Binary Adsorption of Reactive Red 120 and Yellow 81 on *Spirogyra majuscula*

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**Abstract:** Binary adsorption of Reactive Red (RR) 120 and Reactive Yellow (RY) 81 on *Spirogyra majuscula* was performed as function of initial dye concentration, pH regimes and contact time. Characterization of this alga was confirmed by FTIR spectrum. Increasing initial dye concentrations caused to increase ($p < 0.01$) in dye uptakes. Pseudo second-order kinetic model was well to describe the adsorption behavior of reactive dyes. Freundlich and Redlich-Peterson models were more suitable for the adsorption of RR 120, while for the adsorption of RY 81, Langmuir was more appropriate model. Therefore, it could be concluded that the adsorption of RR 120 could be accomplished due to heterogeneous adsorption phenomenon, whereas the adsorption of RY 81 was due to a homogeneous adsorption with monolayer surface coverage. Despite adsorption of these dyes in binary system, this alga could be used as an adsorbent for textiles wastewater treatment without adding extra cost.

**Key words:** Adsorption • Reactive Red 120 • Reactive Yellow 81 • Spirogyra majuscula

**INTRODUCTION**

Various industries such as plastic, food, cosmetic, carpet, paper etc. have used over 10 000 commercially dyes with production of over $7 \times 10^4$ metric tons per year to color final products [1-3]. Reactive dyes are extensively used in textile industries, regarding favorable characteristics such as having bright color, water-soluble and simple application techniques with low energy consumption [2]. About 10-15 % of the dye is lost with a large volume of effluent from textile dyeing processes [1]. It is known that series environmental problems increase with the disposal of such effluents into natural aquatic ecosystems. Apart from the toxicological properties of some dyes, color is one of the first signs of contamination in wastewater. Since a very small quantity of dyes in water can be visible, it often affects the aesthetic merit and water transparency which has adverse influences on photosynthetic activity due to reduction of light penetration [4].

Degradation of dyes are very difficult because they are designed to stable and to resist for fading against exposure to sweat, light, water and oxidizing agents [5]. Therefore, the removal of dyes from wastewaters has been a main target in many researches in the last few years, not only due to their toxicity, but also because of its visibility. Hence, their removals from industrial effluents have a great importance for aquatic ecosystems before discharging into receiving waters. Physical, chemical and physico-chemical processes are used for treatment of industrial wastewaters [2]. These methods are very costly and create disposal problems due to accumulation of concentrated sludge. Besides, use of excessive chemical reagents provides a secondary pollution problem and requires additional cost for regeneration of treatment system [6, 7]. Therefore, the search for efficient, eco-friendly and cost effective remedies for wastewater treatment has been initiated. In the last few years, studies have focused on adsorption of pollutants present in wastewater on biomaterials.
The use of inactive microbial mass and organic materials as biosorbent has many advantages for wastewater treatment; (i) they do not require a continuous supply of nutrients, (ii) they are not affected by toxic wastes and (iii) these materials have been shown to accumulate more pollutants than that of organisms. Therefore, researchers have focused on biological methods for the treatment of effluents. Biological processes such as bioaccumulation and biodegradation have been proposed as to be a potential process for removing of dyes from bearing wastewaters [4]. The use of biomaterials as adsorbents for the treatment of wastewaters provide as an alternative method to the conventional treatment. They have many advantages over conventional treatments such as, economic, nontoxic to environment and widespread [3, 8].

Interactions between dyes and biosorbent depend on the nature of dye, specific surface properties of biomass and environmental conditions (e.g., pH, temperature, ionic strength). The binding mechanisms of dye to biosorbent vary from physical to chemical [5, 6]. The cell wall of algal species has various functional chemical groups such as carboxyl, hydroxyl, amino, thiol, sulphate and phosphate, which are responsible for the sequestration of dye, metal and salt from wastewaters [2, 6]. Adsorption of dye on algae depends on the cell wall properties which changes from species to other one. Distribution of these functional groups on the surface of cell and their activities may be different for each dye.

