e-ISSN No.: 2582-4228

Journal of Indian Association for Environmental Management

Journal homepage: www.http://op.niscair.res.in/index/php/JIAEM/index

Utilization of Agriculture Waste (Mustard Straw) for Treatment of Industrial Waste Water

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Submitted:22November 2023 Revised:29November 2023 Accepted: 30November 2023

***Abstract*:Present Study focus on Reactive dye adsorption using activated carbon. Activated carbon(AC) of mustard straw was prepared by thermo chemical two step activation process using H3PO4, ZnCl2 and KOH as activation agents. Best sample of prepared AC was used for further study. Make use of this agricultural waste for treatment of industrial effluent is economical and feasible. It was seen that Activated Carbon essentially microporous, BET surface area obtained is 1350 gm/m2. The dye adsorption experiments using prepared sample was explored for Reactive Dye. Study of physicochemical characteristics, absorption study, Kinetic study and thermodynamic study is conducted. Experimental data was best fitted with Langmuir Adsorption Isotherm with correlation coefficient of 0.99 from four models under study (Langmuir, Freundlich, Temkin and Dubinin–Radushkevich), which shows monolayer adsorption predominant. The kinetic data were tested for the models of pseudo-first order, pseudo-second order and intraparticle diffusion. The adsorption process follows the pseudo-second order model, which suggests that the process was controlled by chemisorption.**

***Keywords*:Adsorption, AC, Biomass, Biochar, Isotherm, Kinetics**

I. INTRODUCTION

There is growth in dye and textile industries in Gujarat. With development of textile and dye industries, due to presence of minute concentration of coloring substance makes water unsuitable for several applications. There are many methods used to eliminate dye from such polluted water like air flotation, biological, chemical oxidation, froth flotation, adsorption, membrane separation, extraction etc. The dye molecules are complex so, difficult to degrade. It makes unfavorable effects due to its structural stability [8]. Apart from all processes listed here, adsorption is the simplest, economical, comparatively practical option for removal of dye from wastewater. The use of AC as an adsorbent is feasible due to its good thermal constancy, large surface area with large porous structure[7] [5]. Commercial AC is typically derived from traditional sources. There is an exhortation for research scholar to seek the best possible stock to replace conventional once. The ability to absorb dye from wastewater depends primarily on the characteristics of the predecessor, material and process. This study has focused on the preparation of activated biochars from agricultural residues. There are usually two approaches for the preparation of activated carbon-physical and chemical activation. The key benefits of chemical activation over physical activation are the creation ofa rich porous structure resulting in carbon, high yield and lower activation temperature

Fig. 1 Images of Mustad Straw and Powder

requirements [19] [34]. In this study, AC was produced from mustard straw as biomass by chemical activation. The carbonized material is impregnated with an activating agent first then the mixture was heated in inert or oxygen free atmosphere. Apart from Phosphoric acid one can use Zinc chloride, potassium hydroxide, phosphoric acid, sulfuric acid and caustic soda [5][6].

Indian mustard is predominantly cultivated in Rajasthan, UP, Madhya Pradesh and Gujarat. Average production of mustard is around 3.00 lakh tones per year in Gujarat. Residue to Product ratio (RPR) is appx. 1.80[3] [2]. The straw is generally burned in the field and not used as animal feed and has been selected as a precursor to AC. The food chain is not affected by the use of mustard straw as a precursor to the preparation of AC. In this work, straw mustard was used as activation for the preparation of porous AC via $ZnCl_2$, KOH and H_3PO_4[5][6] The objectives of this research was to examine adsorption, kinetics of the adsorption and thermodynamics to describe adsorption behavior. The dye chosen for the study was Reactive Dye.

II. MATERIAL & METHODS

Materials

The mustard straw was collected from an agricultural field of northern region of Gujarat, a state in western India. It was first thoroughly washed with water to remove clay and other water soluble impurities and then dried naturally [34]. Analytical grade Phosphoric acid, Potassium hydroxide and Zinc chloride were used as an activating agents Nitrogen gas was used to create oxygen free atmosphere[5][6].

Preparation of Activated Carbon

Dry mustard straw powder was filtered through a 60-80 mesh screen. It was then infuse with activating agent for 24 hours at room temperature and then dried around 101°C - 105°C. The infused biomass was then inserted in a tubular batch reactor made from of stainless steel (0.07 m internal diameter and 0.69m length).

