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The Case Study of Isothermal Adsorption of Phenol, O-cresol on Natural Charcoal's and Applications

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Abstract: ***This Activated carbon (AC) was synthesized using inexpensive Coconut shell and Coconut coir's by a simple and efficient method. The adsorption experiments were well incorporated on the AC surface by the reactions of Aromatic chemical (o-cresol and phenol) at low temperatures. The complete formation and distribution nanoparticles on the surface of AC have been analysed and explained by different analytical techniques such as TG-DTA, NTA, UV-VISIBLE, DRS, FTIR, XRD. Further, the adsorption property was investigated through adsorption kinetics, adsorption isotherms, and thermodynamics parameters. The maximum adsorption capacity (Q max) of adsorbent was displayed at Coconut shell charcoal rather than coconut coir's charcoal for both aromatic compounds. The application of the Coconut shell charcoal and coconut coir's have made to adsorption study of Mithi river and Industrial water inlet collected from industry. The importance and role of the AC were examined by a comparative adsorption study of the previous work done. The adsorption studies confirmed that adsorption of adsorbates takes place via the formation of hydrogen bonding, π-π interactions and metal coordination between adsorbent and pollutants. Overall results suggested that AC-Coconut shell and Coconut coir's is a prominent adsorbent for efficient removal of pollutant from polluted water.***

Keywords: Activated charcoal, Coconut shell charcoal, Coconut coir's charcoal, Aromatic compound, Adsorption Isotherms, Characteristics.

I. INTRODUCTION

A major problem in the development of suitable treatment technologies for pollutants is the large number of compounds to be taken into consideration consisting essentially of infinite combination of these substances which may be found in any given water stream. Activated charcoal is charcoal that has been treated with oxygen at very high temperatures to make it more porous. This treatment changes its internal structure, reducing the size of its pores and increasing its surface area.

So much additional surface area is created during the activation process that 50 grams of activated charcoal (which is about the weight of 20 U.S. pennies) has 17.5 times more surface area than a full-size football field, according to a 2016 study in the British Journal of Clinical Pharmacology. The charcoal's porous texture has a negative electrical charge, which causes it to attract Positively charged molecules, such as toxins and gases. When liquids or gases pass through this activated charcoal, they bind to it through a process known as adsorption. Activated charcoal may help filter water by removing contaminants, suspended solids, and microorganisms like bacteria - all without affecting the water's pH or taste. Adsorption is a reversible reaction; at a given solute concentration. Adsorbates can attach themselves onto surfaces in two ways. In physisorption (physical adsorption), there is a weak van der Waals attraction of the adsorbates to the surface. During the process of physisorption, the chemical identity of the adsorbate remains intact. Physisorption is a spontaneous process (ΔG & lt; 0), since ΔS is negative, so ΔH be exothermic. In chemisorption (chemical adsorption), the adsorbates stick to the solid by the formation of a chemical bond with the surface. This interaction is much stronger than physisorption and in general, chemisorption has more stringent requirements for the compatibility of adsorbate and surface than physisorption [1-4].

II. MATERIALS AND METHODS

A. Materials

All the chemicals used were of the analytical grade (AR) and with of highest purity. Coconut shell activated charcoal powdered, Coconut coir's activated charcoal powdered,5-6 stoppered reagent bottles, thermostat, burette, 4 pipettes, measuring flask, 4 funnels, 4 burettes (50 ml), filtering papers, rubber stoppers, 4 titrimetric conical flasks, 0.1M NaOH, Phenol, o-cresol, Oxalic acid, Sodium Hydroxide, phenolphthalein.

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B. Methods

1) Preparation of Natural Charcoal: Coconut Shell-CS, Coconut Coir's-CC

a) Pre-treatment:

- CS and CC weighted about 193.47gm and 14.739 gm respectively.
- Three washing treatment to CS and CC with minimum distilled water
- Took desired quantity of CS, CC with minimum distilled water for heating/boiling for 30 min and 10 min respectively
- Dried naturally for 1 hr 30 min under sunlight.

b) Carbonation:

Table 1: Carbonation Of CS At 400^0C And Carbonation Of CC At 250^0C For 1hr 30 Min And 1hr Respectively.

Sr.No.	Material	Weight of Raw Material in gm	Weight of Carbonated Charcoal in gm	Weight loss in gm
1.	CS	9.669	7.126	2.543
2.	CC	4.599	2.815	1.784



Fig. 1 Coconut Shell Charcoal Powder and Coconut Coir's Charcoal Powder

2) Preparation of Stock Solution of Phenol in water: Approximately 0.1 M solution of phenol was prepared by dissolving 4.5429 ml of purified phenol in freshly boiled and called distilled water and diluting to 500 ml in volumetric flask.

3) Preparation of Stock Solution of o-cresol in water: Approximately 0.1 M solution of o-cresol was prepared by dissolving 5.6076 ml of purified phenol in freshly boiled and called distilled water and diluting to 500 ml in volumetric flask.

4) Preparation of Phenolphthalein indicator: Weigh 0.05 g Phenolphthalein add 50 ml 95% ethanol (9.5ml ethanol + 0.5ml distilled water) and add 50 ml distilled water to get Solution of Phenolphthalein indicator.

III. EXPERIMENTAL

A. Standardization of NaOH

Prepare 250 cm^3 approximately 0.1M Sodium hydroxide (0.45g) solution and 100 cm^3 0.05M oxalic acid(1g) solution.

B. Prepare Aqueous Solution Of Phenol/O-Cresol Into Numbered Flasks Following The Scheme Given In The Table

Table 2: Total Volume Of Each Solution Is 50 Ml. Use Flasks Fitted With Stoppers.

Flask no.	V(Phenol) or (o-cresol) [cm^3]	V (distilled water) [cm^3]
1	20	0
2	15	5
3	10	10
4	5	15

C. Procedure

1) Weigh out accurately about 2g finely ground activated charcoal in each of the thoroughly cleaned and dried bottles, numbered as 1to 4.
2) Prepare 500 ml approximately 0.1M Phenol/o-cresol solution by taking distilled water and diluting it to 500cm^3 in a volumetric flask.
3) By means of burettes, add 20, 15,10, 5 cm^3 of the acid solution and 0, 5, 10,15 of distilled water in bottle no.s 1,2,3,4 respectively.
4) Shake the bottles vigorously and leave them in a thermostat at the desired temperature for about one hour.
5) Titrate standard oxalic acid solution with sodium hydroxide solution and find the exact concentration of the latter.
6) Then titrate phenol by using phenolphthalein indicator with standardised NaOH, and determine the exact concentration of the solution.
7) Filter the solutions of each of the bottles through different small dry filter papers, and collect the filtrates in properly labelled flasks.
8) Reject first 5-10 cm^3 of the filtrate in each case.
9) Take an aliquot of each filtrate (5 cm^3 from the first and 2^{nd} ,10cm^3 from 3^{rd} and 4^{th} and titrate with standardised solution.
10) Calculate the equilibrium concentration of the phenol/o-cresol in each bottle.
11) Tabulate your observation n results.