Many types of microorganisms have high capacity of dye uptake, such as algae [9], Bacteria [10] and Fungi [11]. Among these, microalgal biomasses are largely employed as biosorbent for several reasons: such as available in large quantities in nature; largely cultivated worldwide and its processing is relatively cheap. Therefore, microalgal biomass has been successfully used as adsorbent for removing of wastewaters. The binding of dye on inactivated algal biomass depends on the number and type of functional groups on the surface of species and the chemical composition of dye. Even though thousands of algal species are known, only a few of them have been investigated for wastewater treatment [12, 13].

Different algal species have been proved to be effective biosorbent for the treatment of wastewaters such as dyes and heavy metals [9, 14]. However, there is no detail studies related with binary adsorption of reactive dyes on Spirogyra majuscula. A widespread freshwater alga S. majuscula was selected as an effective, natural and cheap adsorbent for removing of reactive red (RR) 120 and reactive yellow (RY) 81 dyes, commonly used in textile industry, as function of initial dye concentration and pH regimes.

**MATERIALS AND METHODS**

**Adsorbent:** The alga used in this study, *Spirogyra majuscula* [15], was obtained from a freshwater pool in University of Gaziantep, Turkey. The species was filamentous green alga and appeared as an elongated filament composed of cylindrical cells. The alga had spiral chloroplast, end cell wall plane, conjugation tubes occurred from both gametangia and lens-shaped smooth zygospore [16].

Collected *S. majuscula* was washed twice with tap water in order to remove adhering larvae of organisms, soil, etc. After that, it was dried in an oven at 80°C for 24 h. Powder of this alga was obtained after grinding and sieving (100-75 µm mesh sizes). For the adsorption studies, the adsorbent suspension was prepared as 10 g L⁻¹ by using the algal powder.

The infrared spectra of the adsorbent were analyzed using a Fourier transform infrared (FTIR) spectrum (Perkin-Elmer Spectrum 100 FTIR Spectrometer) to identify the functional groups responsible for the adsorption.

**Batch Experiments:** Stock dye solution of RR 120 (Procion Red HE-3B; C₉₆H₄₈Cl₂N₁₆O₂₁S₅Na₁₄; Sigma-Aldrich Chemical Co., St. Louis, USA) was prepared in distilled water as 1 g L⁻¹. In a similar way, stock solution of RY 81 (Procion Yellow H-E3G, Sigma-Aldrich Chemical Co., St. Louis, USA) was prepared as 1 g L⁻¹ in distilled water. Each test solution had equal concentration of RR 120 and RY 81. For example, the first test solution included 50 mg L⁻¹ RR 120 and 50 mg L⁻¹ RY 81. Other test solutions were prepared in a similar way.

Adsorption studies were performed in 250 mL Erlenmayer flask containing 100 mL of adsorption solution. Adsorption solution included ten milliliters adsorbent suspension having desired pH (2, 3, 4 and 5) and 90 mL of dyes solution at known initial dye concentration having desired pH (2, 3, 4 and 5) in an Erlenmeyer flask. All the final solutions contained 1.0 g L⁻¹ of the adsorbent. The pH of each solution was adjusted to required value with diluted or concentrated HCl and/or NaOH solutions before mixing the adsorbate or adsorbent suspension. The mixtures were agitated on the orbital shaker at 150 rpm for 48 h to ensure equilibrium was reached.
Results and Discussion

Samples (5 mL) were taken before mixing of adsorbent suspension with dye bearing solution, then at different time intervals (0.083, 0.17, 0.25, 0.5, 1.0, 2.0, 3.0, 24.0 and 48.0 h) for the determination of residual dye value in the solution. After that, samples were centrifuged to precipitate suspended biomass at 5000 rpm for 5 min. The concentrations of reactive dyes in the supernatant were determined by spectrophotometer (Jenway 6305). Maximum absorbance was observed at 350 and 515 nm for RY 81 and RR 120, respectively. All kinetic experiments were carried out duplicates. The data were the mean values of two replicate determinations.

Amounts of RR 120 or RY 81 uptake per unit of the adsorbent at time $t$ ($q_t$, mg g$^{-1}$) and at the equilibrium ($q_{eq}$, mg g$^{-1}$) were calculated by using Eq. (1) and Eq. (2), respectively.

$$q_t = \frac{(C_0 - C_t)xV}{M} \quad (1)$$

$$q_{eq} = \frac{(C_0 - C_{eq})xV}{M} \quad (2)$$

Where, $C_0$, $C_t$, and $C_{eq}$ represent at initial, $t$ time and equilibrium concentrations of RR 120 or RY 81 (mg L$^{-1}$) in the solution, respectively. $V$ is the volume of solution (L) and $M$ is the mass of adsorbent (g).

