

Physico-chemical Parameters analysis for the adsorption of *Acid orange II* Dye on activated carbon of waste Leathers

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ABSTRACT

The feasibility of doing away with Acid Orange II dye from aqueous solutions the usage of adsorbent Activated Carbon of waste leather (ACWL) has been analysed. Batch mode adsorption researches are carried out in order to inspect the adsorption capacities of this adsorbent by using various initial dye concentration, contact time, temperature and pH. Results expose that adsorption capability decreases from 95.07% to 76.04% for (ACWL) at 30 °C with an expand in the initial concentration from 10 to 60 mg l⁻¹. The calculated q_e values agree very properly with experimental values. The regression coefficient values above 0.99 verify that adsorption follows 2nd order kinetics. The enlarge in Langmuir adsorption potential (q₀) is 17.66784 mg l⁻¹ (ACWL) on growing the temperature from 30 to 60°C bills for the endothermic nature of the process. The current study confirms the potentiality of plentiful low price solid waste leather and its availability for the elimination of acidic dyes from aqueous solution.

Key words: *Acid orange II, Activated carbon, Adsorbent, Kinetics, Isotherm.*

Introduction

Textile industries ranks first in the usage of dyes when in contrast to different industries like food, paper, cosmetics and carpet industries (Lago *et al.*, 2021). Many physically and chemical techniques such as adsorption, coagulation, precipitation and filtration have been used to eliminate unsafe dyes from colored waste water. Adsorption is the most wonderful and reasonably-priced approach for elimination of dyes. Many researchers have proved countless low price substances such as pear millet husk carbon (Han *et al.*, 2020). *Aspergillus niger*

(Ahammad *et al.*, 2021) rice husk, banana pith, cotton waste, kaoline, coir pith, guava seeds (Husen *et al.*, 2021) and neem noticed dust (Esad Behrami *et al.*, 2022), clay (Abdoulaye Demba *et al.*, 2021) and mango seed kernel (Cleary *et al.*, 2019) as appropriate adsorbents for the elimination of dyes. Activated carbon was prepared with the aid of carbonization of waste leathers accompanied by means of acid digression 0.1M HCl with H₃PO₄. to find out about the suitability activated carbon for the adsorption of acid orange II from aqueous solution used to be performed by using batch mode adsorption studies.

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Materials and Methods

Preparation of Dyes

The dyes chosen for the adsorption research in the existing work had been Acid orange II. Stock solution with a concentration of 1000 mg l⁻¹ were prepared through dissolving the required quantity of substances, as shown in Table 1. The test solutions were prepared by suitable dilution of this solution. The pH of the solutions had been adjusted by using addition of 0.1 M solution of HCl or NaOH

Preparation of adsorbent

Activated carbon of waste leather (ACWL) accumulated from the leather-based industries, Pallaavaram area, have been overwhelmed with laboratory-scale crushers, powdered with a disk pulverizer, and sieved to 0-63 mesh (ASTM). The powdered adsorbent used to be washed, dried at 105 °C for 10 hr in an oven, and saved in high-density polythene (HDPE) bags. Powdered adsorbent used to be soaked in HCl 0.1 M solution for 24 h, accompanied by means of filtering and washings with distilled water. Afterwards, it was dried in an oven at 105 °C for 10 h and saved in HDPE bags.

Determination of Carbon Characteristics

The activated carbon samples have been characterized by the use of FT-IR SHIMADZU spectrophotometer. The surface morphology of the adsorbents has been found with XRD and SEM.

Proximate Analysis of Carbon

Activated carbon samples have been prepared from the waste leathers collected from the leather industries. About 500 g of samples have been amassed in an air tight aluminum container. Samples for proximate analysis, have been pulverized to a mesh dimension < 100 µ and dried for 12 hours in a desiccators. For proximate analysis for the dedication of moisture content, volatile matter, ash content material and constant carbon laid down. (Indian Standard IS: 1350 (Part- I)-1984 (Benjelloun *et al.*, 2021) was followed.

