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Removal of Reactive Red 4 Dye from Aqueous Solution Using Chitosan Adsorbent

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Abstract: Dyes are chemical compounds that can connect themselves to surface or fabrics to impart color. Dye effluents released from numerous dye-utilizing are harmful toward the aqueous environment and living things. Reactive dyes are widely used in textile industry and have caused harm to human health such as respiratory sensitization, itching and skin irritation. A range of treatments has been studied for the removal of dye in wastewater and adsorption is the most used technique since it is effective and convenient. Batch adsorption experiments were conducted using Reactive Red 4 (RR4) dye solution and chitosan adsorbent under varying contact time, initial dye concentration, adsorbent dosage, temperature and pH conditions. The residual dye concentration was determined using a UV-Vis spectrophotometer at 517 nm. Adsorbent based on chitosan have drawn a lot of interest for the adsorption of dyes due to its abundance and low price as well as rich amino and hydroxyl groups. Because of their excellent chelating power, chitosan-based adsorbents have a very high ability to tightly bind the pollutants present in contaminated water and wastewater. Studies with batch experiments indicated that chitosan achieved an adsorption capacity of 63.64 mg/g under optimum conditions of 30 minutes of contact time, initial RR4 dye concentration of 50 mg/L, adsorbent dosage of 0.01g, temperature of 27°C and solution pH of 3. In conclusion, chitosan was effective and promising adsorbent for removal of reactive dye.

1. Introduction

Since dyes are synthetically stable in water and do not biodegrade, they have been identified as a major threat to the environment. Ten to fifteen percent of the colors are released with effluents during the dyeing process [1]. Since dyes are mostly utilized to add color in textiles, the amount of dye in the water was lost in the wastewater, leaving the water with noticeably high dye concentration. Dyes can be natural or synthetic, and they can be classified based on their structure, solubility, and applications. Dyes also can be categorized based on their particle charge upon dissolution in aqueous application medium such as ionic dyes that include cationic dyes and anionic dyes, and non-ionic dyes [2].

Reactive dyes are the most popular dyes used in the textile industry. Certain reactive dyes have nickel, chromium, and copper bonds. When these dyes decompose, hazardous heavy metals are released into the surrounding environment [3]. Nevertheless, the adsorption results indicate that the electrical attraction between anionic dyes and positively charged surfaces of the adsorbent was associated to the adsorption process because reactive dyes are soluble in solution and have a larger negative charge density [4]. Additionally, reactive dyes frequently cause respiratory sensitization, which impairs breathing and frequently has an immune system impact on the person. Sneezing, coughing, wheezing, itching, watery eyes, and skin irritation are among the symptoms and side effects [5].

Given the hazardous nature of dye stuff, numerous techniques have been attempted to eliminate them from aqueous environments, including chemical coagulation, adsorption, and ion exchange. It has been discovered that the adsorption method gives better results than others [6]. This method is recommended for the removal of various dyes due to its positive outcomes, high adsorption capacity, and simplicity of use [7].

Adsorption is the property of all solids that allows molecules of gases or solutions they encounter to bind to their surface [8]. The difference in concentration between the adsorbate in liquid or gas and adsorbent surface phases drives the mass transfer process of adsorption [9]. In order to remove components of a gas or liquid solution from the solution, certain solids have the capacity to adsorb contaminated materials into their structure. Adsorption is considered as a well-developed method for removal of dye from wastewater since it is less complicated and expensive than alternative techniques [10]. Surface enrichment or adsorption is the process of atoms, ions, and molecules attaching to the active centers of a solid surface. Adsorption is primarily driven by the adsorbent's affinity, its capacity to react with the pollutant and the mechanism of adsorption between the sorbent and the pollutant's functional group [4]. Adsorption results from the

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uneven or residual attractive forces present in the surface particles of the adsorbent, which are not in the same state as the bulk adsorbent particles, which have all the forces balanced together [11].

A natural polymer such as chitosan has been investigated to be a good alternative due to their adsorption capacity for several pollutants [12]. In recent years, chitosan-based adsorbent has gain increased interest in the treatment of water and wastewater because of its affordability, availability, and abundance of amino and hydroxyl groups [13]. Chitosan, the second most abundant biopolymer in nature, is a cationic biopolymer that is obtained through alkaline deacetylation of chitin. Given that its amine and hydroxyl groups can operate as an active site for the removal of dyes, chitosan has a great potential for the removal of dyes [14]. Chitosan exhibits as high dye adsorption capability in neutral solution. Chitosan's ability to absorb dyes has been explored for the direct, acid, reactive, azo and disperse dyes. Chitosan presents a high affinity for anionic dye adsorption [15].

Reactive Red 4 (RR4) is an anionic reactive dye which commonly used in textile dyeing processes due to its high color intensity and strong affinity towards fibers [3]. However, large quantities of RR4 may be discharged into wastewater during dyeing operations due to incomplete dye fixation [16]. The presence of RR4 in aquatic environments may reduce light penetration and may pose adverse effects on aquatic organisms and water quality [17]. Hence, effective treatment methods are required to remove RR4 from wastewater before discharge. Many studies and review have been published on dye adsorption onto chitosan and its derivatives. However, there is no study has investigated the adsorption of RR4 dye on chitosan. Therefore, the objective of this study is to investigate the adsorption performance of chitosan on the removal of RR4 dye. This work aims to optimize its process by varying several parameters such as contact time, initial dye concentrations, adsorbent dosage, temperature, and solution pH.