Table 3: Titration For Coconut Shell Charcoal: Phenol

Bottle no.	Amount of Charcoal in gm (m)	Initial conc.of phenol solution mol/dm^{-3} (Co)	Vol.of filtrate taken (V)	Vol.of 0.1M NaOH added	Equilibrium conc. Of acid in mol dm^{-3} (Ce)	Phenol adsorbed x g	x/m	Logx/m	Log Ce
1	2	0.1	5	0.4	4	0.5236	0.2618	-0.5820	0.6020
2	2	0.1	5	0.6	6	0.8054	0.4027	-0.3950	0.7781
3	2	0.1	10	0.4	2	0.514	0.257	-0.5900	0.3010
4	2	0.1	10	0.1	0.5	0.288	0.144	-0.8416	-0.3010

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Calculate the actual concentration of Phenol after adsorption (Ce) in the flask no.1-4, respectively equation: $Ce = Xi^{o} C_T / V$ [mol dm^{-3}]

Where, Xi^{o} - is the volume of the titrant before adsorption (NaOH)- $50cm^3$

C_T – is the volume of the titrant (NaOH)

V – is the volume of the analyte from tab

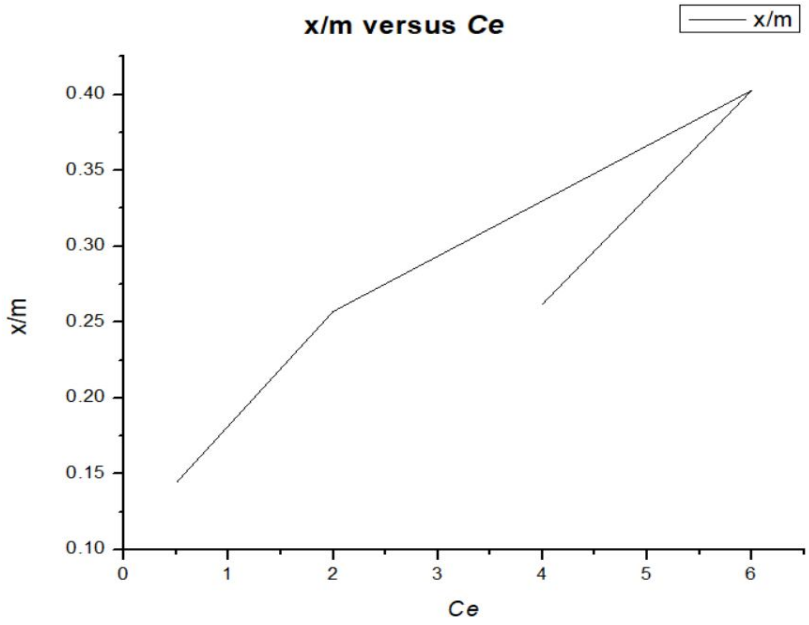


Fig 2 : Plot X/M Values Versus Corresponding Ce Values As Abscissae

In order to test the validity of Freundlich adsorption isotherm, plot log x/m values as ordinate against log Ce values as abscissae. The slope and the intercept of the plot will give 1/n and log K respectively;

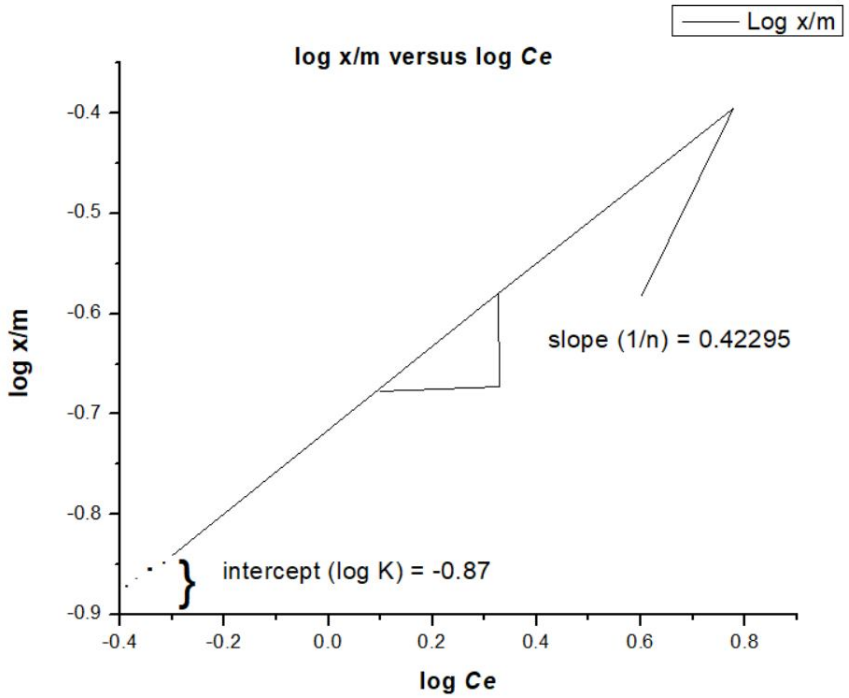


Fig 3: plot log x/m values as ordinate against log Ce values as abscissae. Hence n and K can be calculated.

