



## Removal of Crystal violet dye in textile effluent by coagulation using algal alginate from brown algae *Sargassum* sp.

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### ABSTRACT

The enhancement in efficient wastewater treatment processes is one of the greatest significant problems mainly in textile dyeing units. Textile dyeing wastewater holds huge quantities of chemically diverse manmade dyes, surfactants, and other mineral substances, which are accountable for a severe toxicity on municipal wastewater treatment facilities. In this study, the colored wastewater contains mainly a Crystal violet dye from a local dyeing unit was treated by batch coagulation test using algal alginate as a coagulant extracted from *Sargassum* sp. marine brown algae. The raw algal alginate and dye loaded alginate was characterized by Fourier transform infrared spectroscopy and scanning electron microscopy techniques. The effect of various parameters like alginate dose, calcium dose, and settling time was investigated. It was noted that the maximum color removal of 97.3% at optimum conditions of 50 mg/L of alginate dose, 6 g/L of calcium dose was achieved. The sludge volume index (SVI) was examined at the maximum dye removal condition and compared with alum. The preliminary kinetic study on dye removal process shows that, it follows a second-order kinetic equation. The present study reveals that the algal alginate, a biopolymeric compound extracted from brown algae *Sargassum* sp. can be an efficient coagulant for Crystal violet dye removal in the wastewater from the textile dyeing unit.

*Keywords:* Textile effluent; Crystal violet dye; Coagulation

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### 1. Introduction

Increasing urbanization, industrialization, and exhaustive agricultural farming caused a substantial release of wastewater contains organic and inorganic chemical substances to water streams, which are not easily removed by the traditional water treatment. Conventional treatment methods like coagulation, flocculation, sedimentation, rapid sand filtration, and chlorination have a slight effect on some chemical contaminants which leads to the undesired characteristics of wastewater [1]. The textile and clothing sector is considered as the engine of growth for many developing countries. The

textile industry share in developing countries total manufacturing value-added has been between 15% and 3% and in employment between 20% and 40%. The contribution of other industries such as electronics, telecommunications, steel, light engineering goods, leather goods, processed foods, etc., have also gained prominence [2]. However, the role of the textile industry occupies an important place in economic growth all over the world. Among all continents, South Asia has been traditionally a major cotton-growing region. Most of the countries in South Asia have well-established cotton mills and have been exporting raw cotton and cotton products to

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the international market. It constitutes about 7% of the total value of world exports of natural textile fibers [3]. All the conventional textile processing units need a dyeing unit which mainly discharges a large quantity of colored wastewater. The chemical nature of a dye is determined by the chemical and physical properties of the fibers of the textile to be colored. The four main types of fibers are protein, cellulosic, regenerated (based on cellulose or derivatives), and synthetic. The term regenerated is used when a natural polymer is reacted chemically to form an alternative polymer. For example, the fibers are classified as natural and artificial fibers, the natural cellulose fibers extracted from plants was reacted with acetic anhydride, forms a polymer cellulose acetate called rayon [4]. The conventional textile processing unit consumes a large quantity of water and the wastewater generated was holding adverse characteristics. Mainly, among the various stages in textile processing, the dyeing stage consumes a large quantity of water [5]. The textile processing sequence ends with dyeing and printing which consumes a variety of large quantities of organic and inorganic dyes which are mentioned in Table 1. There are different kinds of classifications for dyes. Generally, these dyes are categorized based on the dyeing process and its applications. They can be classified in terms of their chemical structure, color, and application techniques. However, due to the complications involved in the chemical structures of dyes, classification based on the application is frequently discussed in literatures [6,7]. It has been observed that each class of dye has very distinctive chemistry, structure, and a particular way of bonding. When some dyes can react chemically with the substrates and some others can be held by physical forces.

Conventionally, all the dyes used in the textile dyes release toxic substances when reacting with the substrates, which in turn the toxicity adhere in the effluents of the textile industry. These adverse characteristics of the effluent need intensive treatment to make it usable. Among all the

wastewater treatment methods coagulation is the most commonly used one because of its simplicity and ease.