Ultimate Analysis

The Ultimate evaluation of activated carbon is used for determination of carbon, hydrogen, sulphur, nitrogen and ash content as observed in gaseous products for entire combustion. The trendy technique is

defined in (IS:1350, phase II-2000) (Benjelloun *et al.*, 2021). The quantity of warmth generated at some point of the combustion of a unit of weight of a activated carbon pattern is described as calorific value. Activated carbon calorific values and their traits have been decided in accordance to IS: 1350-1974, 1975 in a bomb calorimeter.

Adsorption Experiments

The adsorption studies were carried out at 30 ± 1 °C and pH of the solutions were adjusted with 0.1M HCl and 0.1M NaOH. A known amount of adsorbent was added to system and allowed sufficient time for adsorption equilibrium. The batch experiments had been carried out in Erlenmeyer glass flasks of 100 ml capacity. To assist mixing of the options with the adsorbents, the combination used to be shaken in a mechanical shaker (Remi make) at appropriate (100-300) rpm for three hours. Then the mixture was filtered and the remaining dyes concentration was determined in the filtrate using Spectro UV-Vis Double Beam UVD- 3500, Labomed Inco photo meter at suitable λ max. The effect of various parameters on the rate of adsorption process were observed by varying mesh dimension of adsorbent, contact time (t), initial concentration of dyes (Co), adsorbent dose, initial pH of solution and temperature. The solution volume (V) was kept constant 50 ml. The dyes adsorption (%) at any instant of time was determined by the following equation:

$$\text{Dyes adsorption (\%)} = (C_o - C_e) \times 100 / C_o$$

Where

C_o is the initial concentration and

C_e is the concentration of the dyes at equilibrium.

To increase the accuracy of the data, each experiment was repeated three times and average values were used to draw the graphs.

Adsorption isotherms

Adsorption research has been made at distinct initial concentrations, ranging from 10 mg l⁻¹ to 60 mg l⁻¹, at most efficient pH, for a contact time of 180 min. at room temperature. From the information obtained, the mechanism of adsorption used to be studied, by the utility of four isotherm equations, particularly these of Langmuir (eq: 1), Freundlich (eq: 2), Redlich-Peterson (eq: 3) and Dubinin-Kaganer-Radushkevich (DKR) (eq: 4) have been plotted by way of the use of fashionable straight-line equations and corresponding parameters had been calculated from their respective graphs.

$$C_e/X = 1/K \cdot K_L + C_e/K \quad \dots (1)$$

$$K_L = b \cdot q_0$$

$$\log q_e = \log K_F + 1/n \log C_e \quad \dots (2)$$

$$q_e = K_R C_e / (1 + b R C_e \beta) \quad \dots (3)$$

$$\log q_e = \log X_m \beta \epsilon^{2/2.303} \quad \dots (4)$$

Kinetics of adsorption

The % of adsorption of dyes used to be decided at more than a few time intervals, particularly 10, 20, 30, 40, 50 and 60 mins. The information had been fed into 4 specific kinetic models and the equilibrium adsorption capacities and different beneficial parameters had been calculated, they are Lagergren's pseudo-first order kinetics(eq:5), Pseudo-second order kinetics(eq:6), Elovich kinetics(eq:7), and intra-particle diffusion model(eq:8).

$$\log (q_e - q_t) = \log q_e - k_1 t / 2.303 \quad \dots (5)$$

$$t/q_t = 1/k_2 q_e^2 + t/q_e \quad \dots (6)$$

$$q_t = 1/\beta \ln \alpha \beta + 1/\beta \ln t \quad \dots (7)$$

$$q_t = k_p \cdot t^{1/2} \quad \dots (8)$$

Thermodynamics of the adsorption

Four vital thermodynamic parameters had been deduced for the adsorption process, specifically the alternate in Gibbs free energy (ΔG° , eq:9), Enthalpy changes (ΔH° , eq:9), change in Entropy (ΔS° , eq:10) and the activation energy (E_a , eq:11)

$$\Delta G^\circ = -RT \ln K_c = \Delta H^\circ - T\Delta S^\circ \quad \dots (9)$$

$$\ln K_c = -\Delta H^\circ / RT + \Delta S^\circ / R \quad \dots (10)$$

$$\log K = \log A - (E_a / 2.303RT) \quad \dots (11)$$

Results and Discussion

Proximate Analysis

The effects of proximate evaluation for moisture, volatile matter, fixed carbon, and ash are given

