REMOVAL OF METANIL YELLOW AND TARTRAZINE USING CHITOSAN AS AN ALTERNATIVE COAGULANT

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Abstract: The textile industry is one of the industries that heavily use dyes. Wastewater coming from these industries will have a negative impact on the environment if it is released without prior treatment. Examples of synthetic dyes that are toxic to the environment are metanil yellow and tartrazine. In light of the presence of those dyes that might harm the environment, a proper treatment of wastewater containing those dyes is needed. Treatment using nature-based coagulants is one of the methods that can be implemented to reduce the concentration of dyes in the environment. This study investigates the performance of chitosan as an alternative nature-based coagulant compared to the two most commonly used synthetic coagulants, i.e. alum and poly aluminium chloride (PAC), in removing tartrazine and metanil yellow from solutions. The effect of coagulant dosage and settling time were examined. Based on this study, the dosage needed by chitosan as a coagulant to remove metanil yellow and tartrazine with an initial concentration of 100 ppm were 10 and 100 ppm, respectively. The optimum dosage of PAC to remove 100 ppm of metanil yellow and tartrazine was 50 and 200 ppm, while the optimum dosage of alum to remove those dyes was 50 and 500 ppm. At their respective optimum dosage, the percentage of metanil yellow being removed by chitosan, PAC, and alum were 17.165%, 51.009%, and 14.284%, respectively. As for the coagulation of tartrazine, the optimum removal percentages by chitosan, PAC, and alum were 90.559%, 84.770%, and 29.178%, respectively. Chitosan and PAC exhibited more efficient coagulation in terms of the settling time needed to have optimum results, which was 60 minutes for both coagulants. Alum needed a longer settling time as within the timeframe being studied, the removal of dyes by alum had yet reached equilibrium.
Keywords: coagulation; chitosan; dyes

Abstrak: Industri tekstil merupakan industri yang menggunakan bahan pewarna sebagai bahan baku untuk hasil produksinya. Limbah hasil produksi ini terkadang masih mengandung zat warna dalam jumlah besar tentunya sehingga dapat berdampak negatif bagi lingkungan. Contoh zat warna sintetik yang dapat mencemari lingkungan adalah metanil *yellow* dan tartrazin. Terkait keberadaan kedua macam zat warna tersebut yang dapat menurunkan kualitas lingkungan perairan, diperlukan metode pengolahan yang ramah lingkungan atas zat warna tersebut, misalnya dengan koagulasi menggunakan koagulan berbahan dasar alami. Penelitian ini bertujuan untuk mengetahui perbandingan kinerja koagulan kitosan, tawas, dan PAC dalam menurunkan kadar tartrazin dan metanil *yellow* (MY) dalam larutan. Koagulasi terhadap sampel zat warna dilakukan dengan

menggunakan seperangkat instrumen *jar test* standar, dan konsentrasi zat warna diukur dengan menggunakan spektrofotometer UV Vis. Variabel yang diamati dalam penelitian ini meliputi dosis koagulan dan waktu sedimentasi. Hasil penelitian menunjukkan bahwa dosis optimum kitosan untuk menurunkan kadar MY dan tartrazin dalam larutan dengan konsentrasi awal zat warna 100 ppm adalah sebesar 10 dan 100 ppm. Dosis optimum PAC untuk menurunkan kadar MY 100 ppm dan tartrazine 100 ppm adalah 50 dan 200 ppm, dan dosis optimum tawas untuk menurunkan kadar MY 100 ppm dan tartrazine 100 ppm masing-masing adalah 50 ppm dan 500 ppm. Pada dosis optimum tersebut, penurunan kadar MY oleh kitosan, PAC dan tawas masing-masing adalah sebesar 17,165%; 51,009% dan 14,284%. Untuk koagulasi tartrazin, penurunan kadar tartrazin oleh kitosan, PAC dan tawas pada dosis optimum masing-masing adalah sebesar 90,559%, 84,770%, dan 29,178%. Kitosan dan PAC merupakan koagulan yang lebih efisien ditinjau dari waktu sedimentasi yang dibutuhkan untuk memperoleh hasil yang optimum, yaitu sebesar 60 menit. Tawas membutuhkan waktu yang lebih lama, dimana dalam kisaran waktu yang diamati, persentase penurunan kadar zat warna masih belum mencapai kestabilan. Hasil penelitian ini selanjutnya dapat dikembangkan sebagai alternatif untuk pengolahan limbah industri, terutama limbah industri yang menggunakan zat warna dengan intensitas tinggi.
Kata kunci: koagulasi; kitosan; zat warna

