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# Treatment of Textile Wastewater using Biodegradable Flocculants of Chitosan and Extracted Pandan Leaves

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**Graphical abstract**

**Abstract**

The study investigated the performance of chitosan and extracted pandan leaves towards treatment of textile wastewater by using flocculation process. Pandan leaves were extracted by using solvent extraction method. Flocculation process was conducted using a Jar test experiment. The effect of dosage, pH, and settling time on reduction of COD, turbidity and color of textile wastewater was studied. The results obtained found that chitosan was very effective for reduction of COD, turbidity, color and indicator for color. The best condition for COD and turbidity removal was achieved at 0.2 g dosage, pH 4 and 60 minutes of settling time. Under this condition, about 58 and 99% of COD and turbidity was removed, respectively. However, the results obtained using extracted pandan was opposite compared to the chitosan. Extracted pandan was not able to remove both COD and turbidity of the waste.

*Keywords*: Pandan leaves; chitosan; textile wastewater; flocculation

**Abstrak**

Kajian ini telah mengkaji keberkesanan kitosan dan ekstrak pandan terhadap rawatan air sisa tekstil dengan menggunakan proses flokulasi. Daun pandan telah diekstrak dengan menggunakan kaedah pengekstrakan pelarut. Prosess flokulasi telah dijalankan menggunakan ujian balang. Kesan dos, ph dan masa pemendakan terhadap penurunan COD, kekeruhan dan warna air sisa tekstil telah dijalankan. Keputusan yang diperolehi mendapati kitosan adalah sangat effektif bagi penurunan COD, kekeruhan, warna dan penyerapan-UV. Kondisi yang terbaik untuk penurunan COD dan kekeruhan dicapai pada dos, 0.2g, ph 4 dan masa pemendakan, 60 minit. Pada kondisi ini, lebih kurang 58 dan 99% COD dan kekeruhan dapat diturunkan, masing-masing. Walau bagaimanapun, keputusan yang diperolehi menunjukkan keputusan yang sebaliknya apabila ekstrak pandan digunakan. Ekstrak pandan tidak dapat untuk menurunkan COD dan kekeruhan air sisa tekstil.

*Kata kunci*: Ekstrak pandan; kitosan; air sisa tekstil; flokulasi

## ■1.0 INTRODUCTION

A lot of industries such as textile, paper, plastics, and dyestuffs consume extensive volume of water and at the same time use chemicals during manufacturing and dyes to color their products. Because of these activities, a considerable amount of polluted wastewater was generated (Mahmoodi, *et al*. 2009). The unused materials from the processes are discharged as wastewater that is high in color, biological oxygen demand (BOD), chemical oxygen demand (COD), pH, temperature, turbidity, and toxic chemicals. Thus, water becomes limited resource because of the industrialization and urbanization that caused the pollution in the water environment (Lu, *et al*. 2010). Furthermore, if the discharge water untreated, the toxic effluents from the industries become the major source of aquatic pollution and will cause considerable damage to the receiving waters (Ali, *et al*. 2009).

The main challenge with the textile wastewater is to eliminate the colour, which is due to the remaining dyes. Effluent from textile industries contains different types of dyes because of high molecular weight and complex structures, shows very low biodegradability (Hsu and Chiang, 1997; Pala and Tokat, 2002; Kim, *et al*. 2004; Gao, *et al*. 2007). In order to cope with these new restrictions and due to ineffectiveness of conventional biological treatment method in decolorization and degradation of textile wastewater (Vandevivere, *et al*. 1998), the research interest to find the other alternative which is simple and low-cost technologies for the on-site treatment of wastewaters. Therefore, coagulation and flocculation is a promising technique in order to reduce COD and color removal. Some researcher have reported that coagulation-flocculation is widely used for dyes removal (Zhuang, *et al*. 2006; Kim, *et al*. 2007; Golob, *et al*. 2005).

