# REMOVAL OF CONGO RED DYE ONTO COCONUT (Cocos nucifera) SHELL AND BAEL (Aegle marmelos) EXTRACTS USING TAGUCHI APPROACH

A Project submitted to the National Institute of Technology, Rourkela

In partial fulfillment of the requirements of

**Bachelor of Technology (Chemical Engineering)** 

by

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#### **CERTIFICATE**

This is to certify that the thesis entitled "REMOVAL OF CONGO RED DYE ONTO COCONUT (Cocos nucifera) SHELL AND BAEL (Aegle mammelos) EXTRACTS USING TAGUCHI APPROACH" to National Institute of Technology, Rourkela is a record of bonafide project work under my supervision and is worthy for the partial fulfillment of the degree of Bachelor of Technology (Chemical Engineering) of the Institute. The candidate has fulfilled all prescribed requirements and the thesis, which is based on candidate's own work, has not been submitted elsewhere.

Supervisor Dr. A Kumar Department of Chemical Engineering National Institute of Technology Rourkela - 769008 INDIA **ACKNOWLEDGEMENT** 

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#### **Abstract**

In the present paper adsorption technique was employed for removal of Congo-Red dye. Congo-Red dye is a carcinogenic dye which comes in the effluents of textile industries during dyeing and rinsing processes. The adsorption of Congo red from solution was carried out using low cost adsorbents like coconut (*Cocos nucifera*) shell extract and bael (*Aegle marmelos*) fruit extract with different contact times, temperatures, and pHs. .2 gm of adsorbent was taken in the dye solution for the adsorption process. The Taguchi experimental design method was applied for the systematic and effective investigation to determine the optimal conditions of the operation variables. In case of coconut it was observed that pH is the main factor for determining the rate of adsorption while in case of bael extract it was found to be time and bael showed characteristics of being an endothermic adsorbent.

Keywords: Adsorption, Coconut (Cocos nucifera) shell, Bael (Aegle marmelos), Taguchi method

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# CHAPTER-1

# INTRODUCTION

#### 1. INTRODUCTION

The introduction of waste products in the environment is a world wide problem that has been highlighted by various environmentalist groups. Dyes are widely used in industries such as textiles, rubber, paper, plastics, cosmetics, etc., to colour their products. The dyes are invariably left as the major waste in these industries. Due to their chemical structures, dyes are resistant to fading on exposure to light, water and many chemicals and, therefore, are difficult to be decolourised once released into the aquatic environment (Sharma et al,2008). Many of the organic dyes are hazardous and may affect aquatic life and even the food chain (sheikh et al, 2009). Release of these dyes in water stream is aesthetically undesirable and has serious environmental impact. Due to intense color they reduce sunlight transmission into water hence affecting aquatic plants, which ultimately disturb aquatic ecosystem; in addition they are toxic to humans also.

congo red dye (1- Naphthalenesulfonic acid, 3, 3'-(4, 4' biphenylene bis (azo) bis 4-amino) di sodium salt) is a benzedene based dye, known to metabolize to benzedene, a known human carcinogen and can also cause allergic dermatitis and skin irritation. The release and accumulation of dyes in suspension solution form in inland waters from tanneries, textile, paper and other industries produce tremendous chemico-azo stress on aquatic organism including fishes and some time results in their mass mortality. The removal of dyes from industrial waste before they are discharged into the water bodies is therefore very important from health and hygiene point of view and for environmental protection.

Methods for treating textile dye wastewaters consist of various chemical, physical and biological processes. These include: adsorption (Bilal et al., 2004), nanofiltration (Chakraborty et al., 2003; Capara et al., 2007), colloidal gas aphrons (Roy et al., 1992), ultrasonic decomposition (Jiantuan et al., 2003), electro coagulation (Alinsafi et al., 2004), coagulation and precipitation (Liu et al., 2003), advanced chemical oxidation (Arslan et al., 2000), electrochemical oxidation (Torres et al., 2010), photo-oxidation (Patricia et al., 2003), predispersed solvent extraction (Mahmoud et al., 2007), ozonation (Mohammed et al., 2007), supported liquid membrane (Mahmoud et al., 2007) and liquid-liquid extraction (Venkateswaran et al., 2005).