Mainly three Parameters:

1. Holding time

2. Impregnation ratio and

3. Activation temperatures are important for preparation of AC.

All above parameters were optimized using response surface methodology for quality carbon [27] [28]. Upper and lower limit of parameters were set as: Impregnation ratio 2:1 to 4:1, Activation temperature 600° C to 800 °C and Holding time of range from 60 min. to 120 min. All prepared samples were washed with water and neutralized around pH 7. At last the samples were dried in oven at 110°C for 1 h. The yield (on dry basis) of the AC was calculated by using [35] [34]

$$\textbf{Yield} = \textbf{Wi / Wo} \times \textbf{100}$$

Where Wi = Mass of the raw carbon after activation
Wo= Mass of the raw carbon before activation.

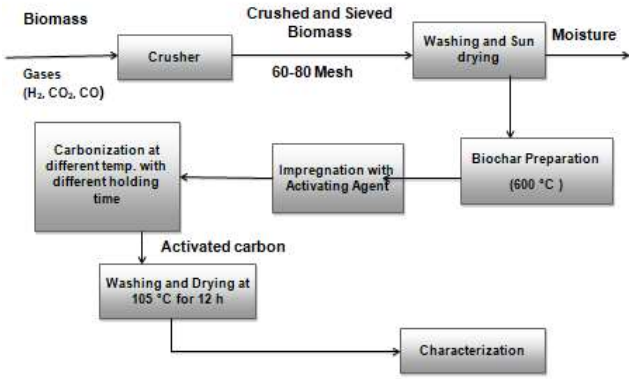


Fig. 2 Diagram for preparation of activated Biochar

Characterization of AC:

The proximate analysis of the Prepared Optimum AC was measured as per standard test methods 1. Moisture: ASTM D3173; 2.Ash: ASTM D3174, 3. Volatile matter: ASTM D3175 and 4.Fixed carbon: by difference ASTM E1131-08[16].

Ultimate analysis was carried out by using ultimate analyzer (LICO-CHNS 932). BET surface area analysis was carried out by 3 Flex Surface Characterization Analyzer. A field emission Scanning Electron Microscope characterized the surface morphology of porous AC[8] [11].

Adsorption study

In the present study, Reactive Dye was used as adsorbate for adsorption abilities of the activated carbon. Experiments on adsorption were conducted by using 250 mL conical flasks with 0.025 grams of AC and 50 mL of specified Reactive Dye solvents. The ranges of selected experimental parameters as follows: Reactive Dye concentrations range from 60 ppm to 150 ppm and temperature ranges from 30 °C to 60 °C. The mixtures were then filtered and the UV-VIS spectrophotometer was studied at 665 nm. Adsorption capacity of AC per unit mass of Reactive Dye was calculated by following equation [8] [11],

$$q_e = \frac{(C_o - C_e)}{w} \times V \qquad (1)$$

Where C_o = Initial concentration of Reactive Dye (mg/ltr)
C_e = Equilibrium concentration of Reactive Dye(mg/ltr)
w = mass of AC (gms.)
V = volume of Reactive Dye solution (mL).

The removal percentage of Reactive Dye was calculated from the following equation[8][11],

$$R = \frac{(C_o - C_t)}{C_o} \times 100\% \qquad (2)$$

Where C_o = Initial concentration of Reactive Dye(mg/Ltr)
C_t= t-time concentration of Reactive Dye(mg/Ltr) and
R = Removal percentage (%)

A) Adsorption Isotherm model

In order to optimize the design of the adsorption system, the most appropriate balance curve correlation is required. In this study, four adsorption isotherms: Langmuir, Freundlich, Temkin and Dubinin–Radushkevich (DR) isotherms were studied using equilibrium Reactive Dye adsorption data [8] [11]. Langmuir isotherm assumes monolayer adsorption on a surface containing adsorption sites for uniform adsorption strategies without shifting of adsorbate to the surface plane. The linear form of the Langmuir isothermal equation is given as[8] [11],

$$\frac{1}{q_e} = \frac{1}{Q_o} + \frac{1}{Q_0 K_L C_e} \qquad (3)$$

Where, C_e = Equilibrium concentration of the Reactive Dye, (mg/Ltr)
q_e= Amount of Reactive Dye adsorbed per unit mass of adsorbent, (mg/g)
Q_o = Langmuir constants related to adsorption capacity
K_L = Rate of adsorption respectively.

