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PREPARATION OF ZINC CHLORIDE-ACTIVATED CARBON FROM *Abutilon indicum* STEM AT VARYING ACTIVATION TEMPERATURES, POWER LEVELS, AND ITS APPLICATION IN THE ADSORPTION OF Cu^{2+} AND Zn^{2+} IONS FROM AQUEOUS SOLUTIONS

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ABSTRACT

In this study, activated carbons (ACs) were synthesized from Abutilon indicum stem using $ZnCl_2$ activation through both conventional and microwave-assisted techniques at varying temperatures (400 °C and 500 °C) and microwave power levels (350 W and 600 W). The synthesized activated carbons and their precursor used as adsorbents were characterized and evaluated for their physicochemical properties and heavy metal adsorption efficiency. Key parameters such as percentage yield, pH, methylene blue (MB) adsorption value, iodine number, and adsorption efficiency for copper (Cu^{2+}) and zinc (Zn^{2+}) ions were investigated. The influence of activation temperature on adsorption performance was particularly notable with AISAC500 showing slightly higher adsorption efficiency for Cu^{2+} (96.23%) compared to AISAC400 (95.74%), while Zn^{2+} removal remained consistent at 80% for both temperatures. Surface morphology and functional group analysis were conducted using Fourier Transform Infrared Spectroscopy (FTIR), Field Emission Scanning Electron Microscopy (FE-SEM), and Energy Dispersive X-ray Spectroscopy (EDS). The results revealed that microporosity and carbonization temperature significantly influence the adsorption capacity of the prepared ACs. This investigation demonstrates that ZnCl2-activated carbon derived from Abutilon indicum stem is an efficient and low-cost adsorbent for the removal of Cu^{2+} and Zn^{2+} ions from aqueous solutions.

Keywords: Activated Carbon, Activation Temperature, Power Levels, Zinc Chloride, Abutilon Indicum Stem Sustainable Development Goal-6: Cleaner Water and Sanitation.

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INTRODUCTION

Waste biomass is a good precursor for the synthesis of activated carbons and may be consumed in the creation of cheap activated carbons. The examples of waste biomasses are materials like fruit peel, rice husk, and dead plant parts like roots, stems, and seed shells.[1,2] ACs comprising of high surface area[3,4] and flexible adsorbent material[5,6] being carbonaceous[6] and pore-rich[7] material. A Huge class of contaminants from several sources, including soil, liquids, and air, can be eliminated using ACs as adsorbents. The contaminants like heavy metals, elevated levels in water can pose several risks to human health, like nausea, salivation, and diarrhoea. The main source of zinc and Copper in water is discharging waste streams from zinc and copper metal works industries. The WHO recommended the maximum acceptable concentration of zinc in drinking water as 5.0 mg/l.[9] Carbonization's primary goal is to increase the carbon percentage in activated carbon. Physical and chemical approaches can be used to accomplish this activation process.[10] While in chemical activation, after carbonization sample is impregnated with an activating agent like KOH, $ZnCl_2$, and Phosphoric acid, and once again carbonized in the physical activation method first step is carbonization in an inert atmosphere, then activation with the help of an oxidizing agent like carbon dioxide.[11] The synthesis of activated carbons depends on temperature, so both yield and microporosity also suffer when the carbonization temperature rises.[12] Commonly, $ZnCl_2$ is employed to activate the charcoal, particularly belonging to lignocellulosic material; zinc chloride also serves as a dampening agent that facilitates the removal of volatile matter.[13] Zinc chloride activation changes cellulose's structure and breaks down its molecules, therefore increasing cavities and hence surface area.[14] Bitumen and other fluids that obstruct the surface of AC are produced less by $ZnCl_2$.[15] $ZnCl_2$ an increasing extent, improves the mesoporosity of ACs.[16] Activated carbon mainly utilized as adsorbent for the carrying away the dyes like methylene

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blue,[17,18] Congo red,[19] methyl orange,[20] phenolic compounds,[21] treatment of grey water,[22] heavy metal ions like Cr(IV),[23] Mn^{+2}, Fe^{+2}, Ni^{+2}, Cu^{+2}, Zn^{+2}, Cd^{+2}, As^{+2} and Pb^{+2} removal[9, 24-26] also in cosmetics.[27] While activated carbons have been widely studied for diverse applications, this research uniquely investigates the synthesis and performance of activated carbons derived from Abutilon indicum stems using both conventional and microwave-assisted activation techniques. The study centres on assessing their adsorption capabilities for dyes and heavy metals in aqueous solution. By focusing on this underexplored biomass source, the work fills a significant gap in the literature through a systematic evaluation of its adsorption properties.