Natural materials that are available in abundance, or certain waste products from industrial or agricultural operations, may have great potential as an inexpensive sorbents. Due to their low cost, after these materials have been expended, they can be disposed of without expensive regeneration. The abundance and availability of agricultural by-products make them good sources of raw materials for activated carbons. Many carbonaceous materials such as bark, coal, lignite, coconut shells, wood, dead biomass, seaweed, pecan shell and peat are used in the production of commercial activated carbons.

The adsorption process has been widely used for colour removal. Adsorption is one of the processes, which besides being widely used for dye removal also has wide applicability in wastewater treatment. This process being very low cost method and most simple method for containing these pollutants is being used in this study. The main objectives of this study were to determine the effectiveness and feasibility of some low cost agricultural waste materials (coconut shell and bael extracts) in the removal of congo red dye. The study included investigation of effect of Hydrogen ion concentration (pH), contact time and temp of experimental conditions.

### CHAPTER-2

# LITERATURE REVIEW

#### 2. LITERATURE REVIEW

Color is the first contaminant to be recognized in wastewater and has to be removed before discharging into water bodies or on land. The removal of color from wastewater is often more important than the removal of the soluble colorless organic substances, which usually contribute the major fraction of the biochemical oxygen demand. Color is the first contaminant to be recognized in wastewater and has to be removed before discharging into water bodies or on land. The removal of color from wastewater is often more important than the removal of the soluble colorless organic substances, which usually contribute the major fraction of the biochemical oxygen demand. Many methods have been reported for removing textile dyes from wastewater, among which are membrane filtration, coagulation/flocculation, precipitation, flotation, adsorption, ion exchange, ion pair extraction, ultrasonic, mineralization, electrolysis, advanced Oxidation and chemical reduction. Biological techniques include bacterial and fungal biosorption and biodegradation in aerobic, anaerobic or combined anaerobic/aerobic treatment processes. The use of one individual process may often not be sufficient to achieve complete decolorization. Also the economic condition of the process must be taken into account as we can't spend a lot of money in clearing. So an economic method is also required.

Abdul et al., (2002) studied removal of dye from waste water of textile industry using membrane technology. With the fabrication of a suitable experimental rig, the effect of dye concentration, dye pH, and operating pressure were studied. Data on the flux and rejection together with the average values for each of the parameters studied were presented systematically. The results show that the dye concentration, pH of dye, and the operating pressure was found to affect the filtration process.

Sharma et al., (2008) studied removal of congo red dye from the effluents of textile industry using rice husk carbon activated by steam. The adsorbent was made from rice husk and was investigated under variable system parameters such as agitation time and dose of adsorbent. An amount of 0.08 g/l of RHCAS could remove 10 to 99 % of the dye from an aqueous solution of 25 ppm with the agitation time increasing from 20 min to 200 min. The interactions were tested for both pseudo first- order and second – order kinetics and it was observed that the interactions could be better explained on the basis of first order kinetics.

Bilal Acemio glu (2003) studied Adsorption of Congo red from aqueous solution onto calciumrich fly ash with different contact times, concentrations, temperatures, and pHs The adsorption

was between 93 and 98% under the conditions studied. Kinetic studies showed that the adsorption process obeyed the pseudo-second-order kinetic model From thermodynamic studies, he observed that the adsorption was spontaneous and endothermic. Desorption studies suggested that desorption was 29.18% in the presence of 0.1 N HCl and was 47.21% in the presence of CH3COOH (50% v/v). This indicated that most of the dye was held by fly ash via chemisorption as well as ion exchange.

Senthilkumaar et al., (2005) studied Adsorption of dissolved Reactive red dye from aqueous phase onto activated carbon prepared from agricultural waste i.e. Coconut tree flower carbon and Jute fibre carbon Quantitative removal of Reactive red dye was achieved at strongly acidic conditions for both the carbons studied. The overall rate of dye adsorption appeared to be controlled by chemisorption, in this case in accordance with poor desorption studies

Chatterjee et al., (2006) studied removal of congo red by chitosan hydrobeads. Adsorption process has been found to be dependant on temperature with optimum activity at 30 °C. Both ionic interaction as well as physical forces is responsible for binding of congo red with chitosan.

Mittal et al., (2009) studied removal of Congo red from wastewater using waste materials like bottom ash and deoiled soya and recovery by desorption. Column operations depicted good adsorptive tendencies for Congo red with 96.95% and 97.14% saturation of dye on bottom ash and deoiled soya, respectively. Regeneration of the saturated columns has been made by eluting NaOH solution and more than 90% dye has been recovered in both cases.