After rearranging above equation, When Ce/q_e is plotted against 1/C_e, a straight line with slope of 1/Q_o and intercept of 1/Q_oK_L is obtained. A dimensionless constant known as a separating factor or equilibrium parameter, R_L, determines the basic features of Langmuir isotherm[8] [11].

$$R_L = \frac{1}{1+(1+K_L C_O)} \qquad (4)$$

The equilibrium parameter R_L indicates the shape of isotherms as follows:

TABLE 1
Relation of R_L with type of isotherm in Langmuir model

Value of equilibrium parameter	Type of isotherm
R_L>1	Unfavorable
R_L=1	Linear
0<R_L<1	Favorable
R_L=0	Irreversible

The Freundlich isotherm model is an empirically based equation on adsorption(surfaces supporting various sites affinities). The stronger binding sites are supposed to be occupied first and the binding strength decreases as site occupancy increases. The famous logarithmic form of Freundlich isotherm is provided by the following equation[8] [11],

$$\ln q_e = \ln K_f + \frac{1}{n}\ln C_e \quad (5)$$

Where, K_f = Adsorption or Distribution coefficient
n = Freundlich constants

$K_f$indicates the quantity of dye adsorbed for a unit equilibrium concentration. The value of slope 1/n represents adsorption intensity or heterogeneity of the surface. When the value of 1/n=0, system becomes heterogeneous. The value of 1/n = 1 shows the normal isotherm of Langmuir while 1/n > 1 shows cooperative adsorption. The plot between ln(qe) versus lnCe has a 1/n slope and lnK_f interception[8] [11].

Temkin isotherm includes a factor that takes adsorbent particles mutual interactions into account. Through adsorbent adsorb of all the layer molecules, heat would decrease linearly with surface coverage. The adsorption consists of a uniform distribution of binding energy to a maximum binding energy. The isotherm of Temkin is expressed as[8] [11],

$$q_e = \frac{RT}{b_T} ln A_T + \frac{RT}{b_T} ln C_e \quad (6)$$

Where,
A_T =Temkin isotherm equilibrium binding constant (L/g)
b_T = Temkin isotherm constant
R = Universal gas constant (8.314J/mol.K)
T = Temperature at 298K
B= RT/A_T = Constant related to heat of sorption (J/mol)

Dubinin–Radushkevich isotherm can be defined as[8] [11],

$$ln q_e = ln q_s - K_{DR}\varepsilon^2 \qquad (7)$$

Where ε can be correlated as,

$$\varepsilon = RTln(1 + \frac{1}{C_e}) \qquad (8)$$

Where R= Gas constant (8.314 J/mol K)
T = Absolute temperature (K).

The constant K_{DR} gives the mean free energy E of sorption per molecule of the adsorbate when it is transferred to the surface of the solid from infinity in the solution and can be computed by using the following relationship [8] [11],

$$E = \frac{1}{\sqrt{2K_{DR}}} \qquad (9)$$

A plot of lnq_e versus ε^2 enables the constants E and q_s to be determined from the slope and intercept respectively.

B) Adsorption Kinetics Model

The adsorption kinetics models narrate the rate of adsorbate uptake on AC and it controls the equilibrium time. The pseudo-first-order and pseudo-second-order kinetic models were applied for present adsorption process whereas the intra particle diffusion model was further studied to determine the diffusion mechanism of the adsorption [8] [11].

Pseudo-first-order model

The pseudo-first-order kinetic model has been widely used to forecast sorption kinetics. The model given by Langergren and Svenska is defined as[8] [11],

$$\ln(q_e - q_t) = ln q_e - k_1 t \qquad (10)$$

Where q_e =Adsorbate Adsorbed at equilibrium in (mg/g)
q_t =Adsorbate Adsorbed at any time (t) in (mg/g)
t = Time in Hour (h), respectively and
k_1 = Adsorption rate constant.

Pseudo-second-order model

The pseudo-second-order equation based on equilibrium adsorption is shown as [8] [11],

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (11)$$

Where q_e =Adsorbate Adsorbed at equilibrium in (mg/g)
q_t =Adsorbate Adsorbed at any time (t) in (mg/g)
k_2 = Rate constant of second-order adsorption (g/mg h)

The plot of t/q_t versus t gave straight line. $1/q_e$ as the slope and $1/k_2q_e^2$ as the intercept. This procedure forecast to predict the behavior over the whole range of adsorption [8] [11].