EXPERIMENTAL

Waste plant residues (stem) of Abutilon Indicum were locally collected around the farms in and nearby to Malkapur, District Buldhana, Maharashtra, India, Pin: 443101

Stem Sample Pre-Treatment, Carbonization, and Activation

The stem samples of Abutilon indicum were thoroughly washed with water to eliminate any adhering foreign particles. Following this, the samples were sun-dried for 5 to 7 days and subsequently cut into small pieces. Before carbonization, residual moisture was removed by heating the samples in a hot air oven at 110 °C for one hour. A dried biomass sample was carbonized in a muffle furnace for one hour at 300°C. Afterward, it was allowed to cool to room temperature, washed with doubly distilled water, and dried in a hot air oven at 110° C. The carbonized sample was then ground under moderate pressure using a mortar and pestle and sieved using a 100–200 mesh sieve. Zinc chloride activation was subsequently accomplished using the Girgis et al. (1999) model with minimal modification. 25 g of carbonized material was mixed with an aqueous solution of zinc chloride in a 1:5 ratio. This mixture was steeped for twelve hours. The paste was then placed in the muffle furnace for one hour at 400°C and 500°C, respectively. Finally, the sample was allowed to cool to ambient temperature, cleaned with distilled water, and dried in a hot air oven at 110°C. Afterward, it was cooled to room temperature.[28,29] After additional sieving by a 150-micron mesh, the synthesized AC was stored in an airtight bottle. In the microwave-assisted technique, the carbonized sample was submerged in $ZnCl_2$ activation agent (1:5 ratio). After these carbons were taken in a quartz crucible and kept in the microwave oven at two different powers (350 and 600 Watts) for a radiation time of 10 minutes for activation. Thus obtained activated carbons were cooled and washed with distilled water, dried at 110°C in a hot air oven.[30]

Table-1: List of Carbonized and Activated Carbons of Abutilon Indicum Stem

S. No.	Abutilon Indicum Stem sample	Sample Code
1	Carbonized at 300°C	AIS300
2	Activated at 400°C	AISAC400
3	Activated at 500°C	AISAC500
4	Activated in a Microwave oven at 350 watts	AISMW 350
5	Activated in a Microwave oven at 600 watts	AISMW600

Assessment of Yield

Activated carbon yield was calculated by using the given formula.[31]

$$(\%)\text{Yield} = \frac{\text{Amount of AC after activation}}{\text{Amount of the raw material before activation}} X\ 100$$

Measurement of pH

One gram of the sample was taken into a 250 ml beaker, then 100 ml of distilled water was added and swirled for fifteen minutes. The samples were kept unaltered, and a digital pH meter (Equiptronic EQ-610) was used to find the pH.

Turbidity Removal Tendency

Turbidity in water is caused by suspended particles such as clay, silt, finely divided organic compounds, plankton, and other microscopic organisms. A wastewater sample was collected and left undisturbed overnight to allow the larger particles and clay to settle. Then it was filtered through filter paper, and turbidity was determined by using a digital Nephelometer (Equiptronic EQ-815) before treatment with activated carbon samples. This tendency of synthesized activated carbons was checked after passing the wastewater through the activated carbon column at the rate of 5ml/minute. The eluent was collected and checked for a change in turbidity of wastewater by the literature method.[32] Turbidity removal tendency was calculated by the following formula:

$$\text{Turbidity removal (\%)} = \frac{\left(T_i - T_f\right)}{T_i} \text{X } 100$$

Where T_i and T_f initial and final turbidity NTU or JTU

Iodine Value (IV)

Defined as the milligrams of iodine absorbed by one gram of activated charcoal, iodine value, or iodine number. The synthetic carbon's iodine count was found to be as shown here. Using 0.5ml 1% starch solution as an indicator, 10 ml of 0.1 N iodine solution was titrated with 0.1 N sodium thiosulfate solution until it became colourless. Conical flask V_{blank} matches the burette reading. Then weigh 50 mg of activated carbon and transfer it to a conical flask containing 15 ml of 0.1 N iodine solution, shake the flask for 5 min, and filter it. Then, titrate 10 ml of filtrate with standard sodium thiosulphate solution using 0.5 ml starch solution as an indicator; now the burette reading corresponds V_{sample} One then computed the iodine value by applying the following formula:

$$\text{IV} = \frac{\left(V_{\text{blank}} - V_{\text{sample}}\right).\ \text{Normality}\ .\ (126.9)}{\text{M}} \left(\frac{5}{10}\right)$$

IV is iodine value (mg/g), V_{blank} and V_{sample}Are the volumes of sodium thiosulfate solutions needed for blank and sample titrations in (ml)? N is the normality of sodium thiosulfate solution; 126.9 is the atomic mass of iodine; M is the mass of activated carbon in grams.[33]

