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Adsorption Characteristics Study on the Removal of Therapeutic Drug *Ibuprofen* Pollution on the Acid Digested Carbon of Waste Leathers (RS) - Leathers

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ABSTRACT

The adsorption characteristics study on the removal of therapeutic drug ibuprofen on the acid digested carbon of waste leather were analysed by varying the physico-chemical conditions. The minimum particle size gives maximum number of surface area and adsorbed more, 91.25% for 0-63 micron. Acidic pH ranges were optable for the adsorption of ibuprofen on this ADCL. But different adsorbates preferred different acidic pH ranges of pH from 1 – 6. In this case at pH 5, 93.1% of adsorption takes place. The percentage of adsorption of ibuprofen is directly proportional to the adsorbent dosage and contact time and inversely proportional to the initial concentration of the adsorbate were found out from this study. Order of this adsorption is pseudo second order kinetics and it belongs to the physisorption, because of no chemical bond formation between the adsorbent and adsorbate. The freundlich and Langmuir isotherm model is fit for these studies. The thermodynamic study reveals the negative ΔG° and positive ΔH° and ΔS° values concluded that this adsorption is spontaneous, feasible and physical in nature respectively. The spectral evidence from the FT-IR, SEM and XRD are in favour of above experimental results.

Key words: Activated carbon Waste Leather, Ibuprofen, Kinetics, FT-IR, SEM and XRD.

Introduction

One of the most often used medications in the world is ibuprofen. With an estimated yearly output of many kilotons, it is one of the top five most used narcotics in the United Kingdom, for example (Sebastine and Wakeman, 2003). It is a common non-steroidal anti-inflammatory (NSAID), analgesic, and antipyretic medication used to treat fever, pain, and rheumatic illnesses. It has great mobility in the aquatic environment because although being just marginally soluble in water, it quickly dissolves in organic solvents. Ibuprofen is prevalent in amounts

on the order of ppb (lg dm^{-3}), according to measurements done on German rivers and wastewater (Ternes, 1998). Recent researches on the removal of drugs from drinking water or wastewater have shown that standard processes (coagulation, flocculation, and sedimentation) are not entirely effective (Ternes *et al.*, 2002). On the other hand, procedures based on activated carbon and oxidation with chlorine and ozone appear to be more effective at reducing Drugs (Yoon *et al.*, 2003). The employment of technologies based on activated carbons is then a potential option to remove Drugs from aqueous medium or serve as concentrators of those pollut-

ants for examination. The presence of benzene rings or amine groups in the structure of the bulk of these compounds increase their capacity to be adsorbed by the activated carbon, so that activated carbons may, in reality, remove any drugs.

There are numerous articles in the literature that discuss the use of activated carbons in the treatment of liquid effluents. The adsorption of metals (Koby, 2004), dyes, phenolic compounds, and endocrine-disrupting substances (Westerho *et al.*, 2005) is the main focus of these investigations. We are aware of very few research that address the removal of Drugs by activated carbons, and in each of these investigations, commercially available carbons were used (Cagnon *et al.*, 2005).

It is crucial to develop low cost methods for decontamination procedures while maintaining high removal efficiency due to the amounts of wastewater that must be treated in industrialised countries and the fact that environmental rules are growing more rigorous. Waste-derived activated carbons from industrial or agricultural byproducts are an excellent substitute for inorganic carbon when it comes to adsorption-based technologies. Leather processing is a significant economic industry in India that produces significant quantities of powdered leather trash. In recent years, we have employed this inexpensive, environmentally acceptable material as a precursor to create activated carbons with high apparent specific surfaces and effective performance as noxious volatile organic compound adsorbents (Carvalho *et al.*, 2003). These findings support a review of the leather-based activated carbons' capability for removing contaminants from solutions. For the current investigation, a drug called ibuprofen was chosen.