Statistical Analyses: Analysis of Variance (ANOVA) was performed to determine significant differences for amount of removal dyes as function of initial pH regimes and initial dye concentrations by using the SPSS version 16.0 (SPSS Inc., Chicago, IL, USA). The fitting procedure was performed by using commercial computer software SigmaPlot version 11 (Systat Software, Inc., California, USA) via the Marquardt-Levenberg algorithm.

Effect of Initial pH Regimes: Change in pH regimes closely affects several functional groups such as amino, carboxyl etc. on the surface of algal cell walls, which are responsible for binding of dye molecules [11, 13].

Effect of pH regimes on the adsorption of reactive dyes on S. majuscula is given in Figure 2. Change in pH values strongly affected ($p<0.01$) the equilibrium adsorption of both reactive dyes. Increasing initial pH value decreased the amount of adsorbed dyes on the adsorbent. Higher adsorption for these dyes was observed at pH values from 2 to 5 than that of other pH regimes (Figure 2). Therefore, pH values between 2 and 5 were selected for further experiments.

Influence of initial pH regimes (pH 2-5) on the adsorptions of RR 120 and RY 81 dyes can be seen in Figures 3 and 4. Equilibrium RR 120 uptake on the green alga was found as 186.95±1.07, 216.16±0.86, 152.68±0.99 and 119.88±0.60 mg g$^{-1}$ at pH 2, 3, 4 and 5, respectively. Similarly, adsorption of RY 81 was measured as 163.18±1.46, 191.75±1.29, 138.04±2.12 and 113.46±1.63 mg g$^{-1}$ at pH 2, 3, 4 and 5, respectively. Remarkable adsorption of both reactive dyes was found at initial pH value of 3.0. Likely, Arçca and Bayramoğlu [20] reported that maximum adsorption of reactive red 120 on Lentinus sajur-caju was found at pH value of 3.

Influence of pH on the dye uptake could be explained on the basis of zero point charge (pH$_{zpc}$) of adsorbent [8, 21]. Çelekli et al. [15] reported that pH$_{zpc}$ of S. majuscula was determined as pH 7.7. Surface of adsorbent gets positively charged at pH<pH$_{zpc}$, which increases the adsorption of dye due to electrostatic forces of attraction. At pH>pH$_{zpc}$, its surface becomes negatively charged, which adsorbs less dye due to electronic repulsion. Presence of high concentrations of protons at lower pH could neutralize the negatively charged groups and increase number of positively charged groups on the surface of S. majuscula, leading to increase adsorption capacity. It was reported that electrostatic attraction could be essential mechanism in the adsorption of pollutants on the alga [8, 13]. However, Aksu and Tezer [5] keynoted that it could be very difficult to explain adsorption mechanism with respect to pH. Since, there are many
numbers of variables involve and affect the adsorption of dye such as the number and type of functional groups on the adsorbent surface, water chemistry, ionic strength etc. [22].

**Effects of Contact Time and Initial Dye Concentrations:**

Binary adsorption of RR 120 and RY 81 dyes at different initial dye concentrations were performed on *Spirogyra majuscula* during 48 h of contact time. Adsorption of these dyes increased (*p*<0.01) with increasing contact time. Rapid adsorption was observed during the first 30 min. After then, removal rate of dyes reduced gradually until the equilibrium state. There was no significant difference (*p*>0.05) in the amount of removing dyes in this state.

Adsorption capacity of the adsorbent was increased with increasing concentrations of dyes (Figures 3 and 4), as a result of increase in the driving forces [11, 23]. *S. majuscula* had remarkable potential for the removal of reactive dyes from aqueous adsorption solution in the binary system when compared with previous studies in single adsorption system [20, 22]. Despite there could be a competitive adsorption of reactive dyes on the surface of the alga, the adsorption capacity of *S. majuscula* for RR 120 in binary adsorption system was found to be higher than that of adsorption on *L. sajor-caju* in single dye adsorption system [20]. This could be due to having differences in the surface properties of each adsorbent such as functional groups, surface area, particle size, concentration etc. and other environmental conditions.