Methylene Blue (MB) Value

Methylene blue value helps in understanding the adsorption capacity of activated carbon for methylene blue-like dye molecules. 1000 ppm MB dye solution was prepared by dissolving 0.5g of MB in a 500 ml volumetric flask using deionized water. From this stock solution, different concentrations (2, 5, 7, 9, 10 ppm) were prepared in separate 500 ml volumetric flasks, respectively, and absorbance on a UV-Visible spectrophotometer was recorded. A calibration curve (Absorbance Vs Concentration in ppm) was drawn for the determination of MB values of different activated carbon samples. 0.1g of each prepared activated carbon was transferred to a conical flask containing 10 ml of a ten-ppm solution of methylene blue and kept on a shaker at room temperature for one hour. After that solutions were filtered using filter paper. Absorption of the filtrate of MB was analysed by using a spectrophotometer at its maximum wavelength of 648 nm. The MB values Q_e (mg/g) were calculated by the following formula:[34]

$$\text{Q}_\text{e} = \frac{(\text{C}_\text{o} - \text{C}_\text{e}).\text{V}}{\text{W}}$$

Where C_o and C_e Are the initial and equilibrium concentrations of MB (ppm), respectively, V is the volume of aqueous MB dye solution, and W is the weight of activated carbon (g).

Adsorption of Heavy Metals (Cu and Zn)

Activated carbons are good adsorbents and can adsorb heavy metals. Adsorption of heavy metals by synthesized low-cost adsorbent (ACs) was evaluated under normal conditions, and results were confirmed by atomic absorption spectroscopy (AAS)[35] (SPV-Spectronics AS205). Standard 1000 ppm solution, Copper (Cu) and Zinc (Zn) solutions were prepared by dissolving $CuSO_4 \cdot 5H_2O$ 3.93 grams in one litre of deionized water, and later by dissolving 0.4390 grams in 100ml of deionized water. From both the stock solutions, desired 10 ppm solutions of both metals were prepared by the dilution method. Columns of activated carbons were prepared by using 1g of AC in a cylindrical borosilicate glass tube supported by glass wool. After this, 25 ml of standard 10 ppm solution of metal was allowed to pass through the column at the rate of 5ml/minute, and the resultant eluent was analysed for the respective metal ion on AAS.

Surface Functional Group Analysis

The FTIR spectroscopy (Thermo Scientific iD3) was used to estimate the surface functional groups of activated carbons synthesized at different temperatures. The FTIR spectra of all the samples were recorded in the range of 500 to 4000 cm^{-1}.

Field Emission Scanning Electron Microscopy (FE-SEM) and EDS Analysis

FE-SEM, Carl Zeiss Sigma 500 connected with EDS, was used to identify the topography of ACs and elemental determination, respectively.

RESULTS AND DISCUSSION

Determination of Yield

The yield of the synthesized activated carbons at different temperatures by using $ZnCl_2$ activating agent at an impregnation ratio of 1:5 is shown. In Fig.-1, the percentage of yield of ACs decreases as the activation temperature increases from 400 °C to 500 °C. Similarly, microwave microwave-assisted sample also shows the same trend when the power is shifted from 350 watts to 600 watts. This result is attributed to the burn off and volatilization of tar at higher temperatures, thereby a decrease in the yield of ACs.[36] The % Yields of different ACs are summarized in Fig.-1.

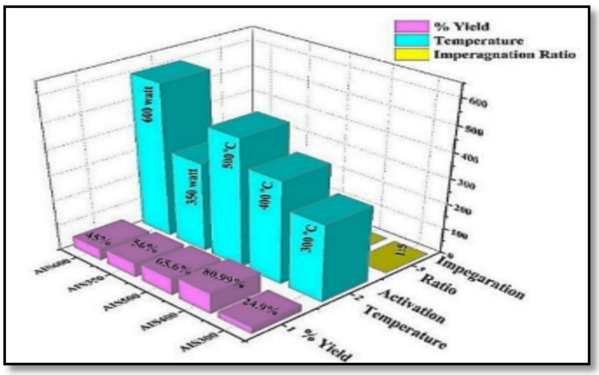


Fig.-1: The % Yields of different ACs at different Activation Temperatures with the Impregnation Ratio

Determination of pH

The observed pH of AIS300 was slightly basic (8.08), and after chemical activation at 400 and 500 °C pH decreased to 6.98 (AISAC400) and 7.12 (AISAC500) similar trend was followed by microwave-assisted ACs AISMW350 and AISMW600, respectively. ACs of pH between 6 to 8 are useful for many applications.[37,38] So, the synthesized ACs could be useful for most processes that involve adsorption from solutions. The observed pH of activated carbons is summarized in Fig.-2.