Fixed Carbon

The fixed carbon content material of activated carbon, aside from moisture and ash, is 91.2%. The constant carbon values rely generally on the C and O values in the activated carbon. It is nicely recognised that the FC content material will increase with the improve of the activated carbon rank (Suo *et al.*, 2019).

Ultimate Analysis

The consequences of analyzed parameters Carbon,

Hydrogen, Nitrogen, Sulphur and Oxygen are tabulated. The consequences of every factor are mentioned under in detail.

Carbon

The Carbon content material in the amassed activated carbon samples from the learn about vicinity is 86.54wt %. The excessive concentrations of C are usually attributed of vitrinite macerals. It is additionally nicely recognised that the C content material in activated carbon will increase step by step with growing activated carbon rank (Adewuyi *et al.*, 2020).

Oxygen

The oxygen content in the samples is 8.5% by way of weight. The decrease O content material is attributing of high-range activated carbon (Abhishek Sharma *et al.*, 2021).

Effect of particle Size

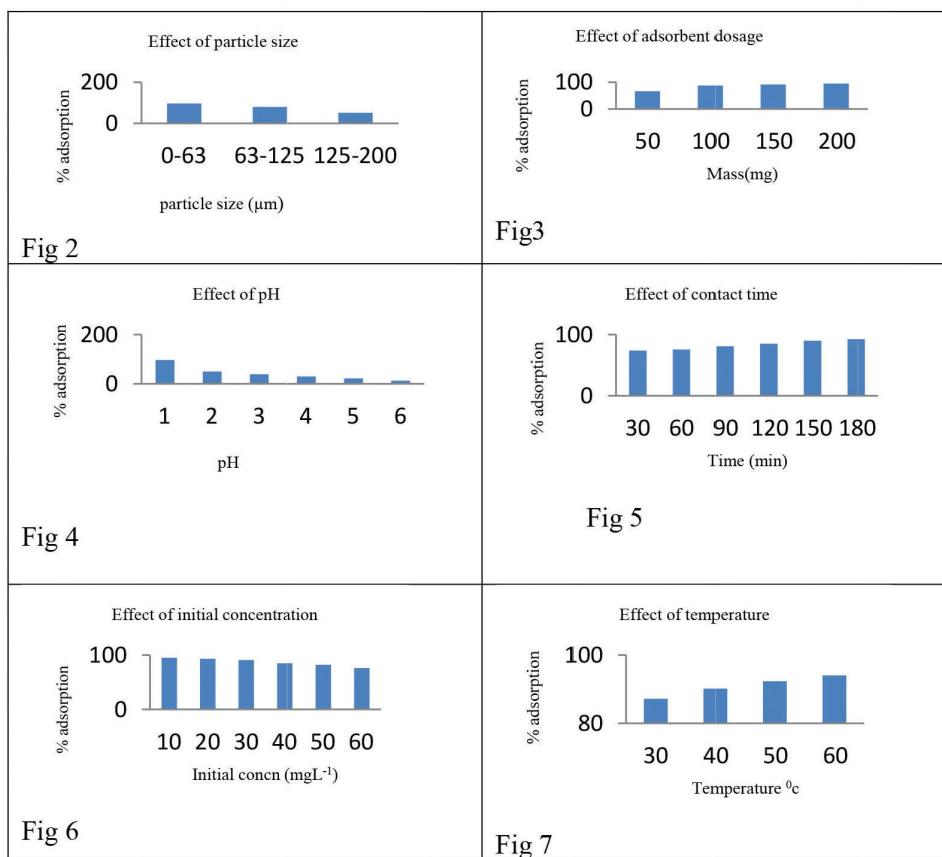
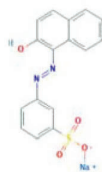
The effect of adsorbent's particle size was studied in the range of 0-200 microns particle size, the maximum adsorption of Acid orange-II at the smallest particle size (0-63) was shown to be best for adsorption, 96.66%, Smallest size of adsorbent presents a larger surface area and the results are shown in Figure 2. Particle measurement of an adsorbent performed a very vital position in the adsorption potential of dye. The relationship of adsorption ability to particle dimension relies upon on two criteria. (i) The chemical shape of the dye molecule (its ionic charge) and its chemistry (its capability to shape hydrolyzed species) and (ii) Its crystalline and porosity (Audrey Tchinsa *et al.*, 2021; Zakariyya Uba Zango *et al.*, 2021).