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Introduction

Synthetic dyes are one of the compounds that are widely used in various industries. The textile and food industries are examples of industries that use many dyes in their production processes. Apart from its use in industry, several side effects of synthetic dyes should be of concern. Most synthetic dyes are compounds that are difficult to decompose in the environment (Gita et al., 2017; Yaseen & Scholz, 2019; Yusuf, 2019). Therefore, the accumulation of synthetic dyes originating from industrial waste will certainly reduce environmental quality and have the potential to have a negative impact on living things.

Tartrazine and metanil yellow are two synthetic dyes widely used in industry, especially the textile and food industries. The presence of these two synthetic dyes in the environment can threaten living creatures. Based on various research results, consuming dyes dissolved in water can cause various diseases, ranging from skin diseases, and digestive, nervous and respiratory disorders to carcinogenic potential (Lellis et al., 2019). In light of this, it is necessary to treat the dyes in industrial liquid waste before it is released into the aquatic environment. Various research results report the adverse effects of tartrazine, such as neurological disorders, respiratory problems, and even DNA damage (Amin & Al-Shehri, 2018; Dehkordi et al., 2021; Leulescu et al., 2018). Metanil yellow is also a synthetic dye whose exposure to living creatures can have a toxic impact on living creatures (Ghosh et

al., 2017; Khan et al., 2020), and can cause tumours in various tissues of the liver, bladder, digestive tract, or skin tissue (Bhernama et al., 2015).

Various methods have been studied to treat liquid waste containing dyes. Coagulation is a method that has the potential to be applied in processing dyes in liquid waste. Coagulation itself is a water and wastewater treatment method that is commonly used because it is easy to apply, even on a large scale. Processing liquid waste containing dyes through coagulation has been widely studied. Even though coagulation is a fairly effective method for treating dye waste, synthetic coagulants such as PAC and alum can potentially increase the environmental burden, partly because the volume of sludge produced is generally quite large. In addition, because most conventional coagulants are aluminium and/or iron salts, their use can potentially increase the concentration of dissolved metals in the water. Chitosan, a multifunctional biomaterial, is one of the coagulants that is widely preferred as an alternative to the synthetic coagulants that are commonly used. Chitosan has been proven to be used as a coagulant in waste treatment and processing of natural water bodies (Irawati et al., 2023; Jadhav & Mahajan, 2013; Lichtfouse et al., 2019; Renault et al., 2009). The use of chitosan as a coagulant to treat waste-containing dyes was reported by, among others, Abdullah & Jaeel (2019); Alibeigi-Beni et al. (2021), Hossain & Hossain (2020) and Watcharin & Wiratthikowit (2019). Despite the numerous research on coagulating wastewater, the use of coagulation to remove certain dyes so that the interaction between the coagulant and the dye can be studied further is still very limited. In this research, the coagulation of tartrazine and metanil yellow was carried out using chitosan as a coagulant. The effect of dosage and sedimentation time was studied to estimate the optimum coagulation conditions for each dye using chitosan.

Methodology

Tools and Materials

The tools used in this material are standard glassware, analytical balances, and magnetic stirrers. Coagulation treatment was conducted using jar-test equipment (VELP Scientifica JLT-6). pH was measured using the HACH sensION+ pH1 pH meter. Dye analysis was carried out using a UV UV-vis spectrophotometer (Genesys 20 Visible).

The materials used in this research were commercial chitosan (DD 70-85%), commercial alum and poly aluminium chloride (PAC), metanil yellow (MY) and tartrazine dyes, as well as reagent grade chemicals produced by Merck. All the materials were used as received, without further purification process unless stated otherwise.