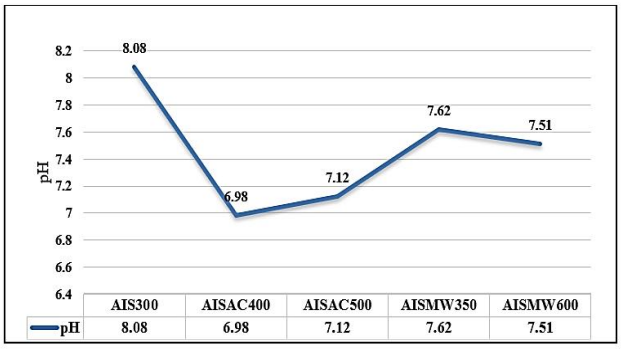


Fig.-2: The pH of different Activated Carbon Samples

Turbidity Removal Tendency

Synthesized Activated carbons are appropriate to remove turbidity and could be an efficient alternative.[39] All the carbon samples exhibit outstanding turbidity removal tendency for the samples AIS300 (82.91%), AISAC400 (82.750%), AISAC500 (81.660%), AISMW350 (83.33%), and AISMW600 (83.08%), respectively, and the maximum value reaches 83.33% in normal conditions. These results highlight the effectiveness of the prepared carbons in improving water quality by reducing particulate contamination. This directly aligns with Sustainable Development Goal 6, which emphasizes ensuring access to safe and clean water for all.

Iodine Number or Iodine Value

The iodine value is a method used to estimate the adsorption capacity of activated carbons (ACs) and serves as an indicator of their porosity. It is defined as the amount of iodine adsorbed by one gram of carbon, reported in mg/g. A lower iodine value corresponds to a lower surface area.[40] The observed iodine values are summarized in Fig.-3.

The highest Iodine value obtained for the AIS300 was 456.84 mg/g at a carbonization temperature of 300 °C, whereas after activation at 400 °C, the Iodine value was lowered to 342.63 (AISAC400), 304.56 (AISAC500), respectively. Contrary to iodine value, in microwave-assisted ACs increases from 350 watts to 600 watts.

Methylene Blue Value (MB Value)

MB values are helpful to know the adsorption efficiency of ACs for molecules that mimic the methylene blue, which helps estimate the surface area. The activated carbons (ACs) contain more mesopores for those higher MB values.[4142] For microwave-assisted AC sample AIS600MW, the MB value achieved is the greatest. Figure-4 summarizes the range of 335 to 423 (mg/g) that all observed for MB values.

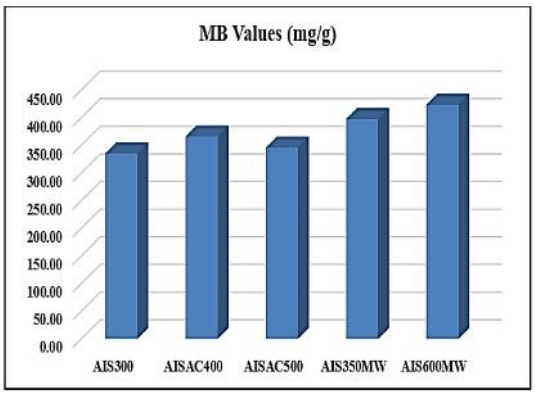


Fig.-3: The Iodine Value Trend of Different Activated Carbon Samples

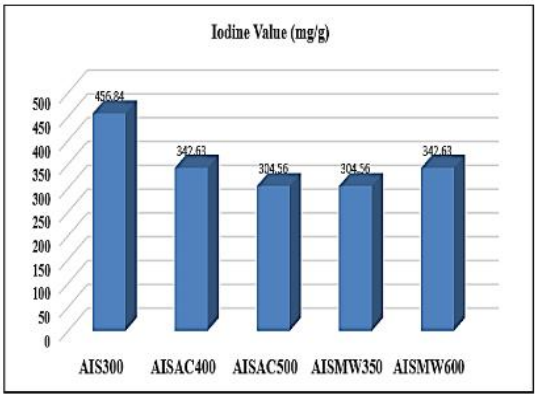


Fig.-4: The Methylene Blue Value of different Activated Carbon Samples

Adsorption of Heavy Metals [Cu (II) and Zn (II)]

Common contaminants in industrial wastewater are heavy metals, which have dangerous effects on living entities; hence, their disposal into the environment is a major problem. Many standard techniques, including coagulation, filtration, ion exchange, reverse osmosis, and adsorption, are applied[43] to lower the concentrations of these dangerous heavy metals in water effluences. Among these techniques, adsorption on activated carbon (AC) is regarded as one of the most sensible and cheap ones for water treatment.[44] Large surface area, porosity, rough surface, and availability of diverse functional sites make AC a flexible adsorbent for the adsorption of heavy metals.[45] Adsorption of Cu^{+2} and Zn^{+2} was studied under normal conditions, and the results and trend of adsorption of adsorbate (Cu^{+2} and Zn^{+2}) on adsorbent were summarized in Fig.-5, which indicates that AIS300 prepared at 300 °C adsorbs 95.05% of Cu (II) and 95.6% of Zn (II) after activation at 400 °C.