Materials and Methods

Adsorption Experiment

The studies were conducted at 30°C, with 0.1M HCl and 0.1M NaOH used to change the acidic and basic properties of the solution. The adsorbent and adsorbates were allowed to come into contact with each other for a set amount of time to allow for adsorption, and the amounts of adsorbent and adsorbates were recorded. For this experiment, a 100ml stopper bottle was used, and the mixture was agitated for 180 minutes with a mechanical shaker set to the right rpm (100-300). It is filtered after the equilib-

rium with watt man 40 filter paper and filtered. A UV-spectro photometer set to a suitable filter range was used to check the drug adsorbent solution concentration in the filtrate. The experiment was performed with the physical and chemical parameters changed, and the adsorbate volume was 50 ml.

$$\text{Adsorption (\%)} = (C_0 - C_e) \times 100 / C_0$$

Where C_0 is the IC is initial concentration and C_e is the concentration of the drug adsorbate at equilibrium.

Results and Discussion

Effect of particle Size

Ibuprofen was adsorbed on ADCL using several particle sizes ranging from 0-63, 63-125, and 125-200 μ . The percentage of elimination was reduced as particle size increased. The reduction in particle size increases the Ibuprofen uptake at a constant adsorbent dosage. Smaller particles were able to reach pores more easily and had a larger surface area for bulk adsorption per unit mass of the adsorbent, which led to an increase in uptake. Results are displayed in Fig.1 and reveal that the smallest particle size (μ) was found to be the best for adsorption. In each experiment, 50 ml of a 10 mg/l⁻¹ Ibuprofen solution containing 150 mg of adsorbent was agitated until equilibrium was reached after 180 min. The adsorbent was then separated, the supernatant solution was tested for Ibuprofen content, and the results are shown in Fig. 1 (Afkhami *et al.*, 2007).

Table 1. Ibuprofen

Particle Size (μ)	% Adsorption
0-63	91.25
63-125	75.96
125-200	49.89

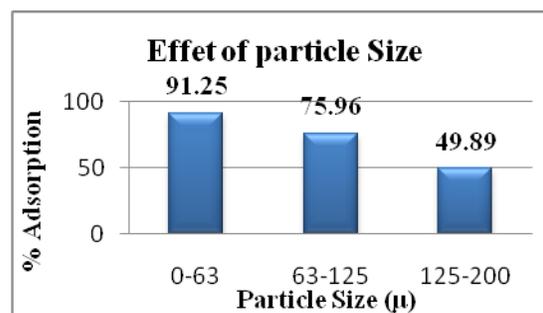


Fig. 1.

Effect of pH

The pH of the solution has a significant impact on the absorption and percentage removal of Ibuprofen from the aqueous solution, as shown in Fig. 2. When the pH rises from pH:1 to pH:5 as shown in Table 2, the absorption of Ibuprofen increases from 30.03% to 93.01%. At pH 5, Ibuprofen sorption is seen to considerably increase, increasing its adsorption capacity by 93.01%. Ibuprofen molecules were positively charged, as was the adsorbent surface and the adsorption capacity is then marginally reduced in the pH range above 5, and it is greatly reduced due to electrostatic repulsion in 6 and above. Similar patterns are seen at pH levels higher than 6, when the negatively charged ADCL surface deters the anionic molecules of Ibuprofen. The observed reduction in Ibuprofen adsorption with increase in pH is an as yet unexplained phenomenon that portends that other adsorption processes and molecular characteristics play major roles. Therefore, it is likely that hydrophobic interactions control Ibuprofen's adsorption to ADCL as well (Ania and Beguin, 2007).

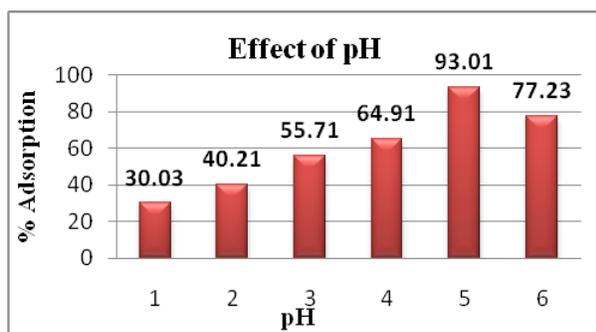


Fig. 2.