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Table 4: Titration For Coconut Shell Charcoal: O-Cresol

Bottle no.	Amount of Charcoal in gm (m)	Initial conc.of o-cresol solution mol/dm^{-3} (Co)	Vol.of filtrate taken (V)	Vol.of 0.1M NaOH added	Equilibrium conc. Of acid in mol dm^{-3} (Ce)	o-cresol adsorbed x g	x/m	Logx/m	Log Ce
1	2	0.1	5	1	10	0.2878	0.1439	-0.8419	1
2	2	0.1	5	0.6	6	0.35	0.175	-0.7569	0.7781
3	2	0.1	10	0.8	4	0.3894	0.1947	-07106	0.6020
4	2	0.1	10	0.4	2	0.3298	0.1649	-0.7827	0.3010

Table 5: Titration For Coconut Coir's Charcoal: Phenol

Bottle no.	Amount of Charcoal in gm (m)	Initial conc.of phenol solution mol/dm^{-3} (Co)	Vol.of filtrate taken (V)	Vol.of 0.1M NaOH added	Equilibrium conc. Of acid in mol dm^{-3} (Ce)	o-cresol adsorbed x g	x/m	Logx/m	Log Ce
1	2	0.1	5	1	10	0.2062	0.1031	-0.9867	1
2	2	0.1	5	1.2	12	0.4172	0.2086	--0.6806	0.0791
3	2	0.1	10	1.8	9	0.3016	0.1506	-0.8221	0.9542
4	2	0.1	10	1.2	6	0.235	0.1175	-0.9299	0.7781

Table 6: Titration For Coconut Coir's Charcoal: O-Cresol

Bottle no.	Amount of Charcoal in gm (m)	Initial conc.of o-cresol solution mol/dm^{-3} (Co)	Vol.of filtrate taken (V)	Vol.of 0.1M NaOH added	Equilibrium conc. Of acid in mol dm^{-3} (Ce)	o-cresol adsorbed x g	x/m	Logx/m	Log Ce
1	2	0.1	5	0.8	8	0.0816	0.0408	-1.3893	0.9030
2	2	0.1	5	1.6	16	0.2416	0.1208	-0.9179	1.2041
3	2	0.1	10	1.6	8	0.238	0.119	-0.9244	0.9030
4	2	0.1	10	1.2	6	0.2212	0.1106	-0.9562	0.7781

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IV. RESULTS AND DISCUSSIONS

Activated carbon (AC) was synthesized using inexpensive of Coconut shell and Coconut coir's by a simple and efficient method with very high adsorption capacity. The adsorption experiments were well incorporated on the AC surface via the reactions of Aromatic chemical (o-cresol and phenol) at low temperatures. The complete formation and distribution nanoparticles on the surface of AC have been analysed and explained via different analytical techniques such as TG-DTA, NTA, UV-VISIBLE, DRS, FTIR, XRD. Further, the adsorption property was investigated through adsorption kinetics, adsorption isotherms, and thermodynamics parameters. The maximum adsorption capacity (Q max) of adsorbent was displayed at Coconut shell charcoal rather than coconut coir's charcoal for aromatic compounds - phenol and o-cresol.

The application of the Coconut shell charcoal and coconut coir's have made to adsorption study of Mithi river and Industrial water inlet collected from National Peroxide Ltd. Hydrogen peroxide production industry. Moreover, the importance and role of the AC were examined by a comparative adsorption study of the previous work done.

A. TG-DTA (Thermogravimetry Differential Thermal Analysis)

1) *TG-DTA of the Activated charcoal:* The TG-DTA of the Activated charcoal is shown in the fig. which brought from market for study.

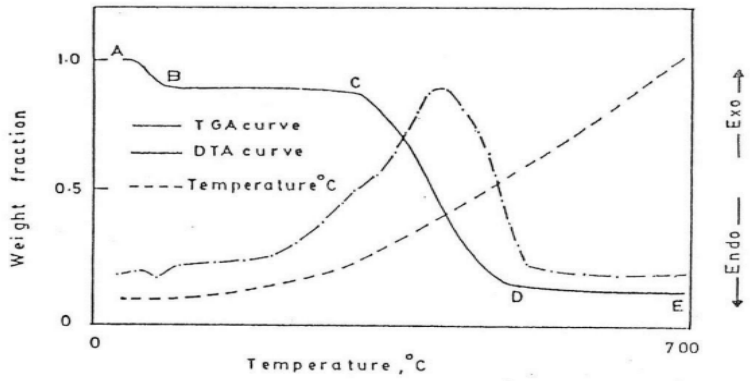


Fig.4 TG-DTA of Activated Charcoal

2) *Coconut shell TG:* Indicates that the sample of coconut shell charcoal is formed at 400^{0}C. Due to this result a suitable temperature range for calcination was obtained, at 400^{0}C.

DTA Curve endothermic peak about 100 ^{0}C which is due to the carbonation of the coconut shell sample.

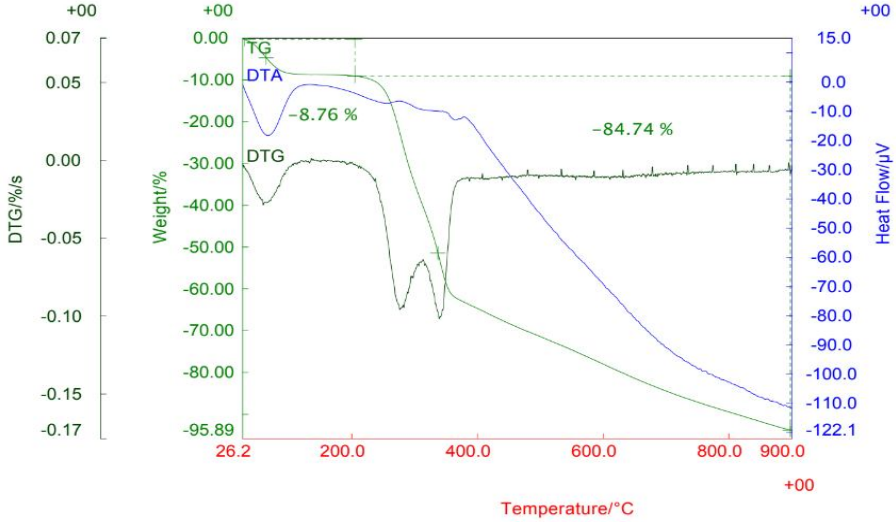


Fig.5 TG-DTA of Coconut Shell

3) Coconut coir's TG: It indicates that the sample of coconut coir's charcoal is formed at 200^{0}C. Due to this result a suitable temperature range for calcination was obtained, at 200^{0}C.

DTA curve endothermic peak below 100 ^{0}C which is due to the carbonation of the coconut coir's sample.