Adsorption of Cu (II) increased to 95.74% (AISAC400), but Zn^{+2} adsorption was decreased at higher temperatures to 80%. Further, the activation temperature was increased to 500°C at this stage, AISAC500 shows adsorption of Cu (II) to 96.23% as well as adsorption of Zn (II) to 80%. The percent adsorption of Cu^{+2} increased at higher temperature, whereas Zn (II) remained unchanged. Similarly, AC synthesized by microwave-assisted method AISMW350 and AISMW600 competes

well and shows percent adsorption for Cu (II) to 91% and Zn (II) to 80% respectively. Adsorption percentages of Zinc (II) and Cu (II) are summarized in Fig.-5.

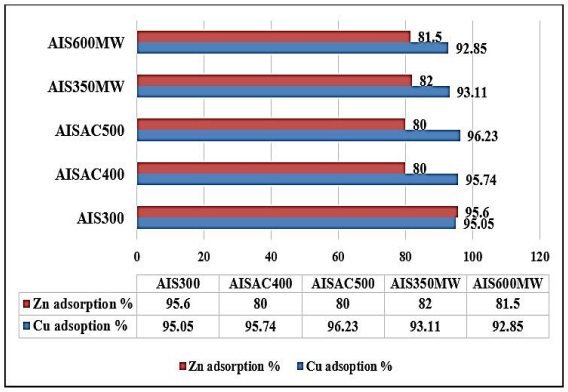


Fig.-5: The Adsorption Percentages of Zinc (II) and Cu (II) of different Activated Carbon Samples

Surface Functional Group Analysis

The following figures depict the FTIR results of carbonized carbon and activated samples, analysed at different temperatures and power levels (in watts).

