

# Removal of Indigo Carmine Dye by Using Palm Wood Cellulose Activated Carbon in Aqueous Solution: A Kinetic and Equilibrium Study

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**Abstract:** Mechanism of Indigo carmine dye adsorption on palm wood cellulose activated carbon (PWCAC) was studied. The use of low cost ecofriendly adsorbent has been investigated as an ideal alternative to the current expensive methods of removing dye from aqueous solution. The study is done by batch adsorption techniques. The quantitative adsorption kinetic and equilibrium parameter for indigo carmine dye were studied using uv-visible adsorption spectroscopy. The effect of initial dye concentration, pH, adsorbent dose, temp The kinetics and erature ,particle size were determine to find the optimal condition for adsorption. The percentage removal of dye was found to be most effective at pH<sub>7</sub> and contact time 140 min and at an adsorbent dose 4.8 gm/lit of dye. The study indicates that's, the percentage removal of dye increases with increasing initial dye concentration, adsorption dose and contact time and attains equilibrium at optimum conditions.

The mechanism of adsorption of indigo carmine dye on to palm wood cellulose activated carbon was investigated using pseudo first order and pseudo second order kinetics models .The adsorption kinetics was found to follow pseudo first order kinetic model. The equilibrium adsorption data of indigo carmine dye on PWCAC were analyzed by Langmuir and Freundlich adsorption model. The result shows that the Langmuir model provides the best correlation.

The adsorbent was also characterized by FTIR, EDAX SEM and XRD studies. The results indicates that the PWCAC could be employed as low cost alternative for the removal of dye indigo carmine from aqueous solution.

**Keywords-** Indigo carmine, Adsorption kinetics, equilibrium study, activated carbon , FTIR, EDAX, SEM, and XRD

## I. INTRODUCTION

The dyes are widely used in many industries to color their products. The dyes using industries are Dye stuff, Textile, Cosmetics, Leather, Plastic, Paper etc. The dyes are harmful to mankind [1]. The dyes have complex molecular structure and they are resistant to aerobic digestion and are stable to oxidizing agent and so they are very recalcitrant and difficult to remove and degrades [2]. At present various methods and their combinations are used to treat dye waste water, like

biological treatment [3], Ozonation [4], Chemical coagulation [5], Electro coagulation [6]. The adsorption technique has been proved as an outstanding capability for efficiently removing a broad range of adsorbent [7]. Removal of dyes by adsorption has become a great importance. Due to their chemical and biological stability to the conventional water treatment method. Activated carbon is one of the most widely used adsorbent materials due to its effectiveness and versatility.

Indigo carmine has IUPAC name disodium (2E)-3-oxo-2-(3-oxo-5-Sulfonato-1H-indol-2-ylidene)-1H-Indoll-5-Sulfonate [8]. The indigo carmine is blue colored water soluble di sulphate derivative of the dye indigo. The indigo has major industrial application as a textile coloring agent [9]. The indigo has been employed as an additive in Pharmaceutical tablets and capsules, and as a coloring agent in confectionary food items and cosmetics. It also used in a diagnostic Aid (In kidney function test) and a redox indicator in analytical chemistry. It is also used as a microscopic stain in Biology [10]. The indigo carmine is an Acidic dye [11]. The indigo carmine has been used to dye protein fibers, wool and silk [12]. A limited number of indigo carmine dye adsorption studies have been carried out for its removal from industrial dye waste water by using activated carbon adsorption technique and so in this paper an efforts are made to study the removal of indigo carmine dye from aqueous dye solution using PWCAC carbon.

## II. MATERIALS AND METHODS

The precursor material was collected from the field of coconut plants. The material is converted into small pieces and then washed several time by distilled water and then put in sunlight to dry for 24 hours. The small pieces are converted into different size particles by grinding in boll mill. The material is sieved to an average particle size. The obtained material is activated chemically. A 50% zinc chloride solution is used for activation. The material was soaked in ZnCl<sub>2</sub> solution for 24hours. Then after decantation the sample were pyrolyzed in Muffle Furnas in absence of air at 600° for 60 min. The obtained material is used as an

adsorbent. The physical characterization of the resulting activated carbon was then analyzed in terms of SEM analysis. The material is further used without purification as an adsorbent.

### 2.1. Adsorbate

The acidic dye, indigo carmine (Acid blue 74, C.I.73015) is blue colored water soluble powder. The indigo carmine has molecular formula  $C_{16}H_8O_8N_2S_2Na_2$  and molecular weight 466.37 and wave length 611nm was used as adsorbate. It is supplied by SD fine chemicals Mumbai. The indigo carmine has structure.

