids, which will undergo physical, chemical, and biological changes hence they can cause health problems because they produce toxic substances or create a medium for the growth of microorganisms in the human body [6].

Wastewater left on land will be absorbed by the ground and can accumulate with well water sources. Liquid waste flowing into rivers can contaminate and cause health problems in

the form of itching, diarrhea, cholera, intestinal inflammation, and other diseases, especially those related to blood. In addition, the river water will become dirty and cause poor sanitation [7]. Therefore, it is necessary to treat tofu industrial liquid wastes before disposal in order to achieve the quality standards determined by the Indonesian Minister of Environment Number 5 of 2014 [5].

Industrial waste treatment can be performed either by physical, chemical, or biological means. Physical wastewater treatment is carried out in various stages such as filtration, grit chamber, sieving, sedimentation, and equalization. Chemical wastewater treatment includes chemical deposition, gas transfer, adsorption, disinfection, and dechlorination. Meanwhile, the biological treatment is divided into three models, including aerobic, anaerobic, and facultative treatments [8].

The jarrest method is a wastewater treatment method whose processes include coagulation, flocculation, and sedimentation. It is used to remove suspended solids that can cause turbidity problems [9]. Coagulation is the process of treating water or wastewater by stabilizing colloidal particles to ensure particle growth during flocculation. Meanwhile, flocculation is a type of wastewater treatment that contacts the colloidal particles resulting from coagulation to become larger flocs hence they can settle quickly [6]. The next process is sedimentation also known as the deposition of coagulant particles. In this process, all streams of solids whose density is greater than the density of water (or liquid) will settle for a certain time and during that time, they are automatically separated from the water. The time required for the

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particles to settle is dependent on their sizes as well as the position of the high and low particles from the bottom of the sedimentation tank [10]. Parameters measured in the tofu industrial liquid waste treatment process with the addition of coagulant include wastewater pH, Biological Oxygen Demand (BOD), Chemical Oxygen Demand (COD), and Total Suspended Solid (TSS) [11].

Subsequently, the coagulation and flocculation processes are influenced by the pH level of the wastewater and the type of coagulant used. The appropriate waste pH makes the coagulant work well. The selection of the types of coagulant and flocculant used should be adjusted to the type of colloid contained in the wastewater. These types of coagulants and flocculants are generally oppositely charged to the ionic charge in the wastewater and they function to bind ions in wastewater in order to reduce the repulsion between colloidal particles, hence flocs can be formed [12].

Coagulants play an important role in clean water treatment, especially in terms of reducing turbidity, Total Dissolved Solid (TDS), and Total Suspended Solid (TSS). Alum sulfate, poly-aluminum chloride, ferrous sulfate (FeSO<sub>4</sub>), and ferric chloride (FeCl<sub>3</sub>) are examples of commonly used chemical coagulants. Meanwhile, the use of natural ingredients as coagulants is currently being developed because it has several advantages such as being biodegradable, safer for human health, and more economical. Natural coagulants can be found easily because they can be obtained or extracted from local ingredients such as plants or animals [13].

Chitosan is a by-product obtained from the oxidation of chitin hence it can be

used as an herbal coagulant in water treatment [14]. The degree of chitosan deacetylation is generally about 60%, and about 90-100% for fully deacetylated chitosans [15]. Furthermore, the advantages of using chitosan as a coagulant include the fact it is non-toxic, biodegradable, multi-electron cation, and easily interacts with other organic substances such as protein, hence it has the potential as a natural coagulant for water treatment [16].

Aulia *et al.* [17] used 1000 mL of tofu industrial liquid waste with a fast stirring speed of 150 rpm for 2 minutes and a slow stirring of 60 rpm for 15 minutes, followed by precipitation for 30 minutes. The result of their study showed that the addition of a green mussel shell coagulant dose at 0.7 grams or 700 mg/L can reduce the COD and TSS levels to 1339.59 mg/L and 119mg/L with a removal efficiency of 73.09% and 90.846% respectively.