Preparation of coagulant solution

The chitosan coagulant solution was prepared by dissolving 1 gram of chitosan in a 2% acetic acid solution to obtain a solution with a chitosan concentration of 1% (w/v). Alum and PAC coagulants were prepared by dissolving

1 gram of each compound in distilled water to obtain a solution with alum concentration of 1% (w/v) and PAC 1% (w/v).

Optimization of dye measurements

Optimization of dye measurements for both tartrazine and metanil yellow was done by determining the optimum wavelength and optimal pH measurement. To determine the optimum wavelength, the absorbance of a dye solution with a concentration of 10 ppm was measured at a wavelength of 300-500 nm using a UV-Vis spectrophotometer. The wavelength giving the maximum absorbance reading was recorded as the optimum wavelength. Subsequent measurements were carried out at the optimum wavelength for each dye. Optimum pH measurements were conducted by preparing a set of dye solutions with a concentration of 10 ppm. Next, the pH of each solution was adjusted by dripping HCl and/or NaOH until a set of dye solutions was obtained with pH varying in the range 4, 5, 6, 7, 8, 9, 10. The absorbance of each solution was measured using a UV -Vis spectrophotometer at maximum wavelength. The pH value of the solution with the highest absorbance was determined as the optimum pH for measurement. Next, the dye absorbance was measured after the pH of the solution was adjusted until it was at the optimal pH for measurement.

Determination of Optimum Coagulation Dosage

The 1000 ppm dye mother solution was diluted using distilled water to obtain a 100 ppm dye solution. 1% chitosan coagulant solution was added to 1000 mL of dye solution to obtain various chitosan dosages. The dosage of chitosan added to each dye was adjusted to the results obtained from the previous step. Variations in chitosan dosages used for tartrazine coagulation were 25 ppm, 50 ppm, 100 ppm, 200 ppm, and 500 ppm. As for MY, the various dosages used were five ppm, 10 ppm, 25 ppm, 50 ppm and 100 ppm. The coagulation process was conducted using a jar test device, starting with fast stirring at 100 rpm for 1 minute, then slow stirring at 40 rpm for 20 minutes. The flocs formed were allowed to undergo sedimentation for 30 minutes. The solution was then taken ± 4 cm from the surface using a 50 mL pipette, and then the pH of the solution was adjusted according to the optimum pH for measurement using 0.01 M NaOH and 0.01 M HCl. The absorbance of the solution was measured using a UV-Vis spectrophotometer. The same procedure was repeated using 1% PAC and 1% Alum as coagulants.

Result and Discussion

Optimization of dye measurements

To obtain good accuracy, dye measurements must be carried out under optimal conditions. In this research, measurement optimization was conducted by determining the optimum wavelength and optimum pH. Measurements done at the optimum wavelength have higher sensitivity as this wavelength provides maximum absorbance (Pratiwi & Nandiyanto, 2022). The optimum pH needs to be determined

because the speciation and ionization level of the dye will be determined by the pH of the solution in dye solutions. Shifts in the pH of the solution will affect the dye's absorbance readings as Sakare et al (2022) reported. Therefore, dye concentration measurements with a UV-vis spectrophotometer must be done at a pH where the dye composition is dominated by species that absorb electromagnetic radiation at the measurement wavelength. The results of determining the optimum wavelength and pH measurement for MY are shown in Figure 1. Figure 2 displays the results of determining the optimum wavelength and pH measurements for tartrazine.