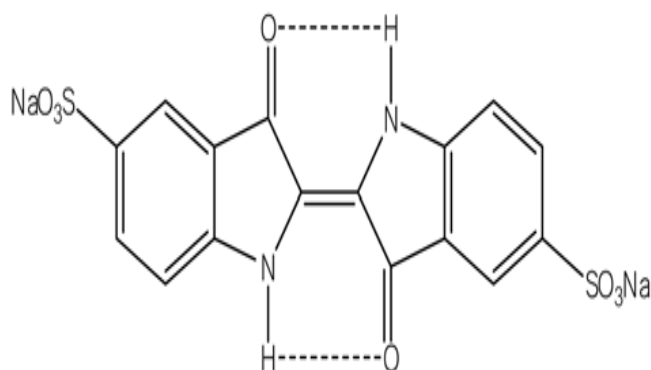


Fig.1. Chemical structure of Indigo carmine

1000 ppm stock solution of indigo carmine dye was prepared by dissolving 1gm of dye in double distilled water and from that stock solution a required ppm solutions are prepared for the experimental study.

### 2.2. Experimental method and measurement

The adsorption experiments were carried in a batch process at room temperature by using an aqueous solution of Indigo carmine dye. In each experiment an accurately weighed amount of PWCAC was added in 50ml of the dye solution in 100ml stoppered conical flask and then mixture was agitated in the mechanical shaker for a definite time at a room temperature. The adsorbent was separated from the solution by Centrifugation. The absorbance of the supernant solution was estimated to determine the residual dye concentration .The residual dye concentration was determine before and after treatment at 610 nm wave length with spectrophotometer (systronics -118) using quartz's cell of path length 1cm.The experiment were carried out at initial Ph values ranging from 2 to 8 pH and were measured by using pH meter equiptronics model EQ 607.The initial PH was controlled by addition of 0.1NHCl and 0.1NaOH solution. The kinetic of adsorption was determined by analyzing adsorptive uptake of dye from aqueous solution at different time intervals [13]. The FTIR, SEM, XRD elemental analysis (C, H, and N) of adsorbent was also carried out

## III. RESULTS AND DISCUSSION

### 3.1 Adsorbent characterization

For structural and morphological characteristic the FTIR, SEM and EDAX and XRD of adsorbent CSAC techniques have been used.

#### 3.1.1 SEM analysis

The scanning electron microscope (SEM) is widely used to study the morphological features and surface characteristics of adsorbent material. It is useful to determine particle shape, porosity and appropriate size distribution of the adsorbent mate

#### 3.12.FTIR

The FTR spectra were obtained for PWCAC before and after adsorption process shown in (fig3). The broad intense adsorption peak around 3834-3726 are indicative of the existing of OH stretching frequency(H-Bonded ),the peak observed at 3045  $cm^{-1}$  is for Aromatic C-H stretching frequency, the peak around 2801 $cm^{-1}$  is for Aliphatic C-H stretching frequency, and 1704  $cm^{-1}$  is for C=O stretching frequency and 1521  $cm^{-1}$  is for C=C stretching frequency. The intense band at 1218  $cm^{-1}$  is for C-O stretching frequency and 956  $cm^{-1}$  is for O-H out of plane bending frequency. Some of these peaks in sample shifted to lower frequency after adsorption, it suggest the participation of the functional group in adsorption of Indigo-carmine by PWCAC material

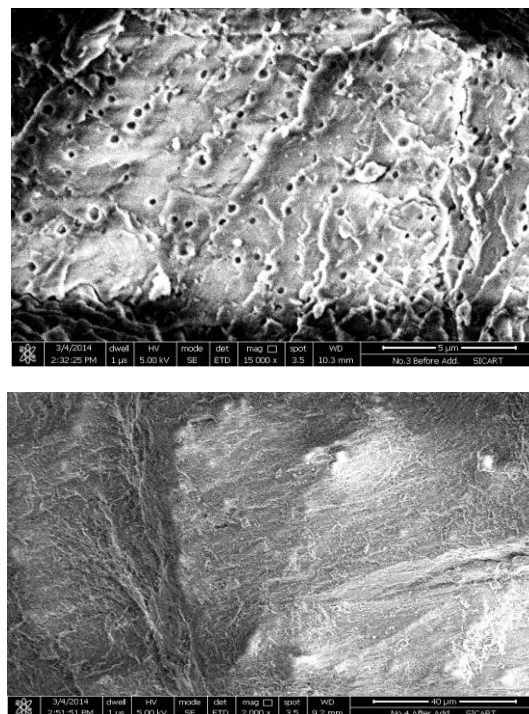


Fig.2.The SEM micrograph of PWCAC and PWCAC dyed by Indigo carmine dye

The SEM image of pure adsorbent shows ,that the adsorbent surface is irregular, rough and highly porous indicating the possibility of its good adsorption properties. The black bright spots shows the presence of tiny holes on the crystalline structure of raw activated carbon.In the dye adsorbed SEM, the adsorbed dye is clearly visible on the surface of carbon.