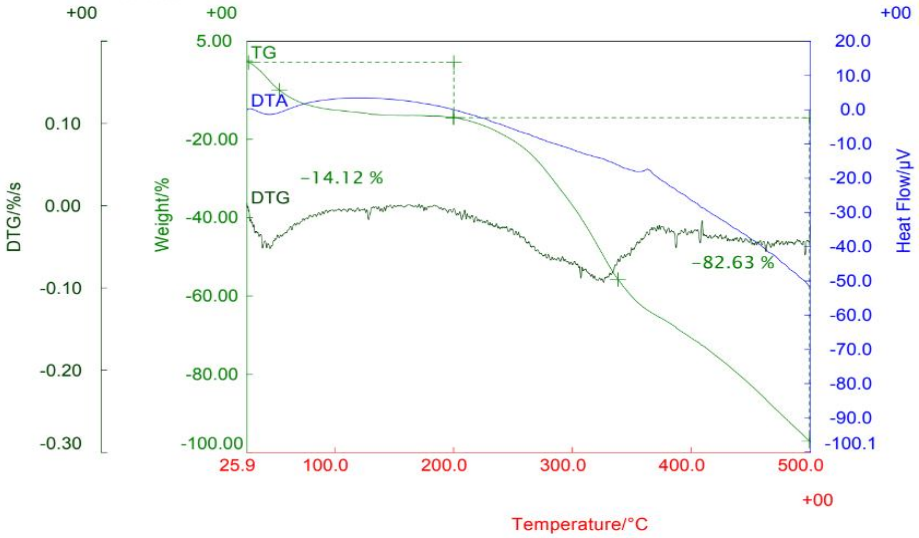


Fig. 6 TG-DTA of Coconut Coir's

B. NTA (Nanoparticle Tracking Analysis)

1) Coconut Shell Charcoal

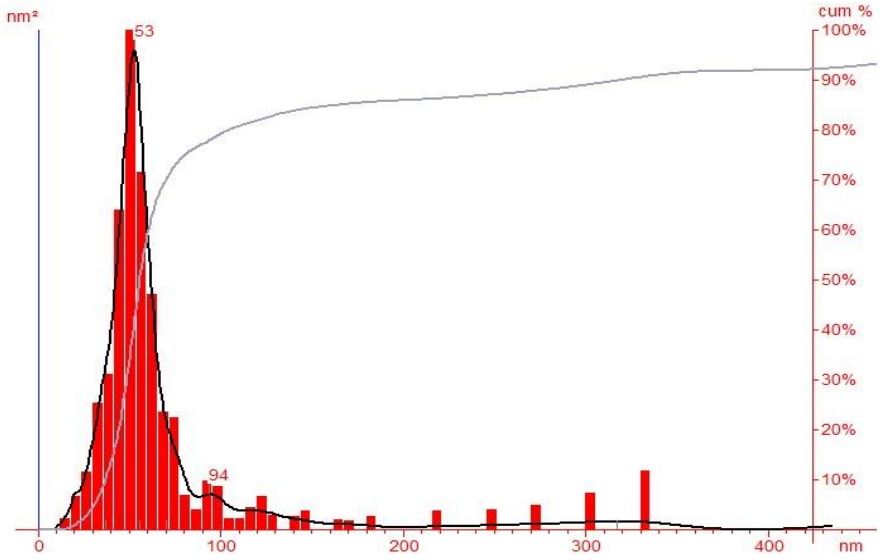


Fig. 7 Particle Size / Surface Area Concentration

The Nanoparticle Tracking Analysis Was performed by using The 'NanoSight NTA LM20' instrument which uses a laser light source to illuminate nano-scale particles. Enhanced by a near-perfect black background, particles appear individually as point-scatterers, moving under Brownian motion. Polydisperse and multimodal systems are instantly recognizable and quantifiable, as are agglomerates and contaminants. The image analysis NTA software suite allows the user to automatically track and size nanoparticles on an individual basis. Results are displayed as a frequency size distribution graph and output to spreadsheet.

Particle size distribution was studied using particle size analyzer. The sample was given pre-treatment before carrying out analysis. The pre-treatment involved sonicating the sample with minimum amount of distilled water in order to reduce the particle sizes for about 10 minutes. Fig.2.1 shows that the particle size distribution is from 0 to 97nm with maximum at 53nm. In the size distribution of nanoparticles, maximum particles are observed to be of size 53nm.

2) Coconut Coir's Charcoal

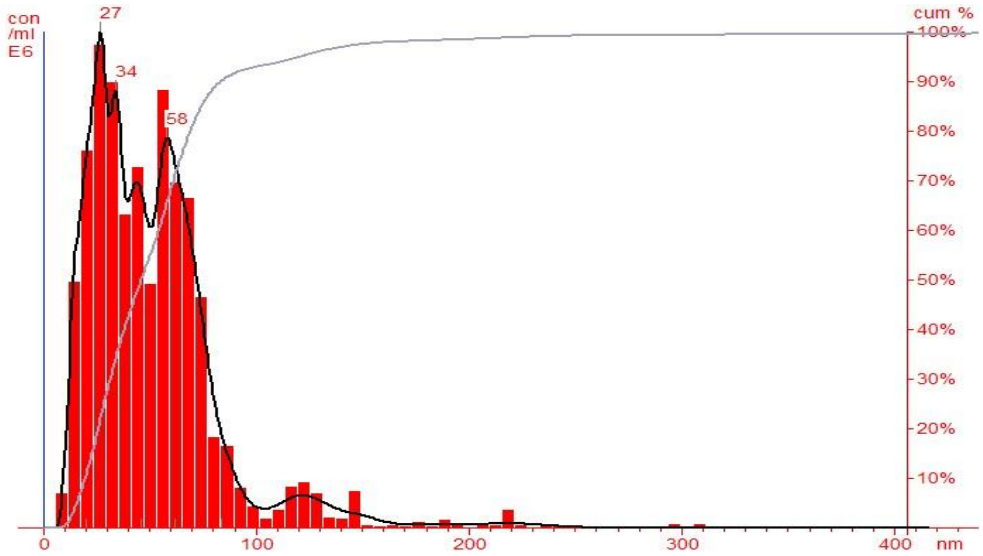


Fig.8 Particle Size / Concentration

Particle size distribution was studied using particle size analyzer. The sample was given pre-treatment before carrying out analysis. The pre-treatment involved sonicating the sample with minimum amount of distilled water in order to reduce the particle sizes for about 10 minutes. Fig.2.2 shows that the particle size distribution is from 0 to 150nm with maximum at 27nm. In the size distribution of nanoparticles , maximum particles are observed to be of size 27nm.

