Journal of Materials and Environmental Sciences
ISSN : 2028-2508
CODEN : JMESCN

J. Mater. Environ. Sci., 2019, Volume 10, Issue 7, Page 657-667

http://www.jmaterenvironsci.com

Activated carbons precursor to *corncob* and *coconut shell* in the remediation of heavy metals from oil refinery wastewater

[1]Kulbir Singh* , [2]Sadiq Abdullahi Waziri

[1]*Department of Environmental Science & Engineering, Guru Jambheshwar University of Science and Technology, Hisar-125001 (Haryana)*

[2]*Department of Civil Engineering, Maharishi Markandeshwar University, Mullana-Ambala, 133207, (Haryana)*

Received 22 May 2019,
Revised 04 July 2019,
Accepted 05 July 2019

Keywords
- ✓ Heavy Metal,
- ✓ Oil Refinery,
- ✓ Corncob,
- ✓ Coconut Shell,

Kulbir.nain@gmail.com
Phone: 9416804433

Abstract

In this research adsorption of heavy metal from refinery effluent was studied using corncob and coconut shell activated carbon. Simultaneous adsorption of six heavy metals cadmium, chromium, copper, lead, nickel and zinc was examined using a batch adsorption process. The activated carbons were produced at different temperature and impregnation ratio. The surface morphology showed a well-defined pore space for both corncob and coconut shell. Freundlich and Langmuir model were applied and the order of adsorption are Cu>Pb>Cr>Zn>Ni>Cd and Ni>Cr>Pb>Zn>Cu>Cd for coconut shell and corncob respectively. Freundlich and Langmuir isotherm represents the sorption data where the R^2 known determination coefficient approaches unity. This research conquers that Corncob and coconut shell activated carbon can be efficiently applied for remediation of pollutant is aqueous state.

1. Introduction

One of nature's best gifts to mankind is water. The deterioration of this best gift due to pollution as a result of release of toxic chemicals is a matter of serious concern. Advanced industrialization and weakness of regulations have collectively compounded the problem. Palatable and high quality water is necessary for human life and satisfactorily clean water is required for domestic, agricultural, commercial as well as industrial activities [1].Oil refinery effluent arising from petroleum refining and processing leads to devastating water pollution and threatens human health. There is a pressing demand on society to solve the difficulty and necessity oil refinery wastewater. These Heavy metals like Nickel, Chromium, Cadmium, Copper, Lead and Zinc are usually present in refinery effluent which are harmful to the environment and are carcinogenic in nature. Heavy metals cannot be biologically broken down into harmless compounds contrary to organic pollutants. [2-3]. Different inorganic and organic compounds such as heavy metals, phenols, dissolved compounds and suspended solids etc. are found in industrial wastewater therefore it is important that industrial wastewater be well treated to an environmentally harmless limit [4-5].

Methods like ion exchange, coagulation-flocculation, membrane transfer and oxidation have been applied for the treatment of heavy metal in polluted water. Adsorption promises to be economical, unambiguous and simple [6-8]. Activated carbon (AC) is a common adsorbent that is used widely, due to its pore volume and high surface area [9-11]. Commercial AC is costly, so research is geared towards producing activated carbon from cheaper and renewable materials for the treatment of environmental pollution [12]. Several agricultural biomasses have been experimented for adsorption such as rice husk ash [13], bamboo [14], date pits [15], silk cotton hull [16], jute fiber [17], groundnut shell [18], corncob [19] and rattan sawdust [20]. In recent times, due to the increasing necessity to search for economical and safe methods for the treatment of heavy metal ions arising from refinery effluent, locally made adsorbent for activated carbon was explored.Cellulosic agricultural residue have been shown to have high potential for the sorption of various pollutants. The materials which constitute these residues are hemicellulose, lignin, lipids, proteins, simple sugars, water, hydrocarbons, and starch, containing variety of functional groups [21-22].

In this study, coconut shell activated carbon (CSAC) and corncob activated carbon (CCAC) were evaluated as adsorbents for removal of heavy metal and physico-chemical parameters from petroleum refinery effluent. The process parameters studied include impregnation ratio, yield capacity, bulk density and temperature effects. Equilibrium studies were conducted using Langmuir and Freundlich models.

2. Materials and methods

2.1. Materials

Coconut shell and corncobs were the precursors utilized for the activated carbon production. These were obtained by the road side from vendors in Barara-Ambala, Haryana, India. Indian Oil and Petroleum Company Ltd. (IOCL), Panipat, Haryana, India was the refinery from there the effluent was obtained.

Other materials utilized in this study constitute instruments such as fire resistant crucibles, muffle furnace, pH meter, laboratory shaker, Glassware and reagents like NaOH, KOH, iodine etc. were AR grade. All solutions were prepared in double distilled water. Methods primarily include measurement of heavy metals.

2.2. Methods

2.2.1. Preparation of Activated Carbon

The materials, has been sun dried after collection, grinded and crushed. Sieve size 1.0 – 1.5 mm was used to sieve the grinded material to obtain a uniform size. Precursor of about 500 g was weighed and then placed in a muffle furnace. It was then pyrolysed.