Intra particle diffusion model

To understand diffusion mechanism in adsorption, intra particle diffusion model based on the theory proposed by Weber and Morris was tested. It is an empirically model. Where adsorption varies almost proportionally with $t^{1/2}$[8] [11].

$$q_t = k_{int} * t^{0.5} + C_i \quad (12)$$

Where k_{int} = Rate parameter of stage i, (mg/g $h^{1/2}$),
C_i= Intercept of stage i,

III. Results & Discussion

Characterization of Mustard straw Biochar and Biomass

The initial characterization of Biochar and biomass from mustard straw is listed in Table II. The mustard straw biomass has relatively high moisture content (7.67 per cent), a high percentage of volatile matter (69.60 per cent), a moderate solid carbon (18.61 per cent) and low ash content (4.12 percent). High volatility usually reduces the yield in pyrolysis, whereas high carbon content AC can produce only when inorganic content low[34]. The ultimate analysis shows that 59,26% of carbon and 5,42% of hydrogen and 30.40% of oxygen for Biomass. The experimental conditions of pyrolysis and activation involved in the preparation of AC are not only the major contributors to the porous structure of the final product; but also play a vital role. A positive saliency of prepared Biochar was the high carbon content (69.57%).

TABLE 2
Proximate and Ultimate analysis of the AC of the precursor, Mustard straw(wt %)

Mustard Straw	BIOCHAR	BIOMASS
Moisture	3.03	07.67
Volatile matter	21.53	69.60
Fixed carbon	69.42	18.61
Ash	6.02	04.12
Carbon	78.93	59.26
Hydrogen	1.45	5.42
Oxygen	18.75	30.4
Nitrogen	0.84	4.65
Sulfur	0.03	0.54

TABLE 3Chemical Composition of Biomass

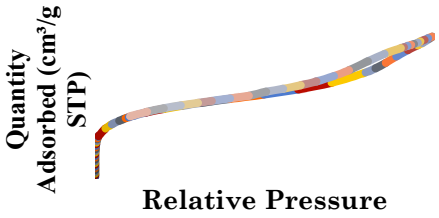
Chemical Composition	% Result
Cellulose	36.7
Lignin	21.6
Extraction Substances	3.46
Mineral Substances	5.6
Cellulose	36.7

Surface area of AC

As observed the chemical activation of the mustard straw resulted in AC having surface area of 1350 m^2/gm, and Pore Volume 0.89 cm^3/gm.

TABLE 4Properties of AC

Properties	Unit	H_3PO_4AC	KOHAC	$ZnCl_2$AC
BET surface area	(m^2/g)	1350	1309	1263
Pore Dia.	nm	5.63	5.7	5.7
Pore Volume	cm^3/gm	0.89	0.86	0.81



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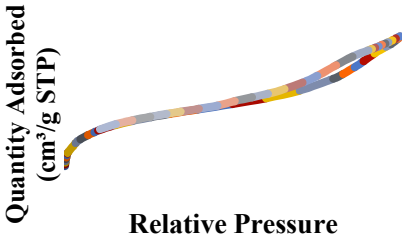

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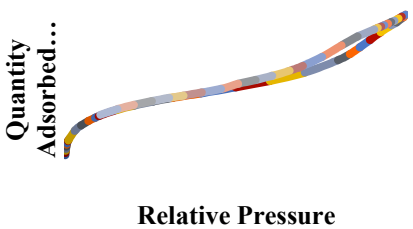
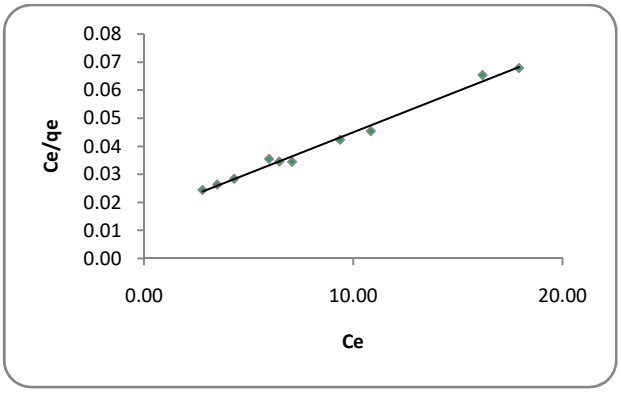