Table 7: NTA max. particle Size of charcoal Sample

SR.NO	SAMPLE	SIZE (nm)
1	Coconut shell charcoal	53
2	Coconut coir's charcoal	27

C. UV-Visible (Ultraviolet)

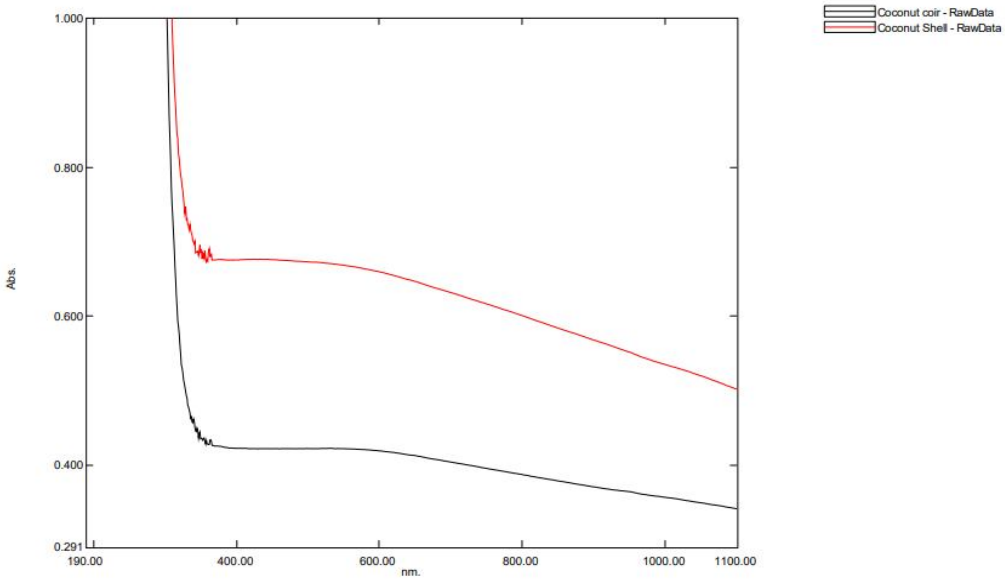


Fig.9 UV-Visible of Coconut Shell Charcoal and Coconut coir's charcoal

The wavelength of the absorption edge of the prepared sample was 500nm . Thus , the band gap energy estimated from absorption edge was about 2.48eV. This indicates that the charcoal sample has some ability to absorb Ultra-violet light.

D. DRS (Diffuse Reflectance Spectroscopy)

1) DRS of Coconut shell Charcoal:

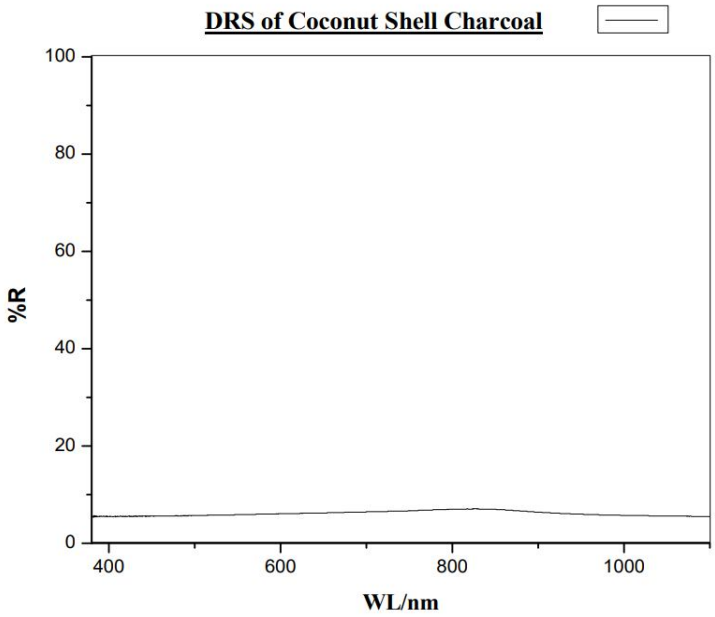


Fig. 10 DRS of Coconut shell Charcoal

2) DRS of Coconut coir's Charcoal:

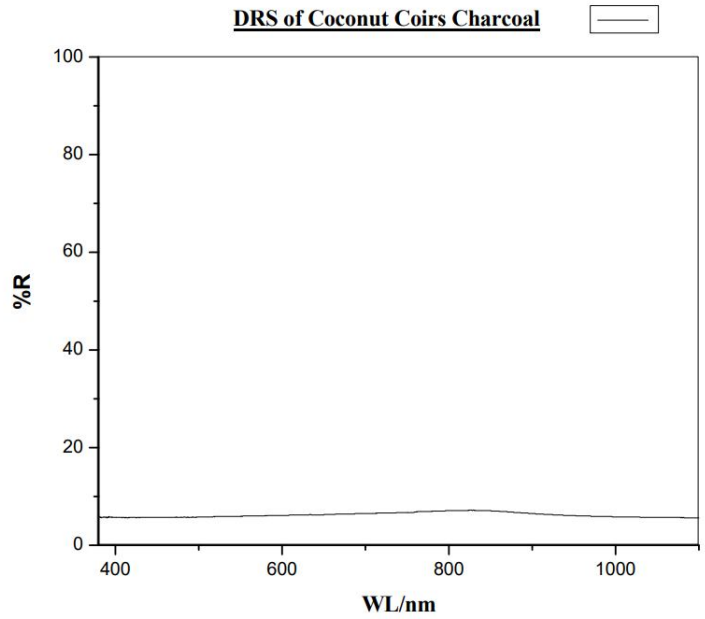


Fig. 11 DRS of Coconut coir's Charcoal

The Instrument used is UV-1900i Shimadzu UV-Visible Spectrophotometer. The range selected for study is 400nm to 1000 nm. The sample holder no.5 and 15 is used to hold the sample inside the instrument. There no such reflectance is absorbed as the black body. The charcoal of both Coconut shell and coconut coir's do not show any reflectance.

