# Removal of Basic Dyes from Aqueous Solutions with Sulfonated Phenol–Formaldehyde Resin

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**ABSTRACT:** In this study, we aimed to investigate the removal of basic dyes, including Safranin T (ST), Nile Blue A (NBA), and Brilliant Cresyl Blue (BCB), from aqueous solution with a sulfonated phenol–formaldehyde resin. This sulfonated resin was characterized by Fourier transform infrared spectroscopy and scanning electron microscopy. The adsorption properties of this resin were investigated under different adsorption conditions with different initial dye concentrations, contact times, and pH values. The adsorption equilibrium data were ana-

lyzed with Langmuir and Freundlich models. The adsorption behaviors of ST, NBA, and BCB onto the sulfonated resol-type phenol–formaldehyde resin were better described by the Freundlich model. The adsorption capacities of the sulfonated resol resin for ST, NBA, and BCB decreased in the following order: NBA > BCB > ST. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 109: 2774–2780, 2008

Key words: adsorption; dyes/pigments; resins

## INTRODUCTION

Synthetic dyes are widely used in many fields of technology, for example, the textile, leather, paper, rubber, plastics, cosmetics, pharmaceutical, and food industries.<sup>1</sup> Synthetic dyes usually have a complex aromatic molecular structure and exhibit considerable structural diversity.<sup>1,2</sup> The chemical classes of dyes preferred more frequently in the industrial field are the azo-based chromophores combined with different types of reactive groups.<sup>1,3</sup> The extensive use of dyes often poses serious pollution problems because of the effluent of industries contain a number of contaminants, such as acid or caustic dissolved and suspended solids, toxic compounds, and coloring pigments.<sup>4</sup> In addition, some dyes or their metabolites are either toxic or mutagenic and carcinogenic.<sup>2,5</sup> Many dyes are also toxic to some organisms, which causes the direct destruction of aquatic communicates.<sup>4</sup> According to the regulations of Ministry of Environment and Forestry of Turkey, the limit of these materials in wastewater should be lower than 10 mg/L because dyes are considered "dangerous and harmful materials".6

The conventional methods for the removal of dyes from wastewater include coagulation and flocculation, oxidation or ozonation, membrane separation, and adsorption.<sup>7–11</sup> Activated carbons have the advantage of exhibiting a high adsorption capacity

for organic pollutants such as dyes. The adsorption of different dyes from aqueous mediums onto activated carbons has already been investigated,<sup>12,13</sup> and the removal of various dye categories has been discussed.14-17 According to these studies, the adsorption capacities of different dyes onto activated carbon changes from 50 to 90%, depending on the properties of the activated carbon, the initial dye concentration, and the type of dye.<sup>18</sup> Pereira and coworkers<sup>20,21</sup> reported that the percentage of dye removal with activated carbon for different types of dyes changed in the range 5-80%. However, a relatively high price; high operating costs, and problems with regeneration prevent its large-scale application. Therefore, there is a growing need to find low-cost, renewable, locally available materials as sorbents for the removal of dve colors.<sup>2</sup> Some low-cost materials have also been directly used as sorbents, such as chitosan, zeolites, clay, fly ash, coal, natural oxides, and industrial wastes, for dye adsorption.<sup>22</sup> There are also many articles about removal of basic dyes with different types of sorbent. These studies have shown that the maximum dye uptake capacities of different types of sorbents change in a large scale from 50 to 200 mg/g.<sup>4,23,24</sup>

In recent years, research interest in the production of alternative sorbents to replace conventional sorbents has intensified because of their low cost and local availability.

The aim of this study was to investigate the possibility of the dye adsorption of sulfonated phenolic resin. A literature survey did not yield any research on the use of sulfonated phenolic resin as a sorbent.

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Phenolic resins are the oldest industrial polymers that are still used because of some of their superior properties and their low manufacturing cost. In addition, the reactive hydroxyl groups of resol resin can be modified easily by various modification agents containing -SO<sub>3</sub>H and -COOH, -NH<sub>2</sub> groups. Materials that have these functional groups exhibit high adsorption properties. Consequently, we attempted to incorporate the -SO<sub>3</sub>H group into the resin by a sulfonation reaction. Then, this resin was used as a sorbent for the adsorption of basic dyes from aqueous solutions for the first time. The dyes selected as sorbates were three cationic dyes: Safranin T (ST), Nile Blue A (NBA), and Brilliant Cresyl Blue (BCB). These were selected because of their extensive use in the textile industry as colorants. Furthermore, these basic dyes were considered as model compounds to represent the dyes that are released in the effluents of the textile industry.