Bhattacharyya et al., (2004) attempted removal of congo red from solutions using Azadirachta indica leaf powder (neem leaf) as adsorbant. An amount of 0.6 g of the Neem leaf powder (NLP) per litre could remove 52.0–99.0% of the dye from an aqueous solution of concentration 2.87 £ 1022 mmol 121 with the agitation time increasing from 60 to 300 min. The results point to the effectiveness of the Neem leaf powder as a biosorbent for removing dyes like Congo Red from water.

Kamel et al. (2009) decoloured congo red dye soln by direct UV photolysis at 254 nm and at pH 6.6 of the solution. It has been observed, that decolouration process of Congo red solutions, became slow when concentration increased in the medium for the lowest concentration (10 ppm), about 70% of dye decolourization was reached after 7 h of irradiation. Improvement of this process was obtained when systems like  $H_2O_2/UV$  and  $S_2O_8^{2-}/UV$  were used.

Khadhraoui et al. (2009) investigated the degradation and mineralization of Congo red, in aqueous solutions using ozone Phytotoxicity and the inhibitory effects on the microbial activity ofthe raw and the ozonated solutions were also carried out with the aim of water reuse and environment protection. From his experiment he concluded that ozone by itself is strong enough to decolorize these aqueous solutions in the early stage of the oxidation process.

Hoda et al. (2006) studied removal of acid dyes (Acid Blue 45, Acid Blue 92, Acid Blue 120 and Acid Blue 129) from aqueous solutions by adsorption onto activated carbon cloth. It was found that the adsorption process of these dyes onto ACC follows the pseudo-second-order model.

Vimonsesa et al. (2009) performed Kinetic study and equilibrium isotherm analysis of Congo Red adsorption by clay materials (bentonite, kaolin and zeolite). Thermodynamic investigations showed that the adsorption is an exothermic and spontaneous process. Sodium bentonite demonstrated the best adsorptive capacity followed by kaolin clay, and they can be employed as low-cost alternatives for recalcitrant dye removal from industrial wastewater.

Yuzhu et al. (2002) investigated removal of Congo red, from an aqueous solution bybiosorption on dead fungus, *Aspergillus niger* Pretreatment with NaHCO<sub>3</sub> was found to be the most effective with a biosorption capacity of 14.72 mg/g compared with 12.10 mg/g of living biomass for Congo Red. The initial pH of the dye solution strongly affected the chemistry of both the dye molecules and fungal biomass in an aqueous solution .This study showed that it is possible to develop systems for dye removal using *A. niger* biomass which occurs as a byproduct in waste streams of fermentation industries.

Tor et al. (2006) studied removal of congo red (CR) anionic dye, from water by using the acid activated red mud in batch adsorption experiments. They observed the pH of the dye solution strongly affected the chemistry of both the dye molecules and activated red mud in an aqueous solution.

Zhenhu et al. (2010) attempted removal of congo red dye using cattail root as adsorbent Removal efficiency increased with increase of cattail root dosage and ionic strength, but decreased with increase of temperature revealing that the removal of CR from aqueous solution by cattail root was a spontaneous and exothermic adsorption process.

Namasivayam et al. (2002) Removal of Congo Red from water by adsorption onto activated carbon prepared from coir pith, an agricultural solid waste. The adsorption capacity was found to

be 6.7 mg dye per g of the adsorbent. Acidic pH was favourable for the adsorption of Congo Red. Desorption studies suggest that chemisorption might be the major mode of adsorption.

Purkait et al. (2007)attempted Removal of congo red using activated carbon and its regeneration. The zero point of charge of the activated carbon is found about 6.6. About 90% dye is removed for initial concentration of 50 and 100 mg/L, it is about 80% at pH 7.0. Maximum adsorption (about 100%) of dye is observed at pH 2.0 for the concentration range studied here.

Mohan et al. (2002)studied removal of dyes from waste water using flyash as adsorbent .They found that adsorption increased with increase in temp which indicated that the process was endothermic in nature.

# CHAPTER-3

### MATERIALS AND METHODS

#### 3. MATERIALS AND METHODS

#### 3.1 Adsorbate

Congo red, is an anionic azo dye having IUPAC name as 1-napthalenesulfonic acid, 3,3-(4,4-biphenylenebis(azo))bis(4-aminodisodium). Its stock solution was prepared in double-distilled water. All the test solutions were prepared by diluting the stock with double-distilled water.