The objective of the study is to reduce COD and color removal by using biodegradable flocculants which is chitosan and extracted pandan leaves. Pandan leaves were extracted in order to obtain pyrrolline component that was used as biodegradable flocculant. Pyrolline consists of cyclic nitrogen (-N=) and ketone which is functional group that are responsible in metals biosorption (Birch and Bachofen, 1990; Le Cloirec, *et al.* 2003). Chitosan can be considered as one of the most promising coagulation flocculation materials since it is very effective for decolouring acidic and direct dyes (Sanghi, *et al*. 2005). Gandjidoust, *et al*. (1997) reported that chitosan resulted in the highest removal in colour as compared to synthetic polymers such as polyacrylamide (PAM), and polyethylimine (PEI) and chemical coagulant, such as alum. Similar conclusions were reported by Rodrigues, *et al*. (2008) and Wang, *et al*. (2007) for the treatment of paper pulp and paper mill wastewater. Guibal's group (2006) published a series of papers on the ability of chitosan to act as an effective coagulant to treat not only particulate suspensions but also dissolved substances. In particular, they showed that colour can be removed either by adsorption onto solid-state chitosan or by coagulation/flocculation using dissolved-state chitosan. Therefore, in this study, textiles wastewater will be treated by using chitosan and extracted pandan leaves as the biodegradable flocculants in order to analyze the efficiency of the flocculation process towards the textile wastewater.

## ■2.0 EXPERIMENTAL

### 2.1 Materials

The textiles wastewater was obtained from the American and Efird (Malaysia) Sdn. Bhd. which is situated in Kulai, Johor Bahru. The wastewater was obtained for the same batch of textiles wastewater so that the experiment was done on the same composition in the wastewater. The wastewater obtained was stored in the refrigerator to maintain the characteristic of wastewater sample under 0°C. Table 1 shows the raw characteristic of textile wastewater.

**Table 1** Initial characteristic of textile wastewater

| Parameter | Value |
|---|---|
| pH | 11.89 |
| COD | 1189 mg/L |
| Turbidity | 213 NTU |
| Color | Dark Yellow |

Chitosan was purchased from Sigma Aldrich in the form of white fine powder. The chitosan was purified by dissolving in acetic acid solution and was washed by anhydrous alcohol for 3 times and dried at 65°C in vacuum for 48 hours before used. 3 g of chitosan was dissolved in 1% acetic acid solution. Then, it was added with 1 M HCl until the pH reached 4. Ethanol was also purchased from Sigma Aldrich in the form of liquid. The pandan leaves were bought from a local market. The leaves were dried for 48 hours in the oven at 30°C prior to use. Then, the leaves were grounded into small pieces with the grinding mill.

### 2.2 Extracted Pandan Leaves

Approximately, 150 g of leaf mass were immersed for one day in ethanol ($C_2H_5OH$). The pandan leaves were filtered out from the solution. In order to obtain the pure extracted pandan leaf which is pyrrolline, the solution was evaporated by using rotary evaporator at 80°C to evaporate the ethanol in the extract solution.

### 2.3 Flocculation Process

The experiment was carried out by using a conventional jar test apparatus to flocculate the suspended solids in textile wastewater with chitosan and extracted pandan leaves. The apparatus was set with six beakers and the stirrers were set to be on the same speed. The research was conducted by manipulating the dosage of the flocculant (0.1-0.6 g), settling time (15-120 min), and pH (3-11) in order to study the effect of the reaction variables towards the efficiency of the flocculation process. The supernatant was analyzed by its UV, color removal, turbidity and COD. UV, color, turbidity and COD was measured by using UV-vis spectrophotometer with 550 nm wavelength, observation, turbidity meter (HACH model) and COD meter (HACH DRB200 model), respectively.

## ■3.0 RESULTS AND DISCUSSION

### ■3.1 Effect of Chitosan Dosage on Flocculation Process

In flocculation process, flocculant dosage plays an important role in determining the flocculation efficiency. Insufficient or overdosing dosage would give poor result in the performance of flocculation. Thus, it is crucial to determine the optimum dosage in order to minimize the cost and maximize the performance in the treatment. The experiment was conducted in order to determine the optimum dosage that affect to the performance of flocculation process. The experiments were done by varying the dosage of chitosan from 0.1 to 0.6 g. The sample of the wastewater used was 500 mL for every beaker at 3 minutes of mixing time with 200 rpm of mixing speed, then 15 minutes of mixing time with 40 rpm and 30 minutes of settling time. The pH of solution was fixed at pH 4. The sample of wastewater was adjusted from 11.95 to pH 4 due to the chitosan that only applicable and soluble in the acidic aqueous phases (Pan, *et al*. 1999). The pH was controlled either by adding strong acid (0.1 M) which is hydrochloric acid (HCl) or strong base (0.1 M) which sodium hydroxide (NaOH). Figure 1 shows the effects of flocculant dosage on COD and turbidity removal. The results indicated that percentage of COD removal increased substantially with the increased of the flocculant dosage. The highest COD removal was achieved when 0.2 g of chitosan was applied. At this condition, the COD removal and turbidity reduction were at 37.51% and 90.37% respectively. When the flocculant dose was above 0.2 g, the efficiency began to decrease. Clearly, the percentage turbidity and COD reduction increased from a dosage 0.1 g to 0.2 g, with a slightly declined above the point of 0.2 to 0.6 g. Thus, the optimum dosage chosen was 0.2 g.