3.1.3.EDAX

It is energy dispersive x ray spectroscopy and it is a chemical microanalysis techniques used in conjunctions with SEM.EDAX analysis was used to characterize the elemental composition of PWCAC.The EDAX of elemental analysis of PWCAC shows 28.51 % of C,21.8%H before adsorption and 88.40% C and 9.18% O. after adsorption

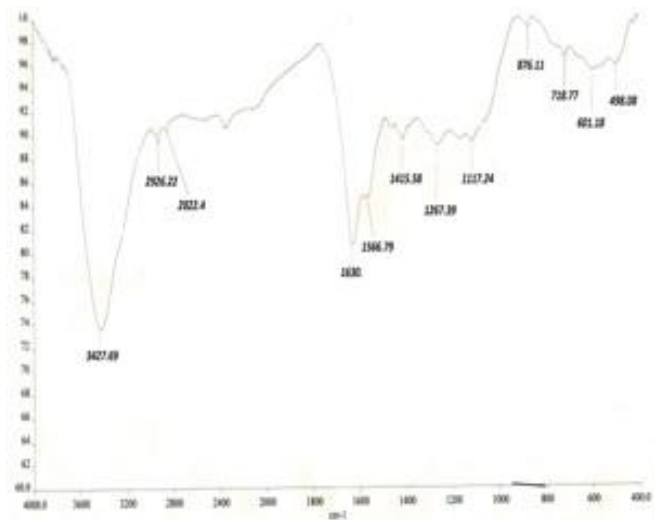


Fig.3. FTIR spectra of pure adsorbent

3.1.4. XRD

The XRD diagram of pure PWCAC (fig 4) and of dye adsorbed PWCAC is shown (fig 5).The XRD of PURE adsorbent shows main peak 2θ of 23.40° and 2θ peak 72.23° respectively.The high intensity of peaks indicates the highly crystalline nature of PWCAC.The average particle of PWCAC is estimated by csherrer formula.The size of partical is 22.2 nm.

The fig 5 shows mean peak at 24.97°,39.84°.72.25°.the observed partical size is 27.76nm. the partical size is increased due to the effect of pH on Indigo carmine on adsorption on to PWCAC. The experiment were carry out at 40 mg of initial dye concentration with 4.8 gm/ L adsorbent dose for 140 min equilibriums time in the range of pH change from 2 to7.Intially when pH increases from 2 to 7 the percentage removal increases from 81% to 87.7% and it slightly decreases up to 86.7% up to pH 12 and then percentage removal remain constant up to pH<sub>7</sub> and hence the pH<sub>7</sub>is selected as a optimum.

Adsorption of dye molecule on PWCAC.

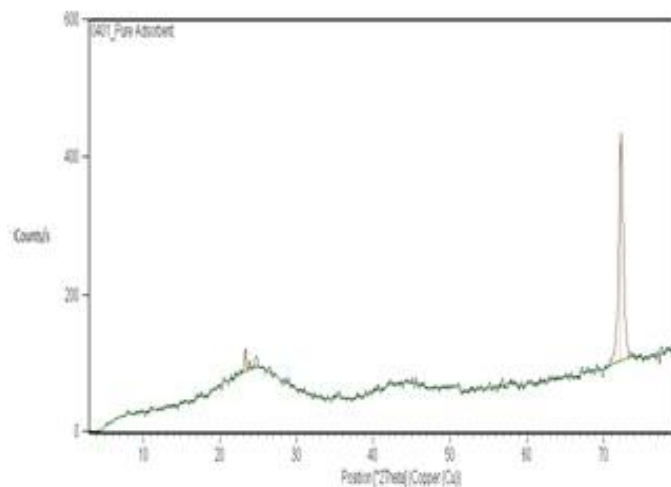


Figure: 4 XRD spectra of pure adsorbent

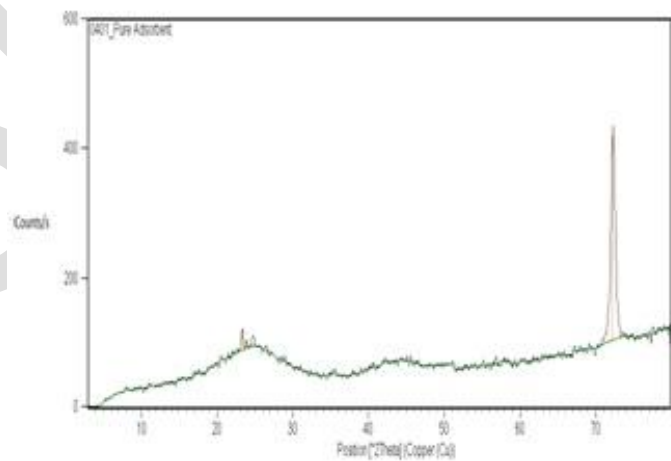


Figure: 4 XRD spectra of pure adsorbent

3.2. Optimization of Condition For Maximum Adsorption Capacity

3.2.1. Effect of PH

The pH values of a solution is an important factor in the adsorption of dye on to adsorbent .The initial pH values of the solution has more influence than the final pH value[14-15].To study the effect of pH on Indigo carmine on adsorption on to PWCAC. The experiment were carry out at 40 mg of initial dye concentration with 4.8 gm/ L adsorbent dose for 140 min equilibriums time in the range of pH change from 2 to7.Intially when pH increases from 2 to 7 the percentage removal