Table 3.1.1 Physicochemical properties of the dye Congo red.

Parameter	Values
Molecular weight	696.68
Molecular formula	$C_{32}H_{22}N_6Na_2O_6S_2$
Absorption maxima	498 nm

1gm of Congo red was dissolved in 1L of double distilled water to obtain stock solution. Later it was diluted by using distilled water according to the concentration required and pH was adjusted by adding .1 M NaOH soln and .1 M HCl soln according to the conditions

$$NH_2$$
 $NH_2$ 
 $N \ge N$ 
 $N \ge N$ 
 $NH_2$ 
 $NH_2$ 
 $NH_2$ 
 $NH_2$ 
 $NH_2$ 

Figure 3.1.1: Structure of congo red

#### 3.2 Adsorbent

Both adsorbent materials, used in the present research work, are waste materials and available easily and abundantly i.e. Coconut (Cocos nucifera) shell and bael(*Aegle marmelos*) fruit extracts used in this experiment were obtained from local sources. The waste materials were washed with tap water to remove soil and dust, sprayed with distilled water, and dried to a constant weight at 105 °C. These extracts were grounded and sieved to obtain particle sizes of 0.25–0.40mm as adsorbent, and then stored in desiccators for use.

#### 3.3 Batch Adsorption Study

The batch adsorption was carried in 250 ml Borosil conical flasks by mixing a pre-weighed amount of the adsorbent with 100 ml of aqueous dye solution of a particular concentration. The conical flasks were kept on a magnetic shaker and were agitated for a pre-determined time interval at a constant speed. The system parameter such as adsorbent amount, agitation time and temperature were controlled during the experiments. After adsorption was over, the mixture was allowed to settle for 10 min. The dye remaining unadsorbed was determined spectrophotometrically. The adsorption experiments were carried out under the following conditions:

• Initial conc. of congo red solution (ppm): 100 ppm

• Amount of adsorbent (gm/l): 0.2 gms

• PH 2,7,12

• Agitation time (min.): 5 mins, 100 mins, 300 mins

• Temperature  $40^{\circ}\text{C}$ ,  $50^{\circ}\text{C}$ ,  $60^{\circ}\text{C}$ 

#### 3.4 Adsorption Studies

Experiments were performed to study the effects of important parameters such as effect of Temperature, amount of adsorbent, time of contact and pH. For this, 50 ml of dye solutions was taken in 250 ml airtight volumetric flasks with .2 gm of adsorbents. The flasks were then subjected to intermittent shaking for proper adsorption. After the fixed time of contact these solutions were filtered with filter (whatman filter size  $0.45 \,\mu m$  PES filter media) and the amount of the dye adsorbed was analyzed at  $\lambda_{max}$  498 nm. In order to determine the uptake of the dye, an entire set of experiments was performed at different time of contact (5, 100 and 300 mins).

temperatures (40, 50, and 60°C) and pH (2, 7, 12) etc. for both adsorbent materials. The amount of Congo red uptake by coconut shell extract and bael extract after each experiment was measured was calculated using the following equation:

$$q = \frac{C_0 - V_e}{W}$$
 (3.1)

where q is the amount of congo red adsorbed by adsorbents (mg/g);  $C_0$  and  $C_e$  are the initial and final dye concentrations (mg/l), respectively, V is the volume of solution (l) and W is the adsorbent weight (g).

#### 3.5 Analytical determination of CR

CR in the aqueous solution was analyzed using UV spectrophotometer (. A standard solution of the CR was scanned to determine the wavelength ( $\lambda_{max}$ ) corresponding to maximum absorbance. The wavelength corresponding to maximum absorbance was 498 nm.

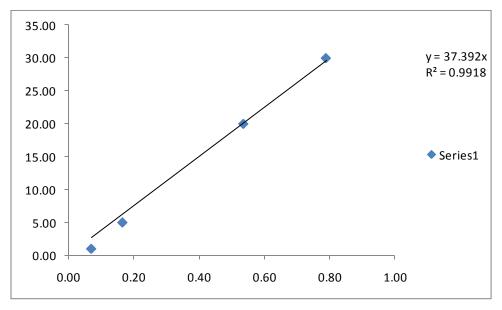


Figure 3.5.1: Calibration curve for congo red

#### 3.6 Taguchi method

Taguchi defined the quality of a product, in terms of the loss imparted by the product to the society from the time the product is shipped to the customer. Some of these losses are due to deviation of the product's functional characteristic from its desired target value, and these are called losses due to functional variation. The uncontrollable factors which cause the functional characteristics of a product to deviate from their target values are called noise factors, which can

be classified as external factors (e.g. temperatures and human errors), manufacturing imperfections (e.g. unit to unit variation in product parameters) and product deterioration.