The pyrolysis of the precursor was done at 300 - 400°C for 1 hour, distillate formed while carbonization were collected to prevent air pollution. The pyrolysed precursor (charcoal or char) was allowed to cool prior to transferring into a crucible. It was then grounded into powder by using pestle and mortar and sieved through of 0.150 mm sieve-size to get uniform particles. Pyrolysed corncob of about 100 g was measured into a beaker with Potassium hydroxide KOH (1N KOH) at different ratios (1.0, 1.5, 2.0) until thoroughly mixed.The paste formed was then poured into a container, dried at 105°C overnight and after that it was placed into the muffle furnace and heated at different temperatures 500^{o}c, 550°c and 600^{o}c for 1 hour thereby enhancing the surface area of the sample and make it a better adsorbent. After it has cooled, it was then washed with mineral free water and the residual filtrate was ensured neutral pH. Further, it was oven-dried at 105°C overnight. Subsequently the adsorbent was stored in polyethylene bag and ready for use [23]. Same process was done for coconut shell, but here sodium hydroxide (1N NaOH) was used as the activation agent.

2.2.2. Yield

Activated carbon (AC) yield was computed on chemical-free nature basis and is regarded as an indication of the process accuracy of the activation. Yield is represented as percentage weight of activated carbon by weight of dried adsorbent.

$$\text{Yield (\%)} \quad = \quad \frac{weight\ of\ activated\ carbon}{weight\ of\ raw\ material} x100$$

2.2.3 Bulk density

A 25 ml glass cylinder filled to a particular volume with powder AC was placed in oven overnight at 80°C. The vessel was then compacted. Bulk density is expressed as g ml^{-1}:

$$\frac{\left(Weight\ of\ dry\ material\ (g)\right)}{\left(Volume\ of\ Packed\ dry\ materials\ (ml)\right)} \times 100$$

2.2.4. Microscopy

Scanning Electron microscope (SEM) was used to analyze the surface morphology.It was done using SEM – JEOL – JSM5800LV.

2.2.5. Iodine number

Iodine number (IN) can be used to determine the relative porosity of the activated carbon. It is micropore indication of the AC (up to 2 nm). ASTM D4607-94 method was used in this study. Iodine number can be definedas the milligrams of iodine adsorbed by 1.0 g of carbon when the iodine concentration of the filtrate is 0.02 N (0.02 mol. /L).

2.2.6. Batch equilibrium studies

Basically the test was conducted on Corncob and Coconut Shell to compare their effectiveness in the treatment of oil refinery wastewater. Batch adsorption studies has been conducted by adding certain amount of activated carbon (Corncobs and Coconut Shell) 1g into 250 mL Erlenmeyer flasks containing 100 mL of different impregnation ratio and temperature. The conical flasks then were placed in a shaking incubator at 120 rpm for 3hrs at 25^{o}C. There after the effluent was filtered with a filter paper and the residual filtrate was analyzed. The amount of sorption at time t and the percentage removal were calculated according to previous study [24].

3. Results and discussions

The activated carbon has been produced successfully, however, analysis of the data and tests conducted comprising adsorption process by batch method and sample analysis before and after adsorption in the wastewater stream are discussed in number table1 and table 2.

Table 1. Activated Carbon Characterization

S/N	Parameters	Coconut shell	Corncobs
1	Bulk density (g/cm^3)	0.667	0.444
2	Yield (%)	91.55	91.94
3	IN (mg/g)	1397	1129
4	Ash Content (%)	8.45	8.06
5	Moisture Content (%)	<1	<1
6	Particle Size (μm)	<150	<150

Table 2. Heavy Metals in oil refinery effluent.

S/No.	Parameter	Value
1	Cadmium	0.015
2	Chromium	0.210
3	Copper	0.740
4	Lead	0.030
5	Nickel	0.304
6	Zinc	1.130

Table 3. Heavy metal effluent characteristics after treatment (600^{o}C).

The wastewater treatment for various heavy metals at temperature of 600^{o}c with various impregnation ratios using coconut shell and corncob activated carbon are given in the table 3.

		Coconut Shell			Corncob		
S/N	**Parameter**	**CS 1**	**CS 1.5**	**CS 2**	**CC 1**	**CC 1.5**	**CC 2**
1	**Cadmium (Cd)**	0.011	0.0006	0.0003	0.0014	0.0006	0.0003
2	**Chromium (Cr)**	0.0294	0.0252	0.0189	0.0294	0.0392	0.0189
3	**Copper (Cu)**	0.126	0.111	0.081	0.096	0.074	0.037
4	**Lead (Pb)**	0.0018	0.0012	0.0003	0.0045	0.0027	0.0021
5	**Nickel (Ni)**	0.073	0.072	0.055	0.055	0.040	0.033
6	**Zinc (Zn)**	0.237	0.226	0.181	0.124	0.091	0.057

Table 4. Heavy metal effluent characteristics after treatment (550^{o}C)

The wastewater treatment for various heavy metals at temperature of 550^{o}c with various impregnation ratios using coconut shell and corncob activated carbon are given in the table 4.

