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ADSORPTION STUDY ON REMOVAL OF REACTIVE BLUE 21 AND REACTIVE RED 180 FROM AQUEOUS MEDIUM USING POLYANILINE CuCl₂ IN THE PRESENCE OF UV LIGHT

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ABSTRACT

The kinetics and isotherm studies of dye–adsorbent system are processed in this work with PANI-CuCl $_2$ as adsorbent material for the treatment of Reactive Red 180 and Reactive Blue 21 from model dye solution. Influence of quantity of dye in solution, the weight of PANI-CuCl $_2$ composite, temperature, pH and stirring speed were surveyed by batch experiments to identify the efficiency of the adsorbent. The ability of the adsorbent is scrutinized using desorption and recyclability experiments. The obtained results are analyzed with two isotherm models and the best fit is found to be Langmuir isotherm model. This adsorption process follows the pseudo-second-order kinetics model. The thermochemical quantities like ΔG° , ΔH° and ΔS° were evaluated. Furthermore, the adsorption of dyes onto PANI- CuCl $_2$ composite is established by conductivity study, FTIR and SEM.

Keywords: Industrial Dye, Adsorption, Polyaniline, Isotherm, Kinetics, Thermodynamics.

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INTRODUCTION

Synthetic dyes are preferred over natural dyes due to its unique shade, fastness, level of dyeing properties and ease of application. A huge number of dyes are being manufactured by industries using chemicals to satisfy the global demand for organic colorants in paint, textile and plastic industries. Dye effluents produced by textile, printing and paper industries are the major pollutant of wastewaters and the synthetic dyes that are unused after the dyeing process are drained into the natural source waterways which affect the quality of water. Various toxic materials like aromatic hydrocarbon derivatives, sulphur, nitrates, acetic acid and certain auxiliary chemicals that are present in the effluent of these dyes are harmful and have lethal effects on human beings and aquatic life. Hence the research community has pushed towards the development of eco-friendly and economically feasible processes for removing the dyes from wastewater to safeguard the populations from pollution. Conventional physicochemical processes for dye removal from wastewater are oxidation¹, photochemical degradation², reverse osmosis³, coagulation⁴, electrochemical process⁵, membrane separation⁶, ion exchange⁷ and adsorption⁸⁻¹⁰. Among these methods, adsorption is an economically viable and better pathway that can be accomplished using a low weight of adsorbents. The various adsorbent used for the removal of dyes are activated carbon¹¹, orange peel¹², clay¹³, agricultural waste¹⁴, etc., but these natural adsorbents are difficult to gather and the level of adsorption capacity is also very less. Limitations of commercial adsorbents are higher cost, substandard adsorption ability and poor recyclability. Polyaniline doped with different compounds is showing good adsorption capacity for the removal of dyes. 15-17 Adding CuCl₂ to polyaniline enhances the photocatalytic activity and creates more polar points to adsorb anionic dyes. 18-19

Reactive Red 180 and Reactive Blue 21 are utilized to color a variety of textile fibers. Exposure to these dyes could trigger skin allergy. The study of removal of RR 180 and RB 21 dyes from the effluent is important since they are commonly used textile dyes. A perusal of literature revealed that very few works are reported on the treatment of RB 21 and RR 180 using natural adsorbent which has poor adsorption efficiency. The present investigation analyzed the successful application of CuCl₂ doped polyaniline for the complete removal of RB21 and RR 180 dyes from wastewater and to study the impact of various



parameters on the removal process. Recyclability of polyaniline CuCl₂ is excellent in successive three cycles.

EXPERIMENTAL

Instruments

Adsorption experiments were carried out using RIS 24-BL orbital shaker that augments adsorption of RR180 and RB 21 dyes onto PANI-CuCl₂ composite in the presence of UV lamp. For the centrifugation of the dye solution, REMI motor was used. The concentration of the dye solution was measured by MAPADA-V-1100D spectrophotometer. pH of the solutions was determined using EQ-610 pH meter. The resistance of adsorbent before and after adsorption was checked using the Four-Probe method DFP-03.

Materials

Analytical grade aniline, hydrochloric acid, ammonium persulphate and copper chloride were procured and utilized without further purification. Dyes were purchased from Colourtex Industries Private Limited to carry out the adsorption experiments.

