



***Dioscorea hispida* Starch as a Novel Natural Coagulant in Textile Wastewater Treatment**

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Highlights:

- Application of *Dioscorea hispida* extracted starch as a natural coagulant for textile wastewater treatment.
- Extraction of starch with novel modification of previous studies.
- Avoiding traditional chemical coagulants to reduce potential severe impact on the environment.
- Achieved 28%, 94% and 64% removal of COD, turbidity and color respectively at pH 7 and 2500mg/L dosage.

Abstract. The process of coagulation-flocculation using *Dioscorea hispida* starch as a natural coagulant was investigated for the purification of textile effluent from several dyeing and finishing mills. The major parameters tested were COD, turbidity, and color. Prior to conducting the experiments, the general physical characteristics of *Dioscorea hispida* starch were investigated. The optimal conditions, pH and coagulant dosage were assessed using a jar test. The results demonstrated that the *Dioscorea hispida* starch yield was 15.38% of *Dioscorea hispida* dry weight. Pulverizing of *Dioscorea hispida* mass also resulted in approximately 10-15% of impurities in the final product. The optimal pH of 7 resulted in maximum COD, turbidity and color reductions of 28%, 94% and 64% respectively. The optimum dosage of *Dioscorea hispida* starch of 2500 mg/L resulted in a maximum reduction of 22%, 93%, 63%, of COD, turbidity, and color, respectively. Overall, this study confirmed that the utilization of *Dioscorea hispida* starch as a natural coagulant offers a relatively good removal efficiency in textile wastewater treatment.

Keywords: *coagulation; Dioscorea hispida; flocculation; native starch; natural coagulant; natural polymer; textile wastewater.*

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1 Introduction

Population growth and increasing industrialization cause extreme contamination of the environment, which is a global problem. It is estimated that more than 100,000 commercially available textile colors are registered and presented in the industry, and about 700,000-1,000,000 tons of dyes are produced annually, while 280,000 tons are disposed into the global environment [1]. Coloring and finishing are two important processes in the fabrication of textiles industries, which use almost 73% of the total water supply in this sector (Table 1). These processes include the coloring of manufactured or natural fibers and their manufacturing into final commercial items [2]. The existence of dyes in wastewater, even at low concentrations, is not acceptable due to esthetic and dangerous impacts on the environment when they are disposed into water bodies. Textile wastewater is well-known to contain a high content of dyes, a significant amount of suspended solids (SS), high COD concentrations and a strongly fluctuating pH [3]. Textile wastewater treatment is a difficult process because of these properties.

Table 1 Water consumption in the textile industry [4].

Purpose	Percentage of Water Use	
	Cotton textile	Synthetic textile
Steam generation	5.3	8.2
Cooling water	6.4	9.5
Demineralization for specific purposes	7.8	30.6
Process water	72.3	28.3
Sanitary use	7.6	4.9
Miscellaneous and fire fighting	0.6	28.0

Textile wastewater treatment systems can essentially be divided into three main processes: the separation and concentration phases; the decomposition and degradation phases; and the exchange phase [5]. A proper mix of these methods is applied in wide-scale treatments to meet acceptable discharge according to treated-water standards. Generally, anaerobic biodegradation (as pretreatment process) is integrated into textile wastewater treatment with high-strength effluents. However, since aerobic processes require the energy of about 1 kWh/kg of degraded BOD, anaerobic processes produce about 0.5-1.5 kWh/kg of degraded BOD and yield a relatively small wasted sludge volume compared with aerobic processes [6]. In general, anaerobic reactor/digester systems are costly, but higher shock loads can be absorbed by these systems in addition to other advantages. In addition to the aforementioned treatment techniques, several works used several different treatment techniques extensively for the treatment of textile wastewater, such as electrochemical [7], adsorption [8], advanced oxidation processes [9], and coagulation [10].

