

# Adsorption of Reactive Orange 13 (RO13) onto Puffed Rice in Aqueous Solution

T.K Saha, Subir Chowdhury

Department of Chemistry, Jhangirnagar University  
Savar, Dhaka, Bangladesh

**Abstract:-** Adsorption of reactive orange 13(RO13) from aqueous solution onto puffed rice was investigated. Batch system and effects of effective parameters include pH, initial dye concentration, ionic strength and temperature was studied. It was found that maximum adsorption of reactive orange 13 onto puffed rice was at pH2, and extent of adsorption increases with increasing initial dye concentration and increasing temperature whereas the adsorption of RO13 decreases with the increase of ionic strength of the dye solution. Based on the result, it can be said pseudo-second order kinetic model is the best model for explanation of adsorption of RO13 onto puffed rice. The experimental adsorption data best fitted with the Langmuir's isotherm model. Which indicates the adsorption is involved with monomolecular. The thermodynamic parameters such as adsorption enthalpy ( $\Delta H$ ), entropy ( $\Delta S$ ) and free energy ( $\Delta G$ ) were evaluated by applying Van't Hoff equation for the system which indicated the process involved with physisorption. The negative values of free energy ( $\Delta G$ ) determined for this systems indicated that adsorption of Reactive Orange-13 was spontaneous at the temperatures under investigation (303-318 K). Puffed rice could be suggested as relatively efficient and low cost adsorbent for dye removal from textile wastewater.

**Key words-** Adsorption, Physisorption, Dye removal, Isotherm models, Low cost adsorbent.

## I. INTRODUCTION

The textile dyeing industry consume huge amount of water and produce a large amount of wastewater [1]. Generally in dyeing processes, huge amount of the dye is lost in the wastewater. This unused residual dyes remain in the process water. Due to high water solubility and characteristic brightness of the dyes [2] create serious environmental pollution when discharges them without treatment. Different methods have been used for dye removal from wastewater include biological methods and physicochemical methods such as electrocoagulation, membrane filtration, ion exchange, coagulation, , floating, filtration and advanced oxidation [3]-[5]. Most of these technologies are luxurious, on the contrary adsorption methods can be considered the suitable one for its potentiality for application in industrial wastewater treatment, because their efficiency is proven in the removal of organic and mineral pollutants and economic considerations [6]-[7]. Various materials have been used as adsorbents for dye removal including orange and banana peels [8], apple peel

and wood [9]. Still scientists are trying to find new cost effective adsorbent.

In this study puffed rice used as adsorbent to adsorb amaranth from aqueous solution.

## II. EXPERIMENTAL

### A. Materials

Puffed rice was collected from local market and was used without further purification although it was grained to make powder. The size distribution of puffed rice was determined using a laser scattering particle size analyzer (LDSA-2400A, Tonochi Computer Applications Japan) equipped with a dry dispersing apparatus (PD-10S, Tonochi Computer Applications Japan). Sample is a composed of large particles and small ones in diameter which is confirmed by the peaks around 32 $\mu$ m, 53 $\mu$ m and 355 $\mu$ m in frequency (Figure not given). The Reactive orange13 (RO13) was obtained from Sigma-Aldrich Germany and was used without purification. The chemical structure of RO13 is shown in Fig: 1. All other reagents and solvents were highest grade of purity. Deionized water was prepared by passing distilled water through a deionizing column (Branstead, Syboron Corporation, boston. USA).

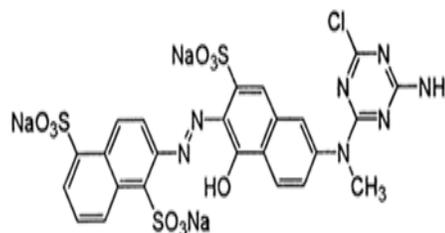


Fig: 1 Chemical structure of Reactive orange 13

### B. Characterization:

Zero point charge ( $\text{pH}_{\text{ZPC}}$ ) of puffed rice was determined. The FTIR spectra of reactive orange13 and puffed rice

before and after reactive orange 13 adsorption were recorded in the frequency range of 400-4000 $\text{cm}^{-1}$  using spectrophotometer (Simadzu, Japan).