2. Method

RR4 was selected as a model synthetic dye to simulate dye-containing wastewater and prepared using distilled water and diluted to the required concentrations for adsorption experiment. 0.01 g of chitosan was weighed and added directly into a 250mL glass conical flask containing 50 mL of 50 mg/L RR4 solution at pH 3. The flask shook at 180 rpm for designated contact time (0, 30, 60, 120, 180 min) at room temperature. Then, the adsorption process further investigated using different parameters including initial RR4 dye concentration (10, 30, 50, 100, 400 mg/L), adsorbent dosage (0.01, 0.03, 0.05, 0.07, 0.1 g). The temperature parameter was conducted at 27, 40, 50, 60 and 70ºC using a temperature-controlled orbital shaker while the solution pH (2, 3, 5, 7, 9) was adjusted using 0.1 M HCl or 0.1 M NaOH and measured using calibrated pH meter prior to adsorption.

After the adsorption experiment, the adsorbent was separated from the solution to measure the concentration of the filtrate at 517 nm using an Ultra-Violet Visible Spectrometer (UV-Vis). The adsorption capacity of RR4 removal was calculated using Eq (1).

$$\text{Adsorption capacity } (q_e) = \frac{(C_o - C_f)V}{m} \quad (1)$$

where C_o is the initial concentration and C_f is the final concentration, V (L) is the volume of the dye solution used and m (mg) is the mass of adsorbent used.

3. Results and discussion

This section explains the variety of conditions of batch adsorption studies, with the effect of contact time, initial RR4 dye concentrations, adsorbent dosage, temperature, and pH solution.

3.1 Effect of contact time

Fig. 1 shows the effect of contact time on the adsorption capacity of RR4 dye solution on chitosan. The adsorbents were in contact to the solution from 0 minutes to 180 minutes while the other parameters remained constant (initial concentration: 50 mg/l, adsorbent dosage: 0.01 g, pH 3, and temperature: 27ºC). The result shows that the adsorption capacity increased continuously with the increasing contact time, from 63.64 mg/g at 30 min to 104.13 mg/g at 60 min and further increased to 124.94 mg/g at 180 min. The rapid increase in adsorption capacity indicated the abundance of available active sites on the chitosan surface. At the initial stage, RR4 dye molecules were readily adsorbed onto the external surface of the adsorbent through strong attractive interactions, resulting in a high adsorption rate. As contact time increased, dye molecules gradually diffused into the internal pores of the chitosan structure, which lead to a further increase in adsorption capacity. The attractive strength between dye molecules and adsorbent materials resulted in quick diffusion onto the outer surface, followed by fast pore diffusion into the intra particle matrix, leading to substantial increase in adsorption capacity with contact time [18]. This trend is similar from the previous study by [19]. The results demonstrate that contact time is an important factor influencing RR4 dye adsorption onto chitosan, with longer contact times promoting greater dye uptake. Since substantial adsorption was achieved within the first 30 min, this contact time was selected for subsequent adsorption studies to reduce experimental duration and improve operational efficiency.

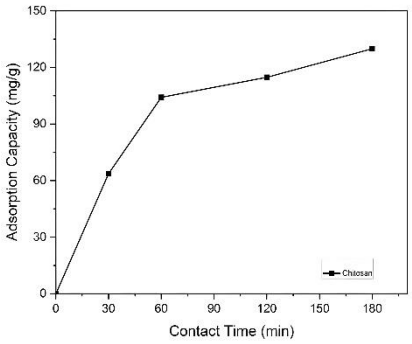

Fig. 1 - Effect of contact time adsorption of RR4 dye solution onto chitosan. (pH 3, 27ºC temperature, 0.01 g adsorbent dosage, and 50 mg/l initial concentration).

3.2 Effect of initial dye concentration

Fig. 2 shows the effect of initial concentration of adsorption capacity of RR4 dye solution on chitosan. The results show that the adsorption capacity increased from 5.05 mg/g to 120.33 mg/g. The result indicates that as the initial concentration increase, the adsorption capacity increase. As stated in previous study by Liu, et al. [20], higher initial concentrations result in more collision between dye and the adsorbent, produce enough driving energy to overcome mass transfer resistance between the aqueous and solid phases.

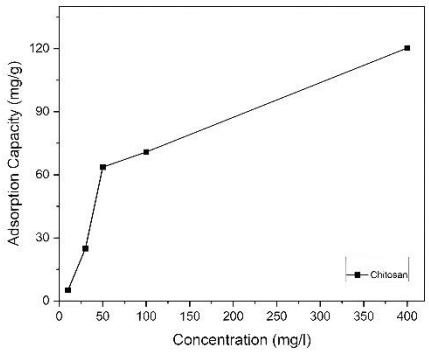

Fig. 2 - Effect of initial concentration adsorption of RR4 dye solution onto chitosan. (30 minutes contact time, pH 3, 27ºC temperature and 0.01 g adsorbent dosage).