Table 2. Ibuprofen

pH	Adsorption %
1	30.03
2	40.21
3	55.71
4	64.91
5	93.01
6	77.23

Effect of Adsorption Dosage

At the ideal pH of 5.0 and at room temperature of 30 °C, the role of adsorbent mass in the adsorption of Ibuprofen was examined. 50mg of the adsorbent was added to 50ml of an Ibuprofen solution with an initial concentration of 10 mgL⁻¹, and the mixture

was agitated for 180 minutes. Ibuprofen concentration in the supernatant was quantified after centrifugation. This allowed for the calculation of the amount of Ibuprofen absorbed on the adsorbent. The results of repeating this technique with 50, 100, 150, and 200mg of the adsorbent are listed in the Table 3. The graphic also displays the impact of adsorbent dose Fig. 3. Adsorption peaked with a dose of 200 mg or higher of adsorbent. As a result, the recommended dosage of the adsorbent was 150mg. The increase in Ibuprofen absorption was clearly visible, going from 65.32% with 50mg of adsorbent to 88.32% with 200 mg. Prior to that, it is evident that the percentage of Ibuprofen removed increases as the dosage of adsorbent rises from 50 mg to 200 mg due to the restricted availability of adsorbing species for a relatively larger number of surface sites on the adsorbent at higher dosage of adsorbent (Ayranci and Duman, 2006).

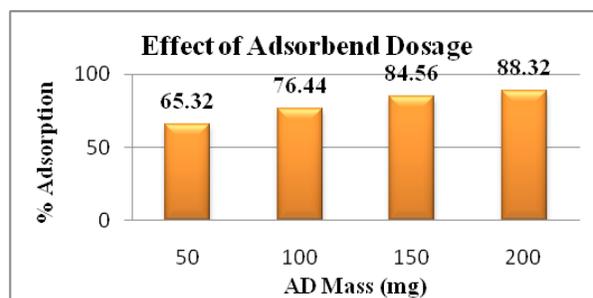


Fig. 3

Effect of Initial Concentration

The results showed that Ibuprofen adsorption capability reduced as the initial concentration increased. Fig. 4 illustrates the influence of initial concentration of adsorbate on Ibuprofen adsorption. The maximum amount of Ibuprofen that could be absorbed was found at a 10 mgL⁻¹ starting concentration. The increased number of exchangeable sites in the adsorbent structure can be used to explain how the initial concentration of the adsorbate affects the percentage of Ibuprofen removal. As the concentration of Ibuprofen increases from 10 to 60 mgL⁻¹, as shown in Fig. 4, the adsorption capacity drops from 89.21%

Table 3. Ibuprofen

AD Mass (mg)	Adsorption %
50	65.32
100	76.44
150	84.56
200	88.32

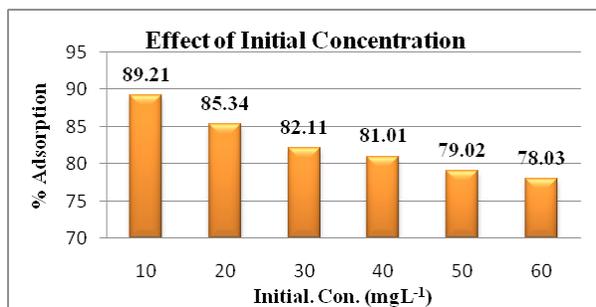


Fig. 4

Table 4. IBUPROFEN

Initial. Con. (mgL ⁻¹)	Adsorption %
10	89.21
20	85.34
30	82.11
40	81.01
50	79.02
60	78.03

to 78.03%. The exchangeable sites in the ADCL structure are saturated as the ratio of Ibuprofen to ADCL drops, which is the explanation for a decrease in the removal percentage with increasing initial concentration of adsorbate in this investigation (Ayrançi and Hoda, 2005).

Effect of Contact Time

According to studies, one of the key elements in the batch adsorption process is contact time. With the exception of contact time, the following parameters were held constant in this stage: temperature, adsorbent dosage, pH, starting Ibuprofen concentration, and agitation speed (150 rpm). In Table 5 and Figure 5, the impact of contact time on Ibuprofen adsorption effectiveness is depicted. As demonstrated, the adsorption rate grew quickly at first, and for any initial concentration, the ideal removal efficiency was attained in 3 hours. Ibuprofen was absorbed in two stages: a quick initial absorption and then a