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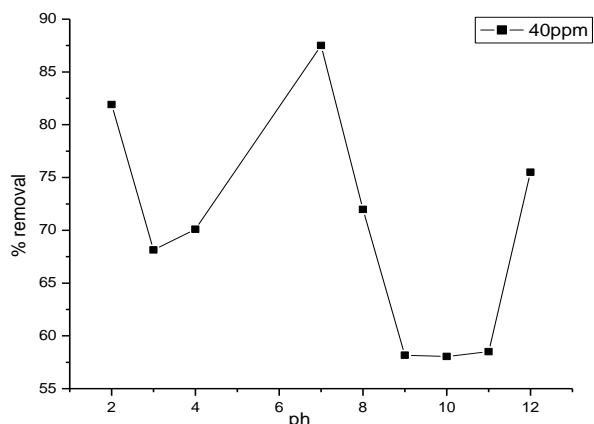


Fig.6. Effect of Ph on percentage removal Indigo carmine dye by activated carbon of PWCAC, Adsorbent dose 4.8gm/lit and contact time 140 min.

### 3.2.2. Effect of Contact Time

The effect of contact time and dye concentration on percentage removal of dye by PWCAC are presented in Figure7. The amount of dye adsorbed qt (Mg/gm) increased with increasing in agitation time and reached to the equilibrium after 140min, for the dye concentration 10.20.30.40 mg/L .Thus time required to achieve a definite fraction of equilibrium adsorption was found to be 140 min and it is independent of dye concentration [14],[15] .The adsorption density qt (mg/gm) also increased with increasing dye concentration. The percentage of dye removal at equilibrium increases from 86.9% to 95.37 % as dye concentration increased from 10 to 40 mg/liter for 4.8 g/L of adsorbent does at Ph<sub>7</sub>.This shows that removal of Indigo carmine depends on concentration of dye.

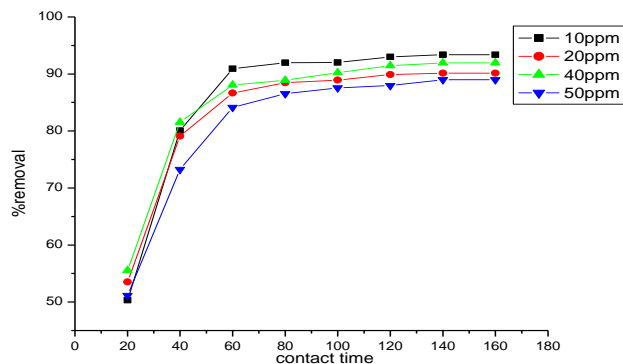


fig.7: Effect of contact time & concentration of indigocarmine on percent removal;adsorbent dose 4.8 gm/l,pH7

### 3.2.3. Effect of adsorbent dose

The effect of adsorbent dose on the amount of dye adsorbed was studied. The equilibrium value of amount of dye absorbed was observed to be increased from 27.57 % to 92% mg/liter with increase in adsorbent dose from 0.02 gm to 4.8 gm /L for 40 mg/L dye concentration ,contact time 140 min and ph7.The percentage removal of Indigo carmine increased with increasing does of adsorbent and that is due to the increased in availability of surface active sites resulting from increased of dose and agglomeration of adsorbent[16],[17].The increase in the extent of removal of Indigo carmine is found to be significant after the does 4.8gm/L and hence it is fixed as a optimum dose for adsorbent.

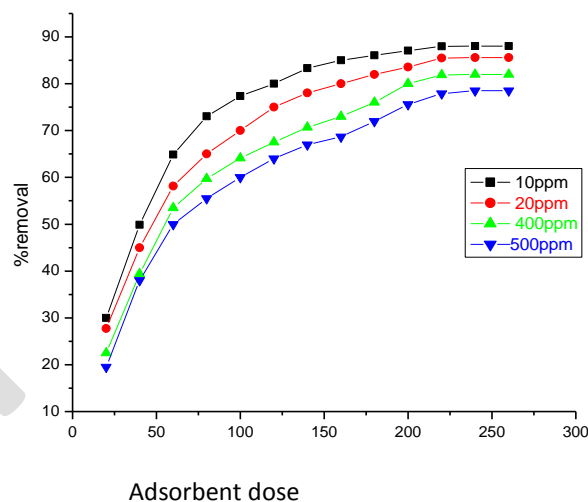


Fig.8.Effect of adsorbent dose on the percentage removal of Indigo carmine at adsorbent dose 4.8gm/L, contact time 140 min. and pH 7.