		Coconut Shell			Corncob		
S/N	**Parameter**	**CS 1**	**CS 1.5**	**CS 2**	**CC 1**	**CC 1.5**	**CC 2**
1	**Cadmium (Cd)**	0.0020	0.0017	0.0014	0.0029	0.0020	0.0015
2	**Chromium (Cr)**	0.0399	0.0357	0.0252	0.0798	0.0672	0.0630
3	**Copper (Cu)**	0.163	0.148	0.111	0.215	0.163	0.133
4	**Lead (Pb)**	0.0036	0.0030	0.0018	0.0090	0.0069	0.0060
5	**Nickel (Ni)**	0.103	0.088	0.064	0.094	0.088	0.079
6	**Zinc (Zn)**	0.339	0.294	0.226	0.215	0.170	0.136

Table 5. Heavy metal effluent characteristics after treatment (500°C)

The wastewater treatment for various heavy metals at temperature of 500°c with various impregnation ratios using coconut shell and corncob activated carbon are given in the table 5.

		Coconut Shell			Corncobs		
S/N	**Parameter**	**CS 1**	**CS 1.5**	**CS 2**	**CC 1**	**CC 1.5**	**CC 2**
1	**Cadmium (Cd)**	0.003	0.0023	0.0015	0.0024	0.0021	0.0018
2	**Chromium (Cr)**	0.042	0.042	0.0315	0.084	0.078	0.0714
3	**Copper (Cu)**	0.222	0.185	0.148	0.222	0.163	0.148
4	**Lead (Pb)**	0.0045	0.0039	0.0024	0.0096	0.0084	0.0063
5	**Nickel (Ni)**	0.122	0.109	0.076	0.106	0.097	0.091
6	**Zinc (Zn)**	0.339	0.305	0.283	0.226	0.192	0.158

3.1.Temperature effect on yield of activated carbon.

The overall efficiency of activated carbon production process can be determined by two important parameters, these are: carbonization and activation temperature. Many researchers have studied the effect of activation temperatures on yield [25-28].

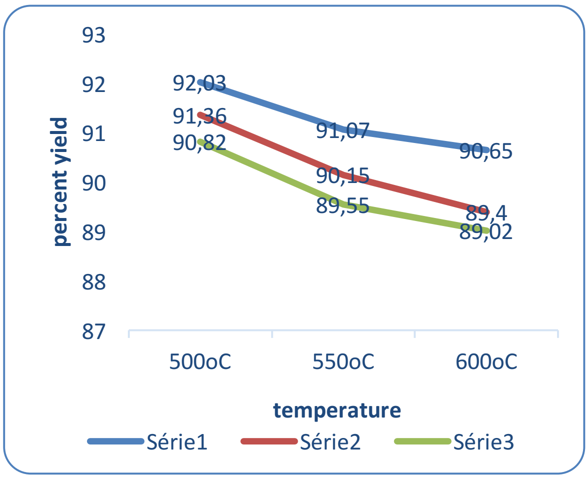

FIG 1: Temperature effect on yield of coconut shell ac

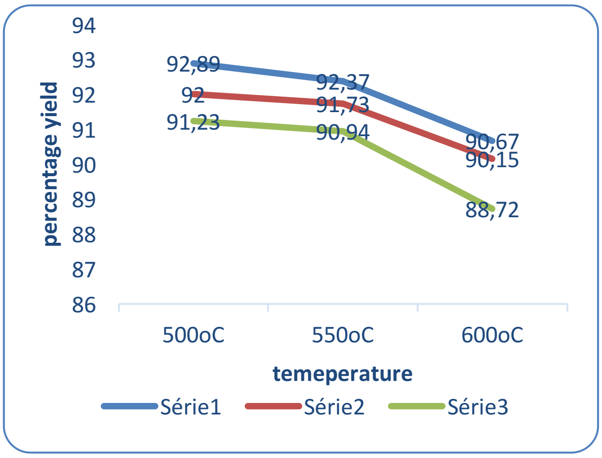

FIG 2: Temperature effect on yield of corncobs ac

Temperature effect on the yield of coconut shell AC is presented in the fig.1 plotted above. It is evident that the more temperature has been increased the yield decreased. At 500°C, 550°C, 600°C the yield were 92.03%, 91.07% and 90.65% respectively. Impregnation ratios of 1, 1.5 and 2 are represented as series 1, 2 and 3 respectively.

Ash formation has increased due to the collapse of internal pore structure when the temperature was raised [29]. When subjected to varying temperatures, it has been observed that corncobs exhibit similar characteristics. At temperatures 500°C, 550°C and 600°C the yield for Corncobs AC was 91.23%, 90.94% and 88.72% respectively. Impregnation ratios of 1, 1.5 and 2 are represented in fig. 2 as series 1, 2 and 3 respectively.

Owabor and Iyaomolere (2013) reported similar result, where weight loss of a periwinkle shell was recorded due to dehydration and elimination reactions.