This could be explained due to the charge density. If compared to the other flocculants such as polyaluminium chloride and polyacrylamide, chitosan has a high charge density (Ahmad, *et al.* 2006). Therefore, chitosan can be categorized as a flocculant that has a high charge density which means only require less amount of dosage to destabilize the particles. The extent addition of the chitosan dosage from 0.3 to 0.6 g resulted to decrement in percentage removal of COD and turbidity. This performance is due to the overdosing of chitosan that gave the phenomenon of excess polymer is adsorbed on the colloidal surfaces and producing restabilized colloids. Thus there were no sites available on the particle surfaces for the formation of interparticle bridges.

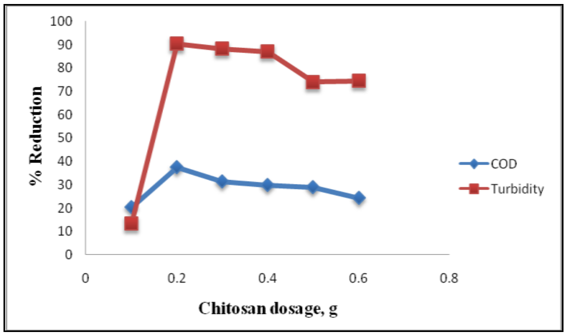

**Figure 1** Effects of chitosan dosage on percentage reduction of COD and turbidity

Figure 2 shows the effect of chitosan dosage on color removal. The removal color rate increased with the increasing in dosage and then became constant whereby almost complete color removal could be achieved. Chitosan was found to be effective to color removal at a low dosage which was at 0.2 g that gave the value less than 0.5. When the chitosan dosage increased from 0.1 to 0.2 g, it was found that the amount of color being removed also increased (as shown in Figure 2). By further increasing the dosage of the flocculant, the amount of color being removed was decreased. The higher the amount of the chitosan dosage is the more likely aggregation between colliding particles that causing destabilization of the particles. When the dosage reached the optimum amount in the suspension, it caused larger amount of dye particles to aggregate and settle. However, overdosing of the coagulant in the suspension would cause the aggregated particle to re-disperse and would also disturb particle settling (Gregory, 1999). This phenomenon also could be explained on the basis of an increase in the repulsive energy between chitosan and textile waste solution, which causes the hindrance in floc formation.

**Figure 2** Final treated wastewater sample with increasing the chitosan dosage. The operating condition was fixed at pH 4, rapid mixing 200 rpm for 3 minutes, slow mixing 40 rpm for 15 minutes and settling time 30 minutes

Figure 3 shows the result of flocculation process by varying the chitosan dosage in the range of 0.1 to 0.6 g. The effect of dosage was analyzed at pH 4, 18 minutes of mixing time and 30 minutes of settling time. The result of the load pollutants and color removal was analyzed as measured by the graph absorbance versus wavelength. It surpassed the color removal ability of the commercial flocculants. From the Jar test experiment, the curve of the graph shows that when the dosage increased, the absorbance peak decreased. This is due to the destabilization of particles was enhanced by the increasing in charged groups (in acidic solutions, there is an increase in the number of protonated amine groups on chitosan) followed by charge neutralization, resulting in a decrease in optimum dosage. Thus the optimum dosage was 0.2 g. However, the overdosing amount of the chitosan could increase the peak of the absorbance. This is because of the excess polymer was adsorbed on the colloidal surfaces and producing restabilized colloids. Therefore, there were no sites available on the particle surface for the formation of interparticle bridges. The restabilized colloidal particles can become more positively charged and cause the electrostatic repulsion among the suspended solids.