### 3.2.4. Effect of initial dye concentration

The adsorbent and dye solution of different initial dye concentration 10mg to 40mg/L was contacted for 140 min at pH 7 and at a constant adsorbent dose 4.8gm/L. The result shows that the percentage removal of dye increases from 86.9% to 95.37 %-when initial dye concentration increases from 10 to 40 mg/L. This shows that the Indigo carmine Removal by adsorption on PWCAC is concentration dependent.

### 3.2.5. Effect of particle size

The adsorption experiment is carried out by using different mesh size PWCAC ranging from 20 to 200 mesh size. It is observed that the adsorption of dye decrease with increase of particle size and increase with decrease in particle size that is due to large surface area availability.

### 3.2.6. Effect of temperature

The experiment is carried out at three different temperatures i.e. from 30, 40 and 50°C. It is observed that the removal of indigo carmine decrease with increase of temperature hence adsorption of indigo carmine is an exothermic process [19].

3.3. Adsorption Kinetics

The kinetics of adsorption study is a significant as it describes the solute uptake rate. It controls the residence time of adsorbent uptake at the solid solution interface. The dye concentration and adsorbent dose are the important parameter because they determine capacity of adsorbent for a given initial concentration of dye solution. The study includes adsorption rate study. The kinetics of adsorption of Indigo carmine on PWCAC were analyzed using pseudo first order (Lagergren), Pseudo 2<sup>nd</sup> order kinetic models. The conformity between experimental data and the model predicated values were expressed by Correlation coefficient (r<sup>2</sup>). A relatively high r<sup>2</sup> value indicate that the model successfully describes the kinetics of Indigo carmine adsorption. The dye removal from the solution is through the mechanism of adsorption (20).

3.3.1. Pseudo first order equation

The Pseudo first order (Lagergren) equation is generally expressed [21] as

$$\text{Log}(q_e - q_t) = \text{Log}e - (K_1/2.303) t \tag{1}$$

where q<sub>e</sub> and q<sub>t</sub> are the adsorption capacity in mg/gm at time 't', respectively, K<sub>1</sub> is a rate constant of pseudo first order adsorption (min<sup>-1</sup>). The rate constant K<sub>1</sub> and q<sub>e</sub> for first order equation are determined from the slope and intercept of the plot of log (q<sub>e</sub>-q<sub>t</sub>) vs. time respectively (fig.9)

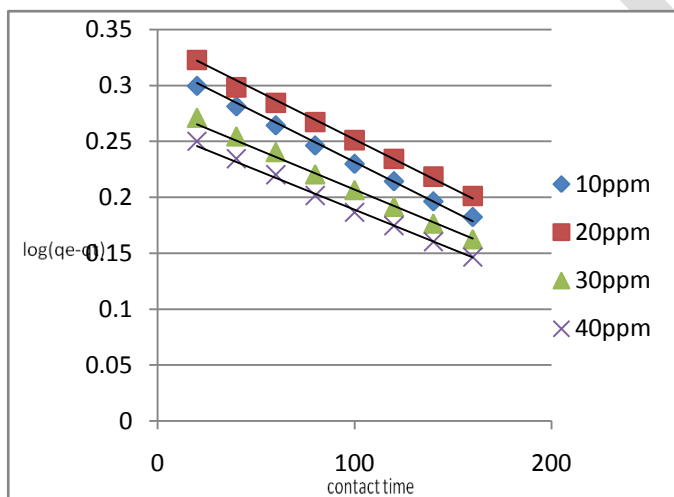


Fig.9. Lagergren 1st order plot for adsorption of Indigo carmine by Palm wood cellulose activated Carbon for different initial dye concentration, adsorbent dose .4.8gm/L pH7

The linear relationship of the plot for 10, 20, 30 and 40 mg/L dye concentration indicates the validity of equation [22]. The co-relation coefficient r<sup>2</sup> of the first order equation is (0.995 to 0.996) the value of q<sub>e</sub> calculated from first order kinetics plots were nearly same as compared to experimental (Table1).

It shows that the rate constant K<sub>1</sub> remains same with increase in initial dye concentration and adsorbent dose. It shows the applicability of Lagergren equation to describe the indigo carmine

3.3.2. The pseudo second order equation

The kinetic data were further analyzed using pseudo second order kinetic model which is expressed as [23]

$$1/q_t = 1/K_2 * q_e^2 + t/q_e \text{ and } h = K_2 q_e^2 \tag{2}$$

Here K<sub>2</sub> is rate constant of second order adsorption (gm/mg/min) the plot of t/q<sub>t</sub> vs. t.