3.2.2. Impregnation ratio effect on yield of activated carbon

One of the most important parameters of activated carbon which magnifies the details of the adsorbent's behavior is impregnation ratio. Expressed as weight of activation chemical to the weight of adsorbent [30-32].

3.2.1. Impregnation ratio Effect on Yield of Coconut shell ac

Yield of coconut shell activated carbon produced at different impregnation ratios has been studied. It was noted that the ratio increased by increasing IR (Impregnation ratio) which afterwards declined with increase IR. This is based on the evidence that in order to form stable compounds, the carbon releases volatile material [33]. Impregnation ratios 1, 1.5 and 2 produces yield of 90.65%, 91%, and 89.02% respectively (fig 3).

3.2.2. Impregnation ratio Effect on Yield of Corncob ac
The change in impregnation ratio gives rise to pyrolytic decomposition and prevention of tar formation. This is also true for Corncob activated AC. For impregnation ratios 1, 1.5 and 2 the yields were 90.67%, 91.15% and 88.72% respectively (fig 4).

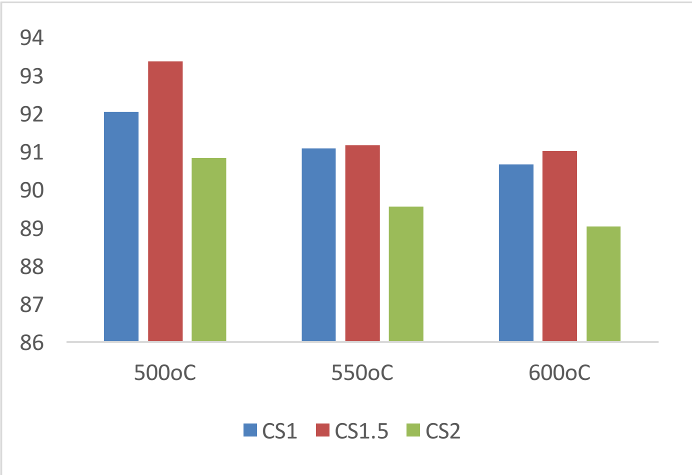

FIG 3: Impregnation ratio effect on yield of coconut Shell ac.

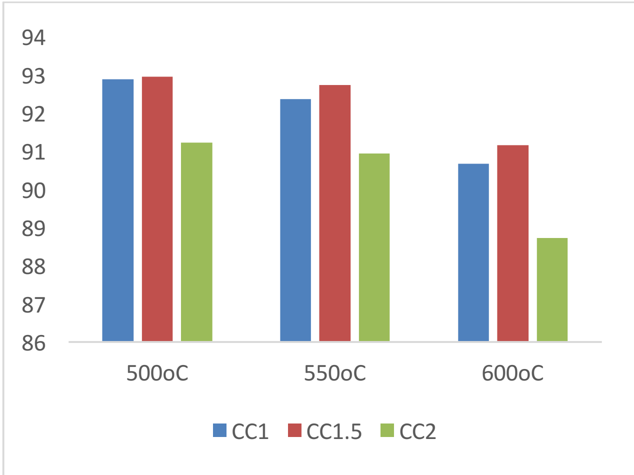

FIG 4: Impregnation ratio effect on yield of Corncob ac.

3.3 Morphology of adsorbent
The morphology of the coconut shell activated carbon was determined using a scanning electron microscope. SEM images provide detailed information and reactions that took place on the surface of the activated carbon micro structure [34]. The SEM micrograph of the coconut shell activated carbon with different magnifications is shown fig.5. Deducting from the images obtained, the CSAC surface is seen to include morphologies having heterogeneous and irregular there by having imperative effect on the process of adsorption [35].