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The coagulation-flocculation method was previously employed in wastewater purification because of the low cost and the high removal efficiency of dyes in wastewater [11]. The formation of flocs through destabilization of colloidal particles as well as charge neutralization of negatively charged colloids in the presence of natural or chemical coagulants followed by amalgamation of impurities is the principle of this process [12]. However, the characteristics of applied coagulants have a crucial influence on the coagulation performance, as they can enhance or obstruct the effectiveness of the treatment [13].

Many coagulants, organic and inorganic, have been recorded by different researchers [11]. Iron and aluminum salts are the most frequent examples of inorganic coagulant application in textile effluent treatment [14]. The main drawback of using inorganic coagulants is the generation of large quantities of toxic sludge, which adversely affects the effluent wastewater pH [15]. Aluminum, for example, is considered a neurotoxic compound that can cause Alzheimer's disease. Moreover, the existing methods of coagulation with chemical coagulants makes the disposal process complex as well as expensive [16] and causes groundwater contamination by metallic toxicity [17]. The application of organic coagulants derived from plants (fruits and vegetables), on the other hand, has received a great deal of attention from researchers for textile wastewater due to their nontoxic behavior, biodegradability, availability, wide variety, and low cost [18]. Several researchers have recently reported the utilization of plant-based natural coagulants for industrial wastewater treatment, for example *Moringa oleifera* [19], *Jatropha curcas* [20] and maize. All of these coagulants have proved their capability in removing different pollutants from wastewater.

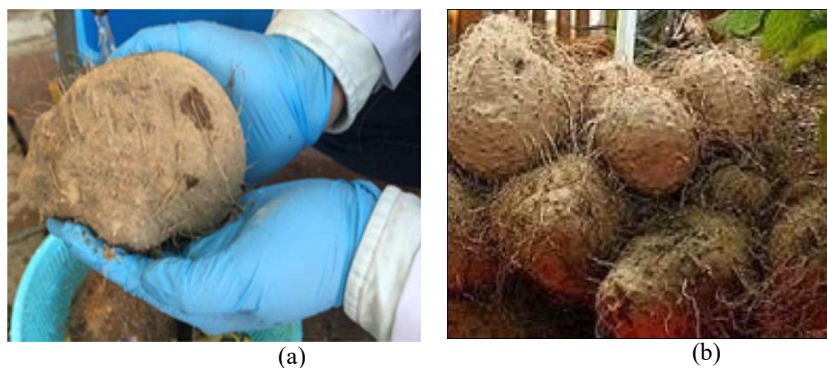


Figure 1 (a) Collected *Dioscorea hispida* tubers during study; (b) typically available tubers [21].

Dioscorea hispida Dennst (Figure 1), generally identified as gadung, is a twister related to the *Dioscorea* genus of the Dioscoreaceae family. This flora with a thorny stem twinning, may reach up to 20 m in height, displaying small pale-yellow flowers. Gadung tubers are rich in resistant starch despite being gluten free [22]. *Dioscorea* genus tubers are a good source of lipids, starch, proteins, crude fibers, vitamins (especially vitamin A and C), and have been utilized as an herbal medicinal plant as well as food [23]. Anti-nutritional ingredients such as tannins, total oxalate, free phenolics, hydrogen cyanide are usually available in *Dioscorea* tubers. *Dioscorea* tubers contain some initial materials for the synthesis of steroidal hormones, i.e. saponins and sapogenins such as diosgenin, which are used as components of anti-inflammatory, androgenic estrogenic, and contraceptive drugs [24]. *Dioscorea hispida* is found scattered in tropical, subtropical, and temperate regions around the world, especially portions of Central America, West Africa, as well as the Caribbean, and Southeast Asia [25]. This paper looks at the applicability of *Dioscorea hispida* as a natural starch-based coagulant for treating textile effluent. Specifically, the removal performance of COD, turbidity, and color was assessed. In this study, the effect of different pH and coagulant dosages was evaluated in terms of the optimum removal performance of COD, turbidity and color. The physical characteristics of *Dioscorea hispida* starch were examined with respect to color, yield and pH.