Reactive Blue 21[RB21] is a Phthalocyanine class dye whose λ_{max} is 624nm and Reactive Red 180 [RR 180] is a single azo class dye whose λ_{max} is 540nm. The structures of dyes are shown in Fig.-1 and 2.

Fig.-1: Reactive Blue 21[RB21]

Fig.-2: ReactiveRed 180 [RR 180]

Methods

Preparation of PANI-CuCl₂ and Stock Dye Solution

The polymerization reaction was processed in a magnetic stirrer by stirring aniline and 1.5N HCl at 400RPM, and then the solution of Ammonium Persulphate was added dropwise followed by CuCl₂ solution at the required amount. Precipitated CuCl₂ doped PANI composite was filtered washed, dried and stored²⁴.100m of a standard solution of RB 21 and RR 180 were prepared by dissolving 0.5g of dye in deionized water.

Experimental Setup for the Adsorption Study

For RB 21 0.8g/L of adsorbent and for RR 180 1.2g/L of PANI-CuCl₂ composite were accurately weighed and transferred into a separate 250ml beaker. 100 ml of the dye solutions having 105mg/L of RB 21 and 55mg/L of RR 180 was poured into the beaker containing the respective amount of adsorbents. The aliquot is stirred at 250 rpm in the presence of UV radiation (6 amp. UV lamp). At required time gap, 10ml of the reaction mixture was taken out and centrifuged. The optical density of the centrifuged dye solution was measured at their respective wavelength and the process is repeated upto the reach of equilibrium. The equilibrium quantity of dyes was calculated using the formula (1).

$$q_s = \frac{(c_o - c_s) V}{W} \tag{1}$$

% Decolourisation =
$$\frac{(c_o - c_t)}{c_o} X 100$$
 (2)

C_o(mg/L) is the concentration of the dye at a time t_o

C_t (mg/L) is the concentration of dye at different intervals of time t(min.)

qe (mg/g) is the amount of dye adsorbed per unit mass of the adsorbent at equilibrium,

W (g) is the weight of the PANI-CuCl₂ composite.

Various sets of experiments were conducted to find the effect of dose of PANI-CuCl₂(0.4g/L to 1.2g/L for RB 21 and from 0.8g/L to 1.6g/L for RR 180), amount of RR 180 (35-105mg/L) and RB 21 (80-144mg/L), a variation of temperature(30°C - 50°C), alteration of pH of the solution(2 - 8) and change of agitation speed(50-250) on the rate of adsorption. Desorption studies were carried out with sodium hydroxide, hydrochloric acid, alcohol and acetone to found out the nature of attractive force between dye molecules and PANI-CuCl₂. Filtered PANI-CuCl₂ composite was washed with water and then stirred in a magnetic stirrer for 15 minutes in water, filtered, dried and required amount of desorbed adsorbent was taken for adsorption study of successive cycles.

RESULTS AND DISCUSSION

Effect of Contact Time

During the process of adsorption, the rate of decolorization of RB 21 and RR 180 increases with augmentation of time up to a particular period, and after that, the rate of removal of the solute becomes constant as shown in Fig.-3a and 3b. It is observed that the concentration of RB 21 and RR180 drops and reaches equilibrium at 120 minutes and 180 minutes respectively. Hence for the present study 120 minutes for RB 21 and 180 minutes for RR 180 is fixed as the equilibration time, which means that this time is sufficient for maximum adsorption of reactive dyes by the PANI-CuCl₂ under the chosen set of experimental conditions. The maximum amount of dye adsorbed corresponding to the equilibration time is found to be 94 % for RB 21 and 98% for RR 180 under UV lamp irradiation with agitation. Same adsorption studies were carried out under agitation alone, by exposure to solar radiation without agitation in winter and summer and in the presence of UV lamp to find out the comparison of the efficiency of uptake of reactive dyes by PANI-CuCl₂ composite, which reveals that agitation in presence of UV lamp helps to decolorize maximum amount of dyes.