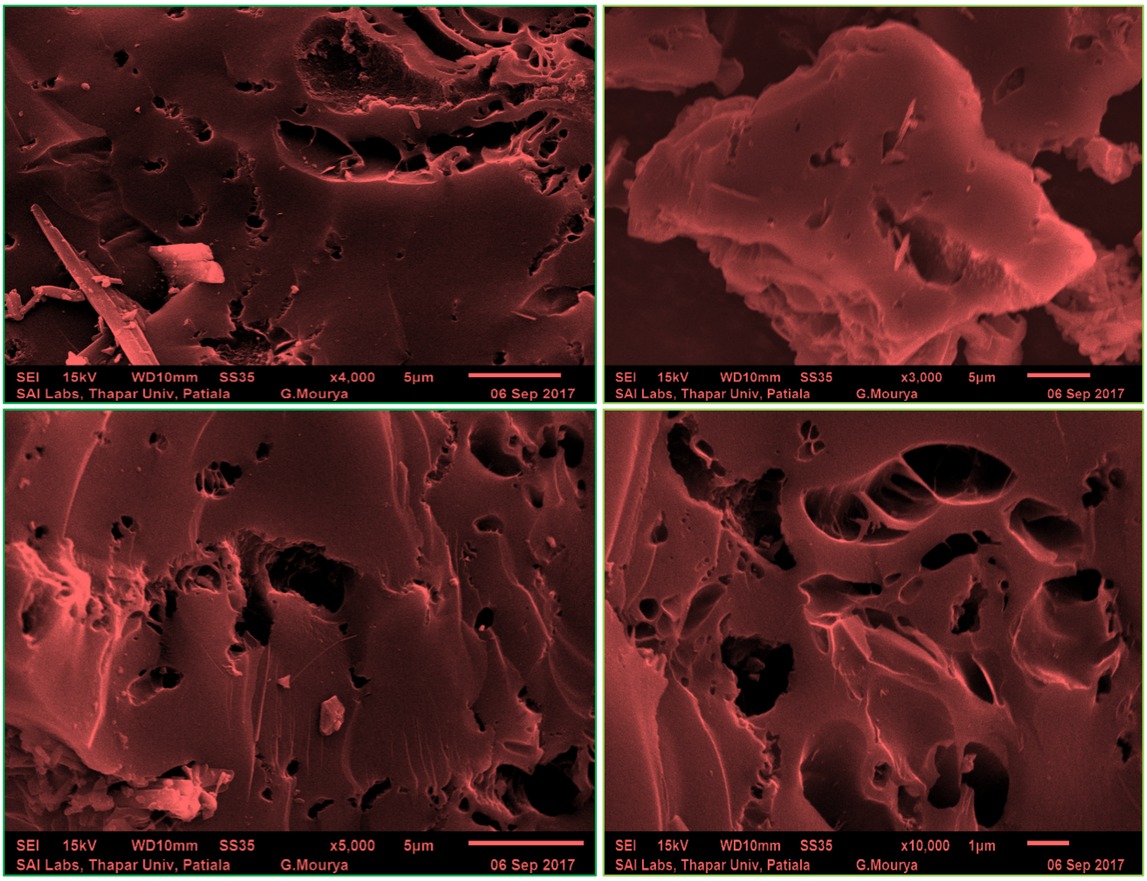

Fig 5: SEM images for Coconut Shell Activated Carbon at various magnification.

The CSAC surface was coarse and rough having uneven formation with varying diameters which points to affirmation of meso and macro-pores which are potential site for the sorption and transportation of metal ions in aqueous solution. The SEM images Corncobs Activated Carbon (fig. 6) shows the presence of rough surface with crevices on the CCAC and these were also evident in the images of CSAC. Additionally, during the thermal activation and preparation of the adsorbent, volatile organic compounds are evolved; this gave rise to the pore network of the corncob activated carbon [36].

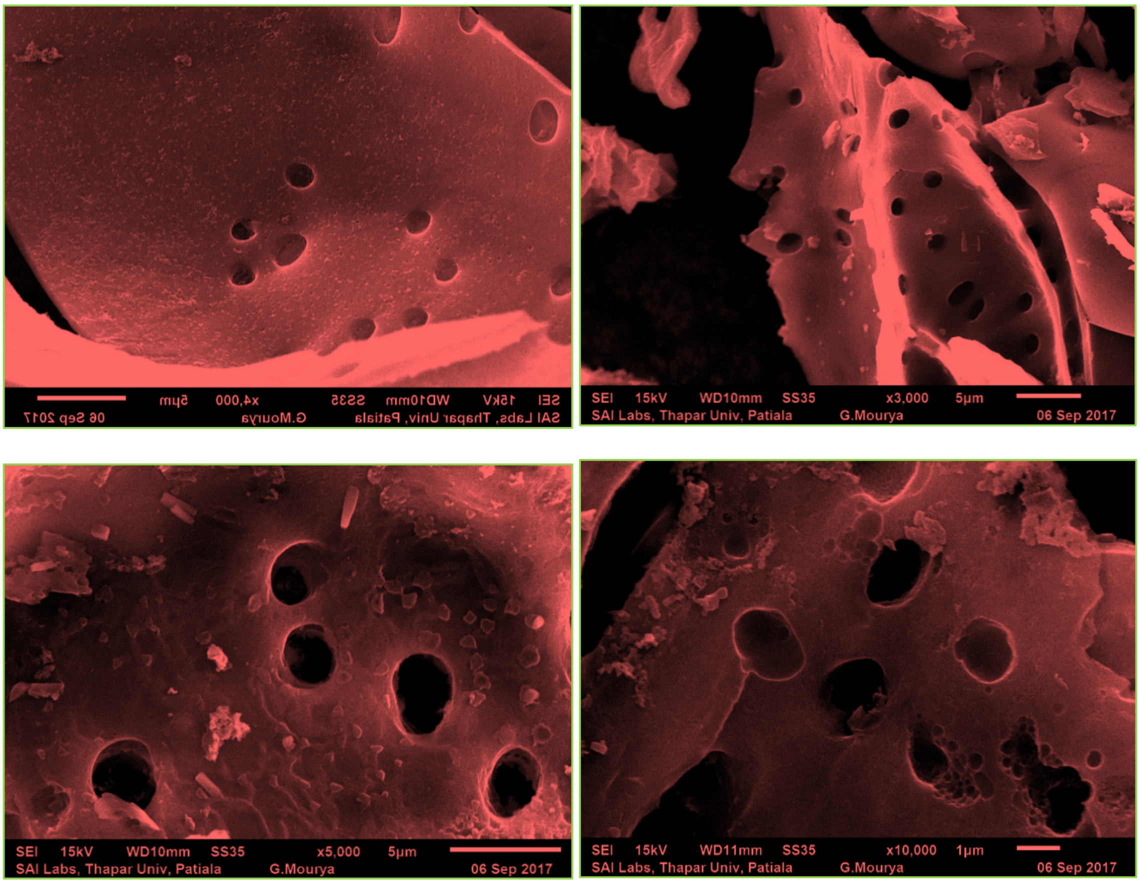

Fig 6: SEM images for Corncobs Activated Carbon at various magnification.