Finally, sulfonated phenolic resins may be a new way to remove some dye molecules from aqueous media because of their low cost and local availability.

## **EXPERIMENTAL**

## Materials and instruments

Phenol (P), formaldehyde (FA; 37% aqueous solution stabilized with 10% methanol), and basic dyes (ST, NBA, and BCB) were all purchased from Merck (Darmstadt, Germany). The rest of the materials were synthesis or analytical grade. The chemical structures of the dyes are shown in Figure 1.

Fourier transform infrared (FTIR (Randolph, MA, USA)) spectra of the samples were recorded on a Digilab Excalibur-FTS 3000 MX instrument in the range 400–4000 cm<sup>-1</sup>. To prepare the sample pellets, the samples were diluted with IR grade Merck KBr (sample/KBr = 1 : 200 w/w).

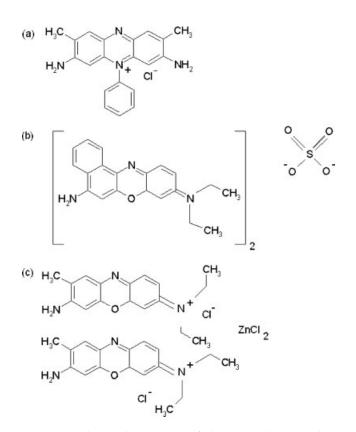
A Jeol JSM-5600 scanning electron microscope (Tokyo, Japan; acceleration voltage = 5 kV) was used to examine the surface morphology of the resins. The micrographs were taken at a magnification of 2500.

Spectrophotometric measurements were also carried out with a Jenway 6105 ultraviolet–visible (UV– vis (Dunmon, UK)) spectrophotometer.

#### Preparation of the phenolic resin sorbent

Step 1: Preparation of the phenolic resin

The polycondensation reaction of P and FA was carried out according to a known procedure<sup>25</sup> with a 1 : 3 molar ratio of P to FA. The reaction system was a 500-mL four-necked glass reaction kettle equipped with a mechanical stirrer, a reflux system, a temperature control system, and a heating mantle. P (47 g), FA (45 g), and NaOH (0.47 g, 0.5 wt %. in the total



**Figure 1** Chemical structure of the basic dyes used in this study: (a) ST, (b) NBA, and (c) BCB.

reaction mixture) as a catalyst were added to the reaction kettle at room temperature and stirred mechanically (150 rpm), and the temperature was increased to 70°C and kept constant. The reaction was followed by the determination of the free FA amount according to the hydroxylamine hydro-chloride method,<sup>26</sup> and the reaction was carried out until a constant free FA value (1.66%) was reached. At the end of the reaction, the resol-type phenolic resin (R) was purified *in vacuo* in a rotary evaporator system. The product was characterized by FTIR spectroscopy.

#### Step 2: Sulfonation of the phenolic resin

The sulfonated resol-type phenolic resin (SR) was synthesized by the sulfonation reaction of R, which was prepared in step 1, with sodium hydrogen sulfite (NaHSO<sub>3</sub>). The sulfonation reaction was carried out in a 500-mL four-necked glass reaction kettle equipped with a mechanical stirrer, a reflux system, a temperature control system, and a heating mantle. At first, 92 g of R was added to the reaction kettle at room temperature, and the temperature was raised to 80°C. NaHSO<sub>3</sub> (25 g) dissolved in 60 mL of distilled water was added to the solution with a burette

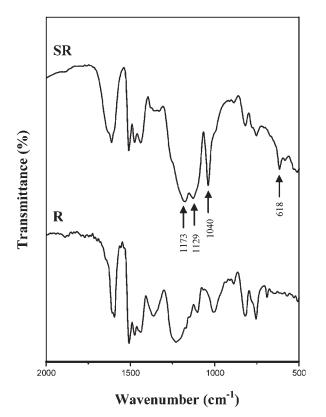


Figure 2 FTIR spectra of R and SR.

where the adding velocity was 2 mL/min. Then, the temperature of the solution was kept at  $80^{\circ}$ C for 80 min. The resin prepared according to this procedure had a solid content of approximately 59–60%. The number of sulfonate groups per unit of the polymeric chain was determined by chemical analysis and was found to be 1 wt %.<sup>27</sup>

Finally, SR was used as a sorbent for the removal of the basic dyes from aqueous solutions after curing at  $150^{\circ}$ C for 1 h.