The above equation gives linear relationship from which q<sub>e</sub> and K<sub>2</sub> can be determine from the slope and intercept of the plot respectively. The linear plot of t/q<sub>t</sub> vs. t shows a good agreement of experimental data with second order kinetic model for different initial dye concentration 10, 20, 30, 40 mg/L and for different adsorbent dose. The calculated q<sub>e</sub>, K<sub>2</sub> and corresponding linear regression correlations coefficients r<sup>2</sup> value are summarised in a table. The values are not greater than 0.99 which indicate the inapplicability of kinetic equation and Pseudo second order nature of adsorption process of Indigo carmine on PWCAC. The q<sub>e</sub> value increases with increase in initial dye concentration and adsorbent dose [24]. The calculated q<sub>e</sub> values are not agreed well with the experimental values. These indicate that adsorption system does not obey the second order kinetic model.

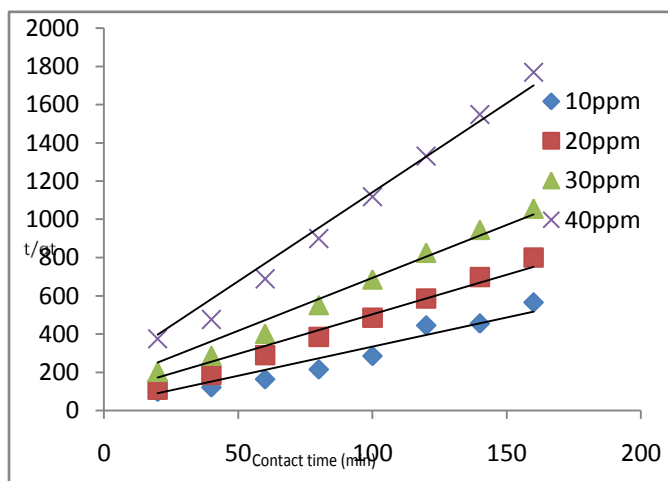


Fig.10. Plot of pseudo 2nd order model at different initial dye concentration for indigo carmine, adsorbent dose 4.8gm/l and pH7

TABLE I The calculated and experimental qe for pseudofirst and pseudosecond order reaction

Adsorbent dose g/L	Conc. of dye mg/L	Pseudo First Order				Pseudo Second Order			
		qe(exp.) mg/g	K <sub>1</sub> min <sup>-1</sup>	qe(Cal.) mg/g	r <sup>2</sup>	k <sub>2</sub> gm/mg/min	qe(cal.) mg/g	h	r <sup>2</sup>
4.8g/L	10	1.81	0.28×10 <sup>-3</sup>	1.82	0.996	0.1073×10 <sup>-3</sup>	0.1074	0.0047	0.984
	20	1.80	0.28×10 <sup>-3</sup>	1.91	0.993	0.2110×10 <sup>-3</sup>	0.1806	0.0071	0.975
	30	2.29	0.2×10 <sup>-3</sup>	2.19	0.965	0.1898×10 <sup>-3</sup>	0.2418	0.0111	0.961
	40	1.85	2.3×10 <sup>-3</sup>	2.19	0.996	0.3088×10 <sup>-3</sup>	0.3285	0.0333	0.934

TABLE I-Comparison of Adsorption rate constant, Calculated and experimental qe values for different initial dye concentration and adsorbent dose for different kinetic model.

TABLE .II Adsorption Kinetic parameter of Indigo carmine on to PWCAC for Elovich model and intrapartical diffusion model.

3.3.3. The Elovich equation

The simplified Elovich equation is

$$Q_t = 1/\beta \ln(\alpha\beta) + 1/\beta \ln t \tag{3}$$

Where α is the initial adsorption rate (mg/gm/min),β is the desorption constant (gm/mg).The adsorption of Alizarin Red-s fits the Elovich model a plot of qt vs. lnt is a linear with a slope 1/β and intercept 1/β ln(αβ). As concentration increases from 10-40 mg/liter the value of β decreases from 6.2893 to 9.6153 for 4.8gm/liter of adsorbent.

Adsorbent g/L	Concentration mg/L	Elovich model		Intrapartical diffusion model	
		β g/mg	r <sup>2</sup>	k <sub>p</sub> mg/g/min <sup>0.5</sup>	r <sup>2</sup>
4.8gm	10	9.6153	0.959	0.013	0.971
	20	17.43	0.944	0.020	0.935
	30	7.4074	0.931	0.027	0.962
	40	6.2893	0.956	0.029	0.981