(c)

Fig. 3 Nitrogen Adsorption Isotherm for (a) H_3PO_4AC (b) KOHAC (c) $ZNCL_2$ AC

Adsorption Isotherms

The best data fitting of the four isotherm models for the adsorption of Reactive Dye by Mustard straw AC are shown here in Fig. 4. The results say that the Langmuir isotherm was better suited in this case than the Freundlich isotherm. As a result, the adsorption of Reactive Dye by AC can be predicted to be monolayer [8] [29] [30] and the surface sites were moderately homogeneous. Also the value of R_L(separation factor) was less than one which indicated a favorable adsorption of Reactive Dye by AC. In addition, the sorption heat value being less than 30 KJ/mol indicated physical adsorption at the respective sites [11] [31]



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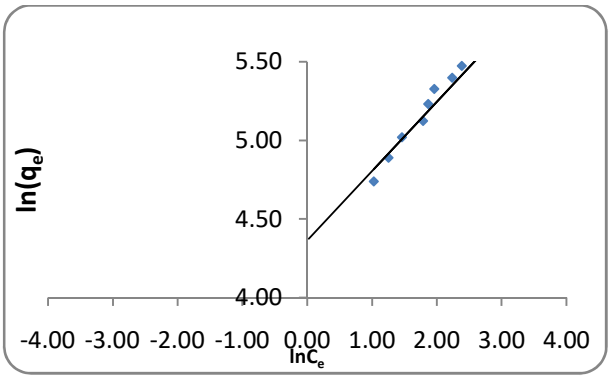

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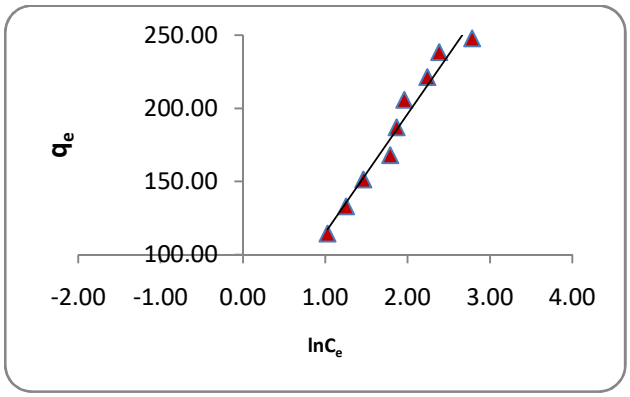

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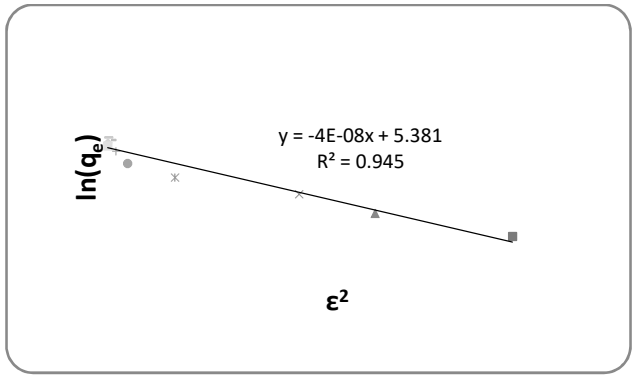


(d)

Fig. 4 Various adsorption isotherm plots (a) Langmuir (b) Freundlich (c) Temkin and (d) DR isotherms

The calculated values isotherms parameters and value of R^2 are also given in Table V

TABLE 5
Isothermal parameters for adsorption of Reactive Dye by AC

SR. No.	1				2			
Isotherm	Langmuir isotherm				Freundlich isotherm			
Terms	Q_m (mg/g)	K_L (L/mg)	R_L	R^2	1/n	K_f	N	R^2
Value	277.78	0.92	< 1	0.99	0.2	149.9	4.6	0.98

3				4			
Temkin isotherm				Dubinin–Radushkevich isotherm			
B (J/mol)	A (L/mg)	b_T	R^2	q_s(mg/g)	E (KJ/mol)	K_{DR} (mol^2/J^2)	R^2
40.23	44.21	63	0.97	217.32	3.535	4 X10^{-8}	0.95