Following this, Farihin *et al.* [18] used 100 mL of PT. Sido Muncul, Tbk liquid waste with variations in the dose of green mussel shell chitosan per liter of waste, such as 150 mg/L, 200 mg/L, 250 mg/L, and 300 mg/L. The best results were obtained at a variation of 250 mg/L with fast stirring of 100 rpm for 1 minute followed by slow stirring of 20 rpm for 15 minutes and precipitation for 30 minutes. Based on the results, this bio-coagulant reduced the COD concentration from 5082.86 mg/L to 1636.43 mg/L, reduce the TSS concentration from 2270 mg/L to 365 mg/L, and set aside the Turbidity concentration value from 621.1 NTU to 194.9 NTU.

Therefore, this research was carried out to determine the efficiency of green mussel shell waste as a coagulant in the tofu industrial liquid waste treatment as

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well as to observe the effectiveness of this natural coagulant in reducing pH, Biological Oxygen Demand (BOD), Chemical Oxygen Demand (COD), and Total Suspended Solid (TSS) levels.

### 2. Research Methods

#### 2.1 Research Place

This research was conducted at the waste treatment laboratory in the UPN "Veteran" Jawa Timur.

#### 2.2 Tools and materials

The raw material used in this research is the green mussel shell waste, which was obtained from the Kenjeran area, Surabaya. Meanwhile, the tofu liquid waste was obtained from the A Hok tofu factory.

The tools used include oven, pH meter, heating reactor, BOD bottle, stative and clamps, burette, Erlenmeyer, COD meter, beaker glass, and sieve. The series of research tools are shown in Figures 1 and 2.

#### 2.3 Research procedure

##### 2.3.1 Green Mussel Shell Preparation

Green mussel shells were washed and dried in the sun, then it was mashed and sieved to obtain a size of 100 mesh.

##### 2.3.2 Chitosan Preparation

###### 2.3.2.1 Deproteination stage

The green mussel shell powder was added to a 3% NaOH solution with a ratio of 1:6 (w:v). The solution was then heated to 80-90°C and stirred using a magnetic stirrer. After which, the solids were washed with distilled water until the pH is neutral and then dried in the oven.

###### 2.3.2.2 Demineralization stage

The result of the deproteination stage in the form of green mussel shell

powder was then added to a 1.25N HCl solution with a ratio of 1:10 (w:v) and also stirred using a magnetic stirrer at a temperature of 70-80°C. The resulting solids were also washed at neutral pH and then dried to obtain dry chitin.

###### 2.3.2.3 Deacetylation stage

Further, the demineralized dry chitin was dissolved in a 45% NaOH solution with a ratio of 1:20 (w:v) at 140°C and stirred with a magnetic stirrer. The resulting solids were then dried at 80°C to obtain the chitosan.

###### 2.3.2.4 Chitosan analysis stage

The water content and FTIR (Fourier Transform InfraRed) of the green mussel shell chitosan were then analyzed.

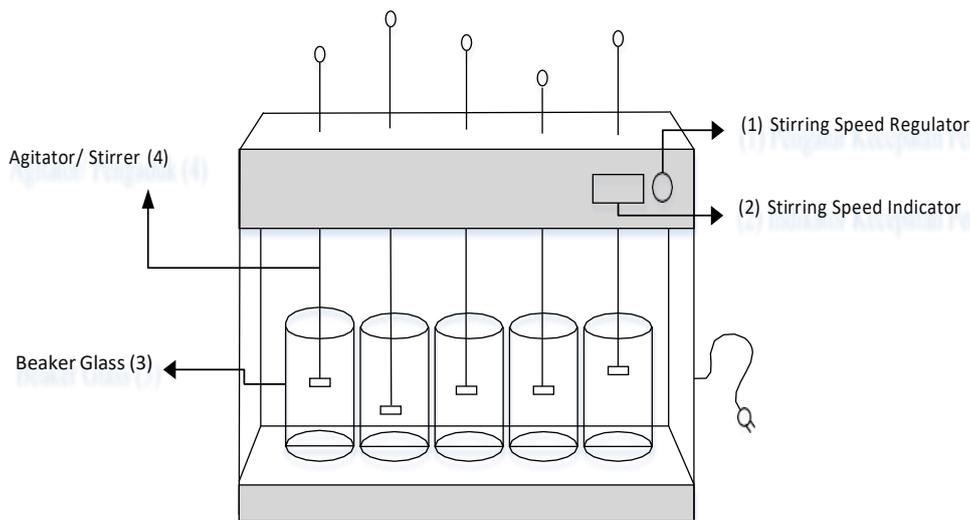
#### 2.3.3 Waste Treatment

The tofu industrial liquid waste sample collected was fresh and still in the temporary waste storage tank. Furthermore, the sample's pH, BOD, COD, and TSS levels were analyzed.