Effect of adsorbent dosage

The results, as plotted in Figure 3, point out that the Acid orange-II elimination proportion marked up from 66.79 % to 95.01 %. The strength might also be attributed to enlarge in the wide variety of adsorption sites. In contrast, with a growing dose of activated carbon, the adsorption potential of Acid orange-II onto ACWL reduced above 200 mg. This reduce ought to be attributed to the break up in the flux or concentration gradient between the solute in solution and solute at the surface of the adsorbent, or to the protecting of adsorption sites following a discount in the whole adsorption surface-area on hand to the dye, and a upward thrust in the diffu-

Table 1. Preparation of stock solution of Dye

Dyes	Molecular Formula of dye	Weight (g) of salts per liter
Acid orange -II	$C_{16}H_{11}N_2NaO_4S$	1.000



sion pathway (Lesley Joseph *et al.*, 2021, Mahmudul Hassan Mondol *et al.*, 2021).

Effect of pH

The impact of pH on the elimination of Acid orange-II dye has been investigated. Solution pH performs an essential position in controlling value of the adsorbent, the degree of ionization of the adsorbate in the solution as nicely as dissociation of various functional groups on the active sites of the ACWL adsorbent. In most cases, pH is termed as the 'master vari-

able'. The adsorption scan of dye Acid orange-II was performed in 1-6 pH vary at 30 °C for a 180 mins. The equilibrium sorption potential used to be lowest at pH 6 and most adsorption of the dye was achieved at pH: 1. Fig. 4 (Pinar Gumus *et al.*, 2021).

Effect of contact time

Figure (5). Adsorption increases as contact time increases and is most (92.41 %) at 3h exhibits that the curve is easy and continuous. Acid orange-II dye to saturation, suggesting viable monolayer coverage of

the dye on ACWL. Nearly 74.00 % adsorption has taken vicinity inside 30 minutes of contact time with the adsorbent which suggests the effectivity of ACWL as adsorbent. More time is required for attainment of equilibrium. In this study, all the batch experiments have been carried out for a time interval of a 180 minutes (Arie Wibowo *et al.*, 2021).

Effect of initial concentration

The adsorptions of Acid orange-II dye on ACWL at a number of initial concentrations are shown in Figure (6). It exhibits that, the true quantity of dyes adsorbed per unit mass of ACWL decreased with enlarge in dye concentration. Adsorption is most when the initial concentration of Acid orange-II dyes has been 10 mg l⁻¹. As the concentration increases, all the adsorption sites are being stuffed up and there stays un adsorbed dye, consequently the

minimize in share adsorption. This end result is in favour of solely monolayer coverage and suggests the utility of the Langmuir isotherm model. Since 95.07 % adsorption happens when the initial concentration was 10 mg l⁻¹, ACWL appears to be very wonderful adsorbent in casting off even traces of dye. Exhibits that adsorption capability decreases from 95.07 % to 76.04% as the dye concentration is increased from 10 to 60 mg l⁻¹ (Natalia Manousi *et al.*, 2021).

