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Development of Isotherms to Evaluate Removal of Dyes Using Adsorbents

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Abstract:

In the recent years the water reservoirs are becoming polluted due to rapid industrialization & increased growth in population. The various pollutants contained in industrial wastewater, colour is considered to be very important from the stand point of aesthetics and is stated as a visible pollutant of various industries like dye making, pulp and paper, tanneries, coffee pulping, pharmaceuticals, food processing, electroplating, distilleries, etc., spew out coloured and toxic effluents to the water bodies rendering them murky, dirty and unsuitable for further use.. Most of the colour bearing waste waters have high COD and BOD ratio and affect the stream water used for drinking purpose. In the present study attempts have been made to access the removal efficiency of adsorbents namely Maize Shell and Orange Peel in removing colours Rhodamine-B and Congo Red from synthetic wastewaters. Experiments were carried out by varying the parameters like initial dye concentration, contact time, pH, adsorbent dosage and grades of adsorbents.

1. Introduction

The environmental pollution is becoming the most challenging threat to the human beings as a result of rapid industrialization and growth population throughout the world. Mostly the areas situated around industrial belts are under stress due to the continuous disposal of the untreated wastes from the various industries. The quality of water is continuously deteriorating due to addition of toxic as well as coloured effluents from various industries where different dyes are used (Khattri & Singh, 1998). The industries, which generate coloured effluent, are the textile

and dye manufacturing industries, pulp and paper mills, tanneries, electroplating factories, distilleries, food processing industries etc (Subramanian et al., 1999). Amongst these industries, the textile industry in India is one of the oldest and largest industries in the country. These mills require volumes of water of high purity and generate equally large volumes of coloured wastewater (Vasanthkumar, 2003). The wastewater from the textile industry is known to be strongly coloured, presenting large amount of suspended solids, pH broadly fluctuating, high temperature, besides high chemical oxygen demand (COD). The textile effluents mainly comprise of carbonate, hydroxide, chloride, peroxide, sulphite, nitrite, silicate, oxychloride and sulphide of sodium, sulphuric acid, hydrogen peroxide, bleaching powder, starch gum etc, used in various wet processing units such as desizing, kiering, bleaching, mercerization, dyeing, printing and finishing in the textile industries (Rao et al., 1993). Colour is the first contaminant to be recognized in this wastewater (10-50 mg/l) is highly visible and reduces the light penetrations in water systems, thus causing a negative effect on photosynthesis. Therefore, the removal of the colour from the textile and/or dyeing wastewater is one of the pressing environmental issues. Among the various techniques used for decolourization namely adsorption, membrane filtration, electrokinetic coagulation, ion-exchange method, ozonation and biological treatment. Adsorption has evolved into one of the most effective physical processes for decolourization of textile wastewater due to its low cost, simple design, sludge free operation and superior colour removal (Shivakumar & Murugesan, 2002). It is generally recognized that conventional biological processes are not totally effective for the colour removal. Biological treatment, although very common, requires large land area and is constrained by sensitivity towards diurnal variation as well as toxicity of some chemicals and less flexibility in design and operation (Arunima & Bhattacharyya, 2001). On the other hand, physico-chemical treatment like adsorption is free from afore related problems.

2. Literature Review On Colour

There is an ever-increasing demand for fabrics and food in the country for the rapid expanding population, which is growing at a rate of 11.04 %. Nearly 10-15% of the synthetic textile dyes, used yearly are lost to waste streams and about 20% of these losses enter the environment through effluent from wastewater treatment plant. The disposal of coloured wastes such as dyes and pigments into receiving waters damages the environment, as they are carcinogenic and toxic to humans and aquatic life.

Table 2.1: Waste Generation in Textile Industry (Khatti & Singh, 1999).