Table 5. Ibuprofen

Time (min)	Adsorption %
30	63.06
60	71.03
90	75.21
120	78.12
150	86.04
180	89.88

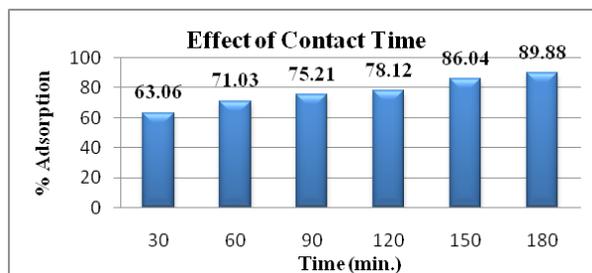


Fig. 5

slower absorption later. When the number of accessible sites is significantly greater than the number of Ibuprofen species that need to be adsorbed, the adsorption process seems to move along quickly. Adsorption rises as contact time rises, peaking at 89.88% after 3 hours. The adsorption phase had reached equilibrium and the equilibrium concentration had not changed significantly. As a result, all tests will have 180 minute contact duration (Azaý's *et al.*, 2006).

Effect of Temperature

The percentage of Ibuprofen adsorption was examined as a function of temperature in the range of 30-60 °C in Figure 6 and Table 6. A higher temperature was shown to boost the adsorption yield. For an initial concentration of 50ppm of Ibuprofen ion solution, the minimum adsorption was 78.11% at 30 °C and the maximum adsorption was 84.22% at 60 °C. Figure 6 and Table 6 illustrate how temperature affects the proportion of Ibuprofen adsorption on ADCL. This is only expected because a rise in temperature gives the endothermic process of adsorption the energy it needs, causing an increase in the

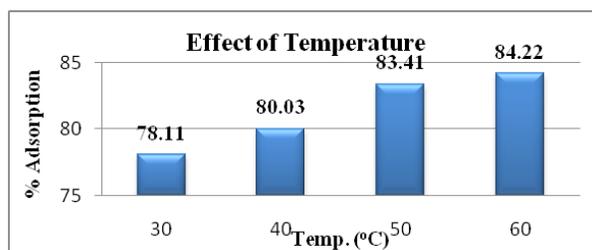


Table 6. IBUPROFEN

Temperature (°C)	Adsorption %
30	78.11
40	80.03
50	83.41
60	84.22

pace of the process. The important thing to understand is that even at room temperature, more than 83.41% of adsorption has already occurred. This in turn confirms the effectiveness of the adsorbent in removing harmful Ibuprofen (Biniak *et al.*, 1997).

Kinetic Study

The adsorption closely matched pseudo-second-order kinetics, implying the model's underlying assumptions of a physisorption rate determining step and the reaction rate being proportional to the number of active binding sites on the adsorbent surface, as shown by the high R^2 values near unity (> 0.98) in Table 7. The solubility of the molecule is one of the factors that affect the adsorption process. If it is high, it interferes with the molecule's affinity for the carbon surface. In this regard, Ibuprofen's increased solubility may be connected to its slower adsorption process. Ibuprofen has a higher affinity for the water molecule than it does for the surface of the adsorbent because of its hydrophilic nature. This is ex-

plained by the fact that Ibuprofen is a smaller molecule and hence packs more effectively on the adsorbent's surface (Cagnon *et al.*, 2005).

Isothermal Study

Using Freundlich and Langmuir isotherm models to match the equilibrium data, the distribution of the adsorbates between the aqueous and solid phases was assessed. Table 8 displays the computed isotherm parameters together with the related R^2 values. The experimental findings had good agreement with both the Freundlich and Langmuir isotherms models. Both postulated multi-mechanistic Ibuprofen adsorption models and the heterogeneous structure of the ADCL surface are explained by these theories. The Freundlich constant, $1/n$, which is between 0 and 1 for heterogeneous surfaces, corroborated the inferred heterogeneity of the surface of ADCL. If there are multiple instances, adsorption is a physical process and numbers falling within the

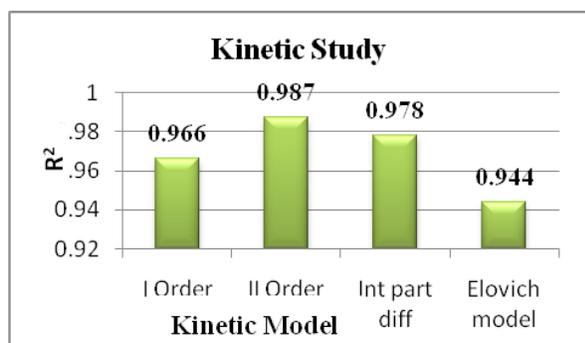


Fig. 7(a)