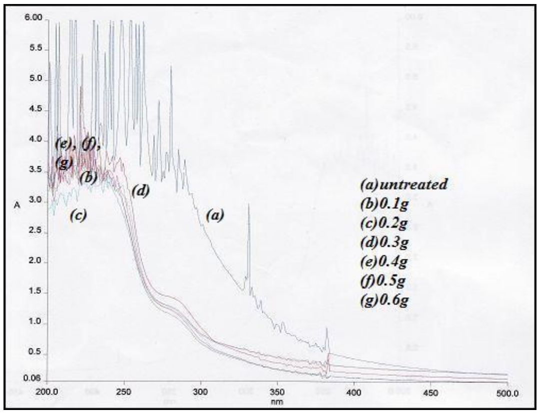

**Figure 3** Graph absorbance vs. wavelength for the effect of chitosan dosage. The operating condition was fixed at pH 4, rapid mixing 200 rpm for 3 minutes, slow mixing 40 rpm for 15 minutes and settling time 30 minutes

### 3.2 Effect of pH on Flocculation Process

pH is a critical parameter in the efficiency of the flocculation process. This parameter influences the solution properties (net charge of the dye) and the behavior of the biopolymer in the solution (charge of the amine groups). The addition of chitosan to the textile waste solution changes the pH of the solution for two reasons: (a) the direct impact of the acidity of the chitosan solution, and (b) the effect of the interaction of the dye with the biopolymer (Agata, *et al.* 2009).

The experiment was conducted to investigate the influence of wastewater initial pH on flocculation efficiency at optimum dosage 0.2g, with rapid mixing 200 rpm for 3 minutes, slow mixing at 40 rpm for 15 minutes and 30 minutes of settling time. Wastewater initial pH was adjusted in the range of 4 to 11 by using hydrochloric acid (HCl) and sodium hydroxide (NaOH). Figure 4 illustrates the effect of wastewater initial pH on flocculation efficiency. The results show that the initial pH largely influenced both the COD and turbidity reduction. By decreasing the initial pH from 11.89 to acidity solution, the maximum percentage turbidity removal increased noticeably until the pH value at 4 but when the pH was increased above this point distinctly reduced percentage of the turbidity and COD removal. Thus, the optimum pH for the turbidity and COD removal was at 4 which the percentage reductions were 87.11% and 58.28%, respectively.

The results obtained were consistent with the study done by Domard, *et al*. (1989). They pointed that there are 90% of the functional group of $HN_2$ on chitosan surface has been protonated at pH 4, and gradually reduced to about 50% as pH increased to 6. Therefore the positive charges on the chitosan surface will significantly decrease as solution pH increased.

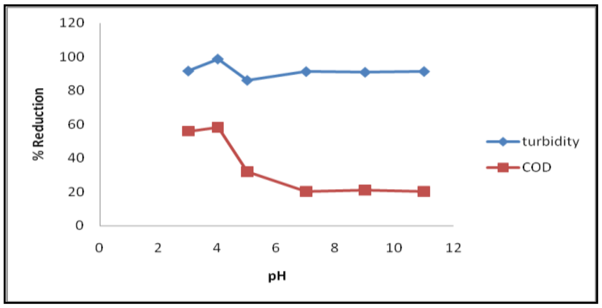

**Figure 4** Effects of pH on the percentage reduction of COD and turbidity by using chitosan as flocculant

Figure 5 shows the effect of pH on color removal. As shown in figure 5, the maximum color removal efficiencies for the textile waste water were at pH 4. Changing the initial pH of the solution seemed to significantly changing the performance of the system. At the lower values the amount of chitosan needed for an efficient coagulation-flocculation was lower than required at higher pH values.

This was explained by the acid base properties of chitosan and the degree of association of the polyelectrolyte (Guibal and Roussy, 2007). At the pH 4 of or less, more that 90% of the amine groups are protonated. The initial pH of 3.0 obviously led to more protonated form of chitosan. By decreasing the pH values, the dye protonation process could reduce the charge density and cause the dye molecules to self aggregate. Thus, less coagulant would be required for destabilize them. The short range of concentration for optimum dye removal increased with initial pH, which renders the process easier to control at a large scale.

Therefore, the interaction between dye molecules and chitosan is basically the combined effect of the charges on the dye molecules and the surface of the biopolymer (Singh Maurya, *et al.* 2006).