2 Materials and Methods

2.1 Textile Wastewater Collection and Sampling

The textile effluent samples in the current work were collected from the Craft Batik Teluk Bahang factory located at Pulau Pinang, Malaysia, in February 2019 using the grab method from the discharge point of the factory. The collected samples were kept in 10 L HDPE bottles with a sealed cap. The collected samples were then transported to a laboratory within 1 hour and stored in a cold room at 4 °C to avoid possible chemical and biological reactions prior to the experiments. The samples were acclimatized at room temperature for 2 to 3 hours before each of the experiments. The major parameters tested were pH, turbidity, COD, and color and analyzed according to the Standard Method of Water and Wastewater [26]. The pH was determined by a portable pH meter (Eutech Cyber Scan pH 510). The turbidity was determined using a turbidity meter (HACH 2100N). Measurement of COD and color was done using a colorimetric method (5220-D).

2.2 Preparation and Extraction of *Dioscorea hispida* Starch

Fresh *Dioscorea Hispida* was collected and used for the preparation of *Dioscorea hispida* starch. The *Dioscorea hispida* was washed carefully to remove dust and impurities. Next, the *Dioscorea hispida* skin was peeled away and the yam was

chopped to a smaller size. The total weight of *Dioscorea hispida* was approximately 8 kg. The physical properties of *Dioscorea hispida* starch, i.e. yield, impurities estimation, appearance, and pH, were investigated.

Dioscorea hispida extraction was conducted following the Modified Dos Extraction Method of Noor, *et al.* [27]. This method has been used previously by several researchers with various adjustments and modifications based on the purpose of the study [28,29]. 300 g of dried *Dioscorea hispida* was soaked in 400 mL aliquots of 0.5% (w/v) aqueous sodium bisulphite solution (NaHSO_3) for 60 seconds. The NaHSO_3 was added to disintegrate the protein/starch medium and inhibit microbial growth [30] and to prevent enzymatic browning [31]. *Dioscorea hispida* was then blended in a blender with distilled water added to form slurry.

The blended slurry was then kept in a fabric bag and pressed to obtain the filtrate, which was put on a plate. The filtrate was then sieved to 212 μm to ensure no contaminants remained and then left for 60 minutes to settle. The starch supernatant was carefully removed to avoid mixing with the starch once again by tilting the plate. The starch suspension was then carefully mixed with a 0.5% of 1 L NaHSO_3 aqueous solution and left for 30 minutes to settle. The supernatant was removed like previously mentioned. The starch obtained from the preceding process was then filtered using vacuum filtration with 1.6 μm optical glass fiber filter paper to clean the extracted starch. The cleaned starch sediment was cleaned with 50 mL of acetone aliquots to remove the water before being placed in an oven at a temperature of 45 °C overnight.

2.3 Coagulation-Flocculation Process by Jar Test

A conventional jar test apparatus (VELP Scientifica JLT 6) was used to run the coagulation-flocculation experiments. This apparatus has impellers fixed with 2.5 cm x 7.5 cm rectangular blades. The samples were taken out from the cold room and acclimatized at room temperature and placed into jar test beakers. The apparatus has six beakers, where each beaker has a volume of 500 mL of wastewater. The pH was adjusted from 5 to 10 by adding required volumes of 3M NaOH and 3M H_2SO_4 solutions.

The jar test was divided into three subsequent stages: first a rapid mixing phase, then a slow mixing phase, and finally a settling phase. The stages were adapted from Asrafuzzaman, *et al.* with a few modifications [32]. During the first stage, 120 rpm was assigned for the rapid mixing stage for six sequence beakers with different coagulant dosages varied between 500 mg/L to 3000 mg/L, while the impellers were kept at rapid speed mixing. After five minutes of rapid mixing, the speed was changed to 20 rpm for 30 minutes. The stirrer was stopped for the settling stage, where the textile wastewater samples were left to settle for 1 hour.

Afterwards, the samples were collected using a pipette 10 cm below the water level for analytical measurements. All experiments were performed in triplicate.

The initial and final concentrations of the textile wastewater samples were measured in order to calculate the removal efficiency of the studied parameters (COD, turbidity and color). The removal efficiency was determined using Eq. (1):

$$\text{Removal (\%)} = \frac{C_i - C_f}{C_i} \times 100\% \quad (1)$$

where C_i and C_f are the initial and final textile wastewater concentration, respectively.