### C. Batch adsorption experiments:

Batch sorption studies were carried out by agitating 25 mL of dye solution of the desired concentration and 0.1g of the puffed rice powder in 220 mL reagent bottle. Agitation was performed at room temperature ( $30 \pm 0.2^\circ\text{C}$ ) with shaking machine at a speed of 120r/min at different time intervals. pH of the solution varied from 2 to 8. The dye adsorption was determined spectrophotometrically (UV-160A, Shimadzu, Japan) by monitoring absorbance changes at the  $\lambda_{\text{max}}$  (488.0nm) of reactive orange 13 (RO13). Same method was used for different initial concentration (65- 910  $\mu\text{mole/L}$ ) and ionic strength (0.01M, 0.05M, 0.10M, 0.15M, 0.20M. 88.33 $\mu\text{mol/L}$ ) of dye solutions and temperatures (30, 35, 40 and 45  $^\circ\text{C}$ ) to study adsorption characteristics. The amount of RO-13 adsorbed onto puffed rice  $q_t$  ( $\mu\text{mole/g}$ ) at any time  $t$  and  $q_e$  ( $\mu\text{mole/g}$ ) at equilibrium were determined from the following relationships:

$$q_t = V(C_0 - C_t)/m \quad (1)$$

and

$$q_e = V(C_0 - C_e)/m \quad (2)$$

where  $C_0$  ( $\mu\text{ mole/L}$ ),  $C_t$  ( $\mu\text{ mole/L}$ ),  $C_e$  ( $\mu\text{ mole/L}$ ), are the liquid-phase concentrations of RO-13 at initial, at any time  $t$ , and equilibrium, respectively;  $V$  (L) is the volume of RO-13 solution and  $m$  (g) is the amount of dry puffed rice powder used. Desorption was also performed by producing complex of puffed rice and RO-13 and 0.1g of the complex was taken into 25 mL of 0.1 mole/L of NaOH solution and then performed batch adsorption experiments for 180 min. Reuse process were also performed to study the feasibility of puffed rice using as a good and commercially viable low cost adsorbent.

### D. Determination of point zero charge ( $\text{pH}_{\text{PZC}}$ )

Determination of Point Zero Charge ( $\text{pH}_{\text{PZC}}$ ) was done to determine the surface charge of Puffed Rice. It is important because this information helps us to explain the surface behavior of the adsorbent under certain pH. 0.1 M KCl solution was prepared and its initial pH was adjusted 2 to 8 (0.5 gap) using HCl and NaOH. Then 25ml of that solution was taken in the 125ml reagent bottle and 0.1g of Puffed rice added to each solution. These bottles were kept for 24 h and then final pH of the solutions was measured with a pH meter (410a Orion pH meter). Graphs were plotted between final pH and initial pH. (Fig is not shown) It was found that the  $\text{pH}_{\text{PZC}}$  of puffed rice is 6.

### E. FTIR Analysis

Which ensure that there is some interaction between OH and NH groups of puffed rice and S=O group of reactive orange 13 dye (Fig are not shown).

## III. RESULTS AND DISCUSSIONS

### A. Effect of pH on the adsorption process:

The pH of the dye solution is an important parameter in the adsorption. The effect of pH on the adsorption of RO-13 by puffed rice was studied in aqueous solution within pH range 2-8 as shown in Fig: 2. It was observed that 120 minutes was taken to reach the equilibrium although data was taken for 180 minutes to ensure the attainment of the equilibrium. pH effect showed that the adsorption of RO13 onto puffed rice increases with decreasing pH. It was also observed that the initial rate of adsorption ( $h$ ) of RO-13 is generally increases with decreasing solution pH. Lower initial rate ( $h$ ) of adsorption observed at basic pH values is due to competition between the excess hydroxyl ions and the negatively charged RO-13 ions for the sorption sites. After 180 min. of adsorption, the equilibrium sorption capacity ( $q_e$ ) was found to be 9.67 – 1.69  $\mu\text{mol/g}$  at pH 2-8 (Table-1). These results suggested that the initial rate of adsorption ( $h$ ) as well as the equilibrium adsorption capacity of puffed rice is suitable at pH- 2 among the observed pH ranging from 2-8 and the initial concentration of RO-13 solution was 40 $\mu\text{mol/L}$ . Similar behavior observed in the adsorption of remazol brilliant violet onto puffed rice [11].