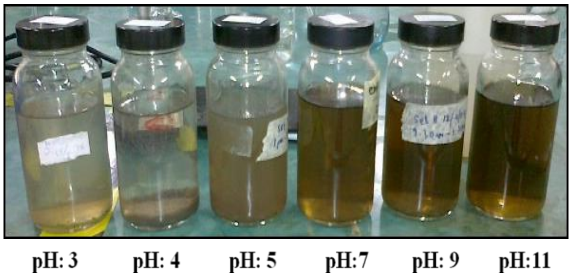

**Figure 5** Final treated of wastewater sample with the effect of pH value. The experiment condition was fixed at 0.2g of chitosan, rapid mixing 200 rpm for 3 minutes, slow mixing 40 rpm for 15 minutes and settling time 30 minutes

Figure 6 demonstrates the pH effect on the absorbance graph versus wavelength of the textile wastewater with a fixed amount of dosage 0.2 g of the chitosan. As the pH increased in the range of 3 to 4, the peak of the absorbance decreased, thus the maximum color and load pollutants removal was at pH 4. As can be seen the percentage reduction in absorbance at the pH 4 was the lowest compared to other pH level. However, the particle removal started to decrease slightly at pH 5, due to chitosan is insoluble and is not applicable in alkaline solution. The main mechanism for dye coagulation with chitosan appears to be charge neutralization at acidic pH Guibal, *et al*. (2007).

In the neutral solutions, the peak in the graph does not give good results and the peak was almost the same with the initial solution wastewater. Huang, *et al.* (2000) also indicated that the chitosan becomes more compact in more acidic solution and therefore lowers the viscosity of the solution. Therefore, in this study pH 4 was appeared to be the best pH for the coagulation process by using chitosan.

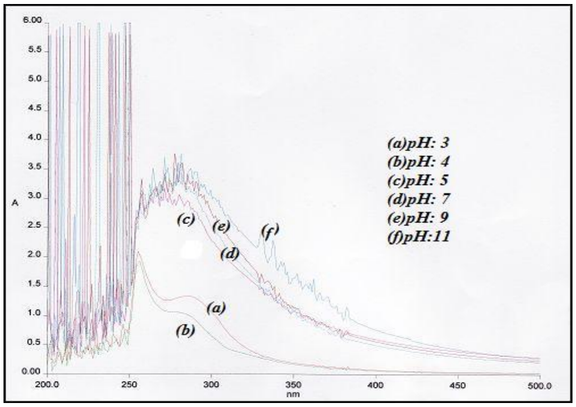

**Figure 6** Graph absorbance vs. wavelength for the effect of pH. The operating condition was fixed at 0.2g of chitosan dosage, rapid mixing 200 rpm for 3 minutes, slow mixing 40 rpm for 15 minutes and settling time 30 minutes

## 3.3 Effect of Settling Time on Flocculation Process

Besides the effect of chitosan dosage and pH, settling time is also the important parameter since this parameter influenced the design of the settling tank that can affect on the overall investment and operating cost. By increasing time for the sedimentation process, it will lead to increase of the flocs settled. On the other hand, if the settling time was too short, the flocs does not have enough time to settle down and to precipitate suspended solids in wastewater. Chitosan coagulants also produced larger flocs of better quality and faster settling velocity Pan, *et al.* (1999.) Thus, a study was conducted on the effect of the settling time in order to obtain high performance in coagulation-flocculation process.

The effect of the settling time was analyzed at the optimum dosage and pH which was 0.2 g and at pH 4 respectively, as well as with the speed mixing at 200 rpm within 3 minutes, slow mixing at 40 rpm in 15 minutes and varied the settling time which was in the range 15 minutes to 2 hours. Figure 7 shows the results on the influence of the settling time on the percentage reduction of COD and turbidity by using chitosan as a coagulant.

By analysing for each curve in figure 7 which are the percentage reduction of COD and turbidity, it proved that settling time had influenced on the coagulation process using chitosan. Moreover from the data obtained, the optimum settling time was taken for 60 minutes which gave the maximum value of the percentage reduction for both turbidity and COD which were 88.31% and 47.77% respectively (i.e. suspended solid was almost removed completely at this condition).

However, after the 60 minutes of settling time the percentage removal for COD and turbidity was decreased. The percentage reduction for turbidity decreased from 88.31% to 86.45% while for the COD the percentage reduction decreased from 47.77% to 42.47%. These results showed that the extent of the settling time does not give any good results to the sedimentation process.