## Characterization of SR

The FTIR spectra of R and SR are presented in Figure 2. The FTIR spectrum of R exhibited the characteristic absorption peaks. In this spectrum, absorption peaks at 756, 820, and 890 cm<sup>-1</sup> (ortho, parasubstituted phenolic ring), 1006–1011 cm<sup>-1</sup> (deformation vibration of -OH due to the methylol groups and phenolic -CO stretching), 1102 cm<sup>-1</sup> (phenolic -CO stretching), 1229 cm<sup>-1</sup> (phenolic -CO stretching), 1229 cm<sup>-1</sup> (phenolic -CO stretching and dimethylene ether bridges), 1363 cm<sup>-1</sup> (deformation vibration of -OH due to the methylol groups), 1437 cm<sup>-1</sup> (asymmetric bending vibration of  $-CH_2$ ), 1474 cm<sup>-1</sup> (1,2,3-substituted aromatic ring and 1,2,3,5-substituted aromatic ring), 1509 cm<sup>-1</sup> (ortho, para-substituted phenolic ring), and 1596 and 1611 cm<sup>-1</sup> (C=C stretching vibration) were present.<sup>28–32</sup>

The spectrum of SR showed additional peaks, which was different from the spectrum of R. These new absorption peaks were observed at about 618, 1040, 1129, and 1173 cm<sup>-1</sup>. The absorption peaks at 618, 1040, and 1129 cm<sup>-1</sup> resulted from the symmetric stretching vibration of -SO<sub>3</sub>H groups and the stretching vibration of the sulfonate anion, respectively. In addition, sulfonic group vibration bands were observed at 1173 cm<sup>-1</sup>, as reported previously.33-36 Moreover, after sulfonation, a significant decrease in the intensity of methylol groups (1360 cm<sup>-1</sup>) supported the occurrence of a sulfonation reaction between the -SO3H groups and the methylol groups of resol resins. These changes observed in the spectrum indicated the incorporation of  $-SO_3H$  groups into the resin structure after sulfonation.

## Dye adsorption experiments

Adsorption experiments were carried out in 100-mL beaker flasks containing 50 mL of synthetic dye solution at 25°C. SR (0.2 g) was added to the ST, NBA, and BCB solutions with concentrations of 500 mg/L for the determination of adsorption capacity. ST, NBA, and BCB solutions were prepared by the dissolution of dye in distilled water to the required concentrations. At different time intervals of 5, 10, 15, 30, 45, 60, 90, 120, 180, 240, 300, and 360 min and 24 h at pH 6, the dye concentration in the solution was analyzed colorimetrically with a spectrometer by the measurement of absorbance at the maximum absorption wavelength, that is, 530, 638, and 622 nm for ST, NBA, and BCB, respectively.

In the equilibrium adsorption isotherm experiments, a fixed amount of sorbent (0.2 g) was contacted with 50 mL of aqueous solutions of ST, NBA, and BCB with different concentrations (50–500 mg/L). The amount of residual dye in aqueous solution was determined with a UV–vis spectrophotometer after 24 h. All of the experiments were carried out in triplicate.

#### **RESULTS AND DISCUSSION**

## FTIR analysis of SR after dye adsorption

Figure 3 shows the FTIR spectra of SR before and after dye adsorption. As expected, the adsorption peaks that were easily seen at 618 and 1129 cm<sup>-1</sup>, which were attributed to the symmetric stretching vibration of  $-SO_3H$  groups and to the stretching vibration of the sulfonate anion, in the spectrum of SR before adsorption disappeared after dye adsorption. In addition, because of the interference between the peaks attributed to methylol groups (1328–1383 cm<sup>-1</sup>)<sup>35</sup> and to a newly formed band at 1330–1420 cm<sup>-1</sup>

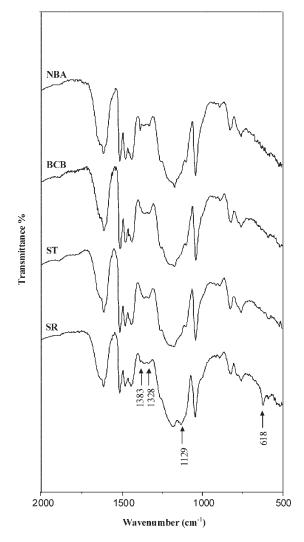


Figure 3 FTIR spectra of SR before and after dye adsorption.