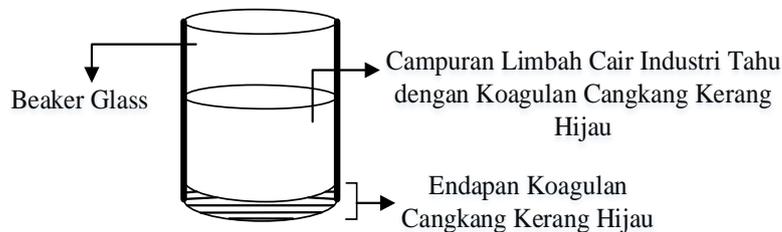
At most 1000 mL of tofu industrial liquid waste and green mussel shell coagulant with weight variations of 0.5 grams; 0.7 grams; 0.9 grams; 1.1 grams; and 1.3 grams was coagulated at a stirring speed of 150 rpm for 2 minutes at a glass beaker. After which, the flocculation process was carried out for 15 minutes by reducing the stirring speed to 65 rpm.

The further stage was the deposition process of the colloidal particles which resulted from flocculation (slow stirring), with variations in the precipitation time of 20, 30, 40, 50, and 60 minutes for each variation in chitosan weight. The treated wastewater was then analyzed for pH, BOD, COD, and TSS.

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**Figure 1.** The series of coagulation-flocculation process tools



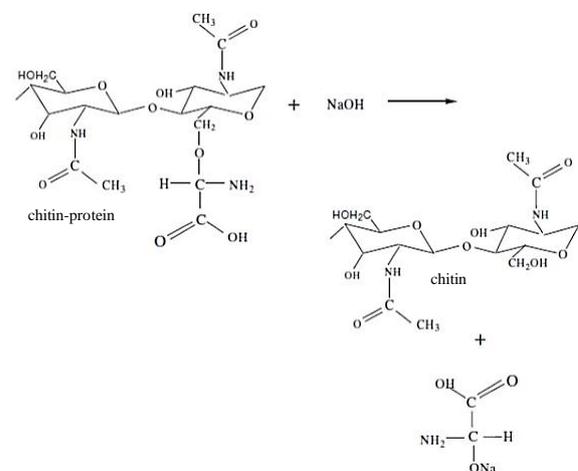
**Figure 2.** The series of sedimentation process

**3. Results and Discussion**

The process of producing coagulant from chitin involves several stages. First, is the deproteination. This stage aims to reduce the protein content with a dilute alkaline solution. Secondly, the demineralization stage is aimed at reducing the mineral content by using low acidity to obtain the chitin. Lastly, the deacetylation stage aims to completely remove the structure of acetyl chitin by heating it in a strongly alkaline solution at a high concentration [19].

Before the deproteination stage, Chitin was a proteinate. To make it protein-free, the material was dissolved in a 3% NaOH solution. The deproteination process uses high temperatures and stirring to accelerate the binding of the ends of the protein chains using NaOH hence the protein degradation and

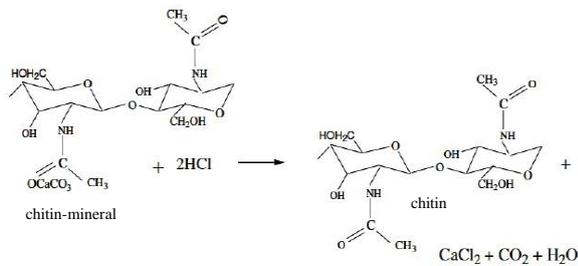
precipitation process were perfect. Furthermore, the purpose of washing with distilled water was to dissolve the Na-proteinate formed during the reaction hence it was lost during the filtering and washing process [20]. The reaction that occurs in the deproteination step is shown in Figure 3.