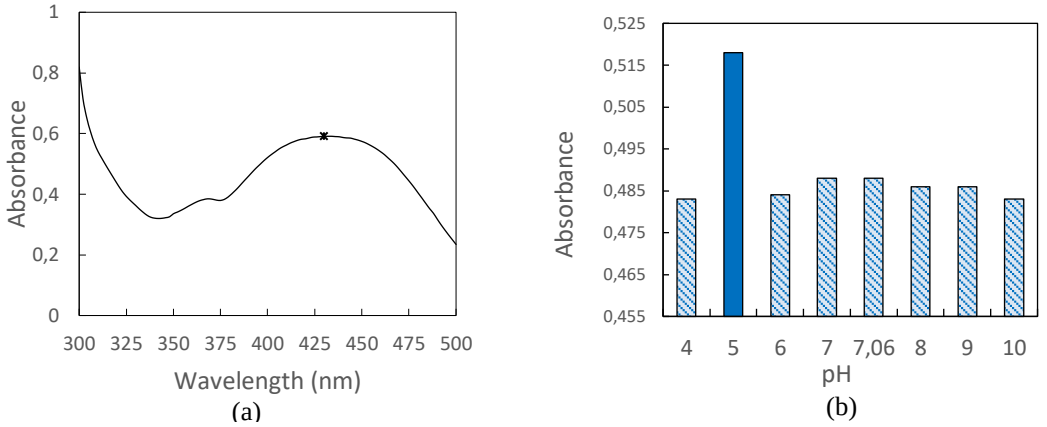

Figure 1. (a) optimum wavelength and (b) optimum pH for MY measurement (7,06 is the initial pH of MY stock solution)

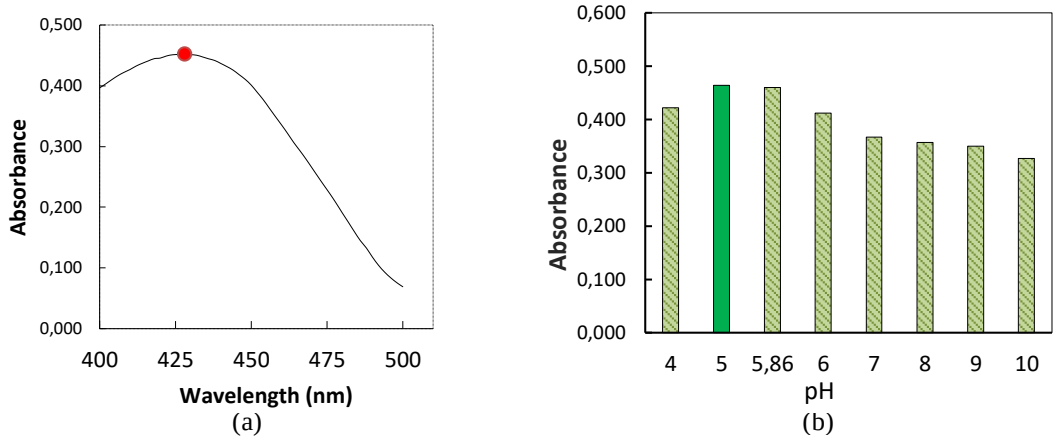

Figure 2. (a) optimum wavelength and (b) optimum pH for tartrazine measurement (5.86 is the initial pH of tartrazine stock solution)

Based on the measurement results, the optimum wavelength for MY is 430 nm, while the optimum wavelength for tartrazine is 428 nm. The optimum wavelength obtained is still in a range that is not much different from several other studies which also used MY and tartrazine in their studies (Arsenault-Escobar et al., 2023; Ashok et al., 2015; Kourani et al., 2020; Lim et al., 2020; Popadić et al., 2021). The optimum wavelength obtained is not exactly the same as several other research results, which can occur due to differences in equipment conditions and

measurement environments. In addition, it is possible that the pH of the solution used in this study was not exactly the same as that of the solution used in previous research, thereby slightly shifting the optimum wavelength. However, the wavelength obtained is still in the wavelength range for yellow compounds. For optimum pH, the measurement results show that both MY and tartrazine provide maximum absorbance at pH 5. The results of this measurement are used to determine the subsequent concentrations of MY and tartrazine.

Effect of Coagulant Dosage in Dye Removal

Determining the correct coagulant dosage needs to be done because the dosage is one of the factors that determine the effectiveness of coagulation. According to Abujazar et al. (2022), if the dosage of coagulant added is too small, it will be difficult for flocs to form. On the other hand, adding coagulant at too high a dosage will cause deflocculation, which reduces the effectiveness of coagulation (Maria et al., 2020; Soros et al., 2019). In this research, preliminary research has been carried out to estimate the effective dosage range for removing dye with an initial concentration of 100 ppm. The range of coagulant dosages used for MY coagulation is 5 ppm, 10 ppm, 25 ppm, 50 ppm, and 100 ppm. The reduction in MY concentration after coagulation for each dosage of coagulant used is shown in Figure 3.