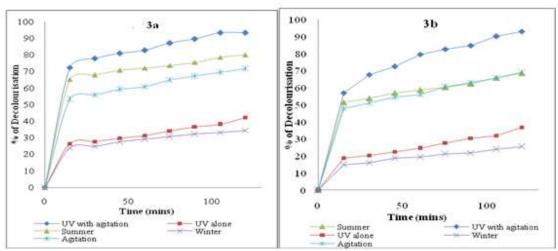


Fig.-3: Contact Time of Reactive Blue 21(a) and RR 180 (b) at Different Conditions

Effect of Initial Dye Concentration

A certain number of adsorbent sites can adsorb only a fixed amount of dye species, thus it is important to know the influence of concentration of dyes on the rate of adsorption. The graph plotted between the initial concentration of dye versus the equilibrium quantity of dye adsorbed (qe) and percentage of

decolorization of RB 21 and RR 180 dye solutions by PANI- CuCl₂ is shown in Fig.-4a and 4b, which unveils that reduction in the percentage of uptake of PANI-CuCl₂ composite on the rise in the initial concentration of dye solution. This can be accounted for that an increase in the concentration of dye solution requires a large number of reactive sites for higher adsorption percentage to occur whereas the increase in the concentration of dye enhances the amount of dye adsorbed (q_e). The higher initial concentration of dye gives a driving force to reduce the repulsion among the solid and liquid phases, thus raising the uptake and increased the adsorption capacity that occurs as a result of the increased mass transfer. Thus adsorption capacity is always higher at high concentration whereas the percentage of decolorization is higher at a lower concentration of dye.²⁵

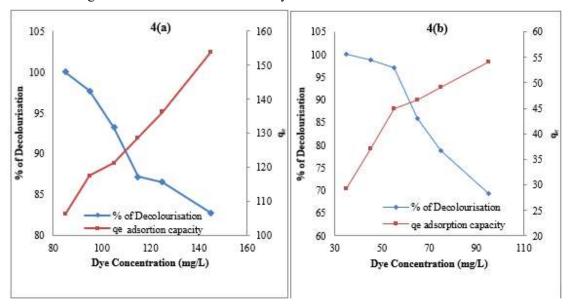


Fig.-4: Effect of Initial Dye Concentration of RB 21 and RR 180

Effect of Dosage of the Adsorbent

The experimental results of adsorptions of RB 21 and RR 180 on to the PANI-CuCl₂ with various dosages of adsorbent weights are shown in Fig.-5. It is found that the adsorption is more favorable with the higher amount of adsorbent dosage for both the dyes; incremental rise in the PANI-CuCl₂ composite weight enhances the surface of the composite and increases the active sites available for the adsorption of the dye molecule.²⁶

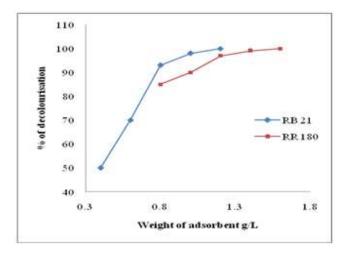


Fig.-5: Effect of Dosage of PANI- CuCl2 on Adsorption

Effect of pH

Experiments were conducted using RR 21 and RR 180 with respective concentrations and the weight of the adsorbent at different pH values and the results are plotted in Fig.-6. Sulphonic acid dyes are ionized in polar solvents like water and the anionic part of dye was moved from solution to the surface of the composite and dye removal occurs due to the electrostatic interaction between oppositely charged dye ions and PANI-CuCl₂ composite. The decrease in the percentage of decolorization at alkaline pH reveals the competition of smaller sized hydroxide ion to the adsorbent site which results in the repulsion of larger sized dye anion.²⁷

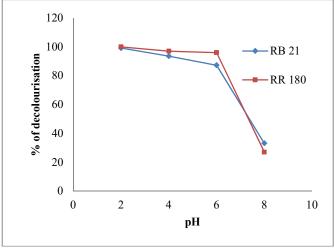


Fig.-6: Effect of pH for RB 21 and RR 180

Effect of Temperature

The study of the effect of temperature on the rate of adsorption indicates that an increase in temperature marginally increases the rate of adsorption of dye molecule on PANI-CuCl₂composite (Fig.-7) which confirms the endothermic nature of adsorption process.²⁸

