The Effect of Acetic Acid and Hydrochloric Acid as a Solvent for Chitosan to Reduce Turbidity in Eutrophic Water

by Utami Irawati

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Utami Irawati*, Fauziah A. Hikmah, and Ahmad B. Junaidi

***Abstract*—In this research, the potential of chitosan as a coagulant to reduce the turbidity of eutrophic water was investigated. To examine the possible effect of the solvent of chitosan on its performance in reducing the turbidity of eutrophic water, different types of acid, i.e. acetic acid and hydrochloric acid, were used to dissolve chitosan, in which the concentration of each acid was also varied. The chitosan dissolved in acetic acid was labeled as Chit-AA while the one being dissolved in HCl was labeled as Chit-HCl. A standard jar test apparatus was used to determine the optimum dosage and coagulation pH for both Chit-AA dan Chit-HCl. Both Chit-AA and Chit-HCl gave optimum results at a dosage of 10 mg/L, where the turbidity reductions after coagulation with Chit-AA and Chit-HCl were 64.78% and 85.33%, respectively. The optimum coagulation pH for Chit-AA was pH 7 while Chit-HCl gave an optimum result at pH 8. The concentration of the acid being used to dissolve chitosan appeared to affect the decrease in turbidity, where a higher acid concentration lead to a higher decrease in turbidity.**

***Index Terms*—Chitosan, coagulant, turbidity**

I. Introduction

Eutrophication is a condition where the excess nutrient content in the water leads to uncontrolled growth of aquatic plants and algae. Nitrate and phosphorus, in the form of phosphate, are the major contributor to excessive nutrients in eutrophication. These nutrients might come from domestic wastewater that has a high load of detergent waste [1]. One of the indicators that water has become moderately eutrophic is when the level of phosphate in said water is between 10–30 $\mu g \cdot L^{-1}$, along with a 500–1100 $\mu g \cdot L^{-1}$ of nitrogen content [2]. When the levels of phosphoric and nitrogen exceed 100 and 2000 $\mu g \cdot L^{-1}$, respectively, the water is considered to be hypertrophic [3]. While eutrophication naturally takes place over hundreds of years, in many places human activities have greatly increased the speed of eutrophication, causing some severe damages to the quality of the water [4, 5]. Surface waters suffering a decreased quality due to eutrophication needed to be treated before they can be used for consumption.

Some techniques have been explored to treat eutrophic water. Yu and Miao *et al*. [6] investigated the ability of six different aquatic plants to decrease the nitrogen and phosphorus levels in eutrophic water. Portland cement has been reported to give promising results in decreasing the level of phosphorus in the samples of river water to an acceptable level [7]. Novais and Zenero *et al*. [8] reported that biochar from poultry manure and sugarcane showed good potential to be used as an adsorbent to reduce the phosphorus content in eutrophic water. Furthermore, biochars that were prepared from those materials had higher adsorption capacity after being modified by doping them with Al ions. Phytoremediation using water lettuce to decrease the nutrient level and improve the quality of eutrophic water has also been proposed [9]. Mousavi and Dehghanzadeh *et al*. [10] suggested that ozonation, catalyzed by activated carbon is also a very efficient technique to treat eutrophic water. They demonstrated that catalyzing the process using activated carbon gave a significantly better result than the ozonation process alone.

Coagulation-flocculation is also a method that has been widely studied as a means of treating eutrophic water. This method is widely used as a water treatment method due to its practicability and economic feasibility. Particularly for water experiencing eutrophication, it should be noted that treatments using coagulation might need some enhancement steps, as a massive amount of algae would significantly increase the concentration of Natural Organic Matter (NOM) in the water. As pointed out by Cheng and Chi [11], the occurrence of eutrophication is very likely responsible to a decrease in coagulation efficiency in removing dissolved organic content in water. When it comes to the treatment of eutrophic water, it has been reported that aluminum-based coagulants exhibit more satisfying results than iron-based ones. In particular, a coagulant that releases more Al_{13} species, for example, Alum [$Al_2(SO_4)_3 \cdot 18H_2O$], gives a better performance compared to the ones which produce more monomeric and dimeric aluminum species, such as PACl [12].