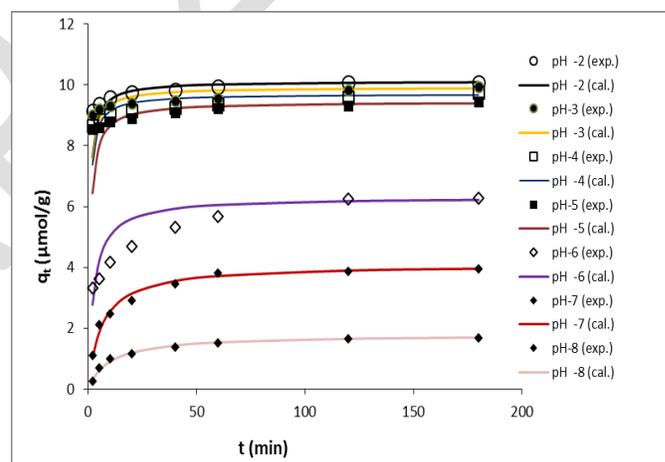


Figure:2 pH effect

### B.. Effects of initial dye concentration on adsorption:

The adsorption studies were carried out at pH 2, 0.1 g of puffed rice and at  $30^\circ\text{C}$  temperature. Results of concentration effect were listed in Table-1. The adsorbent and solution of different initial dye concentrations (65.11– .11 $\mu\text{mol/L}$ ) contacted for 180 minutes. It was found that as the initial dye concentration was increased relative to a fixed sorbent dosage, the extent of adsorption increased.

### C. Effect of salt on dye adsorption:

The effects of ionic concentration on the adsorption kinetics are observed under various ionic strength (0.01M, 0.05M, 0.10M, 0.15M and 0.20M ) of KCl solution and other fixed operating conditions (concentration 88  $\mu$

mole/L, and at pH-2 of the dye solution). The result is shown in Table-1; it indicates that the higher the KCl concentration is, the lower the adsorption capacity. Since the addition of KCl reduces the electrostatic interaction between puffed rice and dye [12].

#### D. Effect of temperature on adsorption kinetics:

Changing the temperature may change the equilibrium capacity ( $q_e$ ) of the adsorbent for a particular adsorbate [13]. A larger amount of RO-13 dye was removed by puffed rice within 20 min of contact time. When the temperature is raised from 30°C to 45°C, the removal RO-13 dye by adsorption onto Puffed rice increased from 15.73 to 15.84  $\mu\text{mole/g}$ , indicated the process is endothermic (Table-1).

#### E. Rate Constant Studies:

In the present study pseudo first-order and pseudo second-order and Elovich models were used to determine the mechanism and rate-controlling steps.

The pseudo-first-order equation can be expressed as

$$(q_e - q_t) = \log q_e - k_1 t / 2.303 \quad (3)$$

The second-order kinetic model can be expressed linearized form as mention bellow

$$t/q_t = 1/k_2 q_e^2 + (1/q_e)t \quad (4)$$

Where symbols are as usual meaning

The Elovich Model is often valid for systems in which the adsorbing surface is heterogeneous. The Elovich model is expressed as

$$q_t = \left(\frac{1}{\beta}\right) \ln \alpha \beta + \frac{1}{\beta} \ln t \quad (5)$$

Where  $\alpha$  is the initial adsorption rate in ( $\mu\text{mole/g min}$ ) and  $\beta$  is related to the extent of surface coverage and the activation energy for chemisorption in ( $\text{g}/\mu\text{mole}$ ). The Elovich coefficients can be calculated from the plot of  $q_t$  vs  $\ln t$ .