The complex phenomenon in the coagulation process is to choose the exact coagulant. Chemical coagulants can be categorized into three groups, they are hydrolyzing metallic salts, pre-hydrolyzing metallic salts, and synthetic cationic polymers [8]. Commonly used inorganic coagulants are aluminum and iron salts such as  $AlCl_3$ ,  $Al_2(SO_4)_3$ ,  $FeCl_3$ , and  $Fe_2(SO_4)_3$  due to their low cost, ease of use, handling, storage, and mixing properties [9]. When added to water, aqueous aluminum and ferrous salts dissociate to their respective trivalent ions, that is,  $Al^{3+}$  and  $Fe^{3+}$  then they are hydrolyzed and produce several soluble complexes retaining highly positive charges, thus adsorbing onto the surfaces of negative colloids [10]. Even though the wide application of these metal salts, their use has been examined because of the iron salts creating brownish color on the surface of equipment, and environmental impacts produced by high concentrations of leftover aluminum in treated wastewater [11].

The intrinsic difficulty in this process is its discharging of bulk magnitudes of chemical sludge that is categorized as the most hazardous waste that must be disposed of in safe landfills. The process also increases the total dissolved salt content in the treated wastewater, increasing desalination costs by a substantial margin. On the other hand, coagulant aids are inorganic materials that, when used alongside the main coagulant improve or accelerate the process of coagulation and flocculation by producing quick forming, dense, and rapidly settling flocs [12,13]. The efficiency of Coagulation process and its operational costs depend on several factors, including the coagulant type and dosage, mixing conditions, pH, temperature, ionic strength, as well as the nature and concentration of the organic matter, the total dissolved solids, the size, and distribution of the colloidal particles in suspension among others [14,15]. To overcome these drawbacks, the

Table 1  
Categories of dye based on dyeing process and applications [4]

Type	Chemical property	Applications
Acid	Water soluble— anionic	Dyeing od silk, wool, nylon, etc.
Basic	Water soluble— Cationic	Coloring of acrylic fiber and paper
Direct	Dyeing is carried with addition of sodium salts in mild alkaline bath	Used on cotton, paper, leather, wool, silk, and nylon. Also used for pH indicators and biological staining
Mordant	Requires a mordant	Used for the navy or black shades in wool
Vat	Insoluble; reduction in alkaline environment produces a water-soluble metal salt which binds to the textile fiber; subsequent oxidation reforms the original insoluble dye	Highly effective for the color of indigo
Reactive	Use a chromophore to directly react with fiber substrate	Dyeing cotton and other cellulosic fibers at home or in the art studio in normal temperature and pH
Disperse	Water insoluble non-ionic dyes used for hydrophobic fibers	Dyeing cellulose acetate fibers, polyester, nylon, and acrylic fibers
Azo	Insoluble dye is prepared in situ in the fiber by treating with both diazoic and coupling components	Useful for dyeing cotton; however, it is obsolete due to toxic nature of diazo compounds
Sulfur	Treating the fabric in a solution of an organic compound and sulfide compound	Inexpensive; used for dark shades in dyeing cotton

use of natural plant-based coagulants gained more attention and recent researches show that the natural coagulants are promising outputs compared to conventional coagulants.

Alginates are polysaccharides mostly found in the cellular wall matrix of brown seaweeds (Phaeophyceae) and composed of  $\beta$ -(1 $\rightarrow$ 4)-D-mannuronic (M) and  $\alpha$ -L-guluronic acid (G) blocks in heterogeneous proportions. The polysaccharide is located in the cell wall matrix in the form of insoluble calcium or magnesium salt, which makes the tissue elastic and strong. Apart from brown seaweed, alginates are also present in Gram-negative bacteria; *Azotobacter vinelandii* and some species of *Pseudomonas* produce alginate as extracellular polysaccharides [16].

Alginate is a natural polyelectrolyte which is highly selective to alkaline metal ions (Ba > Sr > Ca > Mg) and it binds  $\text{Ca}^{2+}$  ion that leads to gel formation. The binding capacity occurs due to the structural characteristics of two  $\alpha$ -L-guluronic acid blocks (GG). This phenomenon is called “egg-box” model [17]. These properties will be very useful for effective coagulation and thereby the present study proves the capability of alginate as a potential coagulant for the color removal process.