E. FTIR Spectroscopy (Fourier Transform Infrared)

1) FT-IR spectrum of Activated Charcoal:

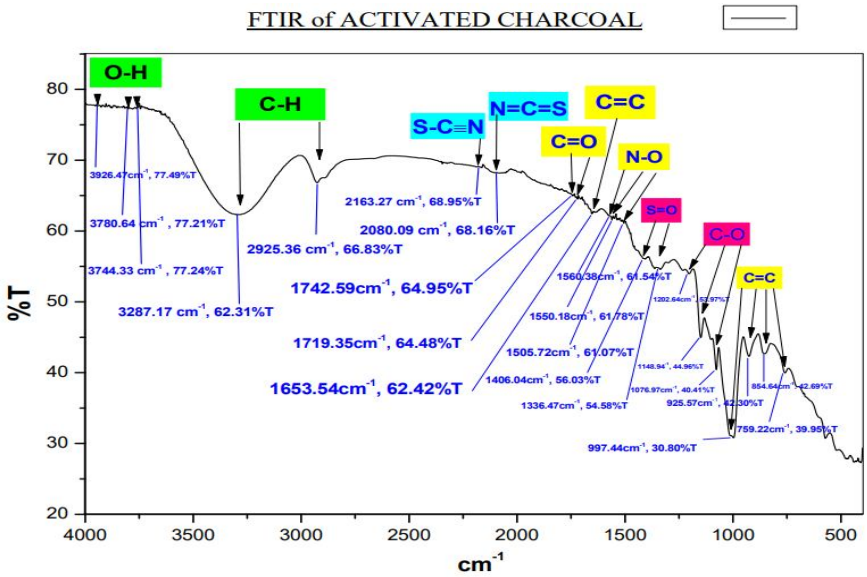


Fig. 12 FT-IR spectrum of Activated Charcoal

In fig.12 FT-IR spectrum of Activated Charcoal shows %T (percentage of transmission) on x-axis and cm-1 (wave-number) on y-axis. The spectrum shows 9 peaks, each having individual characteristics. A broad peak at 3780 cm-1 indicates presence of 0-H bond and peak at 2925 cm-1 indicates C-H bond, 2163 cm-1 corresponds to S-C=N. C=O Stretching having peak at 1742 cm-1. 1653 cm-1 indicates C=C bond, N-O stretching shows peak at 1560 cm-1. The peak at 1406 cm-1 is due to presence of S=O bonding and the peak at 997 cm-1 corresponds to C=C stretching.

2) FT-IR spectrum of Coconut Shell Charcoal:

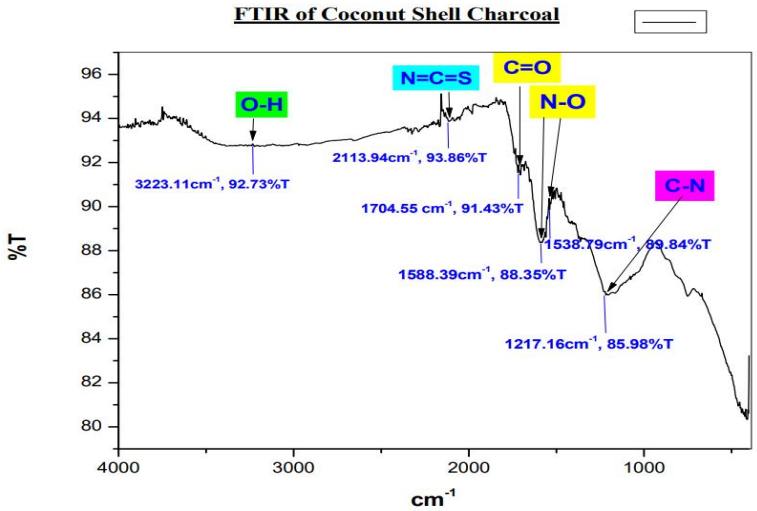


Fig. 13 FT-IR spectrum of Coconut Shell Charcoal

In fig.13 FT-IR spectrum of Activated Charcoal shows %T (percentage of transmission) on x-axis and cm-1 (wave-number) on y-axis. The spectrum shows 5peaks, each having individual characteristics. A broad peak at 3223cm-1 indicates presence of 0-H bond and peak at 2113 cm-1 indicates N=C=S bond, 1704 cm-1 corresponds to C=O. N-O stretching shows peak at 1588cm-1. Peak at 1217 cm-1 indicates C-N bond.

3) FT-IR spectrum of Coconut Coir's Charcoal:

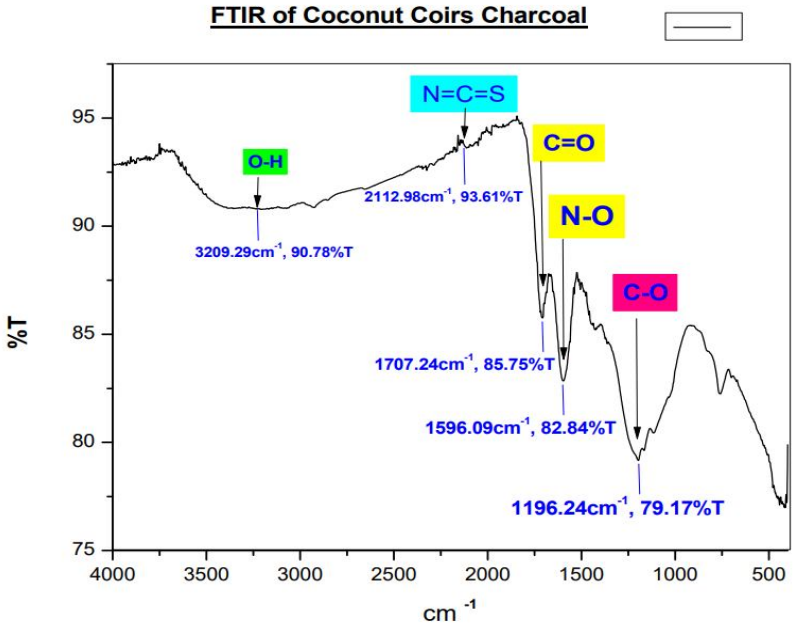


Fig.14 FT-IR spectrum of Coconut Coir's Charcoal

In fig.14 FT-IR spectrum of Activated Charcoal shows %T (percentage of transmission) on x-axis and cm-1 (wave-number) on y-axis. The spectrum shows 5peaks, each having individual characteristics. A broad peak at 3209 cm-1 indicates presence of 0-H bond and peak at 2112 cm-1 indicates N=C=S bond, 1707 cm-1 corresponds to C=O. N-O stretching shows peak at 1596 cm-1. Peak at 1196 cm-1 indicates C-O bond.