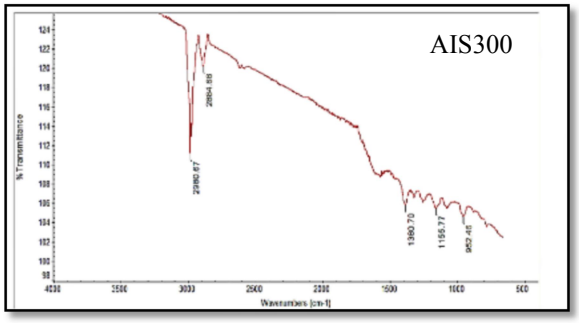


Fig.-6: FTIR Spectra of Carbonized Carbon at 300 °C

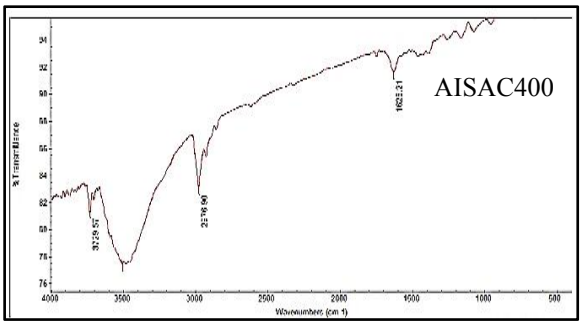


Fig.-7: FTIR Spectra of Activated Carbon at 400 °C

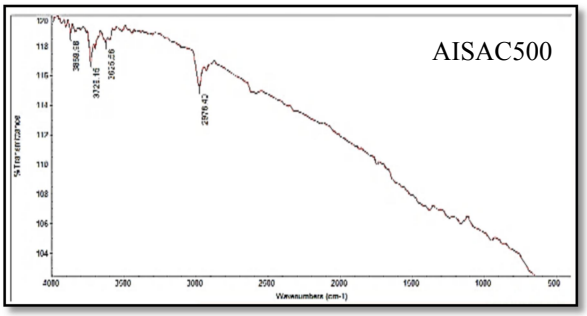


Fig.-8: FTIR Spectra of Activated Carbon at 500 °C

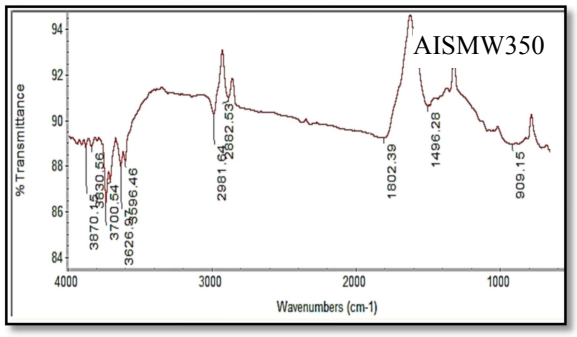


Fig.-9: FTIR Spectra of Activated Carbon at 350 Watts

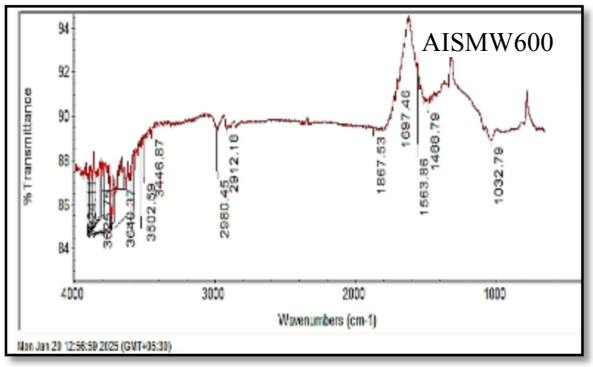


Fig.-10: FTIR Spectra of Activated Carbon at 600 Watts

Figure-6, the FT-IR spectra of AIS300 show the band at 2980.67 cm^{-1}, 2884.68 cm^{-1} shows the presence of aliphatic –C-H stretching.

Figure-7, AISAC400 shows a band between 3625.56 cm^{-1} to 3869.98 cm^{-1} attributed to hydroxyl O-H vibrations, which appeared after chemical activation by $ZnCl_2$ at 400°C. Strong band around 2976.40 cm^{-1} corresponding to -CH stretching. In the Fig.-8, AISAC500 the chemical activation at 500°C screened the -OH bands as well as develops -C=O carbonyl stretching around 1628.21 cm^{-1} which confirms that heat treatment affects the surface functionality of AC, on the other hand Fig.-9 and 10 were the activated carbons which were chemically activated by microwave assisted method found to contain the bands around 3625.75 cm^{-1}, 3625.75 cm^{-1} and 3502.59 cm^{-1} are attributed to phenolic hydroxyl functionality. The stretching's found at 2980.45 cm^{-1} and 2981.64 cm^{-1} are attributed to C-H functionality. Carbonyl functional group stretching was attributed to 1697.46 cm^{-1}, 1802.39 cm^{-1}. The stretching at 1563.86 cm^{-1}, 1496.28 cm^{-1} is due to aromatic C=C in both microwave-assisted ACs. Observed stretching frequencies are tabulated in Table-2.

Table-2: IR Stretching Frequencies Found in Carbon Samples

S. No.	Functional group	AIS300 (cm^{-1})	AISAC400 (cm^{-1})	AISAC500 (cm^{-1})	AISMW350 (cm^{-1})	AISMW600 (cm^{-1})	Ref.
1	Aromatic C=C	--	--	--	1563.86	1496.28	7
2	C-H in CH_3 and CH_2	2980.67 2884.68	2976.40	2976.90	2980.45	2981.64 2882.53	30
3	C=O Str.	--	--	1628.21	1697.46	1802.39	46
4	O-H str.	--	3869.98 3729.15 3625.56	3729.57 3500	3625.75 3648.37 3502.59	3870.15 3700.54 3626.97 3596.46	47

FE-SEM and EDS Analysis

Figure-11(11a-11e) is the SEM micrographs of carbonized carbon and activated samples at different temperatures and 350 watts. SEM has been universally used to characterise the topography of activated carbons. SEM micrograph of AIS300 shows fewer pores and cavities compared to other activated and microwave-assisted samples. During carbonization, most of the organic materials were

evolved, which cause the formation of pores, and a ruptured surface was left behind with a small number of pores[14] SEM image of AISAC400, activation at 400°C shows extensive rough surface with bigger cavities and more pores as compare to AIS300 it may be due to the impact of activator on the topographical characteristics of carbon surface.[47] Also, the grains are observed in AISAC400. SEM micrograph at 500 °C of AISAC500 shows wide cavities with a rough surface with pores of different sizes. Microwave-assisted activated carbon AISMW350 shows oval shape prominent pores with smooth surface, whereas AISMW600, which was activated by increasing 250 watts as compared to AISMW350, showed irregular pores with broken surface.[48] SEM micrographs of the carbonized sample and activated carbons at different temperatures demonstrate the effect of higher temperature and activating agent.