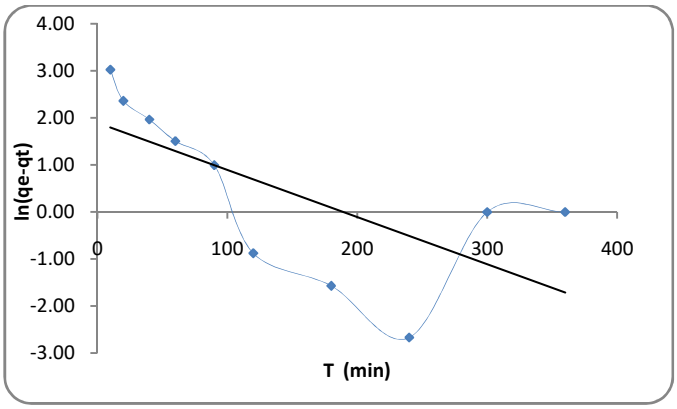
Adsorption Kinetics

The adsorption mechanism depended on the physical or chemical characteristics of the adsorbent. Fig.3.3 shows the linear regression fits of ln(qe-qt) Vs. t (Fig. 5(a)), t/qt Vs. t (Fig. 5(b)) and qt Vs. $t^{1/2}$(Fig. 5(c)). The kinetic parameters obtained from the slopes and intercepts are shown in Table VI. The parameter obtained shows that the pseudo-second-order kinetic model gives better fit with the experimental data of Reactive Dye adsorption by AC compare to pseudo-first-order kinetic model [24].

The pseudo-first order equation predict that the adsorption of one Reactive Dye molecule on one active site over AC surface, while in pseudo-second order model one Reactive Dye molecule is adsorbed onto two active sites. The adsorption phenomenon for AC was rapid at initial stage and then decrease slowly upto equilibrium. At the initial stage of adsorption, the rate of adsorption of Reactive Dye was fast because of high concentration gradient of Reactive Dye [17] in liquid phase and available of large number of active sites on

the surface of AC. These predict that both physical and chemical adsorption might be involved in these phenomena.

The Intra particle Diffusion Model is shown in Fig. 5(c) indicate a linear relationship between the amount of dye adsorbed and the square root of the contact time. Result shows that line do not pass through the origin, which means that the adsorption process is not limited to intra-particle diffusion alone [24].



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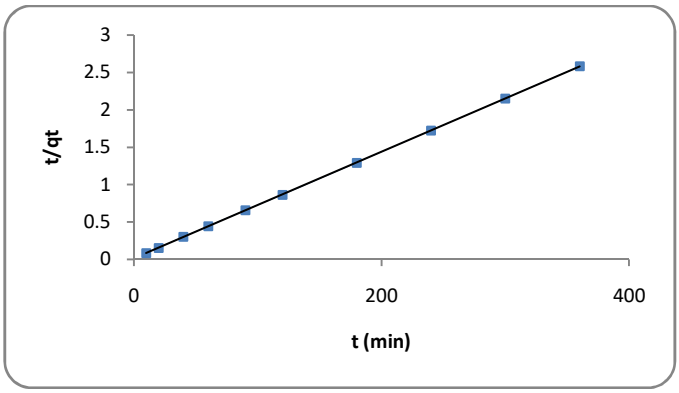

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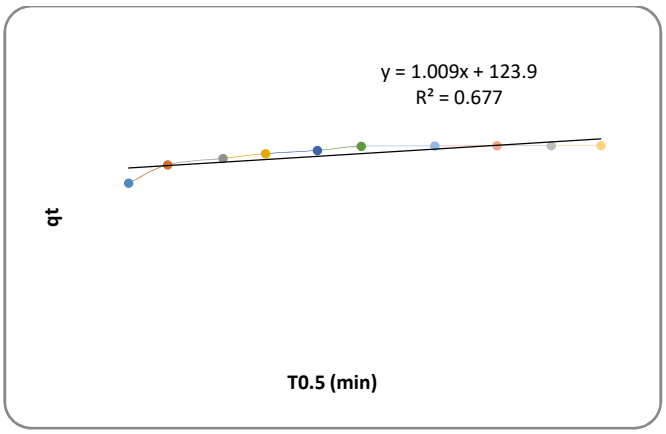