Adsorption isotherm

The equilibrium adsorption isotherms are fundamental in perception the interactive nature between adsorbents and adsorbates, as they are beneficial in designing adsorption systems. The effect of Acid orange-II concentrations (10-60 mg l⁻¹) used to be examined and introduced in figures 8 (a, b, c, d, e) the

Table 2. Figures 8 (a, b, c, d, e)

Isotherm parameters for the adsorption of Acid orange II dye on ACWL at 30° C
pH: 1.0, Adsorbent dosage: 150 mg, Initial concentration: 10 mg l⁻¹

Langmuir parameters KL	q ₀	bL	R ²
6.7659	17.66784	0.38295	0.994
Freundlich parameter KF	n	R ²	
4.9705	1.435956	0.9683	
Dubinin-kaganer-Radushkevich parameters â	b	q ₀	R ²
6.02235	3.05	1122.018	0.85
Redlich Peterson parametersâ	bR	KR	R ²
0.2246	4.45236	2.30187	0.945

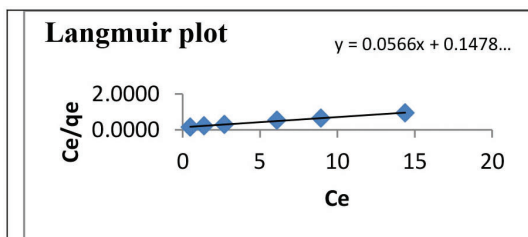


Fig 8a

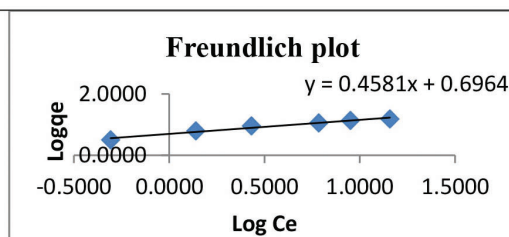


Fig 8b

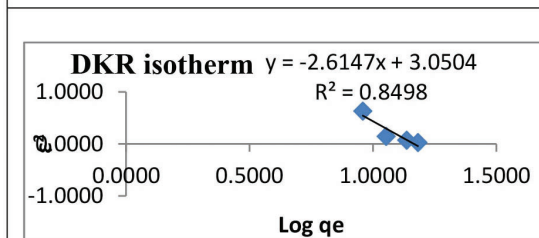


Fig 8c

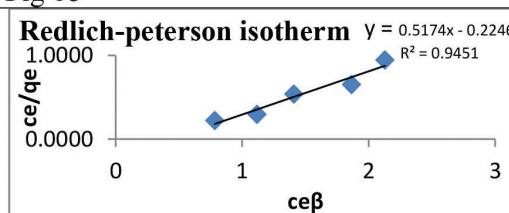


Fig 8d

place equilibrium of the exceptional concentrations in an aqueous solutions, C_e (mg l^{-1}), and the quantity of Acid orange-II adsorbed on the activated carbon, q_e (mg/g), have been plotted as a feature of the equilibrium adsorption of Acid orange-II in aqueous solution. The K_L (sorption equilibrium constant) value for the Langmuir isotherm, i.e. 6.7659 mg/g indicated the excessive adsorption potential of adsorbent towards dye adsorption. This is in flip supported via the values of the dimensionless separation component (R_L), which are much less than one. The correlation coefficient R^2 value 0.994 indicated that the Langmuir isotherm is true for explaining the dye adsorption. The R^2 value calculated for the Freundlich isotherm was determined to be 0.968, indicating that the experimental information can be defined through the Freundlich isotherm. The final adsorption ability value K_F as calculated from the Freundlich isotherm was 4.9705 (Imteaz Ahmed *et al.*, 2021).

Kinetics

The kinetics of adsorption of Acid orange-II dye on

ACWL was observed with the aid of the use of 4 models, Lagergren's pseudo-first order kinetics (eq3.5), Pseudo-second order kinetics (eq3.6). The Elovich kinetics (eq 3.7), the intra-particle diffusion model. (eq 3.8). The kinetics parameters are given in Table 3 and Figures 9 (a, b, c, d, e). Analysis of the kinetics parameters indicates that there is no correlation between theoretical and experimental q_e values for the Lagergren's first order kinetics and therefore the adsorption system is now not in all likelihood to be of the first order. The R^2 values for the 2nd order kinetics suggest excessive precision suggesting that 2nd order kinetics is a first-rate fit (Mushtaq *et al.*, 2019).