The main objective of the present study is to test the alginate extracted from the marine brown algae *Sargassum* sp. as a coagulant for the treatment of textile dye present in the real-time wastewater collected from the textile industry.

## 2. Materials and methods

### 2.1. Sample collection

The colored textile wastewater handled in this study was collected from the local textile dyeing unit situated near Kanchipuram, Tamilnadu, India. The present study focused on the color removing the ability of alginate as a coagulant, the textile wastewater collected from dye bath was used as a target pollutant in this study. The characteristic of textile wastewater is shown in Table 2. The textile effluent from the dye bath primarily contains Crystal violet dye, which is the most commonly used cationic azo dye having a molecular weight of 408 g/mol [18]. The molecular structure of the Crystal violet dye is given in Fig. 1 and the physicochemical properties are given in Table 3 [19]. Crystal violet dye is used in the textile dyeing industry is more hazardous mutagenic and mitotic poisoning nature [20].

### 2.2. Alginate extraction

The marine brown algae (*Sargassum* sp.) were collected from the coastal region of Mandapam at Bay of Bengal (Gulf of Mannar), Tamil Nadu, India. The algae samples

Table 2  
Characterization of textile wastewater

Parameter	Crystal violet
Concentration of dye (approx.)	1,000 ppm
pH	9.4
Absorbance	590 nm
Temperature	30°C

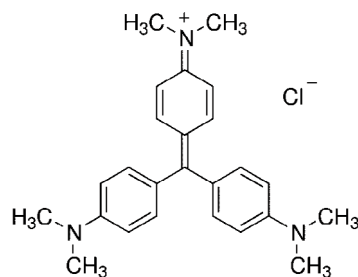


Fig. 1. Molecular structure of Crystal violet.

Table 3  
Physicochemical properties of the dye

Parameter	Crystal violet
Molecular weight	407.99 g/mol
Molecular formula	$\text{C}_{25}\text{H}_{30}\text{ClN}_3$
Absorption maxima	590 nm
Color	Dark green powder with a metallic lusture
Color index number	42,555
Dye class	Triphenyl methane
Solubility	1% in water

were collected by cutting the thallus with a scalpel near the rhizoid. The collected algae samples were washed with plenty of seawater in the coastal area and stored in jute bags with proper ventilation before bringing it to the laboratory for further processing. The sample was washed amply with tap water in the laboratory and dried out for 30 h at 65°C. Alginate extraction from marine brown algae was carried out based on the practice described by Fenoradosoa [21]. Exactly 25 g of dried algae were drenched in 800 mL of 2% formaldehyde for 1 d at room temperature, washed with water after which soaked with 800 mL of 0.2M HCl for 1 d. Later, the samples were washed again with distilled water and the Alginates were extracted with 2% sodium carbonate at 100°C. The soluble fraction from the extract was collected by filtration and polysaccharides were precipitated by 95% of ethanol (v/3v). Sodium alginate precipitated was splashed by 100 mL of acetone, dried at 65°C, and dissolved in 100 mL of distilled water. It was then precipitated again with ethanol (v/3v) and dried at 65°C [22].

### 2.3. Characterization of alginate

Fourier transform infrared (FTIR) spectroscopy and scanning electron microscopy (SEM) techniques were used to illustrate the characterization of alginate extracted from the marine brown algae. The FTIR spectroscopy (Thermo Nicolet, AVATAR 330) was engaged to determine the functional groups that exist in the extracted alginate. FTIR analysis was carried out both for raw and dye loaded samples. The infrared spectrum of algal alginate was documented as KBr discs in the range of 4,000–400 $\text{cm}^{-1}$ . SEM (Anna University, TESCAN - VEGA 3, Czech republic) and (VIT University, ZEISS, US) was used to characterize the surface

structure and morphology of the raw and dye loaded samples of alginate.