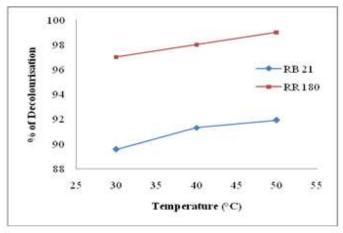


Fig.-7: Effect of Temperature for RB 21 and RR 180

Effect of Agitation Speed

The distribution of the dye molecules on the sites of adsorbent is influenced by an important parameter called agitation speed. Figure-8 shows the adsorption capacity of RB 21 and RR 180 at different agitation speeds. The adsorption efficiency increases almost by a factor of two when the speed of agitation is increased from 50 to 250RPM. The increase in agitation speed reduces the resistance between the dye molecule and the adsorbent.²⁹ If an appropriate speed of agitation is used the mobility of the ion in the

solution is higher and the mass transfer-resistant is lower. The adsorption of RB 21 and RR 180 by PANI-CuCl₂ composite was best studied at 250RPM.

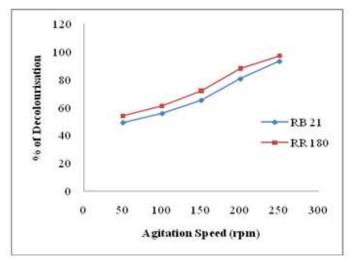


Fig.-8: Variation of Agitation Speed for RB 21 and RR 180

Desorption and Recyclability

The dyes are not desorbed by hydrochloric acid, acetic acid, ethanol and acetone. The desorption trend of RB21 and RR 180 at various strengths of sodium hydroxide was reverse to the adsorption. Increased desorption is observed with an increase in the concentration of sodium hydroxide solution. RB 21 and RR 180 dyes are anionic, removal of dyes in acidic condition and desorption of dye in alkalinity revealed that both dyes are held to PANI-CuCl₂ probably by ion exchange.³⁰ This shows that there is a coulombic attraction between the dissociated dye anion and the positively charged backbone of the composite. Figure-9a and 9b graphically shows the percentage of desorption and percentage of readsorption.

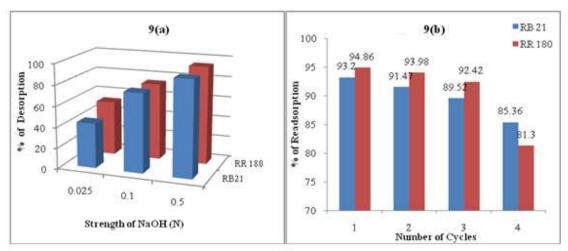


Fig.-9: (a) and (b) Desorption of Recyclability of RB 21 and RR 180

Adsorption Isotherm

The experimental results are evaluated using a linear model of Langmuir and Freundlich isotherms to determine the most fitting correlation for the adsorption system at their equilibrium. The better isotherm model can be predicted from the obtained correlation coefficients values (R^2). The calculated values of Langmuir and Freundlich isotherms are exhibited in Table-1. The results of the analysis are well fitted to the Langmuir model and the plot of C_e versus C_e/q_e is linear (Fig.-10) which shows that the adsorption of

both dyes onto PANI-CuCl $_2$ follows Langmuir adsorption isotherm. The adsorption of RR 180 and RB 21 is found to be a favorable process from the calculated R $_L$ values (0.009 for RB 21 and 0.010 for RR 180). The heterogeneous surface of the adsorbent with irregular distribution of dye molecules is characterized by Freundlich adsorption isotherm (Fig.-11). Values of K $_F$ and n indicate that the adsorption capacity and adsorption intensity respectively. The feasibility of adsorption process can be predicted from the obtained value of 1/n whether it is irreversible (1/n = 0), favorable (0 < 1/n < 1) or unfavorable (1/n > 1). Calculated values of 1/n (0.095 for RB 21 and 0.104 for RR 180) also support the adsorption of RB 21 and RR 180 onto PANI-CuCl $_2$ composite is favourable.