The overall aim of quality engineering is to make products that are robust with respect to all noise factors. The most important stage in the design of an experiment lies in the selection of control factors. As many factors as possible should be included, so that it would be possible to identify non-significant variables at the earliest opportunity. Taguchi creates a standard orthogonal array to accommodate this requirement. Taguchi used the signal-to-noise (S/N) ratio as the quality characteristic of choice. S/N ratio is used as a measurable value instead of standard deviation due to the fact that as the mean decreases, the standard deviation also decreases and vice versa. In less technical terms, signal-to-noise ratio compares the level of a desired signal (such as music) to the level of background noise. The higher the ratio, the less obtrusive the background noise is. "Signal-to-noise ratio" is sometimes used informally to refer to the ratio of useful information to false or irrelevant data in a conversation or exchange. In other words, the standard deviation cannot be minimized first and the mean brought to the target (ghani et al, 2004.). Taguchi has empirically found that the two stage optimization procedure involving S/N ratios indeed gives the parameter level combination, where the standard deviation is minimum while keeping the mean on target. This implies that engineering systems behave in such a way that the manipulated production factors can be divided into three categories:

- 1. Control factors, which affect process variability as measured by the S/N ratio.
- 2. Signal factors, which do not influence the S/N ratio or process mean.
- 3. Factors, which do not affect the S/N ratio or process mean.

In practice, the target mean value may change during the process development. Two of the applications in which the concept of S/N ratio is useful are the improvement of quality through variability reduction and the improvement of measurement. The S/N ratio characteristics can be divided into three categories when the characteristic is continuous:

Nominal is the best characteristic:  $\frac{s}{N} = -10 \log \left(\frac{\ddot{y}}{s^2}\right)$  (3.2)

Smaller the better characteristics: 
$$\frac{s}{N} = -10 \log_{\frac{1}{N}}^{\frac{1}{N}} (\underline{\Sigma} \mathbf{y}^2)$$
 (3.3)

Larger the better characteristics: 
$$\frac{s}{N} = -10\log(1/n)(\frac{1}{v^2})$$
 (3.4)

where .y is the average of observed data,

 $s^2$  the variance of v,

*n* the number of observations,

and y the observed data.

For each type of the characteristics, with the above S/N ratio transformation, the higher the S/N ratio the better is the result.

#### 3.6.1 Design of experiments with Taguchi orthogonal array (L9 array)

In this study, the experiment design is based on Taguchi method to determine the influence of control factors and an optimal condition to maximize the uptake of dye. In this experiment three control factors were taken into account i.e. Time, Temp, pH and were repeated on 3 levels. So L9 orthogonal array was used to form different combinations of factors which should be conducted in order to study the main effects and interactions (Jafari et al, 2008.). The following table shows the various parameters selected and their respective levels in the present study:

Factor	Level 1	Level 2	Level 3
рН	2	7	12
Temperature(°C)	40	50	60
Time(mins)	5	100	300

Table 3.6.1: Various parameters selected and their respective levels

#### 3.6.2 Analysis of variation (ANOVA)

ANOVA was performed in order to see whether the process parameters are statistically significant or not. The results of ANOVA are listed in table. The row which is marked as error refers to the error caused by uncontrollable factor (noise). In general the values should be below 50 % .otherwise the results would not be reliable (Gonder et al., 2010). F ratio is a tool to indicate which parameter has a significant effect on the uptake of dye due to the control factors. The larger the F ratio the greater is the uptake. The use of F-ratio in an ANOVA is only helpful for the qualitative evaluation of factorial effects .Qualitative evaluation can be achieved with using percent contribution (p %).

#### 3.6.3 Data analysis

The objective of experiment is to optimize the adsorption parameters to get better dye removal, the larger the better characteristics was used. Table 2 and 5 shows the actual data for pH, time and temp along with their computed S/N ratio. Whereas Tables 3 and 6 shows the mean S/N ratio for each levels of pH, time and temp respectively and arranged according to its rank obtained from its S/N ratio. These data were then plotted as shown in graphs 2 and 3 respectively.