**Figure 3.** Deproteination stage reaction

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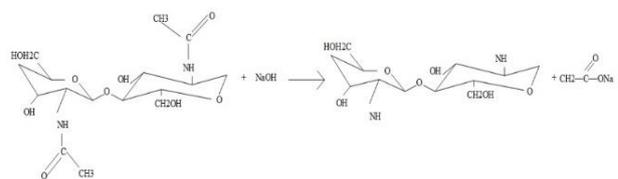
The application of Chitin contained in green mussel shells into the main mineral content of CaCO<sub>3</sub> dissolved in a 1.25N HCl solution will produce chitin solids, calcium chloride, carbon dioxide gas, and water. The demineralization process using HCl dissolved the minerals contained in the green mussel shells, thus drastically reducing the mineral contents in the chitin. When the deproteinized chitin was dissolved in HCl, a CO<sub>2</sub> gas bubble was formed which indicated the ongoing mineral release process [20]. The reaction in the demineralization stage is shown in Figure 4.



**Figure 4.** Demineralization stage reaction

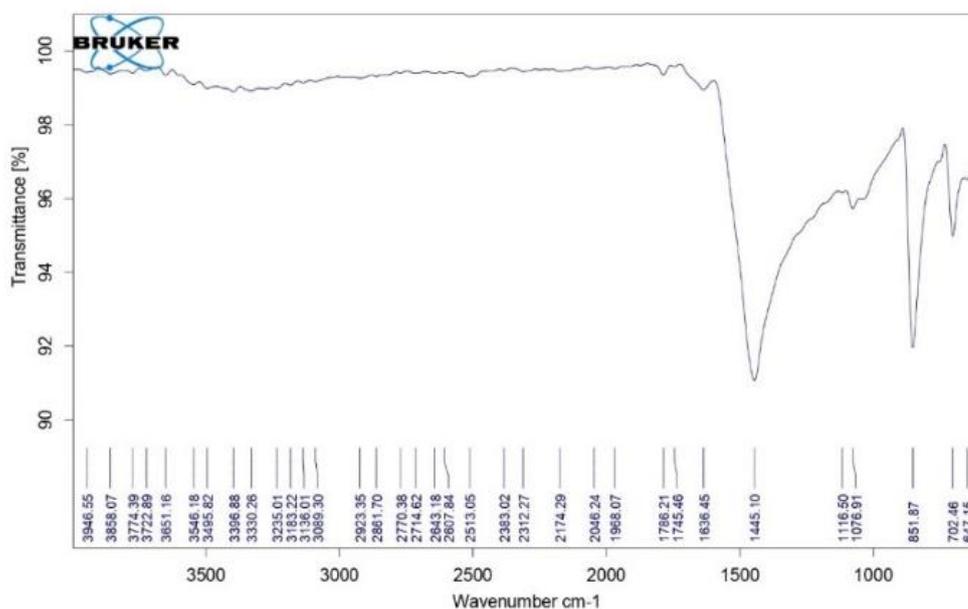
Chitin was the amide and NaOH was the base. At first, an addition reaction

occurred, the OH<sup>-</sup> group entered the NHCOCH<sub>3</sub> group, then the elimination of the CH<sub>3</sub>COO<sup>-</sup> group produced an amine, namely chitosan. Furthermore, the deacetylation process, which was conducted using a high concentration of base and high temperature affected the degree of deacetylation. The formation reaction of the chitosan from chitin was a hydrolysis reaction of an amide [20]. The reaction in the deacetylation stage is shown in Figure 5.



**Figure 5.** Deacetylation stage reaction

The preparation of chitosan from green mussel shells was carried out by analyzing the water content and the degree of deacetylation. Its water content was 1.29% and the degree of deacetylation was obtained from the FTIR spectrum analysis results shown in Figure 6.