F. XRD Analysis (X Ray Diffraction)

1) XRD of Activated Charcoal:

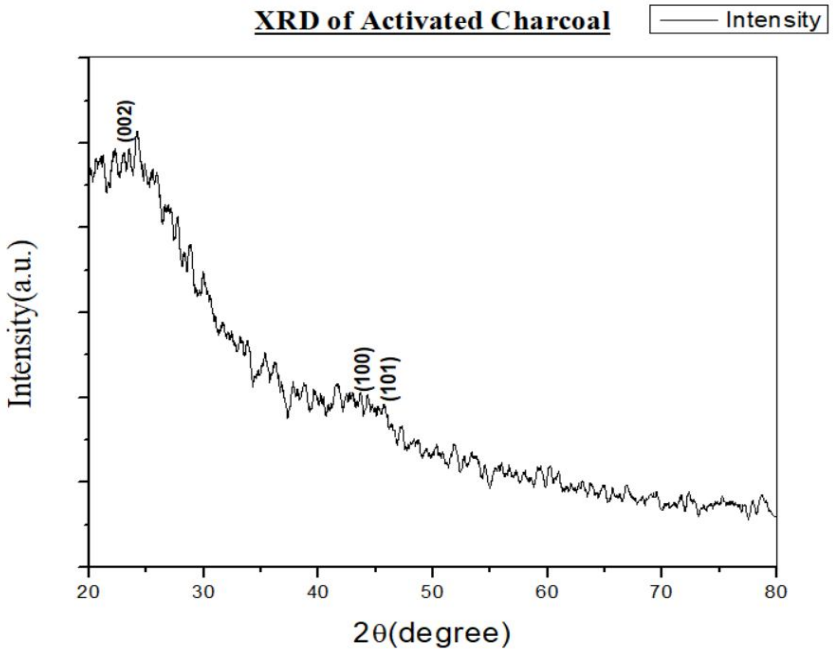


Fig. 17 XRD of Activated Charcoal

2) XRD of Activated Charcoal:

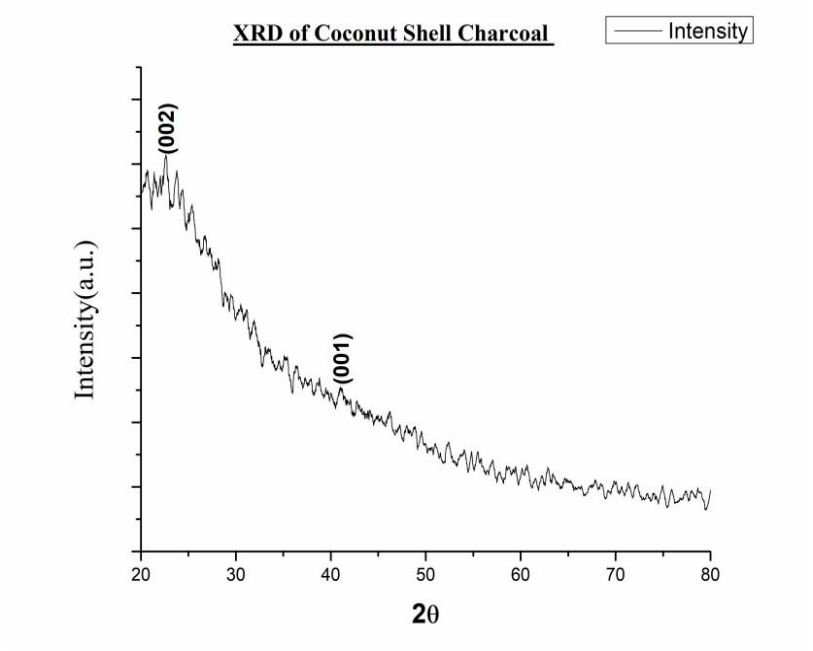


Fig. 18 XRD of Coconut Shell Charcoal

3) XRD of Coconut Coir's Charcoal:

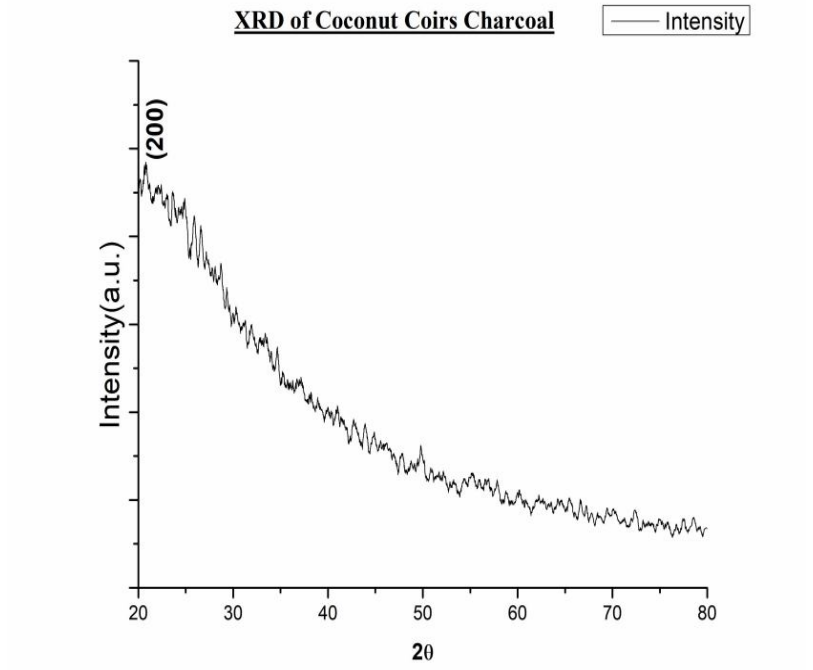


Fig. 19 XRD of Coconut Coir's Charcoal

The XRD of Activated charcoal, Coconut shell charcoal and coconut coir's charcoal
The patterns were obtained over a 2θ range from 20◦ to 80◦ as shown in fig.19 The peaks were in close agreement with JCPDS card numbers 00-0560159 associated to activated charcoal The plane orientation of Activated charcoal was confirmed by the presence of characteristic peaks around 0 0 2), (1 0 0), (1 0 1) planes. There's no such impressive report we get because the sample was amorphous.

V. APPLICATIONS

Applications of AC are enormous. Activated carbon can be used for removal of poisonous heavy metal ions from aqueous solutions. Adsorption in this case is due to the surface complex formation between the metal ions and the acidic surface function group of AC. Adsorption is due to the surface complex formation between the metal ions and the acidic surface function group of AC. The removal efficiency is influenced by various factors, such as solution concentration, solution pH, ionic strength, nature of adsorbate, adsorbent modification procedure, Physical properties (surface area, porosity), and the chemical nature of AC For example lead, cadmium, mercuric ions all are very toxic and carcinogenic. Lead is also a cumulative metabolic poison, acting as a mutagen when adsorbed in excessive amounts. These ions can not be removed from water with classic physical or chemical treatments completely. In order to make it economical, fixed bed systems containing granular carbon could be used in tertiary treatment of wastewater. [5-14] The application of the Coconut shell charcoal and coconut coir's have made to adsorption study of Mithi river and Industrial water inlet collected from industry.