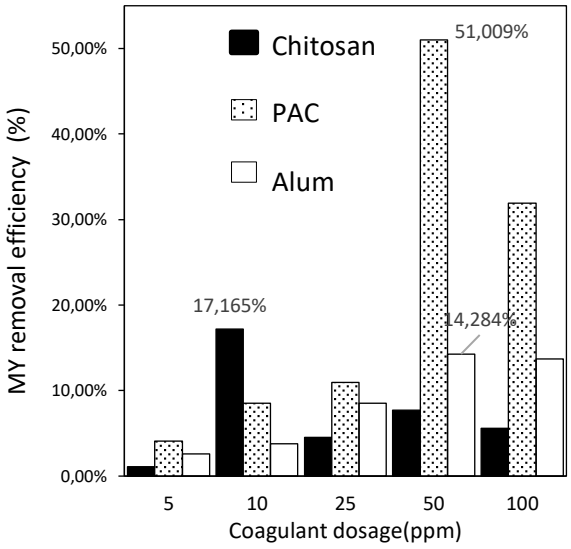

Figure 3. Effect of coagulant dosage on MY removal efficiency

Figure 3 shows that each coagulant provides the best concentration reduction at different optimum dosages, with different concentration reduction values. In this study, PAC was the coagulant with the highest reduction in MY levels compared to chitosan and alum. This is possibly because PAC does not rely only on the charge neutralization mechanism in the coagulation process in removing MY from the solution.

According to Mcyotto et al. (2021) sweep flocculation mechanisms and absorption bridges can also occur in coagulation using PAC. When dissolved in water, PAC will release several Al species, one of which is medium chain Al polymer with an open chain conformation (Lin et al., 2008; Yang et al., 2010). The Al polymer produced by PAC can coagulate MY by forming bridges between MY particles so that they can finally agglomerate to form large flocs. An illustration of the mechanism for forming bridges between MY particles and Al polymer from PAC coagulants is shown in Figure 4.

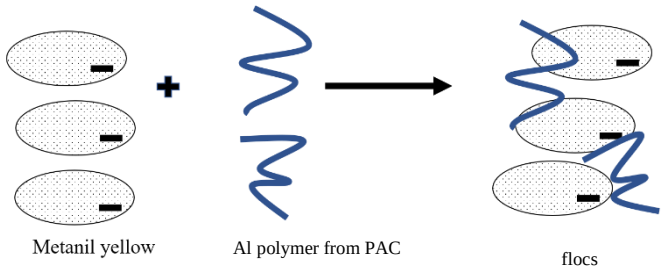

Figure 4. Illustration of the formation of metanil yellow floc through the bridge formation mechanism from PAC

At relatively high PAC concentrations, the presence of Al species released by PAC can also form floc nuclei so that in addition to the bridging mechanism between particles, coagulation-flocculation can take place using a sweeping flocculation mechanism, which further increases the effectiveness of MY removal from the solution. As shown in Figure 3, a reduction in MY levels also occurred when coagulation was carried out using chitosan. Chitosan can function as a coagulant because the chitosan polymer structure contains an active amine group (-NH_2), which in a protonated state becomes -NH_3^+. This protonated amine group can bind negatively charged particles so that these particles will be destabilized to form larger particle sizes or form flocs so they can settle. Several possible mechanisms in the coagulation process are the formation of bridges between particles and charge neutralization. However, determining which mechanism occurs is not easy because both mechanisms can co-occur (Hendrawati et al., 2016). In terms of dye coagulation by chitosan, the mechanisms that would likely dominate the process are charge neutralization and the formation of bridges between polymers (Szyguła et al., 2008; Wang et al., 2017).

Compared with PAC, chitosan's performance as a coagulant has not provided satisfactory results in reducing MY levels in solution. This may be related to the conformation of chitosan dissolved in acetic acid. When dissolved in a carboxylic acid solution, chitosan will tend to coil around each other, forming a closed chain (Dutta & Singh, 2008). This kind of conformation causes the number of MY

particles interacting with chitosan to be less because some of the active groups of chitosan are located inside the closed chain.