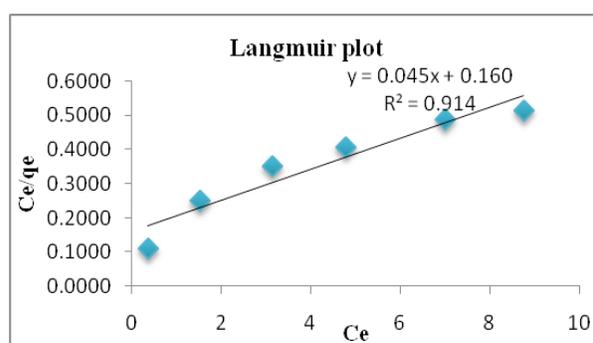


Fig. 8(a)

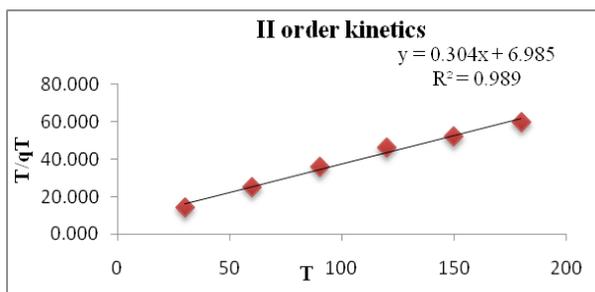


Fig. 7(b)

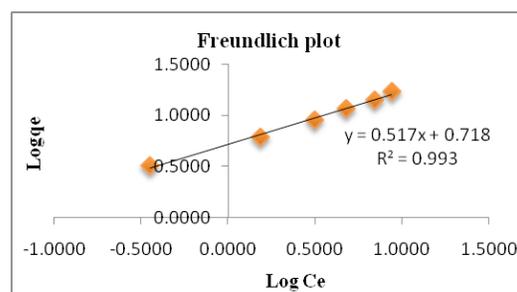


Fig. 8(b)

Table 7. Kinetic parameters for the adsorption of Ibuprofen

I Order	K_1	0.006909	q_e	1.266174	R^2	0.966
II Order	q_e	3.289474	K_2	0.013231	R^2	0.987
Int part diff	K_p	0.109	c	1.487	R^2	0.978
Elovich model	\hat{a}	0.479	\acute{a}	0.415	R^2	0.944

range of 1 to 10 indicate good adsorption. Additionally, the K_f and n values for Ibuprofen are substantially greater, indicating that the drug has a stronger affinity for the sorbent sites. The maximum adsorption capacity parameter for the Langmuir monolayer (Q_o) in theory is a reliable indicator of this Table 8. Last but not least, the dimensionless Langmuir separation constant (R_L) demonstrated that ADCL benefits from the adsorption process.

Freundlich constant (n) values that are much more than one indicate that physisorption controls the adsorption process. The experimental results can be explained by the Redlich-peterson isotherm, according to the Redlich-peterson isotherm's R^2 value, which was calculated and determined to be 0.985. According to the results of this isotherm, the value was 0.266. To characterise the single-solute adsorption isotherms, the Dubinin-Kaganer-Radushkevich (DKR) model was chosen. The experimental results cannot be satisfactorily explained by the DKR isotherm, as shown by the R^2 value of 0.963 that was derived for the DKR isotherm. From this isotherm's calculations, the value was calculated to be 1.2713. The desorption constant, can be measured by looking at the values of the desorption constant in the Redlich-Peterson and Dubinin-Kaganer-Radushkevich isotherms. Its values are smaller than 1, which suggests that adsorption is favourably impacted. To distinguish between physisorption and chemisorption, the sorption energy in the DKR isotherm is an important metric. Physisorption is suggested by lower readings.