3 Results and Discussion

3.1 Textile Wastewater Characteristics

Table 2 summarizes detailed features of the textile wastewater samples. The raw textile wastewater had a dark purple color (645 Pt-Co) with an average pH of 10.98. The high pH value indicates that the textile wastewater was alkaline. Having high alkalinity is intolerable as it is an indication that the textile wastewater has the capacity to neutralize acids [33]. On the other hand, the color is often caused by forms of organic matter content that cause color pollution, inducing synthetic chemical dyes and natural dissolved organics such as lignin and tannins [34]. The color and pH exceeded the acceptable conditions for the discharge of industrial wastewater effluent (MQA, 2009). The COD value was 460 mg/L, which is also unacceptable for discharge without treatment (the acceptable range for COD is 80 to 250 mg/L).

Table 2 Characteristics of raw textile wastewater.

Parameter	Value	Standard*
pH	10.98	5.5-9
COD (mg/L)	460	400
Turbidity (NTU)	21.37	-
Color (Pt-CO)	645	200
Appearance (color)	Dark purple	-

Table 3 Comparison of initial and treated values of textile wastewater parameters.

Parameter	Initial Value	Value After Treatment	Standard*
COD (mg/L)	460	330	400
Turbidity (NTU)	21.37	4.48	-
Color (Pt-CO)	645	232	200

3.2 Physical Properties of *Dioscorea Hispida* Starch

The *Dioscorea Hispida* starch yield was 15.38% of its dry weight. Pulverizing of *Dioscorea hispida* mass also resulted in approximately 10-15% of impurities in the final product. This considerable amount of impurities consisted of fibers and tiny roots that grow inside *Dioscorea hispida*. The physical properties of the *Dioscorea Hispida* starch are tabulated in Table 4. The appearance of the final product of *Dioscorea hispida* starch was white wheat powder that turned yellowish after oven drying. This was due to the temperature of the oven, which was not higher than 45 °C. NaHSO₃ had also been added during starch extraction to prevent enzymatic deterioration prior to starch application and to inhibit microbial growth [30].

Table 4 Physical properties of *Dioscorea hispida* starch.

Parameter	Value
Yield (% initial material)	15.38
Impurities estimation (%)	10-15
Appearance	Wheat white powder
pH (0.1% aqueous solution)	6.78 ± 0.42

The starch yield obtained from *Dioscorea hispida* starch was in the range of 15.36% to 15.4% with an average of 15.38% of the dry mass. Lopez-Diago [35] states that the yield of starch from *Manihot utilissima* (bitter cassava) acquired using a wet method was in the range between 17.2 g and 39.4 g of starch obtained from the original 250 g of wet of *Manihot utilissima* samples, and the yield was in the range between 6.88% and 15.76% with an average of 10.64% of the dry mass. The low crushing speed and the shorter crushing time seemed to be responsible for reducing the starch yield. However, in the current study, the crushing was done manually with no limitation of crushing time. This explains the average of the yield obtained from *Dioscorea Hispida*, which was more by 4.74% of the *Manihot utilissima* starch yield.

3.3 Outcomes from Jar Test

Dioscorea hispida starch was applied as the primary coagulant in the present study. Several studies have been conducted to examine the effect of pH and coagulant dosage on the removal efficiency of turbidity, color and COD. The coagulant dose was set between 500 mg/L and 3000 mg/L, while the selected pH was 5 to 10.

3.3.1 Effect of pH

Figure 2 shows the influence of pH on the removal efficiency of COD, turbidity and color at pH 5. The maximum reduction of COD, turbidity, and color was

47%, 79%, and 63%, respectively. Overall, a proportional relationship was found between the removal efficiency and the coagulant dosage.