From Figure 3, it can be observed that chitosan coagulant shows better performance at a dosage of 10 ppm compared to PAC and alum coagulants at that dosage. This is because, at low dosages, the Al produced by alum and PAC primarily reacts with water molecules so it is not effective in interacting with MY. Coagulation by chitosan can occur because when chitosan is dissolved in acetic acid, the amine group (NH_2) in chitosan will bind H^+ so that the amine group will be protonated to become NH_3^+, which causes chitosan to become polycationic (Bhalkaran & Wilson, 2016). The presence of this protonated amine group can bind to the sulfonate group of the dye. Figure 5 displays the structure of the MY molecule, which shows the presence of a sulfonate group, which is probably to be the group that interacts with the coagulant.

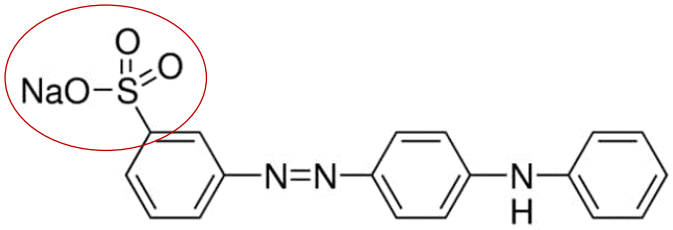

Figure 5. Chemical structure of Metanil *Yellow*. The Sulfonate group was circled in red.

Based on the data obtained, alum coagulant also reached the optimum dosage at 50 ppm. The percentage results of decreasing MY levels using alum at a dosage of 50 ppm were better than chitosan at the same dosage, this was because coagulation using chitosan at a dosage of 50 ppm had occurred deflocculation. These results are in accordance with several other studies showing that chitosan is susceptible to deflocculation if used excessively (Bhalkaran & Wilson, 2016; Renault et al., 2009).

The reduction percentage in MY levels using PAC at the same dosage is better than alum because the coagulation mechanism using alum only relies on the charge neutralization mechanism. In addition, the pH of the coagulated sample solution was in the neutral and slightly alkaline pH range, which was at a pH of 7.06. Under alkaline conditions, $Al_2(SO_4)_3$ undergoes rapid and strong hydrolysis after contact with water, and only a small portion of Al can react with organic materials to form complexes. Most Al salts exist in the form of negatively charged $Al(OH)_3$ and $Al(OH)_4^-$ ions, which provide a weak charge neutralization effect. On the other hand, polymerized Al species released by PAC tend to remain stable during the hydrolysis process even in the alkaline pH range (Lin et al., 2008).

In tartrazine coagulation, the various coagulant dosages used are 25 ppm, 50 ppm, 100 ppm, 200 ppm, and 500 ppm. The decrease in tartrazine concentration after coagulation for each dosage is shown in Figure 6.