Types of Operation	Processing and Wastewater Generation
Slashing	• Yarn is strengthened by loading it with starch or carboxymethyl cellulose.
Desizing	• Desizing is done to remove natural impurities and slashing compounds. • Enzymes are used to hydrolyze the starch. Acids may also be used.
Scouring	• Scouring is done with caustic soda, soda ash, detergents etc. • Almost 50% of the total pollution of the industry is contributed by this operation.
Bleaching	• Bleaching operations use oxidizing chemicals such as peroxides and hypochlorites to remove natural colouring materials.
Mercerizing	• Consists of passing the cloth through 20% caustic soda solution.
Carbonizing	• Hot concentrated acids are used to convert vegetable matter present in wool into loose charred particles which are then dusted.
Dyeing	• Dyeing may be done using different and auxiliary chemicals. Chromium compounds and dye released in the waste are toxic and require special treatment.
Printing	• Thickened dyes along with printing gums are used for printing and subsequent fixation.
Finishing	• Starches, dextrin's, natural and synthetic waxes, synthetic Resins etc are used for the purpose.

Table 1: Waste Generation in Textile Industry (Khatti & Singh, 1999).

3. Quantity of Waste Generated

However, the quantity waste discharged from textile industry depends on many factors connected with the plant and the production method. Typical data extracted from text book are given in Table 2.2. The wastewaters from the textile industry are usually divided into relatively clean water (mostly cooling waters) and polluted effluents arising from production processes, including slightly polluted waters.

Products	Spinning mill and weaving plant, 1/Kg of product	Bleaching mill 1/Kg of products	Dye house 1/Kg of product	Printed fabrics department 1/Kg of product	Dyeing-Bleaching sections 1/Kg of product
Yarn and fabrics	20-40	100-150	50-100	-	150-200
Finished fabrics	-	200-300	150-250	200-350	400-600

Table 2: The Quantity of Wastes Discharged from Cotton Mills (Koziorowaski, 1972)

4. Basic Adsorption Isotherms

Adsorption from aqueous solutions under equilibrium conditions at constant temperature is expressed by adsorption isotherms. Adsorption isotherm is obtained by plotting the amount of solute adsorbed per unit amount of adsorbent (q_e) as a function of the equilibrium constant (C_e) in the bulk solution.

4.1. Freundlich Equation

The Freundlich equation has the following form (Weber, 1972) :

$$\frac{X}{m} = q_e = K_F C_e^{1/n} \dots (1)$$

Where,

$$\frac{X}{m} = q_e = \text{Amount absorbed per unit weight of adsorbent (mg/g)}$$

C_e = Equilibrium concentration (mg/l).

K_F = Adsorption capacity constant.

n = Adsorption intensity constant.

The linearized form of above equation is

$$\text{Log } q_e = \log K_F + \frac{1}{n} \log C_e \quad \dots (2)$$

Equation (2) is used to find K_F and n from experimental data.

4.2. Langmuir Equation

$$\frac{X}{m} = q_e = \frac{Q_0 b C_e}{1 + b C_e} \quad \dots (3)$$

Where,

$$\frac{X}{m} = q_e = \text{Amount adsorbed per unit weight of adsorbent (mg/g)}$$

Q_0 = Amount adsorbed per unit weight of the adsorbent in forming a complete monolayer.

b = constant related to the energy (l/mg)

C_e = Equilibrium concentration of solute in solution (mg/l)

4.3. BET (Branauer, Emmet, Teller) Isotherm Equation

The model is represented by

$$q_e = \frac{b C_e Q_0}{(C_s - C_e)[1 + (b - 1)C / C_s]} \quad \dots (4)$$

Rearranging the above equation to facilitate its application to experimental data, a linear form can be obtained i.e.

$$\frac{C_e}{q_e(C_s - C_e)} = \frac{(b - 1)}{b Q_0} \left(\frac{C_e}{C_s} \right) + \frac{1}{b Q_0} \quad \dots (5)$$