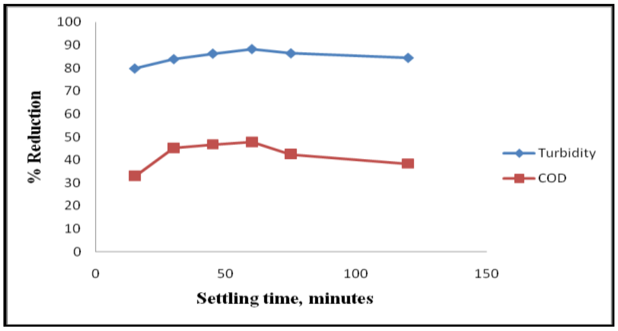

**Figure 7** Effects of settling time on the percentage reduction of COD and turbidity by using chitosan as a coagulant

Figure 8 shows the color removal at the different settling time (15 minutes–2 hours) of the coagulation flocculation process. The dosage of chitosan used was 0.2 g and the pH was controlled at 4. As the settling time increased, ,the color removal increased sharply during the first 60 minutes and after 60 minutes the color removal decreased. The observation showed that after the optimum settling time which was at 60 minutes the suspension turn to cloudy. Thus, the best settling time was achieved at 60 minutes. It shows that considerable sludge reduction was achieved within 60 min of settling. This indicates that chitosan is very effective in removing dye and produces a small amount of sludge that settles very fast. Large amounts of small flocs that produced were consistently observed and would take long time to settle down. Moreover, these small flocs gave the bad results and frequently interfered to the color measurement.

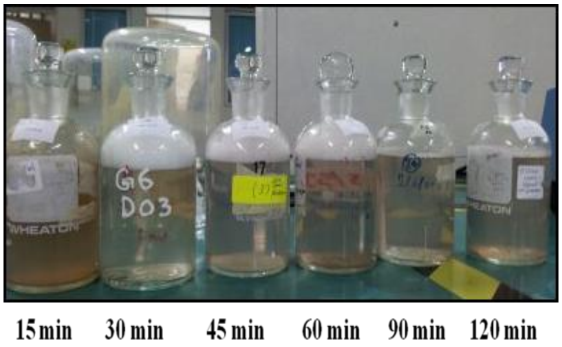

**Figure 8** Final treated wastewater sample with increasing the settling time, the operating condition at 0.2g chitosan dosage, pH 4, rapid mixing 200 rpm for 3 minutes, and slow mixing 40 rpm for 15 minutes

Figure 9 shows the effect on settling time on the graph absorbance versus wavelength. The jar test was carried out with the optimum dosage and pH which was 0.2 g and at pH 4 respectively, by varying the settling time from 15 minutes to 2 hours. As can be seen, the percentage of the color and pollutant load removal increased with the increasing the time of the sedimentation process. However, the extent of settling time above 60 minutes might cause the solution became cloudier and not gives the better result. Therefore the optimum settling time was chosen at 60 minutes. Reduction of absorbance in textile industry wastewater is of utmost importance as this prevents the wastewater from blocking sunlight and thus from interfering with the photosynthesis process of aquatic ecosystems (Lopez, *et al.* 2002).

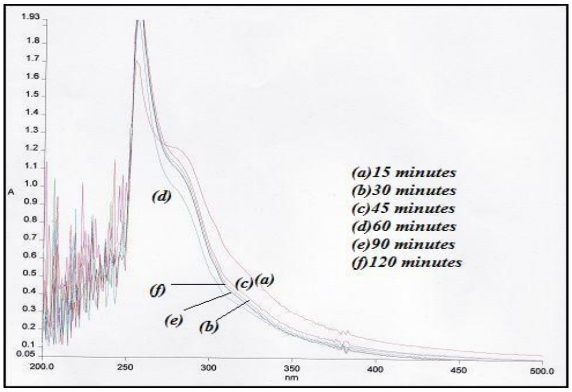

**Figure 9** Graph absorbance vs. wavelength for the effect of settling time, the operating condition at 0.2g chitosan dosage, pH 4, rapid mixing 200 rpm for 3 minutes, and slow mixing 40 rpm for 15 minutes

## 3.4 Effect of Dosage and Ph on the COD and Turbidity by using Extracted Pandan

The experiment was conducted in order to investigate the effect of extracted pandan to the textile wastewater solution by varying the parameter of the dosage and pH. The effect of dosage was analyzed at pH 4, with the rapid mixing for 3 minutes, slow mixing for 15 minutes and settling time 30 minutes. Based on the data obtained from the Jar Test experiment, the extracted pandan does not give any impact to the color and pollutant load removal.