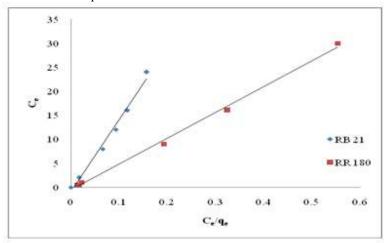


Fig.-10 Langmuir Adsorption Isotherms for RB 21 and RR 180

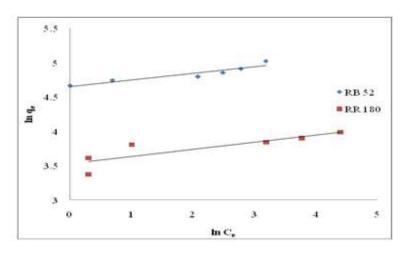


Fig.-11: Freundlich Adsorption Isotherms for RB 21 and RR 180

Table-1: Isotherms and Calculated Parameter Values					
Isotherms	Linear form of	Calculated Values			
	Sotherm Equation				
Freundlich	$\ln a = \ln K + \frac{1}{\ln C}$	RB 21 n= 10.52; $K_F = 105.21$ (L/g)			
	$lnq_e = lnK_f + \frac{1}{72} lnC_e$	RR 180 n= 9.62; $K_F = 34.05 \text{ (L/g)}$			
Langmuir	$C_s = (1 + bC_s)$	RB 21 $Q_0 = 166 \text{ mg/g}$; $b = 1(L/mg)$; $R_L = 0.009$			
8		RR 180 $Q_0 = 55 \text{mg/g}$; $b = 1.63 \text{ L/mg}$; $R_L = 0.01$			
	$q_s = Q_0 b$	2			

Adsorption Kinetics

Kinetics analysis is carried out to assess the model parameters connected with the adsorption process. The plot of t/q_t vs t is a perfect straight line which shows that the adsorption of RB 21 and RR 180 by the adsorbent follows pseudo-second-order kinetics (Fig.-12). The calculated value of q_e from the pseudo-second-order kinetic plot agrees with the experimental data. The comparisons of calculated and experimental values are shown in Table-2.

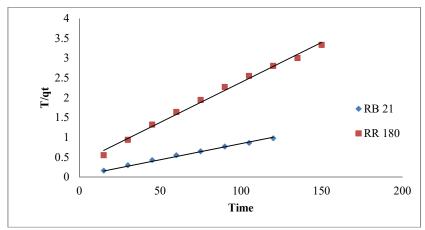


Fig.-12: Pseudo Second Order Model for RB 21 and RR 180

Table-2: Calculated and Experimental Values of RB 21 and RR 180

Dye	q _e (mg/g)	q _e (mg/g)	K ₂	R ²
	Experimental	Calculated		
RB 21	122.5	142.85	5.1×10 ⁻⁴	0.99
RR 180	45	47.61	4.5×10 ⁻⁴	0.99

Adsorption Thermodynamics

Thermochemical quantities like Gibbs free energy ($\Delta G^{\circ}KJmol^{-1}$), enthalpy ($\Delta H^{\circ}KJmol^{-1}$) and entropy ($\Delta S^{\circ}KJmol^{-1}$) are calculated from Van't Hoff equation. ΔH° and ΔS° are computed from the intercept and slope of the plot ln K_d versus 1/T and values are shown in Table-3.

$$ln K_d = - \Delta H^{\circ} / RT + \Delta S^{\circ} / R$$

 K_d is the equilibrium constant. The range of ΔG° for physical adsorption process is from 0 to -20 kJ/mol, and for chemisorptions is -80 to -400 kJ/mol 33 . Thus the adsorption of RB 21 and RR 180 onto PANI – CuCl $_2$ could be considered as physical adsorption. The positive value of ΔS° shows the increase in randomness at the adsorbent-adsorbate interface during the adsorption process. The endothermic process for adsorption of RR 180 and RB 21 onto PANI-CuCl $_2$ composite is indicated by the positive value of ΔH° .

Table-3: Calculated Thermodynamic Parameters and their Outcome Results Thermochemical Calculated Values Calculated Values Outcome Results Quantities for RB 21 for RR 180 -5.965KJ mol⁻¹ -9.587KJ mol⁻¹ ΔG° Spontaneous and -6.430KJ mol⁻¹ -11.684KJ mol⁻¹ feasible in nature -6.955KJ mol⁻¹ -13.942KJ mol⁻¹ ΔS° 18.43KJ mol⁻¹ 4.70KJ mol⁻¹ Entropy driven ΔH° 36.39 KJ mol⁻¹ 59.02 KJ mol⁻¹ Endothermic in nature