Effect of temperature

The elimination of Acid orange-II is studied at extraordinary temperatures viz. 30-60 °C for the determination of adsorption isotherm and thermodynamic parameters. It had been investigated from the scan that with the upward push of temperature from 30-60 °C the quantity of dye uptake is increased from 87.21% to 94.02%. As the value of dif-

Table 3 and Figures 9. (a,b,c,d,e)

Kinetic parameters for the adsorption of Acid orange dye –II on ACWL at 30° C
pH: 1.0, Adsorbent dosage: 150 mg, Initial concentration: 10 mg L^{-1}

Kinetic model	R^2
I order	0.97
II order	0.99
Int part diff	0.96
Elovich	0.89

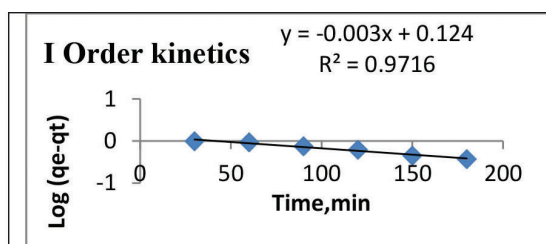


Fig 9a

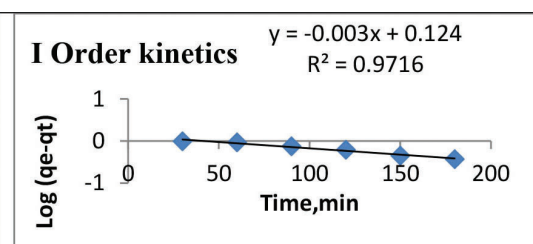


Fig 9b

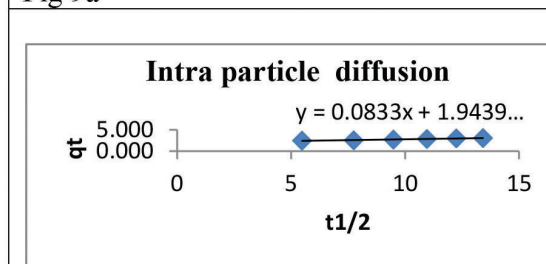


Fig 9c

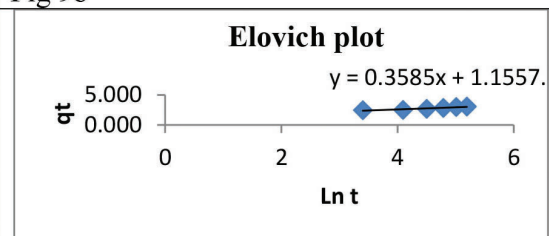


Fig 9d

fusion of the dye molecules is a temperature-controlled process Figure (7) (Harja *et al.*, 2021).

Thermodynamic Parameters

The thermodynamic parameters for the adsorption of Acid orange-II dye on ACWL are given in tables 4 (a, b). For the adsorption of dye on ACWL; the change in enthalpy values is all low confirming that the technique is spontaneous even at room temperature. The entropy change is high quality and explains the enlarge in randomness of the process. The endothermic nature of the adsorption manner is evident from the superb values of enthalpy change. Presumably, the randomness aspect ($T\Delta S^\circ$) overcomes the enthalpy element (ΔH°) and makes the usual technique spontaneous (ΔG° negative). Activation energies for adsorption of Acid orange-II dye on adsorbent used to be calculated. The use of the Arrhenius equation (eq11), plotted in Figure 10(c, d) and tabulated in Tables 4 (a, b). The activation energy acquired in this case, point out that physical forces are concerned in the sorption mechanism and sorption feasibility (Jiang *et al.*, 2020).