In addition, when the amount of the extracted pandan increased, the amount of load pollutant and color also increased (as shown in Table 2). This result was proved by the COD and turbidity levels. The initial amount of the COD and turbidity was 1394mg/L and 6.95 NTU respectively. At dosage of 0.1 g, the value of the COD and turbidity was 1576 mg/L and 7.4 NTU respectively. By addition of the pyrroline dosage to the suspension, the amount of the COD and turbidity also increased. Thus, it showed that the extracted pandan coagulant does not give the good performance on the reduction of the COD and turbidity levels. Therefore, there was no optimum dosage at the pH 4.

Then, the experiment was conducted to investigate the optimum pH that would make the extracted pandan become more applicable and soluble at that pH of the solution. The pH was varied in the range of 3 to 11. The jar test experiment was conducted by adding 0.2 g extracted pandan in each beaker. The speed level for the slow and rapid mixing was constant. The result that presented in the Table 3 shows that when the pH was increased, the level of the turbidity and COD also increased. Means that, either in acidic solution, neutral or alkaline solution the data obtained does comply with the objective which is to reduce of the COD and turbidity level.

Therefore in this study, it can be concluded that the extracted pandan was not suitable to be used as a coagulant to treat textile wastewater. Based on the extracted pandan characteristic, it may be able to be used to remove the heavy metal waste. The pandan lignin contains many oxygen functionalities such as phenols and ketones which can serve as adsorption sites for binding heavy metals (Lalvani, *et al*. 2006). The 2-Acetyl-1-pyrroline might be recover the bad smell produced in the wastewater due to the component inside the extracted pandan that cointains volatile and long lasting aromatic constituents such as 2-Acetyl-1-pyrroline. 2-Acetyl-1-pyrroline is a substituted pyrroline and a cyclic imine as well as a ketone (Adams and De Kimpe, 2006). Pyrolline consists of cyclic nitrogen (-N=) and ketone are functional group that are responsible in metals biosorption (Birch and Bachofen, 1990; Le Cloirec *et al*. 2003).

**Table 2** Data for the study on the effect on extracted pandan dosage

| Dosage (g) | COD (mg/l) | Turbidity (NTU) | pH |
|---|---|---|---|
| 0.0 | 1394 | 6.95 | 4.00 |
| 0.1 | 1576 | 7.4 | 4.07 |
| 0.2 | 1825 | 11.5 | 4.03 |
| 0.3 | 1840 | 15.5 | 4.03 |
| 0.4 | 2478 | 17.5 | 4.08 |
| 0.5 | 2252 | 20.2 | 4.03 |
| 0.6 | 2059 | 23.6 | 4.04 |

**Table 3** Data for the study on the effect of pH

| pH | COD (mg/l) | Turbidity (NTU) | pH |
|---|---|---|---|
| 3 | 1839 | 41.7 | 3.00 |
| 4 | 1666 | 42.9 | 3.94 |
| 5 | 1704 | 39.2 | 4.97 |
| 7 | 1776 | 40.9 | 6.82 |
| 9 | 1949 | 41.8 | 8.34 |
| 11 | 1897 | 30.6 | 10.32 |

## ■4.0 CONCLUSION

This research had demonstrated coagulation flocculation process using chitosan and pyrolline as a coagulant in treatment of textile wastewater. The results showed that chitosan was an effective coagulant for color and load pollutants removal compared with 2-Acetyl-1-pyrroline. The data obtained from the experiment showed that only small amounts of chitosan were required for optimum removal of color, COD, and turbidity. The efficiency of coagulation-flocculation textile wastewater was shown to be highly dependent on the pH control and coagulant dose. At higher pH levels, larger amounts of chitosan were required to reach the maximum color and load pollutants removal. Chitosan can act in two ways; either as a coagulant for the neutralization charge or as a flocculant for bridging, these are depends on the nature of the colloids, the pH of the sample, and the experimental conditions. By decreasing, the pH value, it reduces the dose of chitosan needed for the optimum coagulation-flocculation. Very low of chitosan were needed for destabilization and sedimentation of the sample solutions. The settling time required also was short. To compare with the pyrroline coagulant, it did not have any effect on the color and load pollutants removal, infact the data obtained had showed that the value of treated sample was larger than untreated sample. It can be concluded that, the 2-Acetyl-1-pyrroline was not suitable to be used as a coagulant for the treatment of textile wastewater.

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