**Figure 6.** FTIR spectrum of green mussel shell chitosan

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The degree of deacetylation was calculated by giving infrared light to the chitosan sample, then the light absorption rate was recorded. Additionally, the amide and hydroxyl groups were at wavelengths of  $1655\text{cm}^{-1}$  and  $3450\text{ cm}^{-1}$  respectively. Based on the infrared absorption recording results shown in Figure 6, the absorbance value of chitosan obtained at the two aforementioned wavelengths was 1.047 and 2.252 respectively, hence the degree of deacetylation obtained was 65.04%.

Following this, the obtained chitosan had a water content of 1.29% and a degree of deacetylation of 65.04%. This result is different from that of Aulia *et al.* [17] in whose study crab shell chitosan was used with a water content of 2.21% and a degree of deacetylation of 87.64%. The water content of the chitosan in green mussel shells is, however, lower than that of crab shells. This is influenced by the drying time as well as the amount and surface of the chitosan to be dried [21].

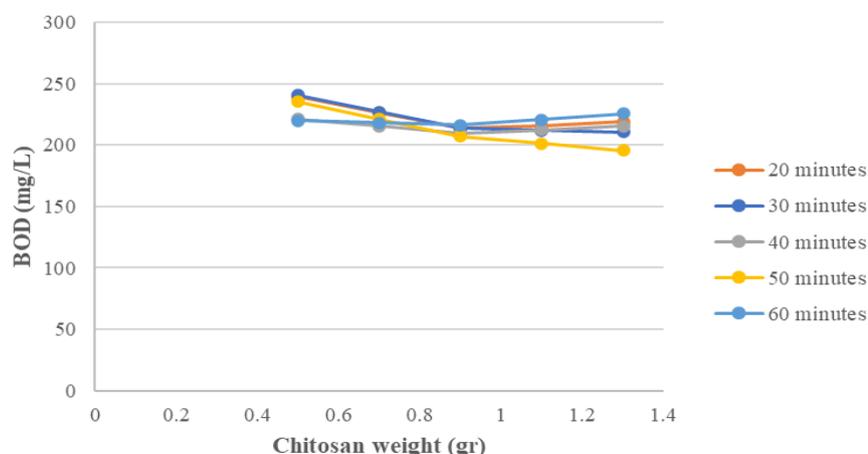
Accordingly, the degree of deacetylation in crab shell chitosan is higher than that of the green mussel shell because of the crab shell coagulant's

ability to separate more nitrogen-bound acetyl groups in the structure of chitin compounds, thereby increasing the ratio of amino groups in the chitosan [16].

**Table 1.** The initial analysis results of the tofu industrial liquid waste before going through the treatment process

Parameter	Unit	Concentration
BOD	mg/L	965.25
COD	mg/L	435.56
TSS	mg/L	395
pH		4

Table 1 shows the initial analysis results of the tofu industrial liquid waste before undergoing the treatment process. The waste had BOD, COD, and TSS levels of 965.25 mg/L, 435.56 mg/L, and 395 mg/L respectively with pH 4. Furthermore, the waste was then processed using the jarrest method with the green mussel shell chitosan as a coagulant. The analysis results of the BOD concentration in tofu industrial liquid waste that had undergone treatment are shown in Figure 7.