It is found that all the experimental data fitted well to the second-order kinetics than that for the pseudo-first-order kinetics model and Elovich model (Table-1).

#### F. Adsorption mechanism:

Intraparticle and film diffusion models are used to determine the rate limiting step. The intraparticle diffusion model equation proposed by Weber and Moris [14].

$$q_t = k_{id} t^{1/2} \quad (6)$$

Film diffusion model equation proposed by McKay [15] can be expressed as :

$$\ln(1-F) = -k_f t \quad (7)$$

$$F = q_t/q_\infty \quad (8)$$

Where  $k_{id}$  ( $\mu\text{mole/g min}$ ) is the diffusion rate parameter in intraparticle diffusion model,  $k_f$  (1/min) is the diffusion rate parameter in film diffusion model,  $F$  is the fractional attainment of the equilibrium and  $q_t$  ( $\mu\text{mole/g}$ ) and  $q_\infty$  ( $\mu\text{mole/g}$ ) are amounts of dye adsorbed after time  $t$  and infinite time respectively.

The values of diffusion kinetic parameters ( $k_{id}$  and  $k_f$ ) and  $R^2$  obtained from applied models are presented in Table-2.

#### G. Adsorption isotherms:

The rise in adsorption capacity is due to the increase in collision frequency between adsorbent and adsorbate, which results in the enhanced adsorption onto the surface of the adsorbent [16]. This enhancement may be due to the creation of new reaction sites or increased rate of intraparticle diffusion of adsorbate molecules into the pores of the adsorbent at higher temperature [17]. Three isotherm models were used at different temperature (30,35,40 and 45°C) to describe the equilibrium characteristic of adsorption and they are Tempkin, Freundlich and Langmuir.

##### Tempkin Model:

$$\text{Nonlinear form } q_e = (RT/b) \ln(K_T C_e) \quad (9)$$

$$\text{Linear form } q_e = RT/b \ln K_T + RT/b \ln C_e \quad (10)$$

Where  $K_T$  ( $\mu\text{mol/L}$ ) is the Tempkin isotherm constant,  $b$  (J/mol) is a constant related to heat of adsorption,  $R$  (8.314J/mol K) is an ideal gas constant and  $T$  is a absolute temperature (K). This isotherm contains a factor that explicitly takes into account adsorbing species-adsorbate interactions. Values obtained from the plot of  $q_e$  vs  $\ln C_e$  are shown in Table-2.

##### Freundlich Model:

$$\text{Nonlinear form } q_e = K_F C_e^{1/n} \quad (11)$$

$$\text{Linear form } \ln q_e = (1/n) \ln C_e + \ln K_F \quad (12)$$

Where  $K_F$  ( $(\mu\text{mol/g})(\mu\text{mol/L})^{1/n}$ ) and  $n$  are Freundlich isotherm constant indicating the capacity and intensity of the adsorption respectively. Values obtained from the plotting of  $\ln q_e$  vs  $\ln C_e$  are listed in the Table-2.

##### Langmuir Model:

$$\text{Nonlinear form } q_e = K_F C_e / (1 + a_L C_e) \quad (13)$$

$$\text{Linear form } C_e/q_e = 1/K_L + (a_L/K_L) C_e \quad (14)$$

Where  $C_e$  ( $\mu\text{mol/L}$ ) is the equilibrium RO-13 concentration in the solution  $K_L$  (L/g) and  $a_L$  (L/  $\mu\text{mol}$ ) are the characteristic constants of the Langmuir equation, and the ratio of  $a_L/K_L$  gives the theoretical maximum adsorption capacity  $q_m$  ( $\mu\text{mol/g}$ ).

Data shows that the Langmuir adsorption isotherm fitted well (Table-2).