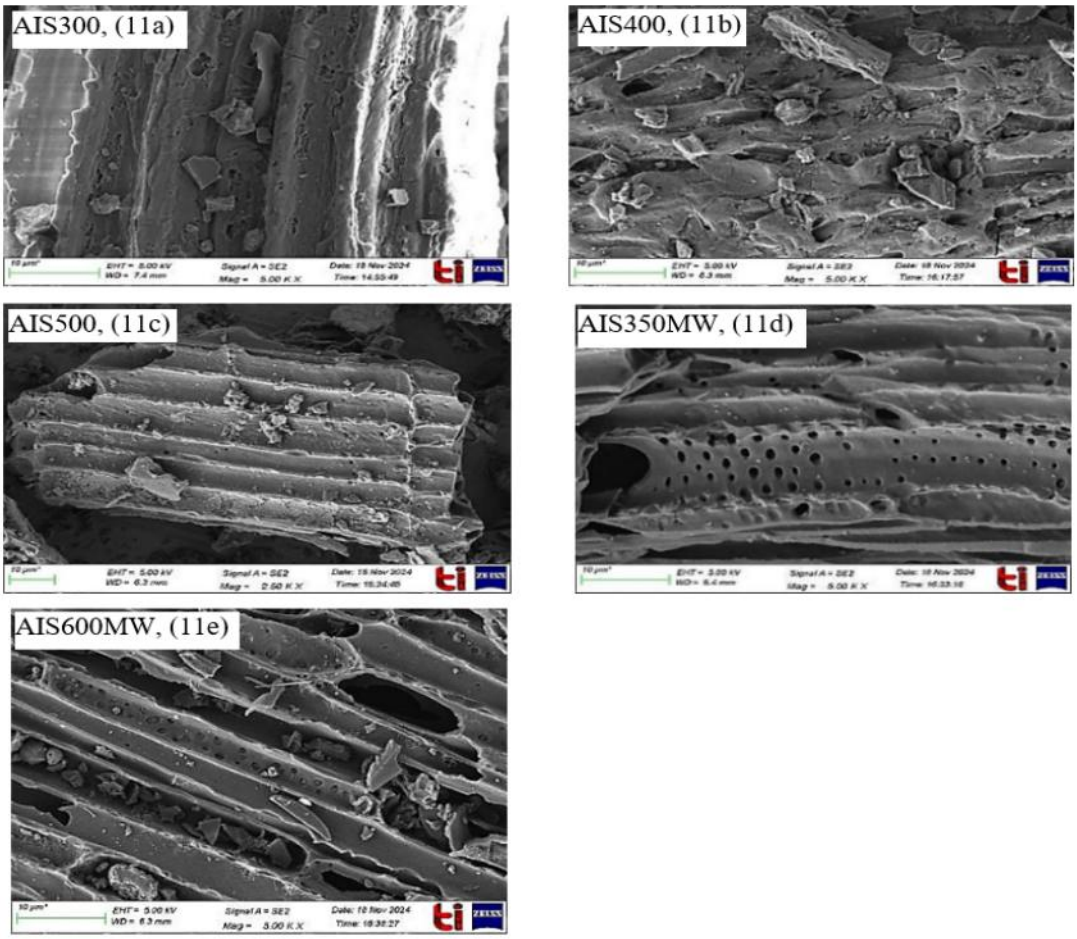
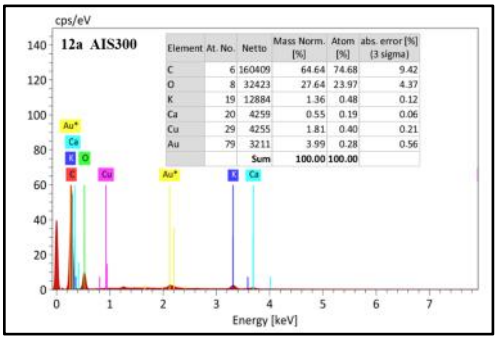


Fig.-11 :(11a-11e) FE-SEM Micrographs of Carbonized Carbon and Activated Samples at different Temperatures and 350 Watts

The following Fig.-(12a-12d) depicts the EDS results, along with the element percentages of carbonized carbon and activated samples, at different temperatures and 350 watts.