# CHAPTER-4

### RESULTS AND DISCUSSIONS

#### 4. RESULTS AND DISCUSSIONS

### 4.1 Results from Adsorption onto coconut shell using Taguchi analysis Main Effects Plot for S/N Ratios

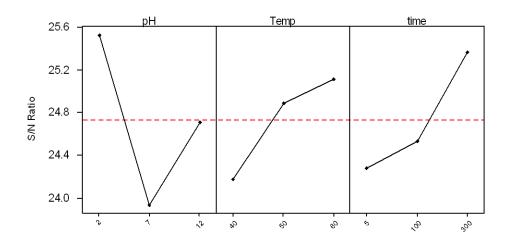


Figure 4.1.1: Response obtained after putting the Experimental results obtained from adsorption onto coconut shell

Table 4.1.1: Response Table for Signal to Noise Ratios for coconut

рН	Temp(°C)	Time(min)	qe(mg/g)	S/N ratio
2	40	5	18.46	25.3246
2	50	100	19.45	25.7784
2	60	300	18.79	25.4785
7	40	100	12.76	22.1170
7	50	300	18.93	25.5430
7	60	5	16.15	24.1635
12	40	300	17.97	25.0910
12	50	5	14.71	23.3523
12	60	100	19.31	25.7156

Table 4.1.2: Order of variables influencing the adsorption process in coconut shell

Level	рН	Temp	Time
1	25.5272	24.1775	24.2801
2	23.9412	24.8912	24.5370
3	24.7196	25.1192	25.3708
Delta	1.5860	0.9417	1.0907
Rank	1	3	2

Table 4.1.3: Analysis of Variance for qe, using Adjusted SS for Tests on coconut

Source	DF	Seq SS	Adj SS	Adj MS	F	P
рН	2	13.101	13.101	6.550	0.68	0.594
Temp	2	4.684	4.684	2.342	0.24	0.803
time	2	6.978	6.978	3.489	0.36	0.733
Error	2	19.136	19.136	9.568		
Total	8	43.900				

From the above results obtained from adsorption of dye onto coconut shell it was found out that pH is the main influencing factor in the process and this result was confirmed by ANOVA result.

### 4.2 Results from Adsorption onto Bael extract using Taguchi analysis Main Effects Plot for S/N Ratios

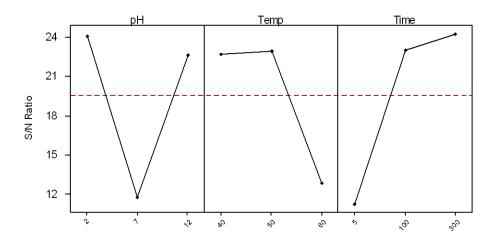


Figure 4.2.1: Response obtained after putting the Experimental results obtained from adsorption onto Bael extract

Table 4.2.1: Response Table for Signal to Noise Ratios for Bael extracts

рН	Temp(°C)	Time(min)	qe(mg/g)	S/N Ratio
2	40	5	15.78	23.9621
2	50	100	17.64	24.9300
2	60	300	15.14	23.6025
7	40	100	9.93	19.9390
7	50	300	17.19	24.7055
7	60	5	0.34	-9.3704
12	40	300	16.74	24.4751
12	50	5	9.18	19.2569
12	60	100	16.55	24.3760

Table 4.2.2: Order of variables influencing the adsorption process in bael extract

Level	рН	Temp	Time
1	24.1649	22.7921	11.2829
2	11.7580	22.9641	23.0816
3	22.7026	12.8694	24.2610
Delta	12.4068	10.0948	12.9782
Rank	2	3	1

Table 4.2.3: Analysis of Variance for qe, using Adjusted SS for Tests onto bael extract

Source	DF	Seq SS	Adj SS	Adj MS	F	P
рН	2	78.62	78.62	39.31	1.56	0.390
Temp	2	28.28	28.28	14.14	0.56	0.640
Time	2	104.86	104.86	52.43	2.09	0.324
Error	2	50.27	50.27	25.14		
Total	8	262.03				

From the above results obtained from adsorption of dye onto bael extract it was found out that time is the main influencing factor in the process and this result was confirmed by ANOVA result.