(c)

Fig.5. Linear regression curves of (a) pseudo first order, (b) pseudo second order and (c) Intraparticle diffusion

TABLE 6
Estimated constants for adsorption of Reactive Dye

Sr No.	1			
Name of Model	Pseudo First order			
Kinetic Parameters	$q_e$exp (mg/g)	k_1 (min^{-1})	$q_e$cal (mg/g)	R^2
Value	120.77	0.015	17.84	0.73
Sr No.	2			
Name of Model	Pseudo Second order			
Kinetic Parameters	$q_e$cal (mg/g)	k_2 (g/mg min)	h (mg/g min)	R^2
Value	121.95	1.85 X 10^{-3}	27.54	0.999
Sr No.	3			
Name of Model	Intra-particle Diffusion			
Kinetic Parameters	K_{int}(mg/g $min^{1/2}$)	C_i	R^2	
Value	1.53	98.77	0.78	

Thermodynamic Modeling

The abstract of thermodynamics commences that in an isolated system, the entropy change is the driving force. The thermodynamic parameters to be considered for adsorption processes were, 1.Changes in standard enthalpy (ΔH^0), 2.Standard entropy (ΔS^0), 3.Gibbs standard free energy (ΔG^0) due to transfer of unit mole of solute from solution onto the solid– liquid interface [13].

The value of ΔH^0 and ΔS^0 could be calculated using the following equation,

$$lnKc = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \qquad (13)$$

Where, R = Universal gas constant,8.314 J/mol K
T = Absolute solution temperature and
K_c= C_i/C_e (L/mg) is the equilibrium constant.

The values of ΔH^0 and ΔS^0 could be calculated, respectively from the slope and intercept of the Van't Hoff plot of lnK_C versus $1/T$ as shown in Fig. 6. ΔG^0 could be calculated using the relation below,

$$\Delta G^\circ = -RTlnKc \qquad (14)$$

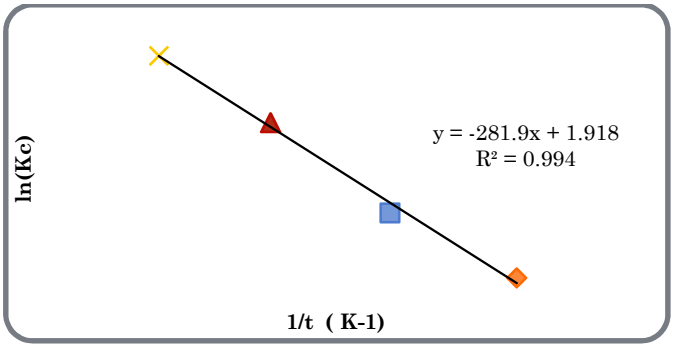

Fig. 6.Van't Hoff plot for adsorption of Reactive Dye

TABLE 7
Thermodynamic parameters for adsorption of Reactive Dye

TEMP (°C)	30	40	50	60
$Ln(K_C)$	0.99	1.01	1.05	1.07
ΔG (J/mol.)	2493.24	2638.20	2811.87	2967.45
ΔH (J/mol)	2344			
ΔS (J/mol.K)	15.94			

The negative value of ΔG^0 shows that the adsorption of Reactive Dye by AC was a viable and spontaneous process[8][12]. The positive value of ΔH^0 was indicating that the adsorption of Reactive Dye by AC was an endothermic process. The negative value of ΔS^0 further indicated smaller disorder degree in the whole adsorption process and affinity of Reactive Dye towards AC[13].

IV. Conclusion

The result of this experiment shows that mustard straw-based Carbon with H_3PO_4as activating agent is shows optimum adsorbent for the removal of Reactive Dye from aqueous solutions over a wide range of concentrations. AC was produced from biomass in a two-step process: (1) Biomass to Biochar (2) Biochar activation. 750° C, impregnation ratio of 4:1 and holding time of 100 min. The BET surface area was 1350 m^2/g and the SEM images were similar to commercial AC. Balance data was best represent by the Langmuir Isotherm models. The kinetics of the adsorption process was derive to follow the pseudo-second-order kinetic model. Thermodynamic parameters have shown that the adsorption phenomenon is spontaneous, endothermic and increases the degree of process disorder.

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