The concentrations of COD, turbidity, and color were still higher than the acceptable range after treatment (Table 3). This can be explained by the low pH, where the coagulant surface and the particles of the suspension are positively charged. Thus, the bridging mechanism has a low occurrence. The same charge for the coagulant surface and the particles of the suspension will result in a repelling force between both [36]. The value of turbidity before treatment was 21.37 NTU. After treatment this value was reduced to 4.48 NTU.

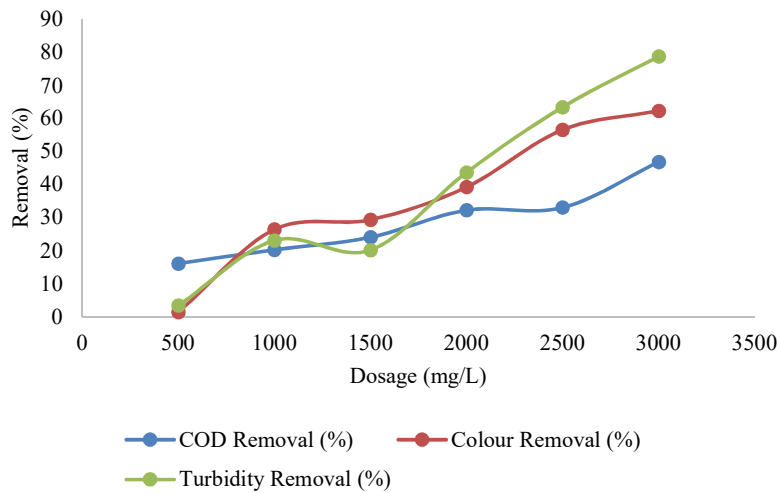


Figure 2 COD, turbidity and color removal performance at pH 5.

At pH 7, reduction of COD, turbidity, and the color was observed at 28%, 94%, and 64%, respectively. As shown in Figure 3, the COD reduction by a coagulant dose between 500 and 2000 mg/L was significantly increased at a slow rate. However, the removal efficiency increased substantially when the coagulant dose was increased to over 2000 mg/L and reached the maximum removal of 28% using 2500 mg/L of coagulant dose.

The removal efficiency of COD, turbidity, and color significantly changed at pH 6. The removal efficiency of COD, turbidity, and color obtained was 32%, 92%, and 67%, respectively, as shown in Figure 4.

Due to increasing floc formation through the bridging mechanism, a portion of dissolved and suspended matters becomes insoluble and settled subsequently,

which eventually accelerates the COD removal [37]. Addition of coagulants enables the destabilization of colloid particles due to having respective functional groups, which form bridges with molecules. Although they have a lower zeta potential value in treated leachate, they still indicate coagulation through the electrostatic patching method [38]. A higher efficiency of removal was observed for turbidity at pH 5 compared to pH 6. The removal efficiency of turbidity was 85% at 500 mg/L of coagulant dosage and increased to 94% at 3000 mg/L of coagulant dosage.

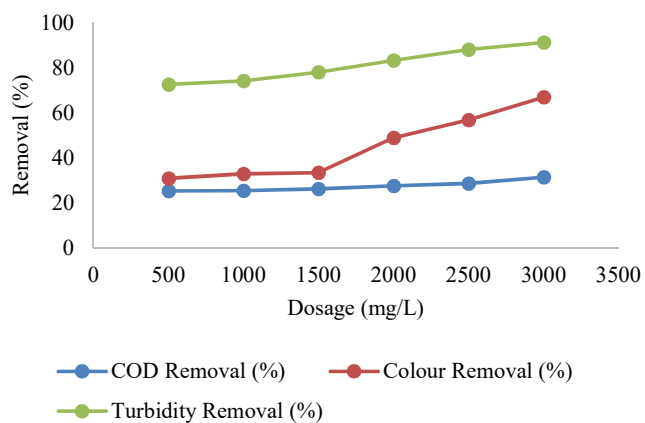


Figure 3 COD, turbidity and color removal performance at pH 6.