**Kinetic Modeling:**

Adsorption kinetics of RR 120 and RY 81 dyes has been carried out to understand the adsorption behavior of dried *S. majuscula* with respect to contact time, initial dye concentrations and pH regimes. In the present study, pseudo first-order [24] and second-order kinetic [25] models (Table 1) were used to describe the behavior of batch adsorption experiments.

Values of pseudo first and secon-order kinetic model are presented in Table 2. Pseudo first-order kinetic model could only describe the adsorption of RR 120 and RY 81 up to 30 min, respectively, where rapid adsorption was occurred [25]. It could be concluded that this model was not suitable to describe adsorption of both reactive dyes on *S. majuscula* for the whole adsorption process. Similar results were also observed in the literature [8, 23].
Table 1: Equations of models and error functions

<table>
<thead>
<tr>
<th>Adsorption kinetics</th>
<th>References</th>
<th>Equation and number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pseudo first-order kinetics</td>
<td>[24]</td>
<td>( \log(q_{eq} - q_t) = \log q_{eq} - \frac{k_1}{2.303} t ) (3)</td>
</tr>
<tr>
<td>Pseudo second-order kinetics</td>
<td>[25]</td>
<td>( \frac{t}{q_t} = \frac{1}{k_2 q_{eq}^2} + \frac{t}{q_{eq}} ) (4)</td>
</tr>
</tbody>
</table>

Equilibrium Models

<table>
<thead>
<tr>
<th>References</th>
<th>Equation and number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Langmuir</td>
<td>( q_{eq} = \frac{b q q C_{eq}}{1 + b C_{eq}} ) (5)</td>
</tr>
<tr>
<td>Freundlich</td>
<td>( q_{eq} = K F_{eq} C_{eq}^{1/n} ) (6)</td>
</tr>
<tr>
<td>Redlich-Peterson</td>
<td>( q_{eq} = \frac{K R P C_{eq}}{1 + g R P C_{eq}^2} ) (7)</td>
</tr>
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</table>

Error function

<table>
<thead>
<tr>
<th>Reference</th>
<th>Equation and number</th>
</tr>
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<tr>
<td>Chi-square statistic</td>
<td>( \chi^2 = \sum_{i=1}^{m} \left( \frac{q_{exp} - q_{cal}}{q_{cal}} \right)^2 ) (10)</td>
</tr>
</tbody>
</table>

Symbols’ of parameters are given in Abbreviation section

Table 2: Kinetic parameters for the adsorption of RR 120 and RY 81 on *Spirogyra majuscula*

<table>
<thead>
<tr>
<th>C, mg L⁻¹</th>
<th>pH</th>
<th>Reactive red 120</th>
<th></th>
<th>Reactive yellow 81</th>
<th></th>
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<tr>
<td></td>
<td>q_{exp}</td>
<td>q_{cal}</td>
<td>R²</td>
<td>q_{exp}</td>
<td>q_{cal}</td>
</tr>
<tr>
<td>50</td>
<td>2</td>
<td>45.96</td>
<td>0.2193</td>
<td>41.377</td>
<td>0.940</td>
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<td>3</td>
<td>49.34</td>
<td>0.0532</td>
<td>42.875</td>
<td>0.815</td>
<td>37.916</td>
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<tr>
<td>4</td>
<td>41.64</td>
<td>0.0569</td>
<td>37.916</td>
<td>0.950</td>
<td>40.381</td>
</tr>
<tr>
<td>5</td>
<td>29.95</td>
<td>0.0368</td>
<td>24.940</td>
<td>0.782</td>
<td>27.268</td>
</tr>
<tr>
<td>100</td>
<td>81.37</td>
<td>0.1110</td>
<td>65.110</td>
<td>0.754</td>
<td>70.557</td>
</tr>
<tr>
<td>3</td>
<td>92.02</td>
<td>0.1071</td>
<td>85.051</td>
<td>0.967</td>
<td>89.655</td>
</tr>
<tr>
<td>4</td>
<td>72.72</td>
<td>0.0225</td>
<td>64.270</td>
<td>0.779</td>
<td>68.260</td>
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<tr>
<td>5</td>
<td>54.07</td>
<td>0.0255</td>
<td>50.272</td>
<td>0.945</td>
<td>55.267</td>
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<td>200</td>
<td>138.08</td>
<td>0.1136</td>
<td>121.388</td>
<td>0.856</td>
<td>129.104</td>
</tr>
<tr>
<td>3</td>
<td>151.16</td>
<td>0.3362</td>
<td>147.674</td>
<td>0.987</td>
<td>147.513</td>
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<tr>
<td>4</td>
<td>111.36</td>
<td>0.1444</td>
<td>101.510</td>
<td>0.943</td>
<td>106.964</td>
</tr>
<tr>
<td>5</td>
<td>92.17</td>
<td>0.0257</td>
<td>83.473</td>
<td>0.931</td>
<td>90.405</td>
</tr>
<tr>
<td>350</td>
<td>186.95</td>
<td>0.0592</td>
<td>175.502</td>
<td>0.944</td>
<td>186.486</td>
</tr>
<tr>
<td>3</td>
<td>216.16</td>
<td>0.0447</td>
<td>188.318</td>
<td>0.874</td>
<td>204.040</td>
</tr>
<tr>
<td>4</td>
<td>152.68</td>
<td>0.0492</td>
<td>137.401</td>
<td>0.892</td>
<td>132.015</td>
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<td>5</td>
<td>119.88</td>
<td>0.2154</td>
<td>104.754</td>
<td>0.904</td>
<td>110.137</td>
</tr>
</tbody>
</table>