#### 2.4. Experimental procedure

The batch coagulation test was conducted in a standard jar test apparatus using alginate as a coagulant for removal of dye from the textile wastewater collected from the dye bath solution. The experiments were carried out in 1 L glass beakers containing 500 ml of real-time textile wastewater collected with an initial dye concentration of 1,000 mg/L, approximately. Calcium as calcium chloride and alginate as extracted algal sodium alginate was added for the experiment. The dosage of calcium varied between 1 and 6 g/L, whereas the dosage of alginate varied between 10 and 60 mg/L. Calcium was added firstly and then sodium alginate was added next for all the experiments. The experiment for each sample was carried out in the following sequence: 5 min of flash mixing at 100 rpm for calcium dosing, 5 min of rapid mixing at 100 rpm for alginate dosing, followed by 20 min slow mixing at 40 rpm and 30 min for settling. The clear supernatant after sedimentation was filtered using Whatman no. 42 filter paper and the filtrate was analyzed using ultraviolet-spectrophotometer. The percentage of dye removal was estimated by using Eq. (1).

$$\text{Dye removal (\%)} = \frac{C_i - C_f}{C_i} \quad (1)$$

where  $C_i$  and  $C_f$  are the initial and final dye concentration, respectively.

The sludge volume index (SVI) in mL/g can be measured from Eq. (2).

$$\text{SVI} = \frac{\text{Settled Sludge Volume, mL/L} \times 1000 \text{ mg/g}}{\text{Suspended solids, mg/L}} \quad (2)$$

The preliminary kinetic study on this coagulation process and the effect of settling time for the effective dye removal were conducted for the optimized parameters in the dye removal process. The experiments were carried out in triplicates and averages of the results are noted.

### 3. Results and discussion

#### 3.1. SEM analysis

The SEM analysis revealed the morphological structure of the alginate surface and the images are given in Fig. 2. The surface of alginate before the coagulation process (Fig. 2a) retains a flaky spine structure and fine perforations on it [23]. After the coagulation process (Figs. 2b and c), it was witnessed that the sludge noticeably shows the lodging of dye particles on the porous surface of alginate. Additionally, it was noted that when comparing the surface of alginate before and after the coagulation test, the samples after the coagulation test becomes smooth and no flakes were noted. The morphological change in the surface of the alginate discloses the removal of dye from aqueous solution through coagulation.

#### 3.2. Fourier transform infrared spectroscopy

The FTIR spectrum of raw alginate from brown algae is presented in Fig. 3a. A broadband at 3,147 and 3,628  $\text{cm}^{-1}$  was assigned to the hydrogen-bonded O–H group [24]. A sharp and strong absorption band at 1,612  $\text{cm}^{-1}$  indicating the C=C stretch. The band at 1,460  $\text{cm}^{-1}$  specifies the methylene C–H stretch [25]. A major band in the region 1,708 and 1,612  $\text{cm}^{-1}$  indicate the existence of a C=O group (carbonyl group) and this endorses the nature of alginate [26]. Fig. 3b represents the FTIR spectrum of sludge containing alginate and Crystal violet dye, the most specific peaks for benzene rings are peak at 1,591  $\text{cm}^{-1}$  corresponding to the C=C stretching of the benzene ring. The broadband of spectrum shows a