Thermodynamic Study

To ascertain the relative thermodynamic viability of Ibuprofen's adsorption on ADCL, adsorption was investigated between 30 °C and 60 °C. The elimination efficiency rose as the temperature was changed from 30 °C to 60 °C, Table 9(a), which is a sign of an endothermic process. As the equilibrium shifts toward desorption and the binding forces between the Ibuprofen and ADCL molecules get stronger, the retention efficiency increases. Using Equation,

$$\Delta G^0 = -RT \ln Kc = \Delta H^0 - T\Delta S^0$$

The thermodynamic parameter ΔG^0 was calculated, and the parameters ΔH^0 and so were determined from the slope and intercept of the Figure in Fig. 9(a), respectively. Negative ΔG^0 changes denote a natural process. It is clear that Ibuprofen adsorption onto ADCL occurs naturally. The reaction is endothermic, which is supported by the positive ΔH^0 values. The magnitude of ΔH^0 values in the current work Table 9 confirms a physisorption mechanism since ΔH^0 values of 23kJmol^{-1} are stated to characterise physisorption mechanism. The improved orderliness at the adsorbent/adsorbate contacts is implied by the positive ΔS^0 value. It is clear that the adsorption processes are energetically stable. The Arrhenius equation, was used to calculate the activation energies for the adsorption of Ibuprofen on the adsorbent.

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

The results are illustrated in Fig. 9(a) and listed in

Table 8. Isothermal parameters for the adsorption of Ibuprofen

L Isotherm	K_L	q_0	b_L	R^2
	2.577319588	26.31578947	0.097938144	0.931
F Isotherm	K_F	n	-	R^2
	2.79898132	2.237136465	-	0.999
DKR	β	b	q_0	R^2
	1.271256	0.681	4.797334486	0.963
RP	β	b_R	K_R	R^2
	0.248	4.032258065	1.072580645	0.985

Table 9 (a,b) and Figure 9(a,b). Thermodynamic parameters for the adsorption of Ibuprofen
IBUPROFEN

ΔG^0	ΔH^0	ΔS^0	$\log_{10} K_a$	1/T
-437.227	19.14714	57.44143	0.07534	0.003299
-753.873			0.125753	0.003193
-1387.28			0.22425	0.003095
-1595.62			0.250187	0.003002

Table 9 (a). IBUPROFEN

Ea	Log A	R ²
12.06844	2.152	0.96

Table 9(b). The activation energy found in this instance suggests that the sorption mechanism and feasibility of sorption are influenced by physical factors.

FT-IR Study

Figures 10 a and b, respectively, depict the FT-IR (NICOLET iS50, Thermo Scientific, USA) spectra of the ADCL samples before and after adsorptions. The existence of surface groups on the adsorbent's surface is demonstrated by the spectra. The peak intensities between them show notable variations. The carbons have noticeable variations in almost every absorption band's intensity, which reflects how differently dense the respective functional groups are. After adsorption demonstrated that no new compounds were forming, there were no new peaks. The figures indicate that the adsorption frequencies

are nearly identical, and it is assumed that this means that the functional groups were not impacted by adsorption, supporting the idea that solely physisorption had occurred. Some peaks in the adsorbate are lost after adsorption due to desorption, while a few peaks are slightly displaced to higher or lower wave numbers due to electrostatic forces.

XRD

Rigaku Corporation, Japan's X-ray Diffractometer 20KV / 30mA, Model XRD 6000 SHIMADZU was used to analyse the adsorbent ADCL before and after Ibuprofen adsorption. Figure 12(a) and (b) shows the diffraction patterns. Adsorbent's 2 Theta value does not significantly differ before and after adsorption. Because weak Van der Waals forces and physisorption affect the adsorbent's surface chemical makeup, adsorption has no effect on it.

SEM

To research the adsorption process. Scanning Electron Microscopy (SEM) was used to analyse the surface morphology of ADCL before and after adsorp-

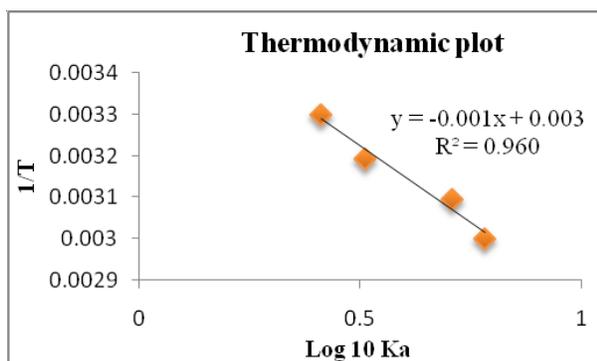


Figure 9 (a)