3.4. X-Ray Diffraction Spectroscopy (Xrd)

In order to determine the level of crystallinity or amorphous nature of an activated carbon, x-ray diffraction becomes indispensable. Goniometer Ultima-IV diffractmeter was used for the study working at a current of 40 mA and voltage of 40 kV. The variation of the diffraction angle (2θ) ranges between $10^o$to 80^o. The adsorbent was studied for their in the matrix [37].

Coconut Shell AC X-Ray Spectroscopy (XRD)

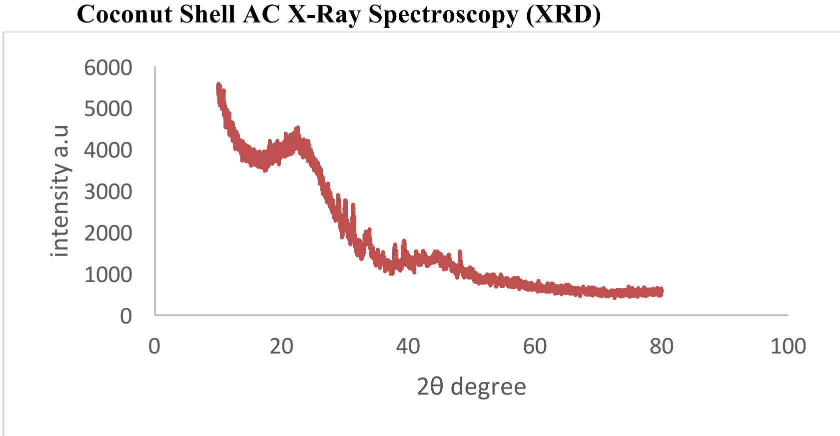

Fig 7: Coconut Shell X-Ray spectra.

X-ray diffraction spectra shown in fig. 7, was the pattern for coconut shell activated carbon, displaying high peak in the beginning at $2\theta=10^{o}$. Subsequently a wide asymmetric peak corresponding to $2\theta=23^{o}$and$2\theta=44^{o}$ was observed. This indicates slight presence of graphite layers which disappears across the spectra. In general, the pattern depicts amorphous behavior [38].

The X-ray spectra in fig. 8 for corncob activated carbon produced using potassium hydroxide as the activating agent. At $2\theta=25^{o}$ and at $2\theta=31^{o}$ sharp crested peaks were noted. These were an indication of crystalline graphite formation within the carbon. These has been as a result of the carbon atoms been better aligned. The spectra exhibit continuous reduction in peak intensity as a result collapse of the graphite layers. This is a typical of amorphous carbon formation [39].

Corncob AC X-Ray Spectroscopy (XRD)

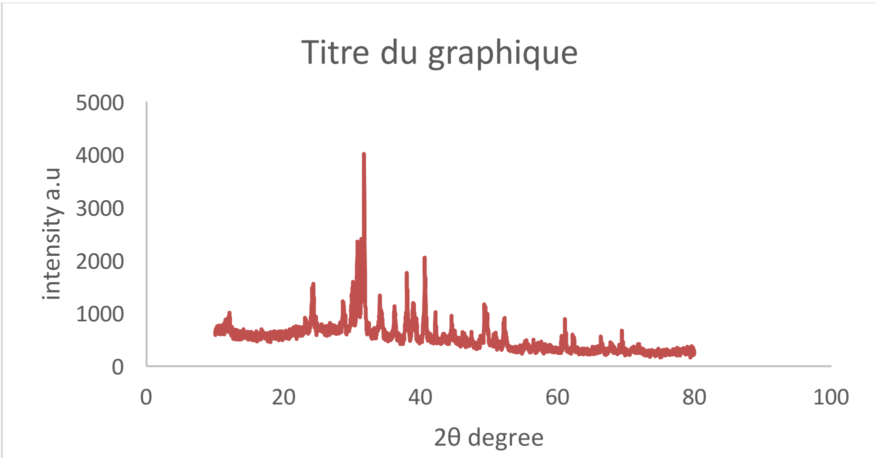

FIG 8: Corncob X-Ray spectra.