The use of coagulation-flocculation has shown promising results in improving the quality of eutrophic water. The turbidity of the water showed significant improvement. The concentration of nutrients is also greatly reduced. Nevertheless, there have not been many reports on using natural coagulants to treat water bodies that suffer from eutrophication. The use of nature-based coagulants is particularly important in light of the raising concerns about the possible detrimental effects of using synthetic coagulants. Numerous studies have also reported that nature-based coagulants show satisfying results in treating water and wastewater [13, 14]. Chitosan is a natural polymer that has been extensively used as a coagulant to the commonly used synthetic coagulants. Chitosan is a very promising biomaterial to be used as a natural coagulant due to its abundance in nature, versatility, and its biodegradability property. Despite the many studies about the use of chitosan

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as a coagulant, not so many of those studies explore the use of chitosan to treat eutrophic waters. Those that study the use of chitosan for eutrophic water mainly focused on removing the algae and cyanobacteria from water [15–17]. The use of chitosan as a coagulant to improve the quality of water experiencing eutrophication in terms of its turbidity and phosphate content has not yet been widely investigated. This study mainly aims to explore the potential of using chitosan as a nature-based coagulant in improving the quality of eutrophic water. Considering what has been suggested by Huang *et al*. [18] that the use of acetic acid as a solvent for chitosan might increase the content of organic matter in the samples being treated, we investigated the effect of using two different acid, i.e. acetic acid and hydrochloric acid (HCl) as solvents for chitosan on the ability of chitosan to reduce turbidity in eutrophic water. The use of a proper solvent to dissolve chitosan is particularly important as eutrophic water has a high content of organic matter.

II. Methodology

A. Materials

Chitosan (85% DD) was obtained from PT. Tokopedia. Chemicals used were of reagent grade and used as received without further purification. Acid solutions were made by diluting concentrated acid using deionized water. Samples of eutrophic water were taken from a lake in Cindai Alus village, Banjar regency, South Kalimantan.

B. Preparation of Coagulant Solutions

Coagulant solutions were prepared by dissolving chitosan in an acid solution. One gram chitosan was carefully measured and then dissolved in 100 mL of 2% acetic acid to have a 1% (w/v) concentration of chitosan. The resulting solution was labeled as Chit-AA. The other coagulant solution, labeled as Chit-HCl, was prepared by dissolving one gram of chitosan into a 2% HCl solution.

C. Coagulation Experiment

Samples were treated by coagulation using chitosan that was dissolved in different acid, to investigate whether the solvent of chitosan holds a role in its efficacy to reduce turbidity. Coagulations on samples were done using a standard jar test apparatus (VELP Scientifica JLT-6) with 6 paddles. Samples were mixed rapidly (100 rpm) for one minute, followed by a slow mixing (40 rpm) for 20 min. The flocs formed were then let to settle for 30 min.

1) Effect of coagulant dose To study the effect of coagulant dose, various doses of coagulants, ranging from 5 to 25 mg/L were added to the samples using volumetric pipette. After the addition of of coagulants, samples were mixed through a rapid mixing and slow mixing using a standard jar-test apparatus as described above.
2) Effect of pH To investigate the effect of pH on coagulation, 0.1 N NaOH and or 0.1 N HCl was carefully added to the samples to set the pH. After the samples has had the intended pH, the samples were separated into two, each with equal volume. One of the samples was left without any coagulant addition to function as a control, while the other one was treated by adding 10 mg/L coagulant into it. After the addition of of coagulants, samples were mixed through a rapid mixing and slow mixing using a standard jar-test apparatus as described above.
3) Effect of solvent concentrationTo study the effect of solvent concentration on the efficacy of chitosan as a coagulant, a set of chitosan coagulants were prepared in acetic acid and hydrochloric acid with different concentrations, ranged from 1% to 5% . One gram of chitosan was dissolved into 100 mL acid on each concentration. Each coagulants was then added into 1000 mL sample where the dose of coagulant was set to be 10 mg/L, and sample was then treated through rapid and slow mixing using a standard jas test apparatus as described above.

D. Turbidity Measurement

After each treatment, samples were taken using a pipette (±4 cm from the surface) and their turbidity was measured. Turbidity measurement was done using a turbidimeter instrument (HACH, model 2100Q).

The turbidity reduction is calculated using the following formula:

$$\%\,Turbidity\ Reduction = \frac{T_i - T_f}{T_i} \times 100\%$$

E. Data Analysis

The turbidity reduction for each variables being studied was tabulated and then presented in a bar graphic to have an easier comparison between each data. A simple t-paired test was conducted for the results on each variables, i.e: dosage. pH and acid concentration to have a statistical point of view on the data.