#### 4.4 Adsorption studies

#### 4.4.1 Effect of agitation time and initial dye concentration

Fig. 4.1.1 and 4.2.1 represents the effects of agitation time and initial congo red concentration on the adsorption of congo red by coconut shell and bael extract which is shown in comparison against the S/N ratio. It was found that the adsorption increases with an increase in the agitation time and attains equilibrium after some time, up to an initial agitation period of 100 mins more than 85% adsorption has been observed. From the above observation, it is evident that for lower initial concentration of the dye, the adsorption is very fast and higher removal of Congo red dye is seen if we increase the contact time. However, increase in initial dye concentration results in the fast attainment of saturation of activated carbon and higher residual dye in the equilibrium solution.

#### 4.4.2 Effect of pH

The effects of initial pH on dye solution of three dyes removal were investigated by varying the pH from 2 to 12. Results as obtained from the S/N ratio graph suggest that coconut shell and Bael extract show almost same type of adsorption behaviors when pH is taken as a factor. In both the cases it was seen that as we increase pH from 2 to 7 there is a significant reduction in S/N ratio suggesting that adsorption gets reduced during this phase of time. Now as the pH was increased from 7 to 12 it was seen that adsorption again increased but it corresponding S/N ratio is bit lower than what was obtained at pH 2. This maybe due to the fact chemisorption takes place. Chemisorption is a classification of adsorption characterized by a strong interaction between an adsorbate and a substrate surface, as opposed to physisorption which is characterized by a weak Van der Waals force. But overall it was observed that acidic solutions had better adsorption results than basic solutions.

#### 4.4.3 Effect of Temperature

To observe the effect of temperature on the adsorption capacity, experiments are carried out for 1000 mg/L congo red at three different temperatures (40, 50 and  $60 \, ^{\circ}\text{C}$ ) using 0.2 g of coconut shell and bael extract per 50 mL of the solution. It has been observed that with increase in temperature, adsorption capacity increases as shown in Fig. 2 and 3 with highest adsorption being observed in the range of  $40 - 50 \, ^{\circ}\text{C}$ . But as obtained from Taguchi's method the temp

factor is ranked as 3<sup>rd</sup> among all other factors which suggest that the reaction maybe exothermic in nature as suggested in an experiment conducted by Mohan et al (2002) and. It's a known fact that increasing temperature increases mobility of ions so helps in faster adsorption but as the reaction was exothermic so we observed degradation in its adsorption ability.

#### 4.4.4 Effect of adsorbent dosage

In this experiment adsorbent dose was taken as a constant parameter i.e. adsorbent dose was fixed(0.2 g/50ml), but it is a known fact that more adsorbent would of course help in faster and better adsorption of the dye from its solution (Bilal et al., 2004).

# CHAPTER-5

### **CONCLUSION**

#### 5. CONCLUSION

After completion of the experiment of removing congo red dye from its solution following conclusions were drawn:

- 1. The amount of dye removed increased with increasing time of contact and temp and decreased with increase in pH for both coconut (*Cocos nucifera*) shell and bael (*Aegle marmelos*) extract.
- 2. After applying Taguchi it was found that in case of coconut shell pH played the most important role in catalyzing the adsorption process while for bael the most important factor was time.
- 3. The optimum condition for adsorption onto coconut shell was found out to be pH=2, temperature= 60°C, time= 300 mins; while for bael extract it was found to be pH=2, temperature=50°C, time = 300 mins

So from this I can deduce from my experiment that

Ranking of the influencing parameters for CR adsorption onto	рН	Time(mins)	Temp(°C)
coconut	1	2	3
bael	2	1	3