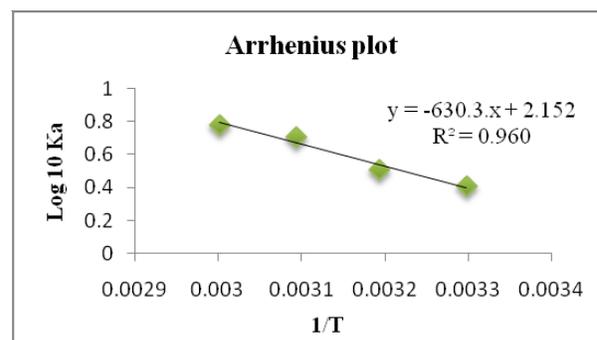
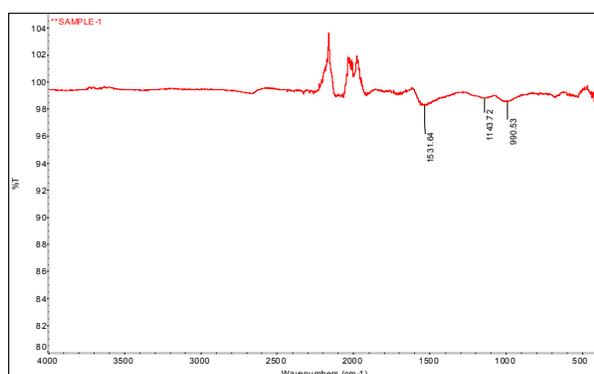
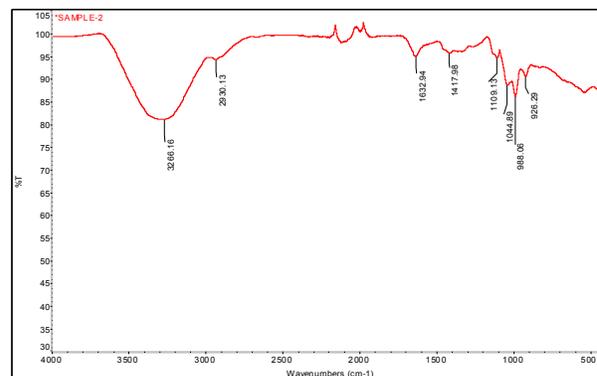


Figure 9 (b)