Symbols’ of parameters are given in Abbreviation section

Pseudo second-order model is related with the adsorption capacity of adsorbent [25]. The straight-line of \( t/q_t \) against \( t \) plots (not shown) indicated the ability of this model to describe the kinetic experimental data. Besides, correlation between the experimental and predicted \( q_t \) data from pseudo second kinetic model can be seen in Figures 3 and 4. It can be concluded that pseudo second-order kinetic model could be regarded as sufficient to describe the adsorption of these dyes on *S. majuscula*. In the literature, this model has adequately described adsorption of dyes [8, 15].

**Equilibrium Modeling:** Langmuir [26], Freundlich [27] and Redlich-Peterson [28] (Table 1) were fitted to describe equilibrium adsorption between adsorbed material on biomass (\( q_{eq}, \text{mg g}^{-1} \)) and unabsorbed pollutant in
Fig. 3: Comparison of experimental points and fitted curves by pseudo second kinetic model for adsorption of RR 120 on *Spirogyra majuscula* at pH 3. Lines show pseudo second kinetic model

Fig. 4: Comparison of experimental points and fitted curves by pseudo second kinetic model for adsorption of RY 81 on *Spirogyra majuscula* at pH 3. Lines show pseudo second kinetic model

Fig. 5: Comparison of experimental symbols and fitted lines by Langmuir, Freundlich and Redlich-Peterson models for adsorption of RR 120 solution ($C_0$, mg L$^{-1}$). Langmuir model can be expressed by means of a dimensionless constant, $R$, [19, 29], whose magnitude provides information about whether the adsorption process is spontaneous or non-spontaneous.

Results of Langmuir, Freundlich and Redlich-Peterson models are summarized in Table 3. Langmuir parameters, $q$, and $b$, were found to be higher at pH 3 than those of other pH regimes for both reactive dyes. Maximum adsorption capacity of *S. majuscula* was calculated from Langmuir model as 273.30 mg g$^{-1}$ and 301.16 mg g$^{-1}$ for RR 120 and RY 81, respectively (Table 3). Difference in dye uptake capacity of *S. majuscula* could be consequence of the properties of reactive dyes such as the structural, chemical composition etc. Despite, there was a competition of reactive dyes to bind the surface of the alga in binary system, *S. majuscula* showed remarkable adsorption capacity compared to the single anionic dye adsorption system in the literature [8, 11, 20, 30]. Values of $R$, were found to be 0.10-0.70 and 0.24-0.78 for RR 120 and RY 81, respectively. Due to its values between 0 and 1, these adsorption processes are favorable, in agreement with findings of previous studies [8, 20, 29].

The highest values of $K_F$ from Freundlich model were found at initial pH 3 for reactive dyes. Magnitudes of $K_F$ and $n$, which showed easy separation of RR 120 and RY 81 dyes from aqueous solution, indicated high adsorption capacity of *S. majuscula*. Similar results were also found in the literature [3, 8, 24].