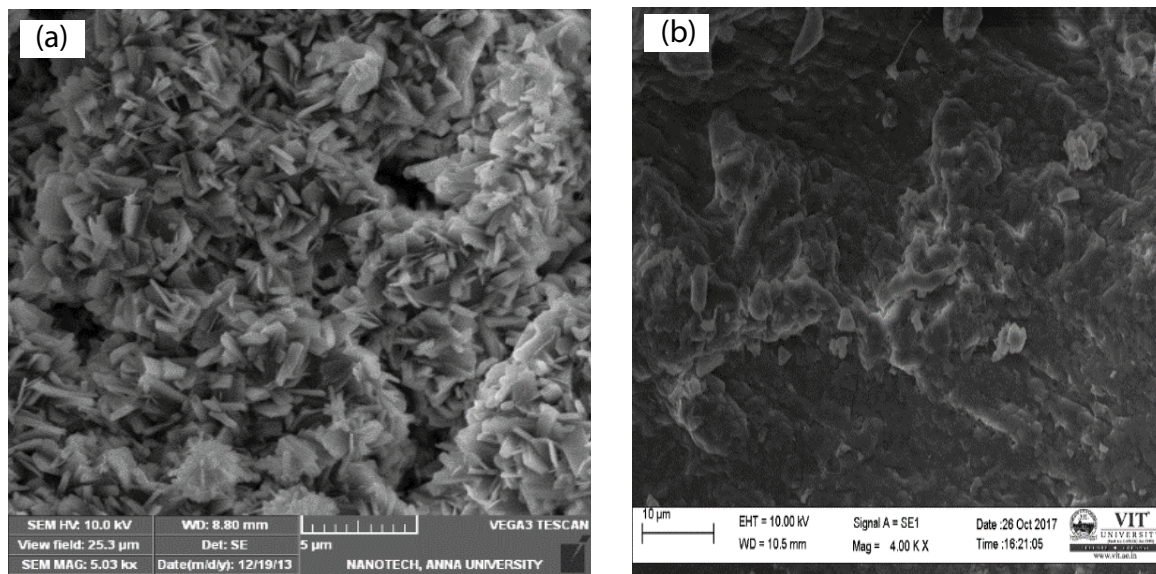


Fig. 2. SEM image of alginate (a) raw and (b) after coagulation with Crystal violet dye.

peak at  $1,708\text{ cm}^{-1}$  which corresponds to the C–N stretching pulsations and peak at  $3,302.13\text{ cm}^{-1}$  for C–H stretching represents the asymmetric  $\text{CH}_3$  group. The asymmetric stretch witnessed in the region between  $2,000$  and  $2,300\text{ cm}^{-1}$  conforming to symmetric and asymmetric stretching of tertiary amine salt. The peak for the C–N stretch of aromatic tertiary amine present in the Crystal violet was observed between  $1,024.20$  and  $1,411.89\text{ cm}^{-1}$  [27].

### 3.3. Effect of calcium and alginate dose

The effect of calcium and alginate dose on color removal of a real-time textile wastewater contains Crystal violet dye was studied and the results are presented in Fig. 4. For real-time textile wastewater containing Crystal violet dye, the maximum color removal of 97.3% was attained at 6 g/L of calcium dose and 50 mg/L of alginate dose. During the color removal process, the role of calcium is very vital, the free calcium ions boosted the gel formation with the alginate, thereby free settling occurs and the effective coagulation was achieved. At lower doses of calcium, the gel formation is not sufficient to remove the dye dispersed in the textile effluent. The optimum condition for the maximum color removal is presented in Table 4. The effective dye removal rate was compared with the literature and found to be satisfactory [28,29].

### 3.4. Sludge volume index

SVI is generally used to witness the settling characteristics of activated sludge and other biological suspensions. In the present work, the SVI can be used to compare the ability of alginate as a coagulant with the conventionally used commercial coagulant (alum). The maximum dye removal (97.3%) was achieved at 6 g/L of calcium dose, 50 mg/L alginate dose; the SVI calculated at this condition was 2.06 mL/g.

For the same color removal percentage (97.3%) the alum dose was found to be 17.2 g/L and the SVI calculated at this condition was 21.43 mL/g. From these experimental results, it was evident that the quantity of sludge produce by alum was much significant compared to alginate.

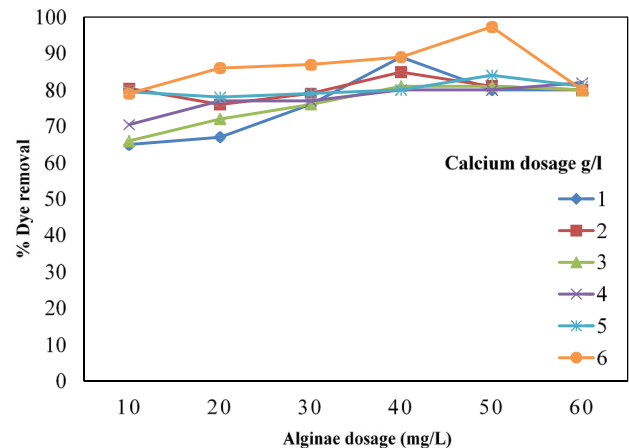


Fig. 4. Effect of calcium and alginate dose on percentage removal of Crystal violet dye.