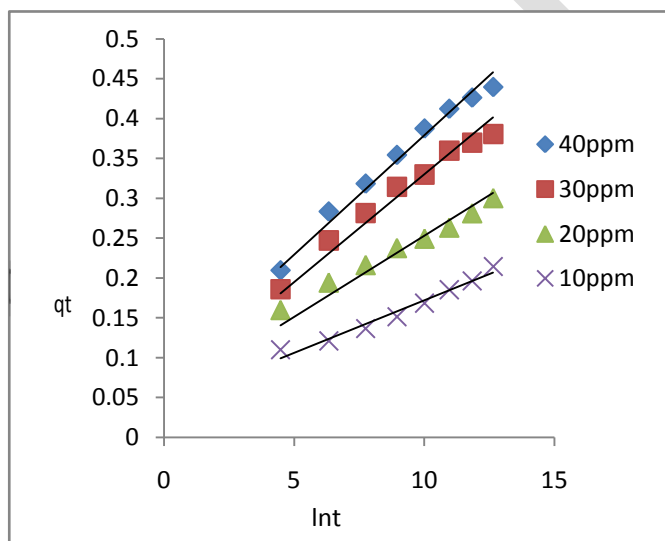


Fig.11. Test of Elovich model for adsorption of Indigo carmine on PWCAC at different initial concentration adsorbent dose 4.8 gm/l and contact time 140 min. and pH7

3.3.4. Intrapartical diffusion model

The Intrapartical diffusion was also involved in the adsorption of Indigo carmine by PWCAC. The kinetic data is well represented by Weber and Morris by the plot of qt vs. square root of time. It was observed that there were two linear portions. The first linear portion ended with smooth curve followed by the second linear portion. The double nature of the curve reflects the two stage external mass transferred followed by intrapartical diffusion [25] and is described by the equation.

$$Q_t = k_p t^{1/2} \tag{4}$$

Where k<sub>p</sub> is intrapartical diffusion rate constant (mg/gm/min) the slope of the linear portion is proportional to the boundary layer thickness. The intrapartical diffusion k<sub>p</sub> at different initial concentration and adsorbent dose are represented in table 2. The values of k<sub>p</sub> were found to be in the range of 0.013 to 0.029 for dye concentration 10,20,30,40 mg/L of dye and adsorbent dose 4.8 gm/L of PWCAC.

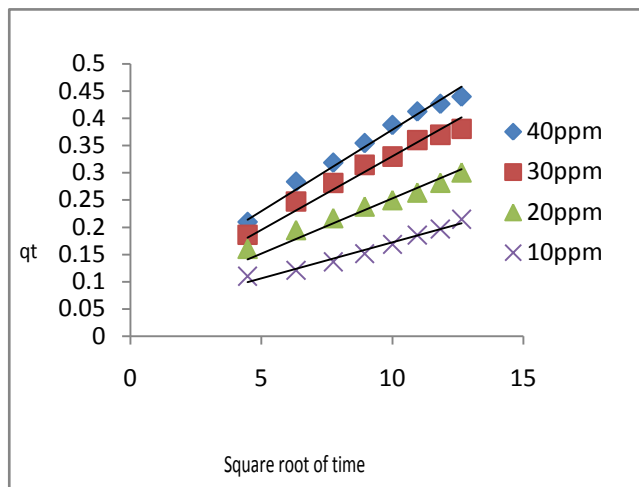


Fig.12. Test of Intraparticle diffusion for adsorption of Indigo carmine on PWCAC

### 3.4 adsorption Isotherms

The distribution of the dye between the liquid phase and the adsorbent is a measure of the position of equilibrium in adsorption process and can be generally expressed in Freundlich and Langmuir isotherm

#### 3.4.1. Freundlich isotherms

The linear plot of  $\log q_e$  vs.  $\log C_e$  confirms the applicability of the model. The logarithmic form of Freundlich isotherms equation is

$$\log q_e = \log k_f + \frac{1}{n} \log C_e \quad (5)$$

Where  $q_e$  is the amount of dye adsorbed per unit mass of the adsorbent (mg/g).  $C_e$  is the equilibrium concentration of the dye (mg/L), the  $k_f$  represent quantity of dye adsorbed in mg/g for unit concentration of dye and  $1/n$  is measure of adsorption density. The linear plot of  $\log q_e$  vs.  $\log C_e$  for 10, 20, 30 and 40 mg/L dye concentration shows that the adsorption also follows the Freundlich isotherms. The  $k_f$  and  $n$  values were calculated from the intercept and slope of the plot and are presented in table 2. The correlation coefficient of the graphs are  $r^2 < 0.9949$  indicate the Where  $b$  is the Langmuir constant and  $C_i$  is the initial dye concentration (mg/l). According to the values of the  $R_L$ , The isotherms shape may be interpreted as if  $R_L > 1$ , unfavorable.  $R_L = 1$  Linear,  $R_L > 0$  favorable and  $R_L = 0$  irreversible adsorption [29-30]. The results given in Table 3 shows that the adsorption of Alizarin red-s on PWCAC is favorable applicability of isotherms.