Isotherms	Parameters			
Tempkin				
Temperature (°C)	30	35	40	45
K <sub>T</sub> (μmole/L)	13.60	8.09	25.23	12.55
b <sub>T</sub> (J/mole)	0.003	0.003	0.003	0.005
R <sup>2</sup>	0.834	0.876	0.849	0.890
Freundlich				
K <sub>F</sub> ((μmol/g)(μmol/L) <sup>-1/n</sup> )	2.19	2.40	2.69	3.31
b <sub>F</sub> (J/mole)	0.591	0.587	0.574	0.607
N	1.69	1.71	1.74	1.65
R <sup>2</sup>	0.778	0.751	0.714	0.673
Langmuir				
K <sub>L</sub> (L/g)	12.24	15.65	20.53	29.85
a <sub>L</sub> (μmole/L)	0.182	0.216	0.273	0.290
q <sub>m</sub> (μmole/g)	67.11	72.46	75.19	103.09
R <sup>2</sup>	0.999	0.999	0.999	0.999

Table-2

Table-2. Freundlich, Tempkin and Langmuir isotherm constants at different temperatures for the adsorption of RO-13 onto puffed rice from aqueous solution at pH2

#### H. Thermodynamic parameters:

To calculate the values of  $\Delta H$ ,  $\Delta S$  and  $\Delta G$  Van't Hoff equation is used.

$$\Delta G = -RT \ln a_L \quad (15)$$

$$\ln a_L = \Delta S/R - (\Delta H/R)(1/T) \quad (16)$$

The values of  $\Delta H$ ,  $\Delta S$  were calculated from the slope and y-intercept of Van't Hoff plot of  $\ln a_L$  vs.  $1/T$  (Equation-16). Generally a value of  $\Delta H$  in between 5-40 kJ/mol is consistent with electrostatic interaction between adsorption sites and adsorbing ion (physical adsorption) while a value ranging from 40-800 kJ/mol suggests chemisorption [18]. The values of  $\Delta H$  and  $\Delta S$  are presented in Table-3.

Table-3 Thermodynamic parameters

Temperature (K)	303	308	313	318
$\Delta G$ (kJ/mole)	-30.52	-31.45	-32.57	-33.25
$\Delta H$ (kJ/mole)	28.51			
$\Delta S$ (J/mole/K)	194.82			

Table-3

The results show that the changes in enthalpy,  $\Delta H$  for the adsorption of RO-13 by puffed rice were 28.51 kJ/mol. A positive enthalpy change,  $\Delta H$  suggests that the interaction of dye adsorbed by puffed rice is endothermic, while a negative adsorption standard free energy change ( $\Delta G$ ) and a positive standard entropy change ( $\Delta S$ ) indicate that the adsorption reaction is a spontaneous process (Table-3). The positive value of  $\Delta S$  indicates that the randomness

increases at the solid--solution interface during the adsorption of dye onto the puffed rice [19].

#### I. Desorption studies:

Experimental results showed dye desorbed in alkaline water. It means ion exchange mechanism is involved [20] in the adsorption process. Desorption study also confirmed the adsorption process is physisorption.

#### J. Reuse

Reuse study showed that puffed rice can be used as reusable adsorbent.

## IV. CONCLUSION

The amount of Reactive Orange 13 sorbed was found to vary with pH, initial dye concentration, ionic strength and temperature. pH2 is favorable for maximum adsorption. The amount of dye uptake (μmol/g) was found to increase with initial dye concentration and solution temperature, and found to decrease with increase in ionic strength. Rate controlling step of the adsorption process was governed by both of the intraparticle diffusion and film diffusion. The adsorption system studied can be well described by the second-order kinetic model. Thermodynamic activation parameter shows that the process is physisorption in nature. Experimental data were in good agreement with Langmuir isotherm. The negative value of the Gibbs energy ( $\Delta G$ ) change of the adsorption indicates that the adsorption is spontaneous. The positive value of the enthalpy change ( $\Delta H$ ) of the adsorption shows that the adsorption is an endothermic process. Reuse study shows puffed rice can be effectively used as a reusable adsorbent for the removal of Reactive Orange-13 from waste waters.