3.5. Adsorption models

The capacity of adsorbent to treat heavy metal pollution up to a certain level has been studied with the help of adsorption model. When there is a contact for a particular time between the carbon sorbent and the pollutant, a stage is arrived when the maximum amount of pollutant is adsorbed and the remainder is known. In an equilibrium conditions for any given process, the mass balance equation employed for the calculation of the materials adsorbed is given below.

$$\frac{X}{M} = (Co - Ce)\frac{V}{M} \quad (1)$$

where *X*/*M* (is milligram of pollutant by gram adsorbent) is the mass of contaminant by the mass of adsorbent, *Co* is the initial concentration of pollutant, *Ce* is the pollutant concentration after equilibrium, *V* is the volume of solution to which the adsorbent mass is applied, and *M* is adsorbent mass. Langmuir or Freundlich models have been used for the study of many mechanisms [40].

Langmuir isotherm is represented by Eq.

$$\frac{X}{M} = \frac{KLCe}{1+aLCe} \quad (2)$$

Where K_L and a_L are model's constants. Linear regression is used for the determination of K_L and a_L values. The linear equation for Langmuir model is given by the equation:

$$q_e = \frac{QbCe}{1+bCe} \quad (3)$$

Linear regression is used for the determination of *Q* and *b* values [41].

The general Freundlich equation is:

$$\frac{X}{M} = \frac{1}{KlCe} + \frac{aL}{Kl} \quad (4)$$

Linear equation for Freundlich model is given by [29].

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

Where *n* and K_F are affinity and adsorption capacity, respectively.

Langmuir and Freundlich models are employed batch adsorption systems where equilibrium is provided enough time between the pollutant in solution and the pollutant adsorbed. In this way most of the contaminants are trapped on the carbon sorbent.

Activated carbon produced from coconut shell has been actively applied for heavy metal adsorption [42-44]. Similarly, in this study also, it is employed in multiple heavy metal ion adsorption and its interaction and properties in the process of adsorption. Fig. 9 above presents the adsorbed, different metals as well as their corresponding performance. Scanty washing or acid removal may be the underlying factor for the poor adsorption of cadmium coconut adsorbent. An average chromium removal was recorded.

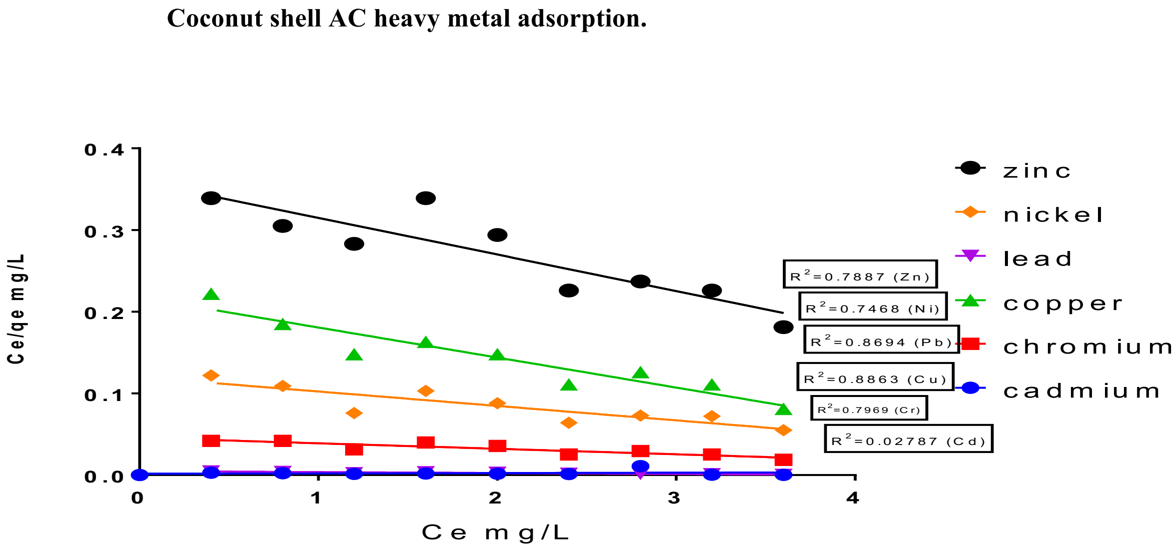

Fig 9: Coconut shell AC heavy metal adsorption.

The R^2 was 0.7969 while Q_{max} was 6.779 showing improved adsorption but the intensity was low. Langmuir model best explain Copper adsorption than Freundlich having R^2 value of 0.8863 and adsorption capacity Q_{max} was 5.295. Langmuir isotherm best explains lead adsorption having R^2 value of 0.8684 with capacity of adsorption Q_{max} of 4.151. Nickel was satisfactorily adsorbed and well explained by Freundlich with R^2 value of 0.7693 and adsorption capacity K_f of 110.2. The adsorption intensity value for nickel was 0.03633, obviously closer to zero showing greater surface heterogeneity. Coefficient of determination R^2 0.7887 with adsorption capacity Q_{max} value of 8.034 was recorded for zinc. A great tendency of removal of multiple heavy metals was exhibited by coconut shell. The preference of removal from the data studied are Cu>Pb>Cr>Zn>Ni>Cd. The model parameters are shown below.

Table 6. Coconut shell AC heavy metal adsorption model and their constant.