### 3.5. Coagulation kinetics

A preliminary study on coagulation kinetics for the removal of Crystal violet dye in the textile wastewater using alginate as a coagulant is shown in Figs. 5a and b. The rate of removal of dye in the coagulation process is proportional to the initial dye concentration and formation of calcium alginate complex. The first-order and second-order rate equation was verified with the experimental data [30]. The solution for the first-order equation is given in Eq. (3).

$$\log\left(\frac{C_i}{C_0}\right) = -kt \quad (3)$$

where  $C_i$  is initial dye concentration,  $C_0$  is the concentration of dye after times minutes and  $k$  is the first-order rate constant ( $\text{min}^{-1}$ ).

The solution for second-order equation is given in Eq. (4).

$$\frac{1}{C_0} - \frac{1}{C_i} = k't \quad (4)$$

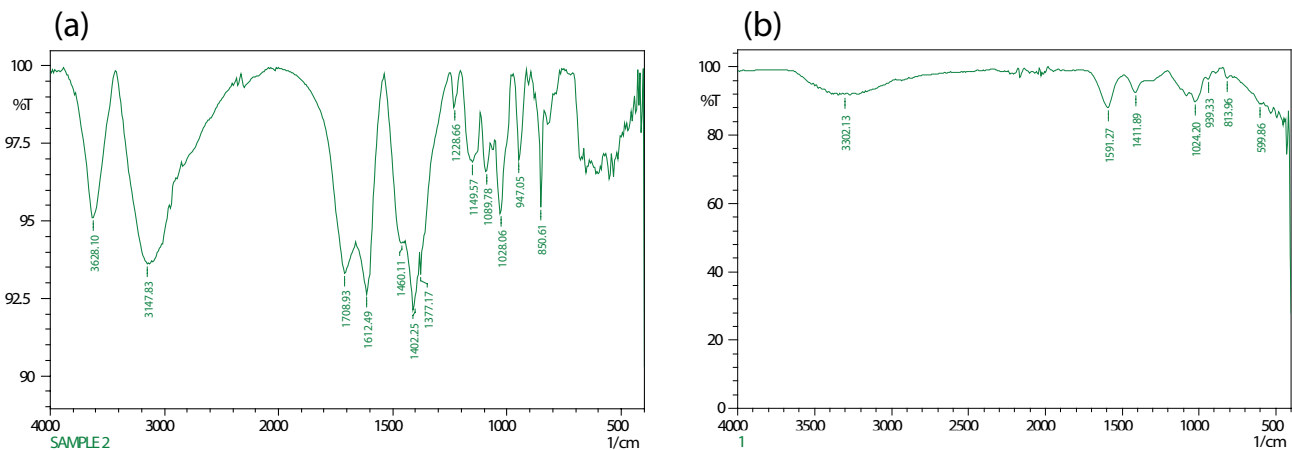


Fig. 3. FTIR spectra of (a) raw alginate and (b) Crystal violet dye loaded alginate after coagulation.