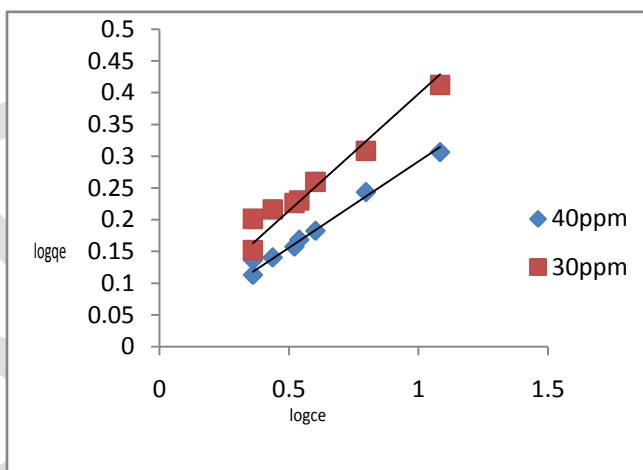


Fig.13. Freundlich plot for adsorption of Indigo carmine by PWCAC, pH and adsorption dose 4.8 gm/L

Table III

Freundlich and Langmuir coefficient for adsorption of Indigo carmine on PWCAC for different dye concentration and adsorbent dose from

Freundlich coefficient					Langmuir Coefficient			
Dye conc. mg/L	$K_f$ L/g	$n$	$1/n$	$r^2$	$a$ mg/g	$b$ g/L	$R_L$	$r^2$
20	1.162	2.6013	0.3844	0.940	5.5555	0.0600	0.4545	0.960
30	1.0615	2.7133	0.3680	0.934	5.3910	0.05700	0.3816	0.965
40	1.0417	3.6734	0.2722	0.982	5.4347	0.05450	0.3030	0.962

### 3.4.2. Langmuir isotherms

Langmuir isotherms is represented by the following equation [26]

$$C_e/q_e = 1/ab + ce/b \quad (6)$$

Where  $C_e$  is the concentration of the dye solution (mg/gm) at equilibrium. The constant signifies the the adsorption capacity (mg/gm) and  $b$  is related to energy and adsorption. The linear plot of  $C_e/q_e$  vs.  $C_e$  shows that the adsorption follows the Langmuir isotherms. The values of 'a, and' b 'were calculated from the slope and intercept of the linear plots and represented in Table 3. The applicability of the Langmuir isotherms suggest the monolayer coverage of the dye on the surface of PWCAC. The essentials characteristics of the Langmuir isotherms can be expressed by the dimensional constant called equilibrium parameter RL [26],[28] and defined by the equation

$$RL = 1 / (1 + bci) \quad (7)$$

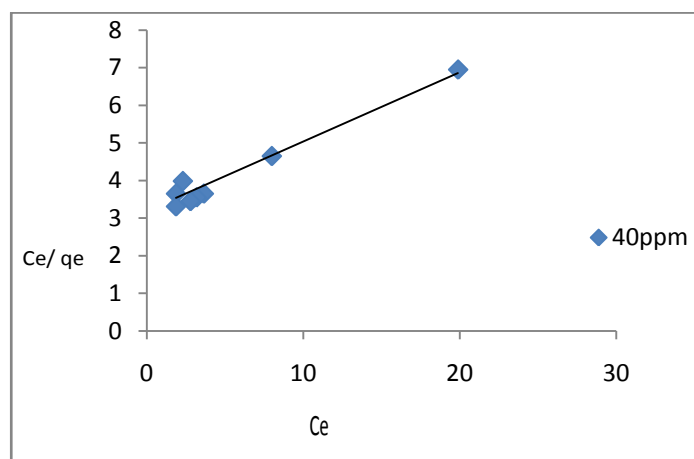


Fig.14. Langmuir plot for adsorption of indigo carmine by PWCAC, pH7 and adsorbent dose 4.8g/L

## IV. CONCLUSIONS

1. The adsorption study indicates that, the agro based material palm wood cellulose activated carbon can be used as an effective adsorbent for the removal of Indigo carmine dye from aqueous solution.
2. The adsorption experiments were conducted in a batch mode for the concentration range 10 to 40mg/L at pH7 and for an adsorbent dose 0.02 to 4.8 g/L and up to contact time 140 min.
3. The equilibrium was achieved in 140 min and maximum adsorption capacity were observed at pH7 and adsorbent dose 4.8 g/L.
4. The amount of dye uptake (mg/gm) was found to be increases with increase in contact time, initial dye concentration and it decreases with an increase in adsorbent dose.

5. The adsorption rate was found to confirm to pseudo first order kinetics with good co- relation.
6. The equilibrium data fit very well in Langmuir isotherms equation. It confirms the capacity of Indigo carmine dye on to PWCAC with a monolayer adsorption capacity.
7. The dimensionless separation factor (RL) called equilibrium parameter showed that PWCAC could be used for the removal of Indigo carmine from aqueous solution.
8. Thus the PWCAC is an inexpensive and easily available material and be a alternative for the most costly adsorbent used for removal of dyes in the waste water treatment processes.

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