5. Specific Reviews

Ajay Vanjara, (1998) studied the adsorptive capacity of refused derived fuel (RDF) for the decolourisation of wastewater containing methylene blue. The effect of system variables such as concentration, temperature, pH, agitation speed and particle size have been studied. Effect of agitation on the adsorption of methylene blue on RDF was studied at initial concentration of 50 mg/dm³ at agitation speed of 50, 100 and 200 rpm. It is observed that, as the speed of agitation increased from 50 to 200 rpm, rate of colour removal progressively increased. Effect of particle size variation of RDF on adsorption of methylene blue at an initial concentration of 50 mg/dm³ was carried out in a similar way. As the particle size increases from 200-400, 400-600 and 600-850 μm , rate constant increased from 12, 14 and 18. Thus the Author concluded that RDF has considerable potential for removing colouring material from wastewater over a wide range of concentrations. AJbanis, et al., (2000) studied the adsorption removal of commercial dyes in aqueous suspensions of fly ash mixtures with sandy clay loam soil of low organic matter content. The commercial dyes acid orange 7, acid yellow 23, dispersion blue 79, basic yellow 28 and direct yellow 28 were used. Batch and column experiments were carried out at equilibrium conditions for concentrations of dyes between 5 and 60 mg/l. The results showed that the amount of dye adsorbed in batch experiments in soil mixture with 20% fly ash content were up to 53% for acid yellow 7, 44.9% for acid yellow 23, 99.2% for direct yellow 28, 96.8% for basic yellow 28 and 88.5% for disperse blue 79. Alok Mittal and Lisha Kurup, (2006) carried out an operational model for the removal of recovery of a hazardous dye "Acid Red-27 by employing power plant waste material, Bottom Ash" and an agriculture waste product, De-oiled soya. They observed that both adsorbents have been meticulously utilized for the removal as well as recovery of hazardous dye like AR-27. They studied desorption by eluting dilute NaOH through the fixed bed of the adsorbents they columns can be regenerated and a quantitative recovery of the AR – 27 can be activated and the percentage removal of the dye was almost similar in both cases. Anamika Patel, et al., (2001) tried to develop an efficient, alternative biological method which is eco friendly and cost effective. They have carried out experiments by the exploitation of Marine Cyanobacteria for the removal of colour from distillery effluent. Amongst the photosynthetic micro-organisms, the cyanobacteria were found to have tremendous capacity to reduce colour, TDS level, SCV2, Ca⁺², nitrogen, phosphorous, heavy metals, etc. Hence, they can act as a very good scavenger for wastewater treatment. They have used marine cyanobacterial isolates for the reduction of colour from distillery effluents. The colour was reduced upto 98% from the effluent after 20 days of incubation period.

6. Objectives

Thus the topic titled "Development of Isotherms to Evaluate Removal of Dyes Using Adsorbents" was selected for dissertation work with the following objectives

- To carry out batch adsorption studies, to assess the removal efficiency of Rhodamine-B and Congo red by adsorbents namely maize shell and orange peel under varied experimental conditions like initial dye concentration, contact time, pH, adsorbent dosage and grades of adsorbents.
- To study the equilibrium concentration (C_e) of the dyes and amount of dyes adsorbed per unit weight of the adsorbents selected (Q_e).

- To develop adsorption isotherms.

7. Materials and Methodology

Issues like adsorbents used and their preparation, colours tried and their concentrations, pH, contact time, adsorbent dosage details of experimental set up carried out are discussed in this chapter.

7.1. Colours/Dyes Tried (C)

Textile industry wastewaters are highly polluted due to the strong concentration of toxic dyes. Various dyes are used in textile industry. Among them in this work, dyes namely, Rhodamine B and Congo red were used for experimentation.

7.2. Colour Intensity Tried

Synthetic colored solutions of intensity 200, 300 and 400 ppm were used for experimentation.

7.3. Preparation of Dye Solutions

An accurately weighed quantity of Rhodamine B and Congo red dyes were dissolved in double distilled water to prepare stock solutions of concentrations 200, 300 and 400 ppm for both the dyes.

7.4. Adsorbents Used (AD)

Adsorbents namely maize shell and orange peel were used for experimentation. Two grades of each adsorbent were used, grade-I being 90 μ and grade-II being 150 μ grain size to access the efficiency of removal of colors, and the adsorbent dosages used were 10, 25 and 40 mg/l.

7.4.1. Preparation of Adsorbents

Adsorbents were prepared as per the procedure given by researchers and are documented in the further subsections

7.4.1.1. Preparation of Adsorbent: Maize Shell

It was prepared as per the procedure given by Chakrapani, et al., (2008). Locally available Maize shell powder was screened and then washed with distilled water till the supernatant solution became clear. It was then soaked in 10 % HCl acid for 24 hours to remove any further impurities and then rinsed with distilled water till the supernatant solution reached 7 pH. The washed powder was dried at 110^o C in oven and was sieved for two grain sizes of 90 μ and 150 μ and used for experimentation.