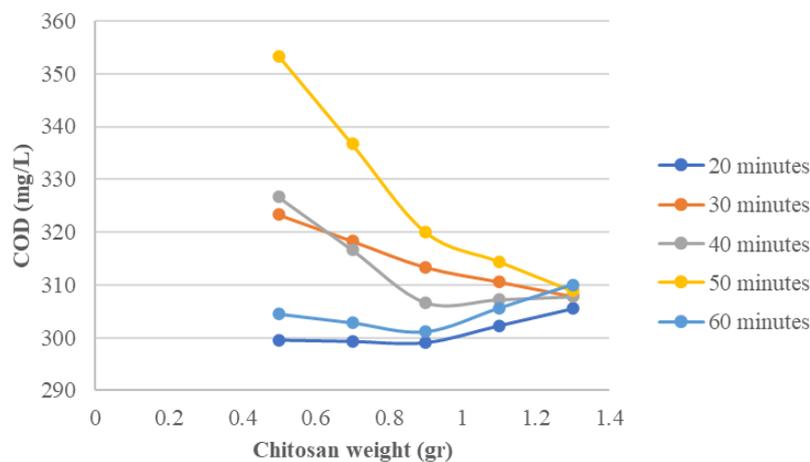


**Figure 7.** Analysis results of BOD level (mg/L) vs chitosan weight (grams)

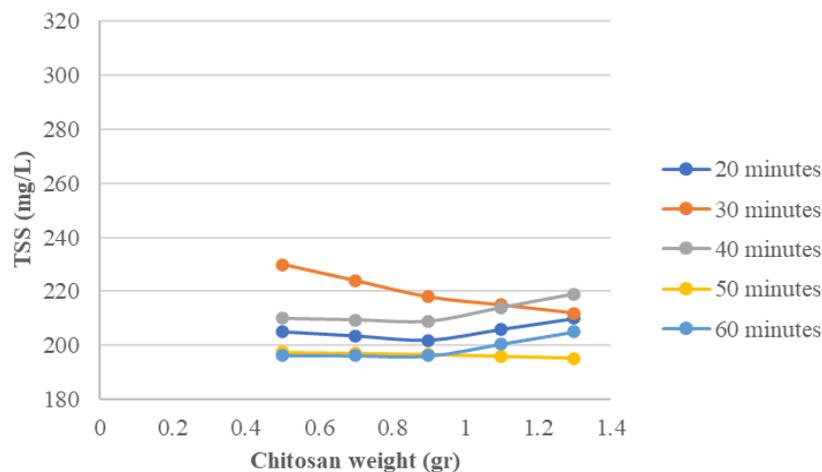
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Figure 7 shows that green mussel shell coagulant can reduce the BOD level. The best results were obtained from the addition of 1.3 grams of chitosan with a precipitation time of 50 minutes, where the BOD level was reduced by 195.56 mg/L from an initial concentration of 965.25 mg/L. However, the BOD produced was still far from the quality standards outlined by the Minister of

Environment Regulation Number 5 of 2014 at 150 mg/L [5]. This was due to the inaccurate speed and delay in stirring, hence the floc was not formed in the wastewater and precipitation occurred easily. In addition, the high BOD level has an impact on the death of aquatic organisms such as fish due to a lack of dissolved oxygen [22].



**Figure 8.** Analysis results of COD level (mg/L) vs chitosan weight (grams)



**Figure 9.** Graph for analysis of TSS level (mg/L) vs chitosan weight (grams)

The COD analysis in Figure 8 show that the green mussel shell coagulant can reduce the COD level. The best results were obtained with the addition of 0.9 grams of chitosan at 20 minutes of precipitation time which reduced the COD

level by 299 mg/L from the initial level of 435.56 mg/L. These results met the quality standards of the tofu industrial liquid waste from the Minister of the Environment Regulation Number 5 of 2014, which is a maximum limit of 300

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mg/L COD [5].

The COD level in the wastewater decreased as the concentration of organic matter decreased. A low COD level indicates that the amount of oxygen present can oxidize the organic matter in the wastewater properly [23].

Figure 9 shows that the green mussel shell coagulant reduced the TSS level of the tofu industrial liquid waste. The best results were obtained with the addition of 1.3 grams of chitosan at 50 minutes of precipitation time which reduced the TSS level by 195.32 mg/L from the initial level of 395 mg/L. This result also met the quality standards of tofu industrial liquid waste determined by the Minister of Environment Regulation Number 5 of 2014, which is a maximum limit of 200 mg/L [5].

## 4. Conclusion

Based on the observation results obtained, it can be concluded that the coagulant of mussel shell waste is quite effective in processing the liquid waste of tofu factories. The initial levels of BOD, COD, and TSS were 965.25 mg/L; 435.56 mg/L; and 395 mg/L respectively with pH 4. However, the processing carried out using the jarrest method with the addition of coagulant produced pH 6 with BOD, COD, and TSS levels of 195.56 mg/L; 299 mg/L; and 195.32 mg/L, respectively. Conclusively, green mussel shell coagulant can reduce the BOD, COD, and TSS levels of tofu industrial liquid waste. The decrease in BOD level, however, did not meet the quality standards of the tofu industrial liquid waste.

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