III. Results and Discussion

Samples used in this study were taken from a village in Cindai Alus area, Banjar regency, South Kalimantan, Indonesia. The sampling location was once a fishing pond, where people intentionally scattered fish feed into it. This caused the pond was heavily loaded with nutrients from the fish feed. The surface of the water is covered by algae, a very distinct character of water suffering from eutrophication. The water is highly turbid with a dark green hue color. Fig. 1 showed the picture of the sampling location. A preliminary examination was conducted to measure the phosphate concentration in water in the sampling area. The result of this preliminary examination showed that the phosphate content in the sampling area was as high as 1.254 mg/L. This high level of phosphate indicated that the area was suffering from eutrophication, considering that a phosphate level between 10–30 µg/L is already considered to be moderately eutrophic [2].

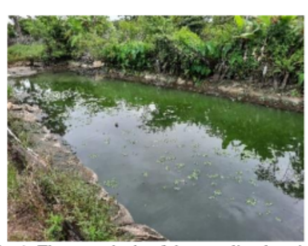

Fig. 1. The water body of the sampling location.

A. Effect of Coagulant Dose on Coagulation

In coagulation, the dose of coagulants added holds an important role in determining turbidity removal. Coagulation will not give a satisfying result if the dose is less than what is needed, and on the other hand, an excessive dose will lead to deflocculation, causing an increase in turbidity instead of reducing it. The range of dose in this study was chosen based on the previous research, that in most cases, chitosan needs relatively low dose to reduce turbidity (less than 50 mg/L) [19–21]. Table I presents the initial turbidity of the samples and final turbidity after the samples being treated using coagulant. The decrease in turbidity for each dose is shown in Fig. 2.

TABLE I: INITIAL AND FINAL TURBIDITY OF SAMPLES

Coagulant Dose (mg/L)	Chit-AA		Chit-HCl	
	Initial turbidity (NTU)	Final turbidity (NTU)	Initial turbidity (NTU)	Final turbidity (NTU)
5	42.3	21.2	52.7	11.8
10	42.3	14.9	52.7	7.73
15	42.3	16.3	52.7	13.3
20	42.3	18.3	52.7	22.8
25	42.3	19.2	52.7	41.2

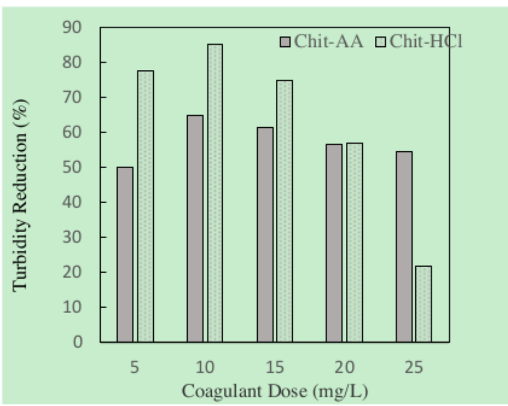

Fig. 2. Turbidity reduction for each coagulant dose.

Although both Chit-AA and Chit-HCl gave optimum turbidity reduction at a dosage of 10 mg/L, the results indicate that Chit-HCl gave a better turbidity reduction compared to Chit-AA. This is probably due to the properties of eutrophic water samples that are rich in organic content. As pointed out by Huang *et al*. [18], using acetic acid as a solvent for chitosan increased the total organic content in the suspension. In this research, the additional organic content coming from acetic acid solution might interact with the already-present organic compounds in the samples, hindering those compounds to be bounded with chitosan. However, it is interesting to note that as a coagulant, Chit-HCl seems to be more susceptible to deflocculation, as indicated by a more significant drop in turbidity reduction once the dose of the coagulant was surpassed. This is probably because HCl is a strong acid and thus, excessive addition of chitosan that is being added gives a considerable increase of the positive charges in the water samples, leading to a significant occurrence of deflocculation. On the other hand, since acetic acid is a weak acid, its addition to samples would not release as much positive charges as HCl. Hence, the deflocculation that took place in samples treated with Chit-AA was not as significant as the ones being treated with Chit-HCl.

From a statistical point of view, the average turbidity reduction for both coagulant within the dose range being investigated might not be significantly different. However, the significant drop in turbidity reduction for coagulation by Chit-HCl must be taken in account as a factor affecting the average value.