	Langmuir			Freundlich		
Metal ion	**R^2**	**Q_{max}**	**K_L**	**R^2**	**K_f**	**N**
Cadmium	0.02787	-3.654	0.0004364	0.401	1.513	-1.164
Chromium	0.7969	6.779	-0.006738	0.7925	267.8	0.01533
Copper	0.8863	5.915	-0.03675	0.7524	94.4	0.03133
Lead	0.8684	4.151	-0.001163	0.8587	355.3	0.01683
Nickel	0.7468	6.792	-0.0767	0.7639	110.2	0.03633
Zinc	0.7887	8.034	-0.04475	0.3251	10.78	0.3345

Corncob AC characteristics towards the adsorption of multiple heavy metal ions are depicted in fig. 10 above. Cadmium recorded a poor adsorption with R^2 of 0.6666 and 0.6694 for Langmuir and Freundlich models. Langmuir isotherm best fits chromium adsorption, R^2 value recorded was 0.8683 improved adsorption and uptake capacity Q_{max} was 4.365. The value for copper was 0.7533 and 0.7673 for respective Langmuir and Freundlich models.

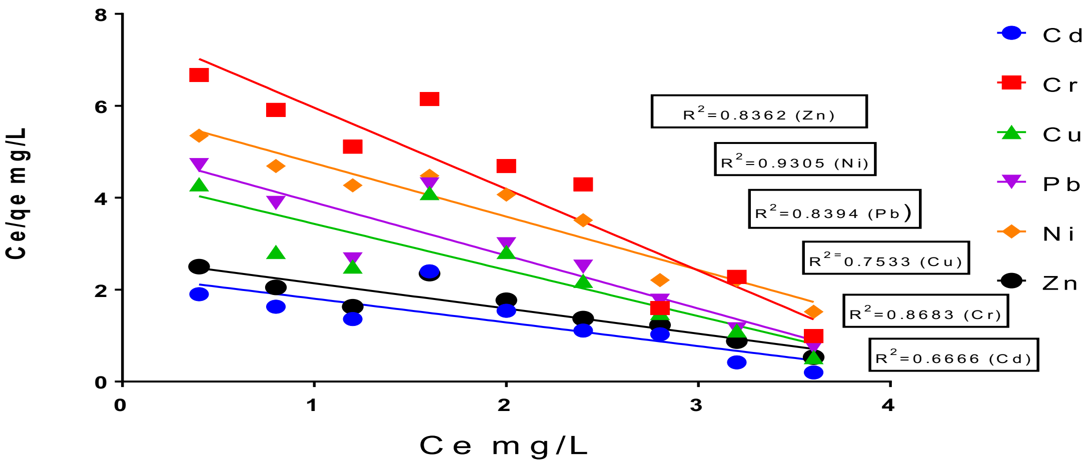

Fig 10: Corncob AC heavy metal adsorption

The adsorption intensity was low, the capacity was 4.418. Lead showed better removal by Langmuir model. The R^2 value was 0.8394 while the adsorption capacity was 4.739. Corncob AC showed great capacity for nickel uptake. The R^2 obtained was 0.9305 and Q_{max} was 5.094. The recorded values of zinc were 0.8362 and 4.897 for R^2 and Q_{max}, respectively. The recorded order of performance is Ni>Cr>Pb>Zn>Cu>Cd, showing good capacity for multiple adsorption of heavy metal by Corncob activated carbon.The recorded data is further shown on table 7 below.

Table 7: Corncob AC heavy metal adsorption model and their constant.

	Langmuir			Freundlich		
Metal ion	**R^2**	**Q_{max}**	**K_L**	**R^2**	**K_f**	**N**
Cadmium	0.6666	4.496	-0.5158	0.6694	353.5	0.019
Chromium	0.8683	4.365	-1.77	0.8537	86.88	0.0613
Copper	0.7533	4.418	-1.004	0.7673	89.89	0.03317
Lead	0.8394	4.739	-1.153	0.7106	204.7	0.03303
Nickel	0.9305	5.094	-1.16	0.92	112.4	0.03533
Zinc	0.8362	4.897	-1.5488	0.8496	126.3	0.01917

Conclusion

Activated carbons from Coconut shell and corncob has been applied for the removal of various heavy metals from oil refinery wastewater under various experimental conditions. The obtained outcome from the batch adsorption study of these heavy metal ions at equilibrium indicated that coconut shell and corncob carbon adsorbent can efficiently be employed in simultaneous heavy metal uptake in refinery effluent and industrial wastewater. Both temperature and impregnation ration affects the yield of the activated carbon. The rise in temperature decreases the yield while the impregnation increases the yield only to certain limit and then reduces. The surfaces of the ACs were coarse and rough having uneven crevices with varying dimensions. These are meso and macro pores which are potential sites for sorption and transportation of metal ions. The XRD diffractogram shows crystalline graphite layers in the carbon structure. There was better and regular alignment which progressively declines as a result of graphite layers destruction. Thereby giving an amorphous orientation to the carbon lattice. The adsorbents showed a promising capacity of the uptake of heavy metal in aqueous solution.

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