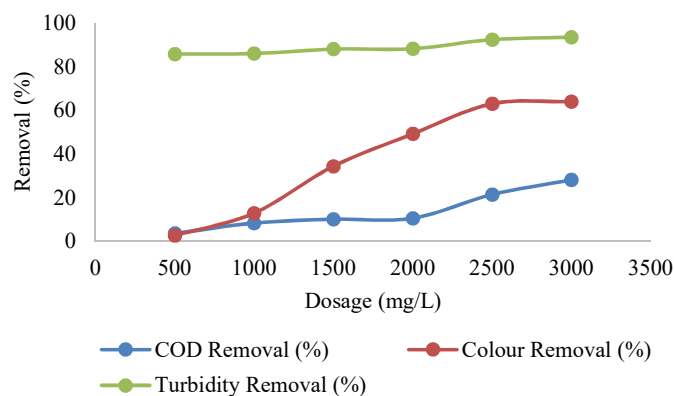


Figure 4 COD, turbidity and color removal performance at pH 7.

When the pH reached 8 (Figure 5), the removal efficiency of COD, turbidity, and color was 27%, 83%, and 62% respectively. This observation can be explained by the excessive addition of sodium hydroxide (NaOH) used for pH adjustment. A high concentration of NaOH can denature some proteins; thus, a reduction in the coagulation activity occurs [39]. Usually, the suspensions need to be destabilized in order to increase the accumulation, resulting in the formation of larger aggregates or flocs to settle. However, the removal efficiency will decrease when the suspensions destabilizes again due to the excess of natural coagulant [40].

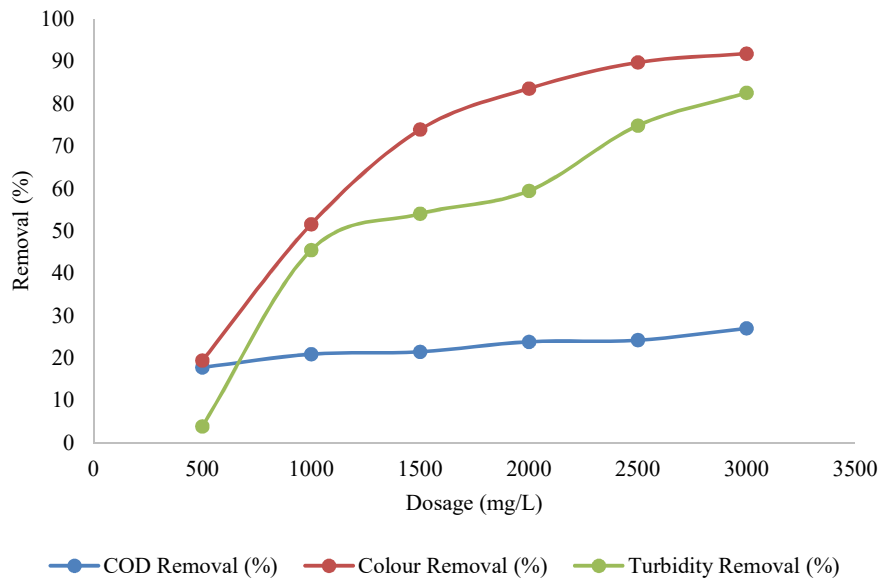


Figure 5 COD, turbidity and color removal performance at pH 8.

As the pH reached 10 (Figure 6), a significant decrease in the removal efficiency of color and turbidity was observed, while the removal efficiency of COD increased. The maximum COD, turbidity, and color reduction at pH 10 was 38%, 56%, and 25%, respectively. The greater alkalinity can explain the increase in COD removal; thus, the degree of potentization of adsorbent decreases.

Variation of pH against removal efficiency is shown in Figure 7. Increased turbidity and color removal was achieved at an optimum pH of 7. An earlier study by Saritha, *et al.* observed that the optimum alum dose to remove turbidity was obtained at pH 7 [41], which is consistent with the present study.