7.4.1.2. Preparation of Adsorbent: Orange Peel

It was prepared as per the procedure given by Kannan et al., (2005). Locally available waste peels of oranges were collected, dried and pulverized. Then it was acid digested (4N HNO₃), heated for about 90 min in hot water bath at with 80^o C and washed several times with water to remove acid. Then the peels were dried in oven at 120^o C for 5 hours. It was powdered and sieved to two constant particle sizes viz 90 and 150 μ and used for experimentation.

7.5. Contact Times Tried

To evaluate removal efficiency of color, experiments were conducted with the various contact times namely 10, 25 and 40 minutes.

7.6. ph Value Considered

To evaluate removal efficiency of dyes, experiments were conducted with the various pH values viz, 4, 8 and 12. The pH values were attained by addition of NaOH or HCl.

7.7. Analyses Of Sampies

The samples were analyzed for their colour concentrations using UV-VIS SL-159(Indian model) spectrophotometer. The wavelengths of 543nm for Rhodamine-B and 497nm for Congo red were used to develop calibration curves. The calibration curves developed are shown in fig 3.2 and 3.3. Effluent colour intensities were read from calibration curves corresponding to absorbance records from UV-VIS spectrophotometer.

7.8. Expeimental Setup and Experimentation

Batch adsorption studies were carried out under varied experimental conditions as discussed above. For a predetermined condition of experimentation 1 liter sample was taken in 6 beakers. The beakers were placed in jar test apparatus for required period of contact time and then the sample was filtered through Whatman no.44 filter paper and it was analyzed for final concentration. By knowing colour intensity in influent and effluent, the efficiency was calculated. Also by knowing the efficiency, mg of colour adsorbed per gm of adsorbent was calculated. Results are tabulated and are represented by linear/ bar charts. Based on observations, inferences are drawn.

Further any attempt has been made for development of Isotherms. The Table 3.1 summarizes materials and methodology adopted in the present work.

CONTACT TIME (min)	pH	ADSORBENT DETAILS			COLOURS	
		Name	Grade	Quantity Used (mg/l)	Used	Intensity Tried (mg/l)
10	4	1.Maize shell	I (90µ)	15	1.Rhodamine -B	200
25	8	2.Orange Peel	II (150µ)	45	2.Congo red	300
40	12			70		400

Table 3: Summary of Experimental Setup and Experimentation

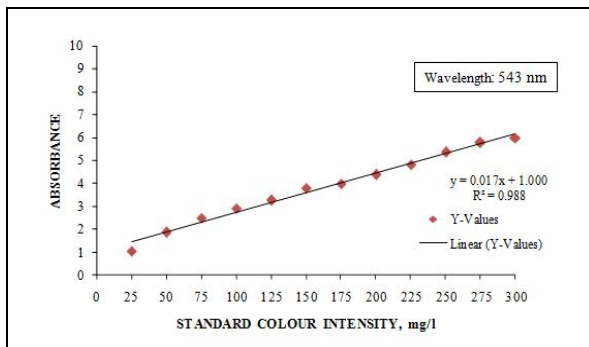


Figure 1: Calibration Curve for Rhodamine-B

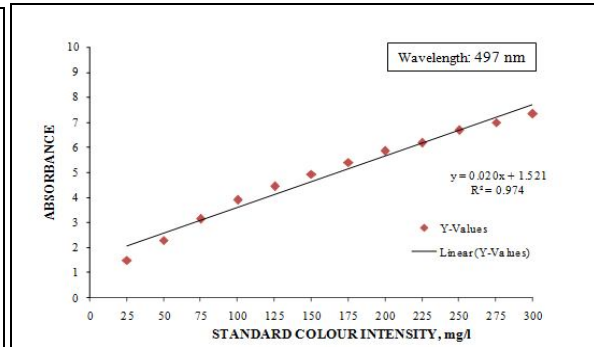


Figure 2: Calibration Curve for Congo red

8. Results and Discussion

An attempt has been made in the present studies to evaluate the removal efficiency of two colours Rhodamine-B and Congo red by two adsorbents namely Maize shell and Orange peel under varied experimental conditions. Effect of pH, particle size of adsorbent, adsorbent dosage and contact time on removal efficiencies has been studied. Results of experimentation are documented. Inferences drawn based on the results are the scope of this chapter.

