

Synthesis and Characterization of Carbon foam using sucrose, chitosan gel and activated carbon

Shekhar Pandharipande¹, Rajendra Ugwekar², Akshata Ramteke³, Shivank Sharma⁴

Abstract—Carbon foam has received significant attention in recent years with researchers developing numerous applications in different field such as aeronautics, agriculture, medical etc. This versatility in applications is possible due to its exceptional properties such as high electrical conductivity, high porosity & light weight among others. This material also possesses high surface area which makes it suitable for application in catalysis. To date, numerous forerunners have been used for creation of 3D organized carbon materials including pitch, carbon nanotubes, graphene foam and polymer foams. In this paper the synthesis of carbon foam by utilizing activated carbon prepared from saw dust and polysaccharides such as sucrose & chitosan has been presented. Two different carbon foams were prepared, one from sucrose resin & activated carbon and the other one from chitosan gel, activated carbon and sucrose resin. For the detection of function groups present, FTIR of the sample was carried and for obtaining the surface morphology SEM was carried out.

Index Terms—Sucrose resin, chitosan, saw dust, activated carbon, porous carbon, carbon foam.

1. INTRODUCTION

1.1. Carbon foam:

Carbon foam is a carbonaceous material with associated 3-dimensional micro structure having special features & properties like high porosity, high temperature resilience, excellent resistance to erosion, low density etc. These properties have enabled the use of carbon foam in wide range of applications in multiple fields ranging from aeronautics to catalysis. However, in spite of its numerous applications, improvement & utilization of carbon foam is significantly limited due to their poor mechanical characteristics & high cost of production. Thus few attempts have been made in order to enhance the mechanical strength of carbon foam through a high weight system or an involute process. Consequently, it is requisite to develop a simple and efficient method to fabricate carbon foams with relatively high mechanical strength.[1]

1.2 Sucrose - Table sugar:

Sucrose, commonly called as table sugar having molecular formula $C_{12}H_{22}O_{11}$ is a disaccharide, a molecule composed of glucose and fructose both monosaccharides. Sucrose, a naturally occurring substance in plants, is processed and refined to obtain table sugar.

1.3 Chitosan:

Chitosan is obtained by carrying out de-acetylation of chitin, which is commonly obtained from shells of crustaceans such as prawns, crabs, fish scales etc. Chitosan finds multiple applications in medical, agriculture & other domains. Chitosan is extensively used in agriculture for seed treatment & as bio pesticides by virtue of its anti-microbial properties helping to avoid spoilage of products.

1.4 Activated carbon:

Activated carbons with abundant micropores (0–2 nm width), mesopores (2–50 nm width) and macropores (> 50 nm width) together with a high specific surface area are widely used as versatile adsorbents for the processing of gaseous and liquid phases. Such activated carbons can be produced from a variety of raw materials. Of these, coal and agricultural wastes such as sawdust and coconut shell are the most commonly used precursors.

2. LITERATURE REVIEW

Huiping Zhang, Ying Yan and Lichun Yang have synthesized activated carbons from sawdust by compound activation with H_3PO_4 . The impacts of different parameters, for example, the impregnation proportion, initiation temperature and time on the nitrogen adsorption isotherms, particular surface regions and pore volumes of the activated carbons were estimated and dissected with a specific end goal to streamline the operational conditions. The nitrogen adsorption limit of the initiated carbons was altogether influenced by the impregnation proportion and the enactment temperature. The most vital

operational parameter in synthetic enactment with H_3PO_4 was observed to be the H_3PO_4 : sawdust impregnation proportion. The particular surface territory of the activated carbon arranged with a 1:1 H_3PO_4 : sawdust proportion and initiated at $500^\circ C$ for 90 min achieved the estimation of $1642 \text{ m}^2/\text{g}$. The test demonstrated that, under the trial conditions, an activated carbon containing all around created micropores and mesopores showing enhanced nitrogen adsorption properties can be obtained by means of a H_3PO_4 actuation process under the ideal task conditions, of 1.0– 1.5 H_3PO_4 : sawdust at $500^\circ C$ for 60– 90 min. [2]

Agata S. Liwak, Noel Diez, Ewa Miniach and Grazyna Gryglewicz have prepared Nitrogen-containing initiated carbons from chitosan, a generally accessible and reasonable bio polysaccharide, by a basic methodology and tried as anode material in super capacitors. The physical initiation of chitosan with CO_2 prompted carbons with a high nitrogen content (up to 5.4 wt. %) and direct surface regions ($1000\text{--}1100 \text{ m}^2 \text{ g}^{-1}$). The chitosan-based carbons with a comparative microporous structure were considered in this examination to assess the impact of the nitrogen substance and dispersion on their electrochemical execution. The N-containing activated carbons were tried in two- and three-anode super capacitors utilizing a watery electrolyte (1 M H_2SO_4), and they displayed prevalent surface capacitance and pseudo capacitance. The low oxygen content and the nearness of stable quaternary nitrogen enhanced the charge engendering on the chitosan-based carbons, which was affirmed by the high limit maintenance of 83 %. The chitosan-based carbons displayed amazing cyclic dependability and kept up 100 % of their capacitance after 5000 charge/release cycles at a present thickness of $1 \text{ \AA} \text{ g}^{-1}$. [3]