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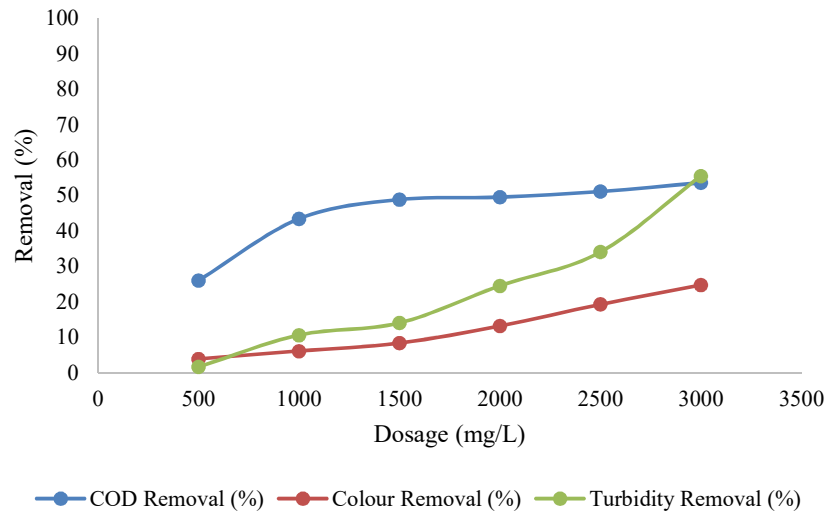


Figure 6 COD, turbidity and color removal performance at pH 10.

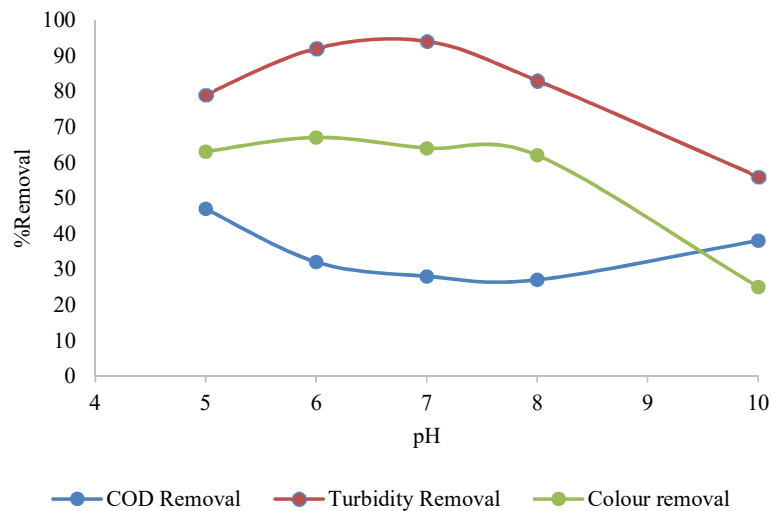


Figure 7 Influence of different pH on COD, turbidity and color reduction.

3.3.2 Effect of Coagulant Dosage

The optimum pH of 7 was used in a set of experiments using different coagulant dosages, as mentioned above. Figure 8 shows COD, turbidity and color reduction using a range of coagulant dosages between 500 and 3000 mg/L at a pH of 7. A

proportional relationship was observed between the coagulant dosage and the removal efficiency for all parameters.

The optimum coagulant dosage was 2500 mg/L, which resulted in 22%, 93% and 63% removal of COD, turbidity and color, respectively. Based on the overall result, textile wastewater requires a higher amount of *Dioscorea hispida* starch to obtain high removal efficiency. This result indicates that the coagulant activities of *Dioscorea hispida* starch are reduced when there are undesirable particles present in textile wastewater. However, an insufficient or an excessive dosage can reduce the performance of the flocculation process. The optimum dosage should therefore be carefully determined to minimize the dosage costs and the formation of sludge and to achieve optimum treatment performance.