#### **REFERENCES**

- 1. Jafari A. ,Tynjälä T. ,Mousavi S.M. and Sarkomaa P. , Computers & Fluids 2008,37(10),1344-1353
- 2. Mahmoud A.S. ,Ghaly A.E. and Brooks M.S. ,American Journal of Environmental Sciences ,2007,3 (4): 205-218,
- 3. Tor A., Cengeloglu Y., Journal of Hazardous Materials, 2006, B138, 409–415
- 4. Alinsafi A., Khemis M., Pons M.N., Leclerc J.P., Yaacoubi A., Benhammou A. and Nejmeddine A., Chem. Eng. Process, 2004, 44:461-470.
- 5. Mittal A., Mittal J., Malviya A., Gupta V.K., Journal of Colloid and Interface Science, 2009, 340, 16–26
- 6. Arslan I., Balcioglu I.A. and Tuhkanen T., J. Environ. Sci. Health, 2000,35:775-793
- 7. Bilal A., Journal of Colloid and Interface Science, 2004, 274, 371–379
- 8. Namasivayam C., Kavitha D., Dyes and Pigments, 2002, 54, 47–58
- 9. Mohan D., Singh K.P., Singh G., Kumar K., Ind. Eng. Chem. Res 2002, 41, 3688 3695
- 10. Kamel D., Sihem A., Halima C., Tahar S., Desalination, 2009, 247, 412–422
- 11. Capara G., Yetisa U., Yilmazb L., Desalination, 2007, 212, 103–113
- 12. Ghani J.A. ,Choudhury I.A. ,Hassan H.H. , Journal of Materials Processing Technology, 2004, 145 ,84–92
- 13. Ge J., Qu J., Journal of Hazardous Materials, 2003, B100, 197–207
- 14. Sharma J. and Janveja B.Vol.1, No.4 (2008),936-942
- 15. Bhattacharyya K.G. ,Sharma A. , Journal of Environmental Management ,2004 ,71 , 217–229
- 16. Liu, R.L.H., H.-M. Chiu and R.Y.L. Yeh, 2003. Intl. J. Environ. Studies, 59: 143-158
- 17. Khadhraoui M., Trabelsi H., Ksibi M., Bouguerra S., Elleuch B., Journal of Hazardous Materials 2009, 161, 974–981
- 18. Riera-Torres M., Gutiérrez M.C., Chemical Engineering Journal, 2010, 156, 114–120
- 19. Purkait M.K., Maiti A., DasGupta S., De S., Journal of Hazardous Materials ,2007 ,145 ,287–295
- 20. Elahmadi M.F., Bensalah N., Gadria A., Journal of Hazardous Materials ,2009 ,168 ,1163–1169
- 21. Hoda N., Bayram N., Ayranci E., Journal of Hazardous Materials, 2006, B137, 344-351

- 22. Patricia A. C. ,Fugivara C.S. ,Nogueira R.F.P. ,Boralle N. ,Zanoni M.V.B. , Portugaliae Electrochimica Acta ,2003 ,21 ,49-67
- 23. Roy D., Valsaraj K.T. and Kottai S.A., Separation Sci. Technol., 1992, 27, 573-88.
- Chakraborty S., Purkait M.K., DasGupta S., De S., Basu J.K., Separation and Purification Technology, 2003, 31, 141-151
- 25. Senthilkumaar S. ,Kalaamani P. ,Porkodi K. ,Varadarajan P.R. ,Subburaam C.V., Bioresource Technology , 2006 ,97 ,1618–1625
- 26. Chatterjee S., Chatterjee S., Chatterjee B.P., Guha A.K., Colloids and Surfaces A: Physicochem. Eng. Aspects, 2007,299,146–152
- 27. Sheikh A., Rana K.S., Biology and Medicine, 2009, 1 (2), 134-138,
- 28. Venkateswaran, P. and Palanivelu K., Journal of Hazardous Materials 2005, 131, 146-152.
- 29. Vimonsesa V., Lei S., Jina B., Chowd C.W.K., Saint C., Chemical Engineering Journal, 2009 ,148 ,354–364
- 30. Yuzhu F., Viraraghavan T., Advances in Environmental Research ,2002 ,7 Ž ,239-247
- 31. Gonder Z.B. ,Kaya Y. ,Vergili L. ,Barlas H. , separation and purification technology ,2010 ,70 ,265-273
- 32. Zhenhu H., Hui C., Feng J., Shoujun Y., Journal of Hazardous Materials ,2010 ,173 ,292–297



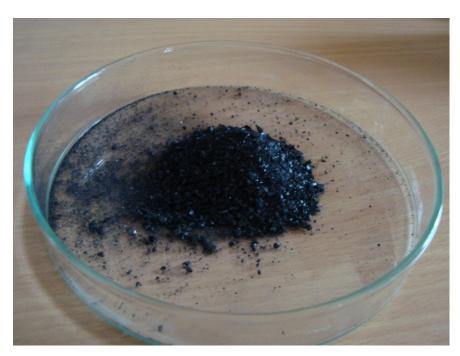
#### **APPENDIX**



A.1: Water Bath Shaker



A.2: Coconut shell sample



A.3: Bael extract sample