3.3 **Effect of adsorbent dosage**

The adsorption process is influenced by the amount of adsorbent and the ratio of adsorbent to adsorbed. Fig. 3 shows the effect of adsorbent dosage on the adsorption capacity of RR4 dye solution on chitosan. The adsorbent dosage decreased from 63.64 mg/g (0.01 g) to 13.24 mg/g (0.1 g). Nga, et al. [21] stated that, increasing the adsorbent dosage leads to increased surface area and availability of adsorption sites. However, increasing the adsorbent dosage showed minimal effect on dye removal. As mention by Tanhaei, et al. [22], the adsorption capacity increased significantly by lowering the adsorbent dosage, which might be attributed to the expanding vacant adsorption active sites following the adsorption of nearly every RR4 molecule, resulting in a decrease in adsorption capacities.

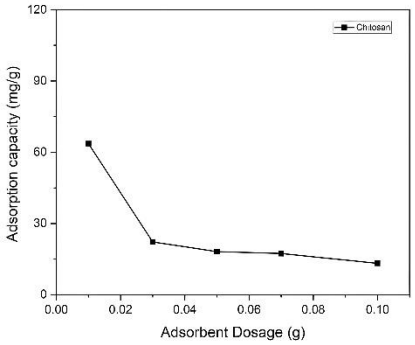

Fig. 3 - Effect of adsorbent dosage on the adsorption of RR4 dye solution onto chitosan. (30 minutes of contact time, pH 3, 27ºC temperature and 50 mg/l initial concentration).

3.4 **Effect of temperature**

The effect of temperature was performed at five different of temperature (27ºC, 40ºC, 50ºC, 60ºC, and 70ºC). Fig. 4 shows the effect of temperature on the adsorption capacity of RR4 dye solution on chitosan. The adsorption capacity decreased from 63.64 mg/g (27ºC) to 48.18 mg/g (70ºC). Chitosan showed that increased in temperature, adsorption capacity decreased. This observation trend in reduction in adsorption capacity with increasing temperature can be attributed to the reversibility of the interaction between the adsorbent and dye. This might be attributed to the exothermic impact of the adsorption process [23].

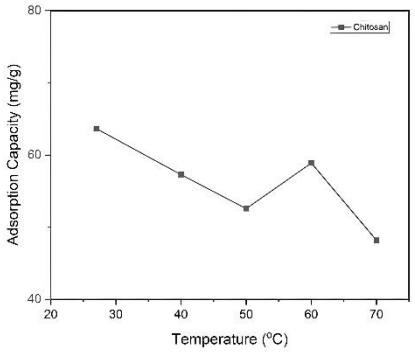

Fig. 4 - Effect of temperature on adsorption of RR4 dye solution onto chitosan. (30 minutes contact time, pH 3, 50 mg/l initial concentration and 0.01 g adsorbent dosage).

3.5 **Effect of pH**

One of the most critical aspects in dye adsorption is the effect of pH shift since the pH value affects the ionization and the surface charge [6]. Fig. 5 shows the effect of pH on the adsorption capacity of RR4 dye solution on chitosan. At lower pH levels, more protons are present. Protonated dyes' amino groups and electrostatic interactions with positively charged active sites lead to increased adsorption on the adsorbent [24]. High pH solutions can reduce electrostatic interactions between dye molecules and adsorbents, leading to their desorption. Anionic dye molecules have a stronger affinity for cationic desorption agents compared to molecular amine chitosan functional groups [25]. Therefore, RR4 dye adsorption capacity decreased as the solution pH increased from pH 3 to pH 9.

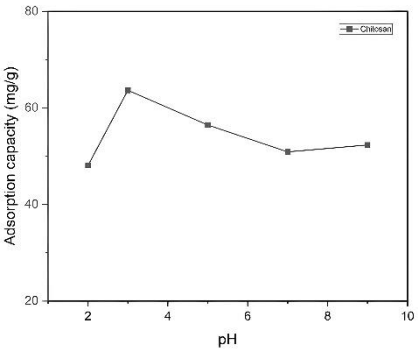

Fig. 5 - Effect of pH on adsorption of RR4 dye solution onto chitosan. (30 minutes contact time, 50 mg/l initial concentration and 0.01 g adsorbent dosage, 27ºC temperature).

4. **Conclusion**

In conclusion the adsorption performance of chitosan on the removal of RR4 dye has been investigated. The best adsorption conditions in batch system were 30 minutes of contact time due to larger size of active sites resulted in high adsorption capacity rates, initial RR4 dye concentration of 50 mg/L due to more collision between dye and the adsorbent, adsorbent dosage of 0.01 g due to increasing surface area and availability of adsorption sites, temperature of 27ºC due to reversibility of the interaction between adsorbent and dye and solution pH of 3 due reducing electrostatic interactions between dye molecules and adsorbents with the adsorption capacity of RR4 dye solution is 63.64 mg/g.

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