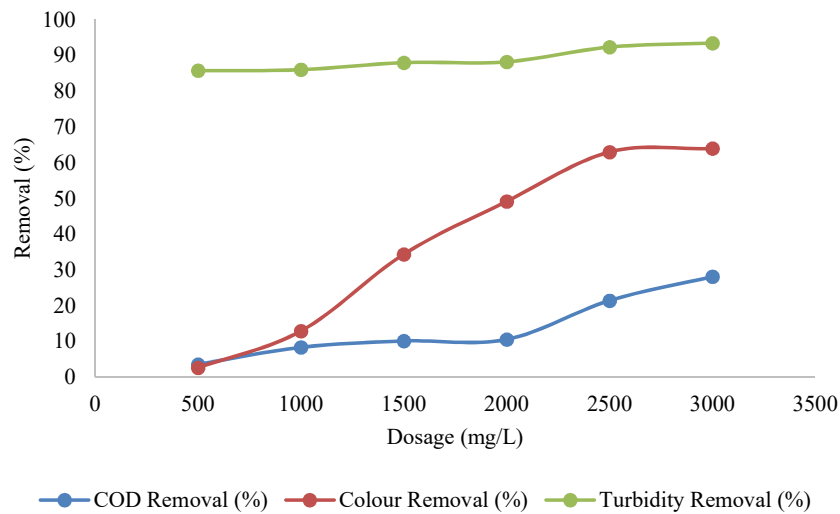


Figure 8 Influence of coagulant dosage on COD, color and turbidity removal efficiency.

Textile wastewater includes a high amount of negative charge particles (due to its high alkalinity), which needs a high volume of coagulant for complete stabilization. Yusoff, et al. used oil palm trunk starch and crosslink oil palm trunk starch as coagulant aids with polyaluminum chloride coagulant in treating landfill leachate [38]. They reported that pH 7 and 500 mg/L dosage were the best conditions for oil palm trunk starch. The optimum conditions for crosslink oil palm trunk starch were pH 8.3 and 1000 mg/L crosslink oil palm starch dose. The outcomes showed 38%, 25% and 45% removal of turbidity, color and COD respectively for oil palm trunk starch (OPTS) application, while crosslinked

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OPTS showed 43%, 29% and 57% removal efficiency of turbidity, color and COD. However, *Dioscorea hispida* performed better in terms of turbidity (94%) and color (64%) removal, compared to OPTS and C-OPTS while, OPTS showed better removal of COD. Thus, Aziz, *et al.* reported that the use of *Dimocarpus longan* seeds as a natural coagulant aid was effective in treating landfill leachate [42]. The maximum removal efficiency of COD, suspended solids and color was 69.19%, 99.50%, and 98.80%, respectively, at pH 4 and the optimum dosage of 2000 mg/L *Dimocarpus longan* seeds. Although *Dimocarpus longan* displayed better performance than *Dioscorea hispida*, *Dimocarpus longan* requires an acidic environment while *Dioscorea hispida* shows optimum removal in neutral condition (pH 7). Overall, it can be summarized that the optimum conditions for *Dioscorea hispida* starch were pH 7 and 2500 mg/L dosage respectively, which gave maximum removal efficiency of COD, turbidity, and color.

4 Conclusion

This study investigated the treatment of textile wastewater using *Dioscorea Hispida* as a novel natural coagulant to remove selective pollutants from textile wastewater in order to comply with the effluent standards for COD, color, and turbidity. The results showed that the *Dioscorea Hispida* starch yield was 15.38% of *Dioscorea hispida* dry weight. Pulverizing of *Dioscorea hispida* mass also resulted in approximately 10 to 15% of impurities in the final product. This considerable amount of impurities consisted of fibers and tiny roots that grow inside *Dioscorea hispida*. The jar test results indicated that at optimum pH 7 this natural coagulant ultimately had better coagulation efficiency and culminated in COD, turbidity and color removal efficiencies of 28%, 94%, and 64%, respectively. Table 3 shows a comparison of the concentrations of the initial and the treated wastewater with respect to the standard. Destabilization of leachate particles by *Dioscorea hispida* occurs mainly through a bridging flocculation mechanism, because the high molecular weight is the key principle of pollutant removal. The optimal dosage of *Dioscorea hispida* starch was 2500 mg/L, resulting in the maximum removal efficiency of 22%, 93%, 63% for COD, turbidity and color, respectively. Overall, this study confirmed that the utilization of *Dioscorea hispida* starch as a natural coagulant offers a relatively good removal efficiency of textile wastewater treatment.

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