CONTACT TIME (min)	ADSORBENT DOSAGE (mg/l)	η AT STATED CONDITIONS			
		INITIAL DYE CONCENTRATION			
		C ₀ =200mg/l			
		Absorb- ance	C _e mg/l	η%	Log Q _e
10	15	1.8738	51.4	74.3	3.99
	45	1.63818	37.54	81.23	3.55
	70	1.46206	27.18	86.41	3.39
25	15	1.629	37	81.5	4.03
	45	1.46546	27.38	86.31	3.58
	70	1.26792	15.76	92.12	3.42
40	15	1.46886	27.58	86.21	4.06
	45	1.23086	13.58	93.21	3.61
	70	1.051	3	98.5	3.44

Table 4

CONTACT TIME (min)	ADSORBENT DOSAGE (mg/l)	η AT STATED CONDITIONS			
		INITIAL DYE CONCENTRATION			
		C ₀ =300mg/l			
		Absorb- ance	C _e mg/l	η%	Log Q _e
10	15	2.56111	91.83	69.39	4.14
	45	2.26174	74.22	75.26	3.70
	70	1.95727	56.31	81.23	3.54
25	15	2.33161	78.33	73.89	4.16
	45	2.05927	62.31	79.23	3.72
	70	1.73899	43.47	85.51	3.56
40	15	1.85629	50.37	83.21	4.22
	45	1.5967	35.1	88.3	3.76
	70	1.3468	20.4	93.2	3.60

Table 5

Table 6: RESULTS OF EXPERIMENTATION					
C: Rhoda mine-B, AD: Maize Shell, pH: 4, ϕ : 90micron					
CONTACT TIME (min)	ADSORBENT DOSAGE (mg/l)	η AT STATED CONDITIONS			
		INITIAL DYE CONCENTRATION			
		$C_0=400\text{mg/l}$			
		Absorbance	C_e , mg/l	$\eta\%$	Log Q_e
10	15	3.49696	146.88	63.28	4.22
	45	3.0842	122.6	69.35	3.78
	70	2.75712	103.36	74.16	3.62
25	15	3.15968	127.04	68.24	4.26
	45	2.75236	103.08	74.23	3.81
	70	2.35116	79.48	80.13	3.66
40	15	2.61636	95.08	76.23	4.30
	45	2.21312	71.36	82.16	3.86
	70	1.84592	49.76	87.56	3.69

Table 6

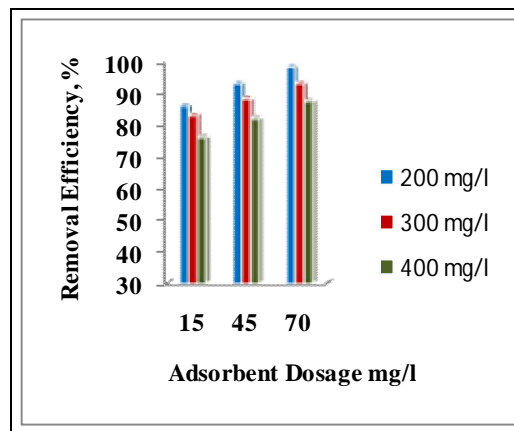


Figure 3: Effect of Adsorbent Dosage on Removal Efficiency (Colour – Rhodamine B, Adsorbent - Maize Shell, Contact Time - 40 min, pH - 4, Size - 90 μ)