B. Effect of pH on Coagulation

Arguably, pH holds a vital role in coagulation as it dictates the speciation of compounds in the solution being treated. In this study, NaOH and HCl were used to have samples with five pH variations. In this study, a control solution was set for each pH being investigated to see how far pH adjustment can affect the turbidity of eutrophic water, and to compare the reduction that took place when chitosan coagulant was added to the samples. The initial and final turbidities of the control solution and treated samples are shown in Table II. The turbidity reductions for each pH being investigated are shown in Fig. 3.

TABLE II: INITIAL AND FINAL TURBIDITIES OF SAMPLES TREATED AT DIFFERENT PHS

pH	Initial Turbidity (NTU)	Final Turbidity (NTU)		
		Control Solution	Chit-AA	Chit-HCl
5	29.7	25.3	9.62	12.8
6	29.7	25.6	5.11	8.47
7	29.7	25.7	4.72	6.08
8	29.7	25.9	18.3	5.21
9	29.7	25.7	27.8	27.6

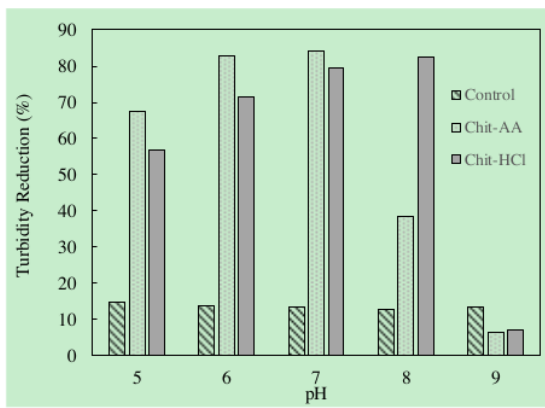

Fig. 3. Effect of pH on turbidity reduction.

As one can see from Table II, when pH was adjusted, the turbidity was only slightly reduced. This indicates that the surface charges of the colloidal particles in eutrophic water are quite excessive that these charges could not be neutralized solely by adjusting the pH of the water. Data in Table II clearly showed that chitosan as coagulant significantly reduced the turbidity of the samples for most of the pH being studied.

From Fig. 3, both Chit-AA and Chit-HCl appeared to give better performances at a slightly acidic and neutral pH. Chitosan has been reported that chitosan has a pKa ranging from 6.17 to 6.51, depending on its deacetylation degree [22]. Hence, at around pH 6, approximately half of the amine groups in chitosan are protonated, enabling chitosan to act as a coagulation agent through the charge neutralization mechanism. However, once the pH is much lower than its pKa, practically all of the amine group will be protonated, leading to an excess of positive charges in the system that will eventually lead to deflocculation.

Fig. 3 also showed that at a pH higher than 7, the turbidity reduction for coagulation by Chit-HCl dropped significantly, and continued to decrease when the pH got even higher. As for Chit-AA, the drop of turbidity reduction occurred at pH 9. Due to this pattern, the difference between average turbidity reduction caused by coagulation using Chit-HCl might not seem to be significantly different from coagulation by Chit-AA. Nevertheless, it has shown a clear indication that pH holds a very important role in coagulation in reducing turbidity.

One thing that is interesting to note is that Chit-AA worked better than Chit-HCl when the samples are in acidic condition. The results also showed that while there is a significant decrease in turbidity reduction by Chit-AA once the system is in an alkaline condition, coagulation using Chit-HCl still gave a relatively good performance, in terms of turbidity reduction. These results implied that there might be differences in the coagulation mechanisms taking place when different acids were used to dissolve chitosan. As summarized by Yang *et al*. [23], it has been reported that the flocculation mechanism by chitosan might involve charge neutralization, or formation of a bridging between particles. Furthermore, as highlighted by Renault *et al*. [24], while flocculation by chitosan might involve both charge neutralization and interparticle bridging, the main mechanism taking place between those two will be determined by a number of factors.

Based on the data obtained in this study, when chitosan is being dissolved in acetic acid, the main coagulation mechanism is probably charge neutralization. On the other hand, when HCl is being used as a solvent for chitosan, the coagulation mechanism might be dominated by the formation of inter-particles bridging instead of solely based on the charge neutralization mechanism. This probability is because as proposed by Belnikevich *et al*. [25], when dissolved in HCl, chitosan tends to have shorter polymer chains, enabling it to have a more extended conformation. This kind of conformation is more effective to function as a bridge between the suspended particles. As the floc formation is dominated by this interparticle bridging mechanism, the coagulation efficacy does not heavily affected by the protonation of amine groups in chitosan. When acetic acid is used as a solvent for chitosan, the longer chains that it has would lead to a more coiled conformation [26]. With this kind of conformation, it is very likely that the charge neutralization is the dominating mechanism of coagulation. As the efficacy of coagulation that takes place through charge neutralization depends on the available positive charge of the coagulant, the amount of protonated amine groups in chitosan will significantly affect the coagulation efficacy. This also comes in line with what has been suggested by.