 $(R-SO_2-OR' \text{ group})$ , the intensity of the peaks in this region increased significantly.<sup>36</sup>

## Scanning electron microscopy analysis of SR

The scanning electron micrograph of the SR sorbent was taken at a magnification of 2500 (Fig. 4). The scanning electron micrograph of the SR sorbent showed its smooth and nonporous surface.

#### UV-vis spectroscopy analysis of SR

At the end of 6 h of adsorption, the adsorption capacities of the SR were determined as about 110 mg/g for all of the basic dyes. Because the SR surface demonstrated a nonporous structure, we can say that the incorporation of  $-SO_3H$  groups into the structure provided sorbent characteristics to the resin.

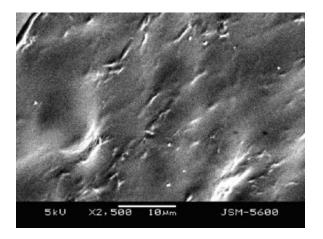


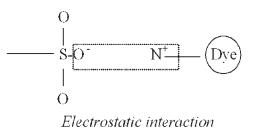
Figure 4 Scanning electron micrograph of SR.

## Adsorption behavior of the cationic dyes

The sulphonated resol-type phenolic resin had many ionizable groups, such as  $-SO_3H$  groups. At pH 6, the  $-SO_3H$  groups were in their ionized form, and electrostatic interaction could occur between the  $-SO_3H$  groups of the resins and the cationic groups of the basic dye molecules (Fig. 5); consequently, the dye adsorption ability became better.<sup>37,38</sup>

## Effect of the contact time and pH

The change in the amount of adsorbed basic dye by SR as a function of time is presented in Figure 6. The residual dye concentrations in solution were determined at different times from initial solutions of 500 mg/L and a sorbent dosage of 0.2 g/L. According to the adsorption studies, which were carried out at different pH values, the optimal pH value was determined as 6, and all experiments were carried out in triplicate at this pH value. This result was compatible with other results given previously in the literature. For example, Güçlü and Keleş<sup>23</sup> reported that pH 6 was the optimal pH value because basic dyes become stable in acidic media (pH < 7). Furthermore, it has also been reported that



Sulfonated Resin

Basic Dye

Figure 5 Interaction between SR and the cationic dye molecules.

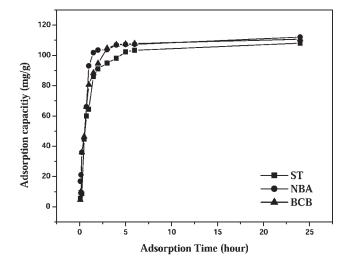


Figure 6 Effect of the adsorption time on the adsorption of the basic dyes onto SR.

the pH values of the basic dye effluent from the dyeing processes are below 7.<sup>39</sup>

The adsorption capacities for ST, NBA, and BCB were increased with the increase of the adsorption time. After the equilibrium adsorption time of 6 h, the adsorption capacities for ST, NBA, and BCB reached about 103, 107, and 108 mg/g, respectively.

As a result, the dye uptake capacities of this sorbent for these basic dyes were compatible and comparable with that other sorbents that have been given in the literature.

## Effect of the initial dye concentration

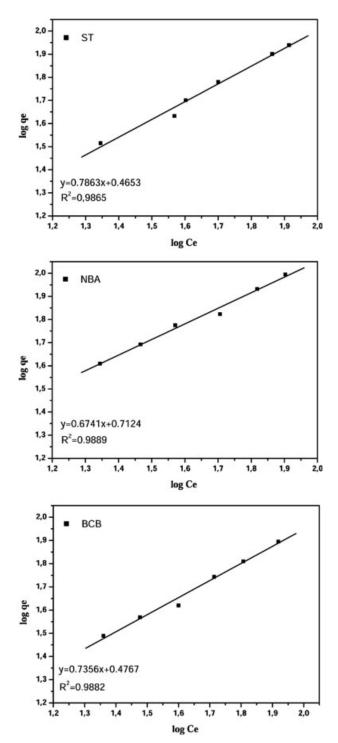
The adsorption isotherms were determined in beaker flasks containing synthetic dye solutions at 25°C and pH 6. A fixed amount of 0.2 g of sorbent was contacted with 50 mL of an aqueous solution of ST, NBA, or BCB with different concentrations. The initial concentrations [ $C_o$ 's (mg/L)] of solution were 50, 100, 200, 300, 400 and 500 mg/L, respectively.