FT-IR Study

Figures 11 (a, b) show the FT-IR spectra of activated

carbon in its natural structure and dye loaded shape respectively. The spectra exhibited countless peaks representing that noticed dirt is composed of more than a few useful groups which perhaps assist in binding of the dye molecules. The spectra of the dye loaded activated carbon confirmed comparable traits as the activated carbon in shape barring for moderate changes. The FT-IR spectrum of the dye loaded adsorbent suggests that the peaks are barely shifted from their function and the intensity gets altered. Thus, the FT-IR of the surface moieties remained unchanged (Liu *et al.*, 2008).

SEM analysis

The surface morphology of ACWL was examined the use of Scanning Electron Microscopy (SEM), before and after adsorption and the corresponding SEM micrographs had been got at an accelerating voltage of 20kV at 10000 magnification and are introduced in figures 12 (a) and 12 (b). At such magnification, the ACWL particles confirmed difficult areas of surface inside which microspores had been truly identifiable. Figures 12 (a, b) indicates the SEM micrograph of ACWL derived activated carbon. It is clear from this picture that the surface is tough and distinctly corrugated (Mushtaq *et al.*, 2019).

Tables 4 (a, b) & Figures 10 (c, d)

Thermodynamic parameters for the adsorption of Acid orange II dye on ACWL at 30°C
pH:1.0, Adsorbent dosage: 150 mg, Initial concentration: 10 mgL⁻¹

ΔG_0	ΔH_0	ΔS_0	$\log_{10} K_a$	1/T
-2069.35	15.3177	68.9297	0.356574	0.003299
-2918.65			0.486859	0.003193
-3732.97			0.603426	0.003095
-4588.12			0.719398	0.003002

Arrhenius parameters for the adsorption of Acid orange II on ACWL

Ea	Log A	R ²
23.3059	4.3722	0.999

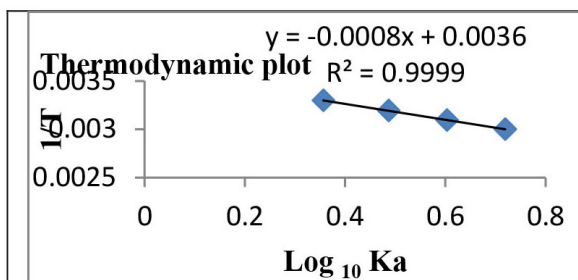


Fig 10c

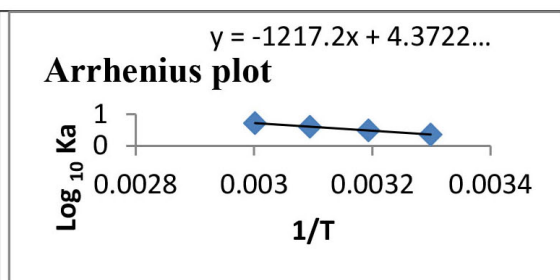
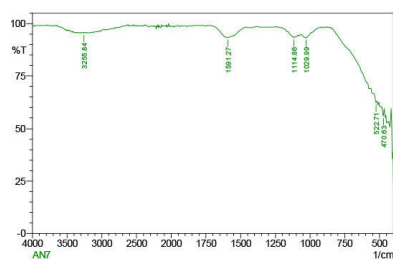
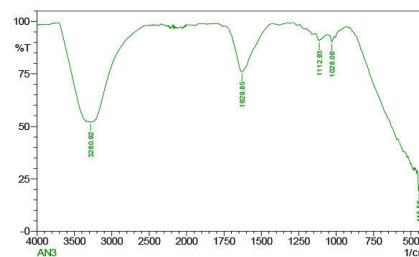