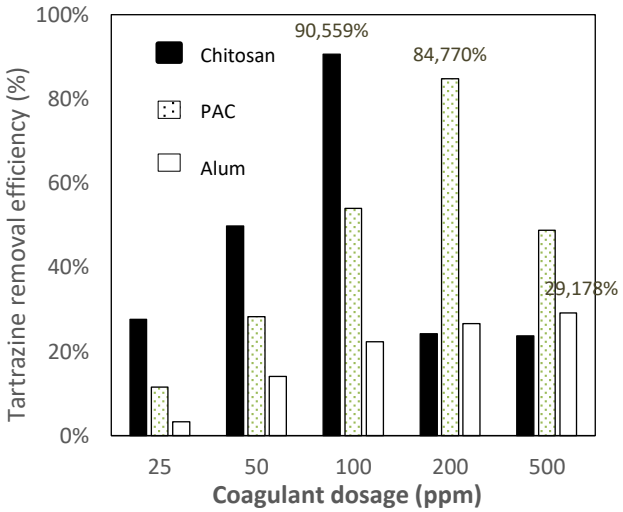

Figure 6. Effect of coagulant dosage on tartrazine removal efficiency

Based on Figure 6, it can be seen that the best reduction in tartrazine levels occurred in coagulation using chitosan compared to PAC and alum. Interestingly, the reduction in tartrazine concentration in solution by the three types of coagulants is better than the reduction in MY concentration. This may be related to the tartrazine structure, which has two sulfonate groups and one carboxylate group which can interact with coagulants. In line with what was reported by Mcyotto et al.(2021), the effectiveness of dye coagulation will be influenced by the structure of the dye and how it interacts with the coagulant. The molecular structure of tartrazine is shown in Figure 7, where the functional groups that can interact with the coagulant are marked with circles. From this structure, it is estimated that the interaction between tartrazine and the coagulant can take place through electrostatic interactions between the negatively charged SO_3^- and COO^- groups and the positive charges on the coagulant (protonated amines in chitosan and positively charged Al species in alum and PAC).

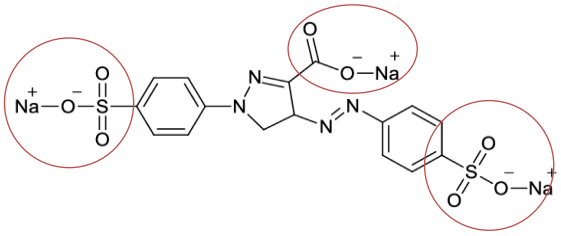

Figure 7. Chemical structure of tartrazine. Functional groups that are likely to interact with the coagulants are circled in red.

Apart from that, the presence of a lone pair of electrons on the oxygen atom in the sulfonate group also allows Lewis acid-base interactions to occur with the protonated amine group on chitosan, where oxygen can donate a lone pair of electrons to be used in forming coordinating covalent bonds. This likely causes the reduction in tartrazine levels in coagulation using chitosan to be more effective than PAC and alum. Similar to MY removal, PAC provides more satisfactory results compared to alum. It is suspected that this is still related to the speciation of Al produced by the two coagulants when dissolved. When dissolved, alum only releases monomeric Al species, namely Al^{3+}, $Al(OH)^{2+}$, $Al(OH)_2^{+}$, and $Al(OH)_4^{-}$. Meanwhile, in water, PAC releases not only Al monomer but also polymer cations, such as $Al_{13}O_4(OH)_{24}^{7+}$ (Lin et al., 2008). The Al species in the form of polymer in PAC makes particle destabilization more effective in forming flocs than monomeric Al produced by alum because it has a higher positive charge. Furthermore, because tartrazine has several negatively charged sites in one molecule, the number of Al monomers required to neutralize the negative charge of tartrazine is greater. As a result, the alum dosage required to obtain optimal reduction in tartrazine levels reached 500 ppm.

Effect of Sedimentation Time

One of the factors that is taken into consideration in the effectiveness and efficiency of the performance of a coagulant is sedimentation time. In coagulation, the formation of denser floc will encourage faster sedimentation, making the application more efficient (H. Cui et al., 2020; X. Cui et al., 2016). To observe the effect of sedimentation time, the coagulant dosage used in MY coagulation was the optimum dosage obtained in previous research, namely 10 ppm for chitosan and 50 ppm for PAC and alum. The observation results for MY coagulation are shown in Figure 8.