The amounts adsorbed by the resin particles  $[q_e's (mg/g)]$  were calculated by the mass balance relation:

$$q_e = \frac{(C_o - C_e) \times V}{W} \tag{1}$$

where  $C_o$  and  $C_e$  are the initial solution concentration (mg/L) and equilibrium concentration (mg/L), respectively; *V* is the volume of the solution; and *W* is the weight of the dry sulphonated resol-type phenolic resin (g).

Several models have been published in the literature to describe adsorption isotherms. The Langmuir and Freundlich models are the most frequently used models.<sup>40,41</sup> In this study, the Langmuir and Freundlich models were used to describe the relationship between the amount of dye adsorbed and its equilibrium concentration in solution. However, when the Langmuir isotherm model was applied to these systems, the regression coefficient ( $\mathbb{R}^{2'}$ s) values were found to be 0.4917, 0.3617 and 0.5389 for ST, NBA and BCB, respectively. Therefore these results are



**Figure 7** Freundlich isotherms for the adsorption of the basic dyes onto SR.

TABLE I
Freundlich Constants of the Adsorption Isotherms for
ST, NBA, and BCB onto Sulfonated Phenolic Resin

Basic dye	Freundlich constant		
	K <sub>f</sub>	п	$R^2$
ST	2.919	1.302	0.9865
NBA	5.039	1.483	0.9889
BCB	2.997	1.359	0.9882

not suitable for describes the adsorption of these basic dyes.

The logarithmic form of the Freundlich model is given by the following equation:

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \tag{2}$$

where  $K_f$  and n are Freundlich constants related to the adsorption capacity and adsorption intensity, respectively. When log  $q_e$  was plotted against log  $C_{er}$ a straight line was obtained with slope 1/n, which showed that the adsorption followed a Freundlich isotherm well. These isotherms are illustrated in Figure 7. The parameters of the Freundlich isotherm,  $K_{fr}$ which is calculated from the intercept of the plots, and n and  $R^2$ , are given in Table I.  $R^{2'}$ s indicate whether the Freundlich isotherm is applicable for a system. The  $R^2$  values were 0.9865, 0.9889, and 0.9882 for ST, NBA, and BCB, respectively. n values between 1 and 10 indicate beneficial adsorption.<sup>42-46</sup> For the adsorption of ST, NBA, and BCB onto the sulphonated resol-type phenolic resin, the n values were above unity, that is, above the critical beneficial adsorption value n = 1. The adsorption capacities of the sulphonated resol-type phenolic for ST, NBA, and BCB had the following order: NBA > BCB > ST.

# CONCLUSIONS

In this work, R was prepared and sulfonated with sodium hydrogen sulfite. Then, this sulfonated phenolic resin was used as a sorbent in adsorption studies for basic dyes such as ST, NBA, and BCB for the first time. The adsorption properties were investigated under different adsorption conditions with different initial dye concentrations, contact times, and pH values. The adsorption equilibrium data were analyzed with the Freundlich and Langmuir models.

Our results show the following:

• After the incorporation of  $-SO_3H$  groups to the resin structure, the adsorption capacity of the resol resin increased noticeably. The adsorption capacities for ST, NBA, and BCB increased with the increase of the adsorption time until a plateau value was reached.

- After the equilibrium adsorption time of 6 h, the adsorption capacities for ST, NBA, and BCB reached about 103, 107, and 108 mg/g, respectively.
- The  $R^{2'}$ s values for the isotherm models indicated that the Freundlich model best described the adsorption of ST, NBA, and BCB onto the sulphonated resol-type phenolic resin, and the experimental data were better fitted to the Freundlich isotherm.
- For the adsorption of ST, NBA, and BCB onto the sulphonated resol-type phenolic resin, the 1/ *n* values were found to be 0.768, 0.674, and 0.736 for ST, NBA, and BCB, respectively. So, these values confirmed beneficial adsorption.
- The  $K_f$  values of the sulphonated resol-type phenolic resin for basic dyes were found to be 2.919, 5.039, and 2.997 for ST, NBA and BCB, respectively, which were compatible and comparable with that of other sorbents given in the literature.<sup>4,23,41</sup> Eventually, these values can be acceptable and workable for new potential and low-cost adsorption systems.

At the end of this study, we can say that the sulfonated resol resin may be used as an alternative sorbent for the removal of some dye molecules from wastewaters.

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