Figures 10(a,b) FT-IR spectrum of ADCL



10 (a) Before adsorption



10 (b) After adsorption

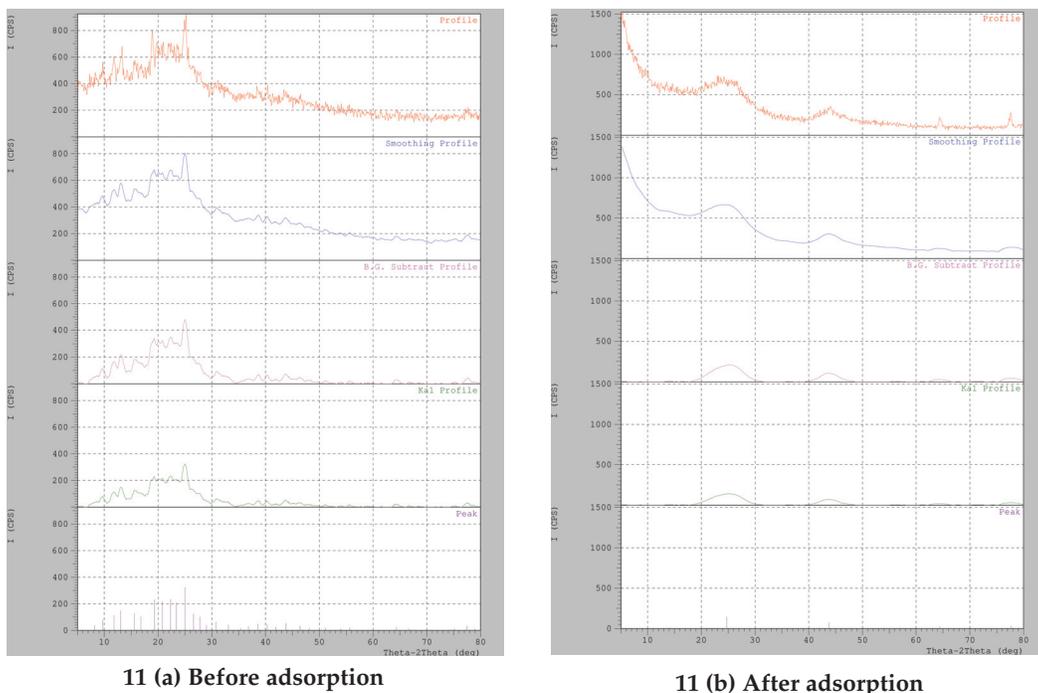


Fig. 11. (a,b) XRD pattern of ADCL

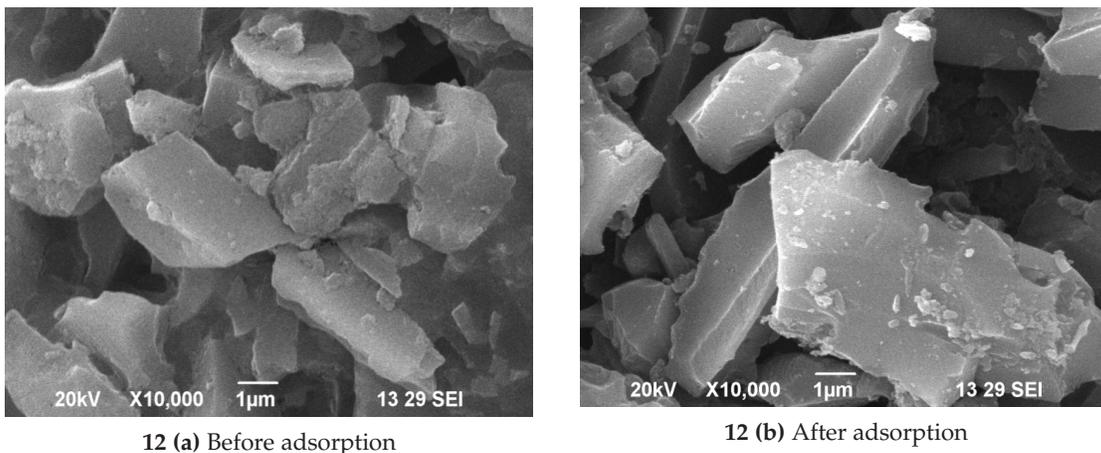


Fig. 12. (a,b) SEM micrograph of ADCL

tion. The corresponding SEM micrographs were acquired at an accelerating voltage of 20Kv JEOL JSM 6390 at 5000 magnification and are shown in Figures 12(a) & (b). The ADCL particles revealed rough surfaces with readily discernible micropores under such magnification. The uneven surface and an increase in porosity are visible in the SEM micrograph of ADCL shown in Fig. 12(a) & (b). These carbonaceous materials often have huge macropores that allow adsorbates to penetrate into microporous systems.

Conclusion

The adsorption characteristics studies on the removal of therapeutic drug ibuprofen on the acid digested carbon of waste leather were analysed by varying the physico-chemical conditions. The minimum particle size gives maximum number of surface area and adsorbed more, 91.25% for 0-63 micron. Acid pH ranges were optimal for the adsorption of ibuprofen on this ADCL. But different adsor-

bate preferred different acidic pH range of pH from 1 – 6. In the basic pH range the adsorption is very pure due to the formation of the hydroxide precipitate. In this case at pH 5, 93.1% of adsorption takes place. The percentage of adsorption of ibuprofen is directly proportional to the adsorbent dosage and contact time and inversely proportional to the initial concentration of the adsorbate. Were found out from this study. Order of this adsorption is pseudo second order kinetics and it belongs to the physisorption, because of no chemical bond formation between the adsorbent and adsorbate. The freundlich and Langmuir isotherm model is fit for these studies. The thermodynamic study reveals the negative ΔG° and positive ΔH° and ΔS° values concluded that this adsorption is spontaneous, feasible and physical in nature respectively. The spectral evidence from the FT-IR, SEM and XRD are in favour of above experimental results and provided the strong applications on the removal of ibuprofen therapeutic drug pollution from the water bodies as well as from the industrial effluence. This is an economically cheapest and eco friendly method for the treatment of water pollution.

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