Table 7 RESULTS OF EXPERIMENTATION					
C: Congo red, AD: Maize Shell ,pH: 4, ϕ : 90micron					
CONTACT TIME (min)	ADSORBENT DOSAGE (mg/l)	η AT STATED CONDITIONS			
		INITIAL DYE CONCENTRATION			
		$C_0=200\text{mg/l}$			
		Absorbance	C_e , mg/l	$\eta\%$	Log Q_e
10	15	3.0694	77.42	61.29	3.91
	45	2.8498	66.44	66.78	3.47
	70	2.5914	53.52	73.24	3.32
25	15	2.7158	59.74	70.13	3.97
	45	2.5118	49.54	75.23	3.52
	70	2.2522	36.56	81.72	3.3
40	15	2.4526	46.58	76.71	4.00
	45	2.1634	32.12	83.94	3.57
	70	1.9486	21.38	89.31	3.40

Table 7

Table 8: RESULTS OF EXPERIMENTATION					
C: Congo red, AD: Maize Shell ,pH: 4, ϕ : 90micron					
CONTACT TIME (min)	ADSORBENT DOSAGE (mg/l)	η AT STATED CONDITIONS			
		INITIAL DYE CONCENTRATION			
		$C_0=300\text{mg/l}$			
		Absorbance	C_e , mg/l	$\eta\%$	Log Q_e
10	15	4.134	130.65	56.45	4.05
	45	3.7728	112.59	62.47	3.61
	70	3.4902	98.46	67.18	3.45
25	15	3.7488	111.39	62.87	4.09
	45	3.4866	98.28	67.24	3.65
	70	3.1314	80.52	73.16	3.49
40	15	3.5304	100.47	66.51	4.12
	45	3.1938	83.64	72.12	3.68
	70	2.8524	66.57	77.81	3.52

Table 8

Table 9: RESULTS OF EXPERIMENTATION
C: Congo red, AD: Maize Shell, pH: 4, ϕ : 90micron

CONTACT TIME (min)	ADSORBENT DOSAGE (mg/l)	η AT STATED CONDITIONS			
		INITIAL DYE CONCENTRATION			
		$C_0=400\text{mg/l}$			
		Absorbance	C_e mg/l	$\eta\%$	Log Q_e
10	15	5.4402	195.96	51.01	4.13
	45	5.0874	178.32	55.42	3.69
	70	4.5954	153.72	61.57	3.54
25	15	5.1906	183.48	54.13	4.15
	45	4.7442	161.16	59.71	3.72
	70	4.2634	137.12	65.72	3.5
40	15	4.9314	170.52	57.37	4.18
	45	4.4402	145.96	63.51	3.7
	70	3.9282	120.36	69.91	3.60

Table 9

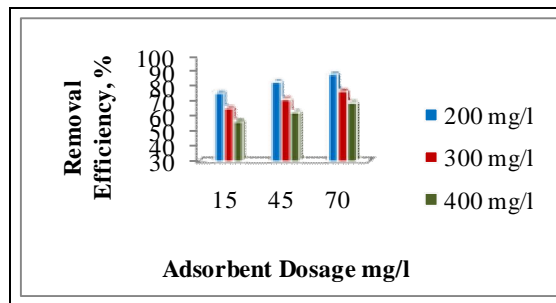


Figure 4: Effect of Adsorbent Dosage on Removal Efficiency (Colour – Congo red, Adsorbent - Maize Shell, Contact Time - 40 min, pH - 4, Size-90 μ)

9. Development of Isotherms

Isotherms development for optimized condition of removal efficiency based on data given in below table-4.25. The data obtained from the batch type adsorption experiment were fitted with the Langmuir and Freundlich adsorption isotherms, respectively by plotting the values of (i) (C_e/Q_e) against C_e and (ii) $\text{Log } Q_e$ against $\text{Log } C_e$.