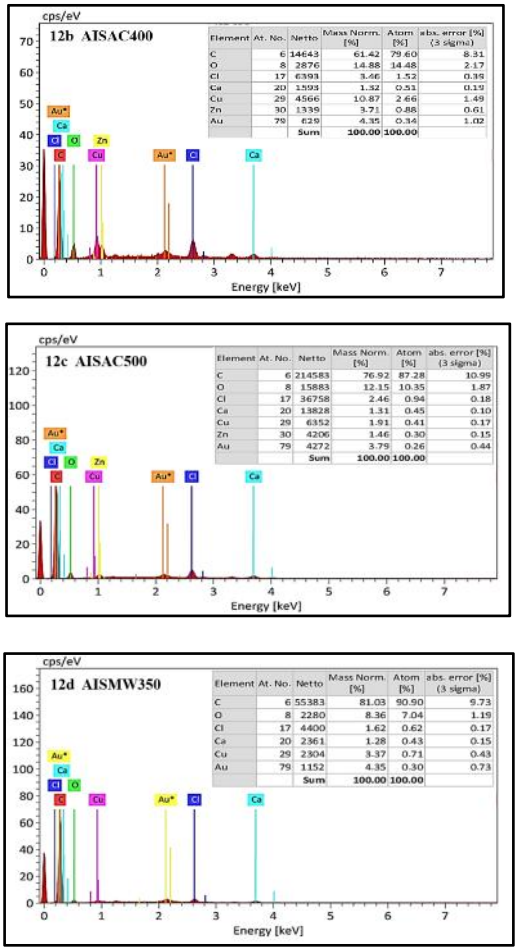


Fig.-12: The EDS Results, along with the Element Percentages of Carbonized Carbon and Activated Samples, at different Temperatures and 350 Watts

Figures-12a-12d show that all the samples contain carbon as a major element, with AIS300 (74.68%), AISAC400 (79.60%), AISAC500 (87.28%), and AISMW350 (90.90%) exhibiting the highest carbon content. In contrast, the oxygen content decreases from AIS300 to AISMW350. From the EDS, it is clear that the chlorine composition percentage was decreased, respectively, which was a remarkable sign of better activation of carbonized samples.[49] The volatile molecules, such as hydrogen and oxygen, were released during carbonization at higher temperatures, resulting in activated carbon that becomes rich in carbon content.[50] Except carbonized sample AIS300, all the activated samples activated by the activating agent $ZnCl_2$ contain Chlorine as a composition element. An increase in activation temperature reveals the activation of the carbonized sample. Appearance of zinc as a composition element in activating samples AISAC400 and AISAC500 indicates involvement of an activating agent containing the zinc element.[51,52] The existing research work focuses on SDG 6: Cleaner Water and Sanitation. Accordingly, researchers can attempt to remove turbidity, dye, $Cu^{2+,}$ and Zn^{2+} from polluted water to make it usable for all.

CONCLUSION

This work reports applications of activated carbons synthesized from waste biomass of Abutilon Indicum stem for turbidity removal tendency, dye removal by MB value study for the adsorptive efficiency of synthesized ACs, and heavy metal (Cu^{2+}, Zn^{2+}) adsorption efficiency to make polluted water clean, and also studies on iodine value to comment on the surface area of ACs. All these applications depend on the surface of the adsorbent, and the reported activated carbons are used as adsorbents to study their effectiveness. Before their applications, these ACs are characterised by FTIR, FE-SEM, and EDS for their surface functionality and topographic characteristics. FTIR, FE-SEM, and EDS studies show ACs were indeed produced. FTIR analysis confirms the presence of hydroxyl and carbonyl groups on the surface of ACs. FE-SEM studies reveal pore-rich and extensive coarse surface for potential adsorption. EDS reported the involvement of the activating agent $ZnCl_2$ in chemical activation. Further higher carbonization temperature increases the carbon content and decreases

oxygen content in ACs, suggesting that better activation was accomplished for adsorption. The physicochemical parameters study supports the potential applications of ACs for adsorption processes. All synthesized ACs have adequate turbidity removal tendency ranging between 81.66 to 83.33, also the pH value ranging between 6.98 to 7.61 is favourable for adsorption processes and may be particularly useful in cosmetic purposes. All ACs show iodine value (IV) ranges from 304.56- 456.84 mg/g and methylene blue (MB) values reach 423.63 mg/g from 345.90 mg/g, which ascribes the sufficient available surface area and sign of good adsorption capacity of ACs, respectively. Heavy metal adsorption study shows adsorption efficiency of ACs for aqueous solutions ranging from 92.85-96.23% for Cu (II) and 80-95.6% for Zn (II) in normal conditions, suggesting these synthesized ACs are outstanding adsorbents for these metal ions and further used to eradicate other heavy metals from water. It can be concluded that ACs from waste biomass of Abutilon Indicum stem activated by zinc chloride produce effective adsorbents for their potential applications in adsorption and act as good and cheaper adsorbents.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-6: Cleaner Water and Sanitation*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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