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Table-1

Table-1 Comparison of calculated and experimental q<sub>e</sub> values and kinetic parameters for the RO-13 adsorption onto puffed rice at various pHs, initial concentrations of RO-13, ionic strength and temperatures.

Parameters	q <sub>e</sub> (ex.) (μmol/g)	Pseudo-first-order kinetic			Pseudo-second-order kinetic			Elovich kinetic model			
		k <sub>1</sub> (min <sup>-1</sup> )	q <sub>e</sub> (cal.) (μmol/g)	R <sup>2</sup>	k <sub>2</sub> (g/μmol/min)	q <sub>e</sub> (cal.) (μmol/g)	h (μmol/g/ min)	R <sup>2</sup>	α (μmole/g/min)	β (g/mole)	R <sup>2</sup>
pH Initial Concentration 41.11 μmole/L											
2	10.08	0.0302	1.27	0.936	0.1504	10.12	16.23	0.999	4.04E+17	4.66	0.980
3	9.94	0.0164	1.17	0.968	0.1566	9.92	15.41	0.999	7.51E+18	5.10	0.957
4	9.75	0.0147	1.06	0.972	0.1633	9.70	15.36	0.999	9.32E+14	4.25	0.985
5	9.44	0.0260	1.07	0.977	0.1136	9.44	10.13	0.999	5.28E+16	4.81	0.978
6	6.29	0.0380	3.60	0.972	0.0623	6.31	2.48	0.997	2.61E+01	1.37	0.994
7	3.96	0.0302	1.96	0.895	0.0400	4.09	0.67	0.999	1.47E+01	1.56	0.969
8	1.69	0.0525	1.38	0.895	0.0559	1.79	0.18	0.999	1.89E+00	3.15	0.970
Initial RO-13 Concentration (μmole/L)(pH-2)											
65.11	16.00	0.0193	1.61	0.803	0.0624	16.03	16.03	0.999	6.56E+08	1.59	0.804
122.22	29.78	0.0550	6.35	0.969	0.0304	29.94	27.25	0.999	6.32E+06	0.67	0.945
188.89	37.67	0.0394	7.07	0.898	0.0197	38.02	28.41	0.999	3.53E+04	0.37	0.897
200.00	36.92	0.0350	6.53	0.935	0.0210	37.17	29.07	0.999	1.76E+05	0.43	0.895
322.22	58.57	0.0184	5.96	0.958	0.0133	58.82	46.08	0.999	3.33E+17	0.78	0.987
911.11	66.67	0.0410	13.62	0.984	0.0063	67.11	53.19	0.999	9.83E+06	0.29	0.961
Ionic Strength (mole/L) Initial Concentration 88.33 μmole/L(pH-2)											
0.01	21.69	0.0497	1.22	0.924	0.2147	21.69	101.01	0.999	6.92E+26	3.12	0.927
0.05	21.64	0.0281	1.90	0.874	0.2093	21.65	98.04	0.999	1.70E+27	3.16	0.836
0.10	21.53	0.0281	1.90	0.874	0.2093	21.55	87.72	0.999	9.33E+24	2.92	0.813
0.15	21.47	0.0329	1.10	0.861	0.1626	21.51	75.19	0.999	7.41E+22	2.71	0.893
0.20	21.44	0.1349	1.18	0.876	0.1349	21.46	62.11	0.999	7.41E+15	1.94	0.819
Temperature (°C) Initial Concentration 65.11 μmole/L(pH-2)											
30	15.95	0.0182	1.58	0.778	0.0619	15.97	15.80	0.9999	8.82E+08	1.62	0.803
35	15.98	0.0159	1.36	0.662	0.0711	15.97	18.15	0.9999	4.06E+09	1.72	0.778
40	16.04	0.0168	1.26	0.683	0.0776	16.03	19.92	0.9999	1.93E+10	1.81	0.750
45	16.06	0.0228	1.12	0.736	0.1054	16.08	27.25	0.9999	2.47E+10	1.81	0.727