Initial Concentration C_0 mg/l	Equilibrium Concentration C_e mg/l	Removal Efficiency %	Log Q_e	Log C_e	C_e/Q_e
200	3	98.5	3.44	0.477	2.43
300	20.4	93.2	3.60	1.309	15.93
400	49.76	87.56	3.69	1.696	38.27

Table 10: Effect of Initial Concentration on Removal of Rhodamine-B by Maize Shell (C- Rhodamine-B, AD-Maize shell, pH-4, size-90 μ , Contact time-40 min, Dosage-70 mg/l)

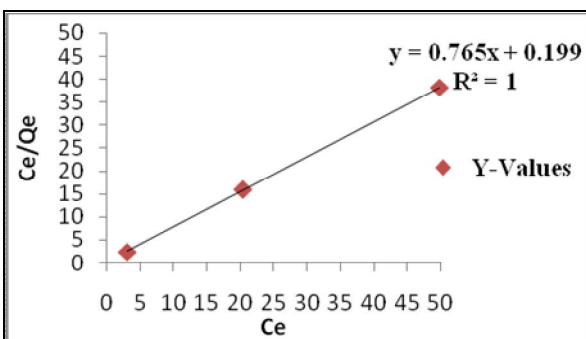


Figure 5: Langmuir Isotherm for Adsorption Rate of Rhodamine-B Dye on Maize Shell

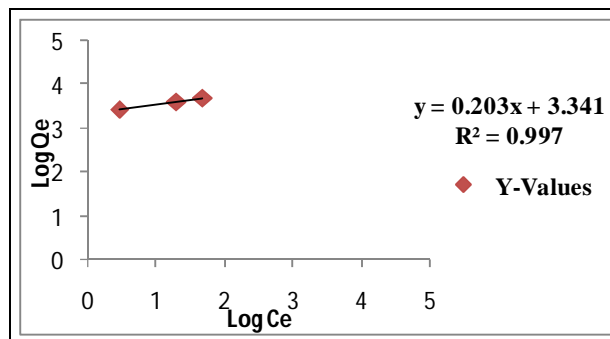


Figure 6: Freundlich Isotherm for Adsorption Rate of Rhodamine-B Dye on Maize Shell

The Langmuir and Freundlich adsorption isotherms are found to be well fitted. When compared with R^2 values of both isotherms, the value of R^2 for Langmuir isotherm is greater when compared to Freundlich isotherm. Hence Langmuir isotherm is best fitted.

10. Conclusion

Based on the results of experimentation carried out under varied experimental conditions and the analysis of the same thereby the following conclusions have been drawn. 1) It is concluded that the size of adsorbent have greater influence on removal efficiency. The better removal efficiency can be achieved with smaller size of adsorbent particles. 2) Contact time and adsorbent dosage found to have direct influence on removal efficiency. On other hand inverse relationship between removal efficiency and pH and initial concentration of dye where observed. 3) The highest removal efficiency of 98.5 % ($C_0 = 200$ mg/l, $T = 40$ min, $pH = 4$, Dosage = 70 mg/l) for Rhodamine-B and 89.31 % ($C_0 = 200$ mg/l, $T = 40$ min, $pH = 4$, Dosage = 70 mg/l) for Congo red with maize shell adsorbent have been recorded. 4) Minimum removal efficiency of 36.23 % ($C_0 = 400$ mg/l, $T = 10$ min, $pH = 12$, Dosage = 15 mg/l, size = 150 micron) for Rhodamine-B and 26.27 % ($C_0 = 400$ mg/l, $T = 10$ min, $pH = 12$, Dosage = 15 mg/l, size = 150 micron) for Congo red with orange peel adsorbent have been recorded. 5) Comparison of removal efficiencies of Rhodamine-B and Congo red colours by maize shell and orange peel adsorbents, it was found that maize shell adsorbent having greater removal efficiency when compared to orange peel. 6) The Langmuir and Freundlich adsorption isotherms are found to be well fitted. Langmuir isotherm is found to be best fitted. 7) Low cost waste materials like maize shell and orange peel may be use to develop effective adsorbents and the adsorbent may be successfully employed for the removal of colours from the coloured effluents. From both of these maize shell is more economical.

11. Limitations of Present Study

The following are the limitations of present study and these limitations are attributed to non-availability of data, lack of infrastructure facility, time shortage, not within the preview of objectives of present study etc. 1) The experiments have been carried out only for set of variables. Confined conclusions can be drawn based only on the results of wide ranges of variables. 2) Refined optimization of variables of experimentation could not be carried out.

12. Scope for Further Study

- The optimization of variables can be taken up for further study.
- Comparative studies to compare coagulation and columnar studies in removing various colours by various adsorbents can be taken up.

13. References

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