Table 8 : Titration for Coconut Shell charcoal: Mithi river water

Bottle no.	Amount of Charcoal in gm (m)	Vol.of filtrate taken (V)	Vol.of 0.1M NaOH added	Chemical adsorbed x g
1	2	5	0.4	0.3428
2	2	5	0.4	1.2508
3	2	10	0.6	0.4872
4	2	10	1.8	0.4016

Table 9 : Titration for Coconut coir's charcoal: Mithi river water

Bottle no.	Amount of Charcoal in gm (m)	Vol.of filtrate taken (V)	Vol.of 0.1M NaOH added	Chemical adsorbed x g
1	2	5	0.2	0.3424
2	2	5	0.3	0.4816
3	2	10	0.3	0.4716
4	2	10	1.6	0.388

Table 10 : Titration for Coconut Shell charcoal: Industrial water inlet

Bottle no.	Amount of Charcoal in gm (m)	Vol. of filtrate taken (V)	Vol. of 0.1M NaOH added	Chemical adsorbed x g
1	2	5	4.2	0.3424
2	2	5	3	0.2676
3	2	10	4.4	0.3024
4	2	10	4	0.3912

Table 11 : Titration for Coconut coir's charcoal: Industrial water inlet

Bottle no.	Amount of Charcoal in gm (m)	Vol.of filtrate taken (V)	Vol.of 0.1M NaOH added	Chemical adsorbed x g
1	2	5	1.8	0.2116
2	2	5	1.4	0.22
3	2	10	2	0.3724
4	2	10	1	0.3256

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VI. CONCLUSIONS

Adsorption process is a powerful technique that can be used for efficient removal or uptake of pollutant materials from gas and liquid phases. Activated charcoal is one of the most important adsorbents that can be employed for these purposes. The use of AC is perhaps the best broad spectrum control technology available at present moment. It is also quite possible to increase the amount of adsorption of inorganics by impregnating the activated carbon with suitable chemicals. The selection of impregnating material should be so that it encourages the adsorption via usual chemical reactions (e.g. neutralization, redox, hydrolysis, precipitation and catalytic reactions). Among the many factors affecting sorption or removal of toxic materials from aqueous solutions, the pH effect is the most prominent especially in the case of inorganics, weak organic acid and bases which their dissociation is highly pH dependent.[15]

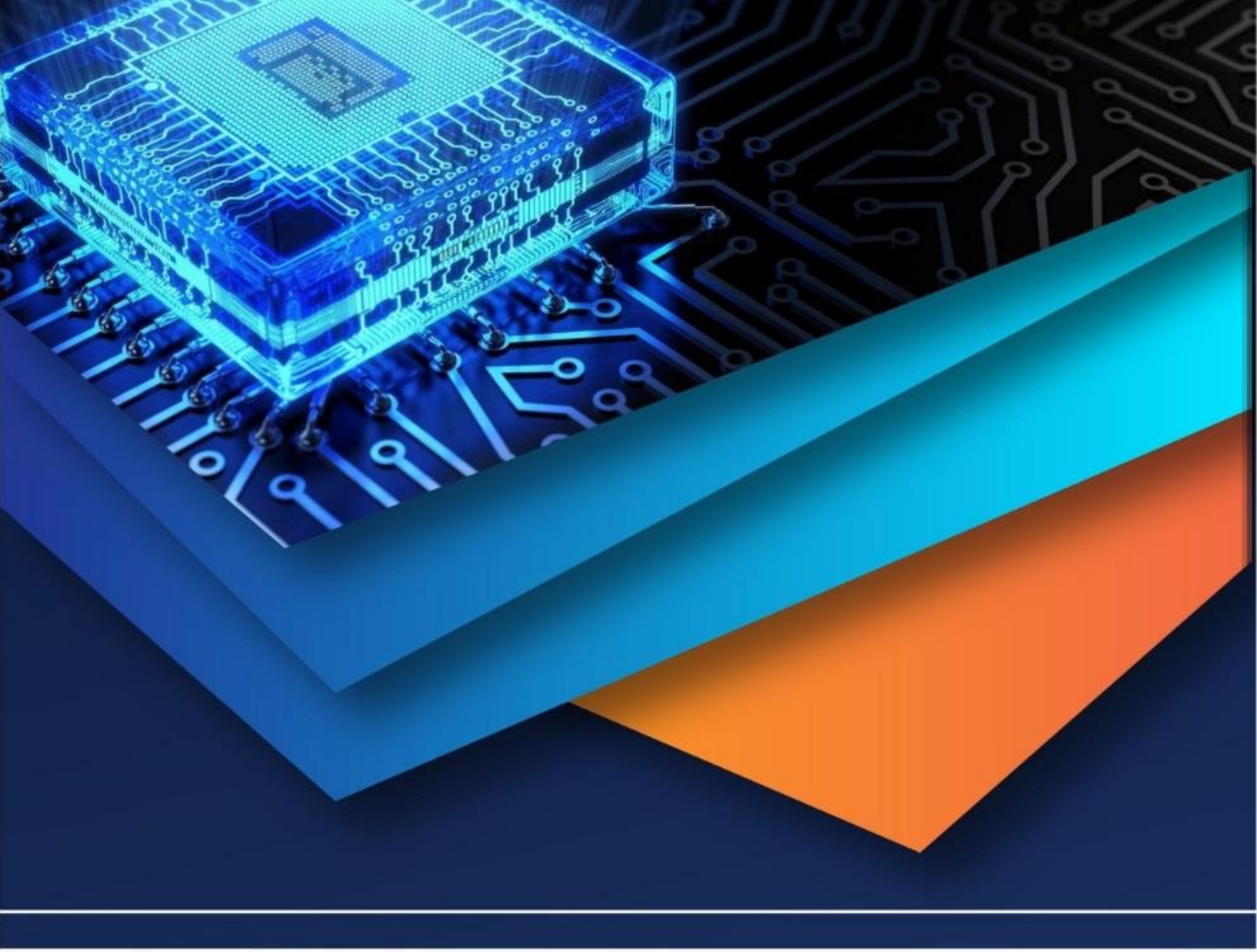
The adsorption studies confirmed that adsorption of adsorbates takes place via the formation of hydrogen bonding, π-π interactions and metal coordination between adsorbent and pollutants. Recyclability test and adsorption of pollutants in synthetic effluents illustrated the sustainability of AC-Coconut shell and Coconut coir's adsorbent. Overall results suggested that AC-Coconut shell and Coconut coir's is a prominent adsorbent for efficient removal of pollutants present in the polluted water.[16]

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