Electrical Conductivity

Electrical Conductivity (EC) of adsorbent before and after adsorption of RB 21 and RR 180 were measured. The voltage is measured by increasing the temperature of the system gradually. From Fig.-13, it is visible that the EC of PANI-CuCl₂ decreases after the adsorption for both dyes. This indicates that the dye adsorbed on the polymer composite surface results in a thin layer of low doped material due to the removal of counter ions which contributes to a decrease in electrical conductivity.³⁴

SEM

Scanning electronic micrographic analyses are commonly used to test the morphological and surface properties of the adsorbent substance before and after adsorption. It also shows the surface structure of the adsorbent material. The change in morphology of PANI-CuCl₂ composite is shown in Fig.-14a, b and c which substantiate the adsorption of dyes by the adsorbent.

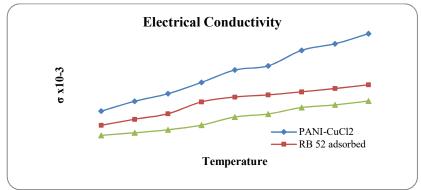


Fig.-13: Electrical Conductivity of PANI-CuCl₂ before and after Adsorption

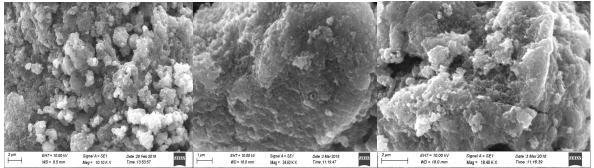


Fig.-14: SEM images of PANI-CuCl₂ before and after Adsorption of RB 21 and RR 180

FTIR Characterization

The stretching vibration of the secondary N-H group is observed at 3434cm⁻¹ for PANI-CuCl₂, after adsorption, it is converted to shoulders with very less intensity for RB 21 and RR 180 respectively. The C=N stretching vibration on PANI-CuCl₂ is observed at 1557 cm⁻¹ which is shifted to 1587.18 cm⁻¹ after vibration. The band at 1293 cm⁻¹ was due to the C-N stretching vibration of aromatic secondary amine was changed to 1299.06cm⁻¹ and 1290.62cm⁻¹. These results reveal that physical forces were involved in adsorption of RB 21 and RR 180 to PANI-CuCl₂ composite. The observed FTIR characterization is shown in Fig.-15a, b and c.

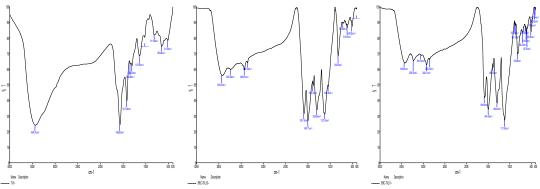
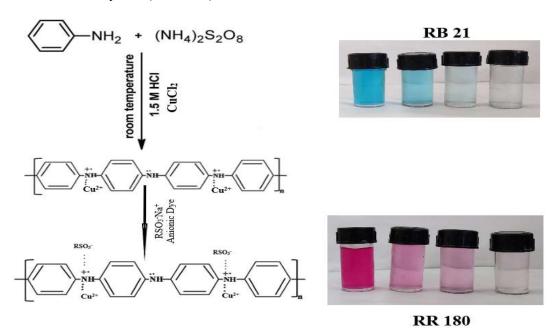


Fig.-15 FTIR Images of PANI-CuCl₂ before and after Adsorption of RB 21 and RR 180

CONCLUSION

This investigation provides evidence that PANI-CuCl₂ can act as a good adsorbent for the adsorption of anionic dyes. An increase in dye concentration increases the equilibrium quantity (q_e) of dye adsorbed whereas an increase in dosage of adsorbent decreases the time required for the removal of dye. Drop-in pH favors the adsorption of dyes and an increase in the speed of agitation also increases the quantity of dye adsorbed. Change in temperature marginally affects the percentage of decolorization. Desorption observed at alkaline pH and the reusability of desorbed adsorbent is excellent. Langmuir isotherm and pseudo-second-order kinetic model describe the adsorption process well. The thermodynamic study indicated that this adsorption is feasible and the type of adsorption is physisorption. The pH of the adsorption-desorption study reveals the electrostatic nature of the interaction between the anionic dye and polyaniline-CuCl₂ composite (Scheme-1).



Scheme-1: Probable binding Mechanism for Adsorption of RB 21 and RR 180

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