Values of $K_{sp}$ obtained from Redlich-Peterson model indicated that adsorption of *S. majuscula* was found to be higher for RR 120 than that for RY 81 except at pH 4 (Table 3). Values of $\beta$ lied between 0 and 1, which indicated adsorptions are favorable.
The present study showed that *S. majuscula* had a huge potential for binary uptake of RR 120 and RY 81 dyes from aqueous solution. Behavior of batch sorption kinetics was well described by pseudo second-order kinetic model. Higher $R^2$ values and lower values of error functions indicated that Freundlich and Redlich-Peterson models were more suitable for adsorption of RR 120, while Langmuir model was more appropriate for adsorption of RY 81. This species could have high potential for treatment of textiles wastewater in industrial scale as effective and natural adsorbent without excessive cost.

### ACKNOWLEDGEMENT

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### Abbreviations

- $a_{b,p}$ Redlich-Peterson isotherm constant (L mg$^{-1}$)
- $b$ Langmuir constant (L mg$^{-1}$)
- $\beta$ Exponent in Redlich-Peterson isotherm
- $C_{eq}$ Equilibrium dye concentration in solution (mg L$^{-1}$)
- $C_o$ Initial dye concentration (mg L$^{-1}$)
- $k_1$ Pseudo first-order rate constant (L min$^{-1}$)
- $k_2$ Pseudo second-order rate constant (g mg min$^{-1}$)
- $K_f$ Freundlich adsorption capacity [(mg/g) (mg L$^{-1}$)$^{1/\beta}$]
- $K'_{b,p}$ Redlich-Peterson isotherm constant (L mg$^{-1}$)
- $n$ Freundlich adsorption intensity
- $q_{eq}$ Amount of adsorbed dye per unit weight of biomass at equilibrium (mg/g)
- $q_{exp}$ Experimental amount of adsorbed dye at equilibrium (mg g$^{-1}$)
- $q_{cal}$ Calculated amount of adsorbed dye for kinetics models (mg g$^{-1}$)
- $q_{m}$ Maximum adsorption dye capacity form Langmuir model (mg g$^{-1}$)
- $q_t$ Amount of dye per unit of biomass at time $t$ (mg g$^{-1}$)
- $RR$ Reactive red
- $RY$ Reactive yellow
- $R^2$ Correlation coefficient
- $R_o$ A dimensionless constant for the Langmuir model
- $t$ time (h)
- $p$ Significance level obtained from ANOVA
- $\chi^2$ Chi-square test statistic

### Table 3: Results of isotherms for the removal of RR 120 and RY 81 on *Spirogyra majuscula*

<table>
<thead>
<tr>
<th>Isotherm</th>
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<th>3</th>
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<th>5</th>
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<tr>
<td>Langmuir</td>
<td>$q_e$</td>
<td>251.94</td>
<td>273.30</td>
<td>189.55</td>
<td>182.94</td>
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<td></td>
<td>$b$</td>
<td>0.0169</td>
<td>0.0248</td>
<td>0.0169</td>
<td>0.0008</td>
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<td></td>
<td>$R_L$</td>
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<td>$R^2$</td>
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<tr>
<td></td>
<td>$\chi'$</td>
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<td>Freundlich</td>
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<td>$n$</td>
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<td></td>
<td>$R^2$</td>
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<td>0.999</td>
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<td></td>
<td>$\chi'$</td>
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<td>Redlich-Peterson</td>
<td>$K_{b,p}$</td>
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<td>$a_{b,p}$</td>
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</tbody>
</table>

Plots of $q_{eq}$ against $C_{eq}$ (Figures 5 and 6) indicated applicability of Langmuir, Freundlich and Redlich-Peterson models. Experimental data were fitted to the aforementioned models which had high correlation coefficients (Table 3, Figures 5 and 6). Applicability of these models were also observed in previous adsorption studies [7, 8, 11]. Results of correlation coefficient and the error functions (Table 3) showed that Freundlich and Redlich-Peterson models were more appropriate to describe the adsorption of RR 120. On the other hand, Langmuir model was the most appropriate model to describe the adsorption of RY 81, which implied a homogeneous sorption phenomenon such as equally available adsorption sites, monolayer surface coverage and no interaction between adsorbed species.

### CONCLUSIONS

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REFERENCES