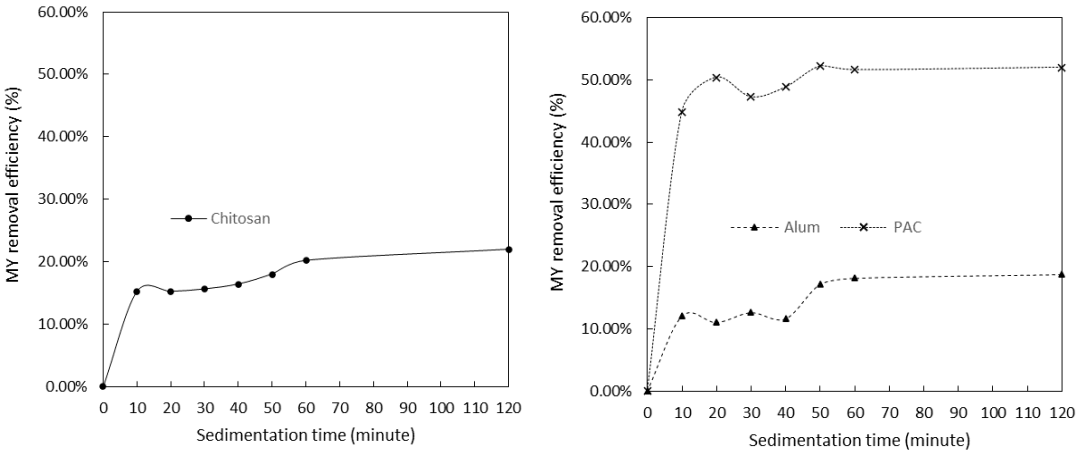

Figure 8. The effect of sedimentation time on MY removal by using: (a) chitosan; and (b) alum dan PAC

As for tartrazine coagulation, the coagulant dosage used to observe the effect of sedimentation time was also adjusted to the optimum dosage previously obtained, namely 100 ppm for chitosan, 200 ppm for PAC and 500 ppm for alum. The percentage of tartrazine reduction for each observed sedimentation time interval is shown in Figure 9.

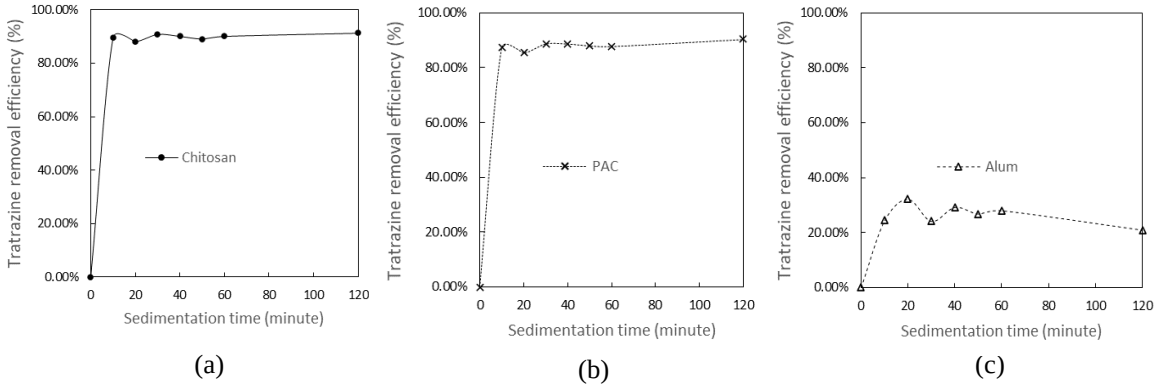

Figure 9. The effect of sedimentation time on MY removal by using: (a) Chitosan; (b) PAC; and (c) alum

Based on Figure 8 and Figure 9, it can be seen that for all coagulants, most of the flocs formed have been sedimented within the first 10 minutes of sedimentation time. For chitosan and PAC, after 10 minutes of sedimentation time, the amount of dye removed from the solution no longer increased significantly. Even after 60 minutes, the amount of dye removed was constant. This is quite different from coagulation using alum, where the amount of dye removed still changes significantly at each observation point. This may be related to the structure of each

coagulant. Chitosan and PAC are coagulants in the form of polymers, thus allowing the formation of flocs, which are larger in size and easier to settle. In contrast to alum, where coagulation by alum is dominated by a charge neutralization mechanism by Al monomers released by alum, the floc formed is smaller in size so it is more fragile and takes longer to settle.

Conclusion

Based on the results obtained in this research, chitosan has the potential to be used as a dye coagulant. Chitosan gave better results as a coagulant for tartrazine compared to when used to coagulate MY, which is probably related to the presence of active groups in the target compound that can interact with chitosan. For the two dyes observed, chitosan is a more effective and efficient coagulant than alum and a better coagulant than PAC for reducing tartrazine levels. Similar to PAC, chitosan requires a sedimentation time of 60 minutes to obtain optimum results in the coagulation of MY and tartrazine. The sedimentation time required by chitosan is better when compared to coagulation of the two types of dyes using alum.

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