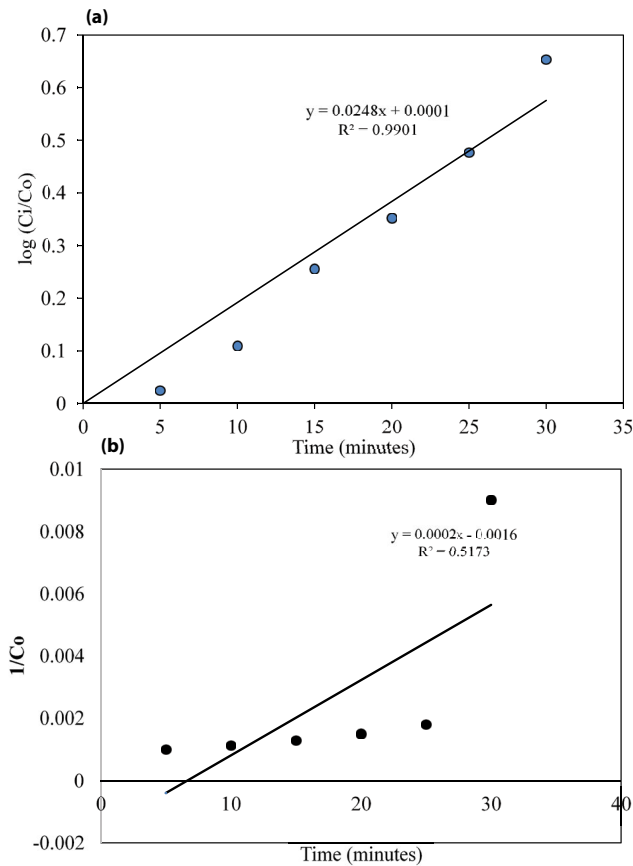


Fig. 5. Coagulation kinetics plot for removal of Crystal violet dye (a) first-order and (b) second-order rate equation.

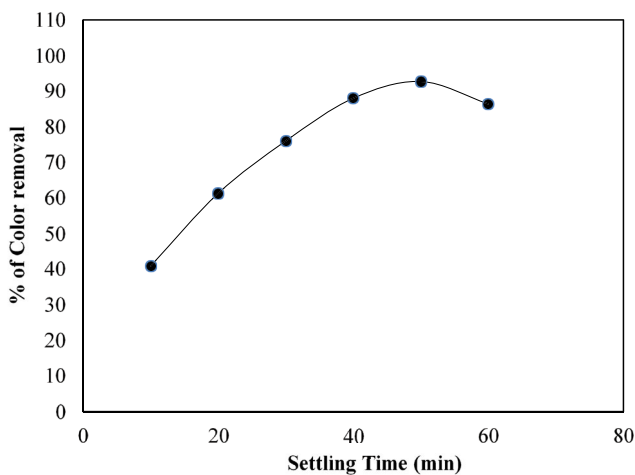


Fig.6. Effect of settling time on percentage dye removal.

where  $k'$  is second-order rate constant (L/mg min).

The first- and second-order kinetics plot to the experimental data with various initial dye concentrations is shown in Figs. 5a and b. From these figures, the first-order equation showed a greater value of intercept which obviously exposes that the coagulation process did not follow first-order

Table 4  
Optimum condition for color removal

Real time textile wastewater containing	Initial dye conc. $C_i$ (mg/L)	Alginate dose (mg/L)	Calcium dose (g/L)	% Dye removal
Crystal violet dye	1,000	50	6	97.3

kinetics. Hence, the coagulation process for color removal of Crystal violet dye solution using alginate as coagulant followed second-order kinetics.

### 3.6. Effect of settling time

The settling time is one of the significant operating parameters during the coagulation process. For the optimum condition for Crystal violet dye removal in real-time textile wastewater given in Table 4, the experiments were conducted up to 60 min. Fig. 6 indicates the effect of settling time on percentage color removal. It can be witnessed from the figure that there is a reliable increase in color removal with an increase in settling time. From Fig. 5 the optimum settling time and maximum color removal were found to be 50 min and 97%.

## 4. Conclusions

The experimental study on the removal of the Crystal violet dye in real-time textile wastewater by algal alginate as a coagulant extracted from marine brown algae (*Sargassum* sp.) was carried out. The maximum color removal of 97.3% was achieved at 50 mg/L of alginate dose, 6 g/L of calcium dose, and 50 min settling time for the pH of 9.4. The free calcium ions involved in the color removal process were boosted and the gel formation with the alginate was enhanced and at lower doses of calcium, the gel formation was not appreciable for the removal of dye. It can be observed that there is a consistent increase in color removal with an increase in settling time. Based on these results, it is evidently recognized that the coagulation ability of the algal alginate for the removal of Crystal violet dye in textile effluent was resourceful.

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