C. Effect of Solvent Concentration

The concentration of acid used to dissolve chitosan has been reported be one of the factors that can influence the performance of chitosan as a coagulant. To investigate the effect of solvent further, the coagulants were then prepared by dissolving chitosan in acid with different concentrations. The coagulants were used to treat samples with a dose of 10 mg/L. Table III shows the initial and final turbidities of samples treated with chitosan being dissolved in different concentration of acid. The turbidity reductions are presented in Fig. 4.

TABLE III: INITIAL AND FINAL TURBIDITIES OF SAMPLES TREATED WITH CHITOSAN DISSOLVED IN DIFFERENT ACID CONCENTRATION

Acid Concentration	Initial Turbidity (NTU)	Final Turbidity (NTU)	
		Chit-AA	Chit-HCl
1%	72.6	35.1	22.5
2%	72.6	22.3	13.0
3%	72.6	15.4	10.4
4%	72.6	11.3	10.2
5%	72.6	8.31	8.21

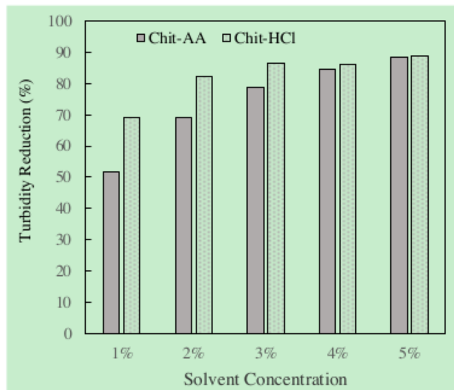

Fig. 4. Effect of solvent concentration on turbidity.

From a statistical point of view, the data obtained in this parameter showed that there was a significant difference of average turbidity reduction between coagulation using Chit-AA and Chit-HCl. From Fig. 4, it can be observed that when used as a solvent for chitosan, a lower concentration of HCl was needed to have a satisfying result than when acetic acid was used as a solvent. A concentration of 2% for HCl was already enough to give more than 80% of turbidity reduction. Increasing the concentration of HCl above 2% did not give a significant increase in turbidity reduction. This is probably due to the nature of HCl being a strong acid, so a 2% concentration was already sufficient to protonate enough amine groups in chitosan to be an efficient coagulant. Acetic acid, on the other hand, is a weak acid that does not completely dissociate to release H^+. For this reason, even when acetic acid and HCl are used as solvents for chitosan at the same concentration of acid, chitosan that is being dissolved in acetic acid might not have as much as protonated amine group as the one being dissolved in HCl. This comes in line with what had been proposed by Rinaudo *et al*. [27], that more amine groups in chitosan are protonated when a strong acid is used instead of a weak acid.

IV. Conclusion

In this study, the effect of the type of acid being used as a solvent on the ability of chitosan to reduce the turbidity in eutrophic water was investigated. Hydrochloric acid seems to be a more efficient acid to be used as a solvent for chitosan, as the chitosan being dissolved in HCl needed a lower dose to give an optimum result in terms of turbidity reduction. When dissolved in HCl, chitosan also still worked quite effectively in a slightly alkaline condition, which was not the case when it was being dissolved in acetic acid. This pattern might be an indication that the dominating coagulation-flocculation mechanism is different when the solvent used is different. As HCl is a strong acid, a lower concentration of acid is needed to have a preferable result, compared to acetic acid. Neverthless, further research is still much needed to have a stronger conclusion on how the type of solvent can influence the efficacy of chitosan as a coagulant. Studies on how solvent of chitosan might affect its performance as a coagulant in improving other important parameters of water quality, such as nitrogen, phosphorus and total organic contents should also be conducted.

Conflict of Interest

The authors declare no conflict of interest.

Author Contributions

Utami Irawati is the main author of this paper who also designed the experiment. Fauziah A. Hikmah did the sampling and analysis. Ahmad B. Junaidi did the data analysis. All of the authors did the data interpretation. All authors had approved the final version.

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