Fig 10d

Figure 11(a). 11(b) FT-IR spectrum of ACWL of Acid orange II

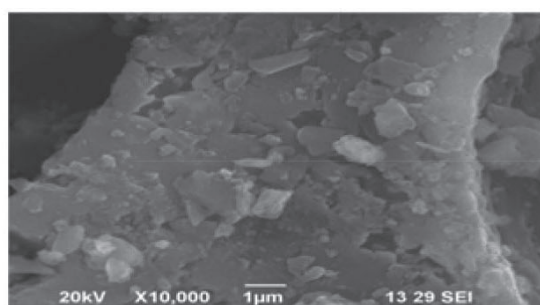


13(a) Before adsorption

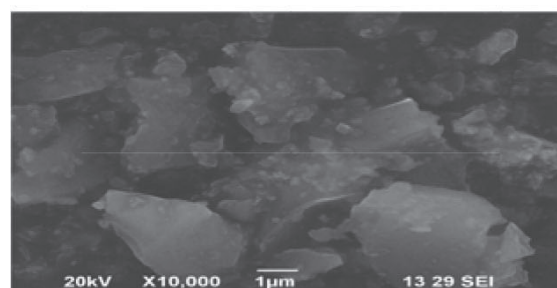


13(b) After adsorption

Figure 12(a).12(b) SEM micrograph of ACWL Acid orange

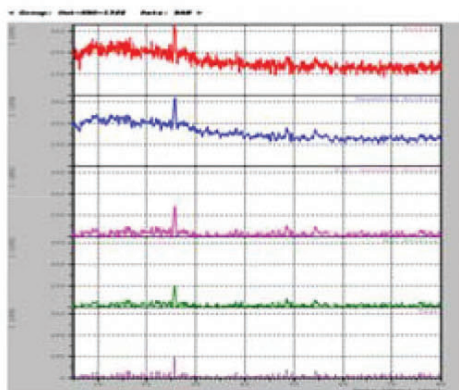


14(a) Before adsorption

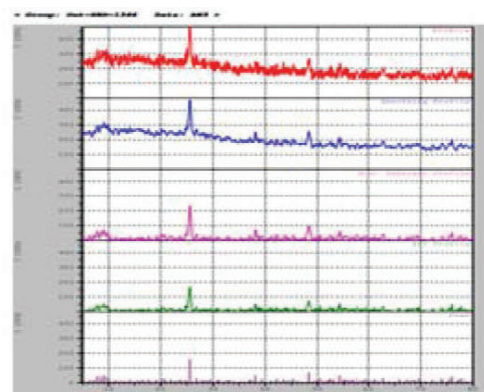


14 (b) After adsorption

Figure 13(a).13(b). XRD pattern of ACWL Acid orange II



15(a) Before adsorption



15(b) After adsorption

XRD Study

The X-ray Diffraction Studies of the adsorbent ACWL, before and after adsorption of dye, had been carried out the use X-ray Diffractometer 40KV / 30mA, Model D/Max ULTIMA III. The diffraction patterns are suggesting in Figures 13 (a) and 13 (b). It is evident from the figures that there is no considerable exchange in the spectra of adsorbent before and after adsorption. This may additionally be due

to the truth that adsorption does not alter the chemical nature of the surface of the adsorbent. The adsorption is ruled by way of vulnerable Van der Waals forces and is physical in nature (Natalia Manousi *et al.*, 2021).

Conclusion

The existing find out about displays that ACWL are

used efficaciously as adsorbents for the elimination of acid orange II from aqueous solution. The quantity of dye adsorbed is observed to differ with preliminary pH, temperature and contact time. Maximum adsorption takes place at pH 1 for acid orange II. Kinetic research predicts that the adsorption of acid orange II onto ACWL follows pseudo second-order kinetics. The adsorption isotherms like Freundlich, Langmuir, Redlich Peterson and Dubinin-Radushkevich had been analyzed for the adsorption of acid orange II onto ACWL. Freundlich isotherm predicts that the adsorption is physisorption in nature. The equilibrium facts match very nicely into the Langmuir isotherm mannequin which suggests monolayer adsorption. The adsorption potential of ACWL increased with upward thrust in temperature indicating endothermic nature of adsorption. Intra-particle diffusion mannequin predicts that pore diffusion performs a important position for the adsorption of acid orange II onto ACWL. On evaluating the results, it is apparent that, for the adsorption of acid orange II, ACWL is an efficient, monetary and alternative adsorbent.

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