R. Narasimman and K. Prabhakaran have contemplated thermo-foaming of activated carbon (AC) powder scatterings in a fluid sucrose sap via carbonization to get carbon foams. The scatterings were described by thickness estimations and sedimentation contemplations. The AC particles adsorbed on the gas– fluid interface balanced out the gas bubbles that brought about foaming of the inadequately cross-connected sucrose polymer pitch having low thickness. The carbon foams have an interconnected cell structure. Cell estimate ($0.138\text{--}0.22 \text{ g/cc}$), thickness ($0.62\text{--}3 \text{ mm}$) and compressive quality ($0.42\text{--}3.4 \text{ MPa}$) of the carbon foams relies upon the AC powder to sucrose weight proportion. Consolidation of the AC powder in the sucrose pitch diminishes the carbonization shrinkage that empowers the readiness of substantial carbon foam bodies without twisting. The carbon foam arranged at an AC powder to sucrose weight proportion of 0.1

demonstrates the most astounding thickness and compressive quality and the least cell measure. [4]

Rajaram Narasimman, Sujith Vijayan, and Kuttan Prabhakaran synthesized carbon foams by the thermo-foaming of scatterings of activated carbon (AC) powder of different normal molecule sizes in a fluid sucrose pitch. The nice, and cozy conduction of the carbon foams ($0.036\text{--}0.049 \text{ W/m-K}$) was less abundant than that of the vitreous carbon foams of comparable densities. The foams exhibited fireproofing, and machinable attributes. [5]

K. Prabhakaran, P. K. Singh, N. M. Gokhale, S. C. Sharma demonstrated a novel procedure for preparation of low thickness carbon foams from sucrose. A sap arranged by fluid acidic sucrose when warmed in an open Teflon form at $120^\circ C$ experiences foaming and after that setting in to strong natural foam. The strong natural foam experiences carbonization in air by lack of hydration at $250^\circ C$ under isothermal conditions. Carbon foams consequently acquired were sintered at temperature in the range $600\text{--}1,400^\circ C$ and indicated good electrical conductivity. The carbon foams contain round cells of size in the range $450\text{--}850 \text{ \mu m}$ and the cells are interconnected through roundabout or oval shape windows of size in the range $80\text{--}300 \text{ \mu m}$. The carbon foam tests sintered at $1,400^\circ C$ indicated compressive strength of 0.89 MPa . [6]

3. PRESENT WORK

3.1 Material and Methodology –

Raw materials and Chemicals: Sawdust, KOH pellets, Sulfuric acid, chitosan, sucrose, activated carbon, Na_2CO_3 , Distilled Water, concentrated nitric acid.

3.2 Process Description:

The present work employed two methods of carbon foam preparation; one using activated carbon & sucrose resin and the other method using sucrose, chitosan gel & activated carbon. The present work is conducted into following 3 parts:

3.2.1. Preparation of Activated carbon from saw dust:

The sawdust was collected from a local furniture store. It was washed with hot distilled water to remove dust like impurities and dried in the oven for 2 hours. The material was finally sieved to discrete sizes. The raw material was then carbonized at $300^\circ C$ under inert atmosphere for 30 min approx. A certain amount of carbonized saw dust was soaked with potassium hydroxide (KOH) at impregnation ratio of 1:1. The mixture was then mechanically agitated for

1 h & kept overnight followed by neutralization & filtration. The retained mass was dehydrated in an oven at 100⁰C for 1 hr. & pyrolysed in a muffle furnace at final temperature of 250⁰C for 30 min. The activated carbon (AC) was cooled to room temperature using decanter.

The block diagram and photographic representation of process steps are given in fig. 1 & 2.

3.2.2. Synthesis of carbon foam using sucrose resin:

- An aqueous solution containing 100 g sucrose in 110 ml water is acidified with concentrated nitric acid.

- The solution was heated to form a dark honey-like viscous resin. The final volume of the resin was 130 ml.

- 15g AC powder prepared from saw dust was added to the sucrose resin and solution containing 1g Na₂CO₃ in 5 ml distilled water was thoroughly mixed with it with constant stirring for 1.5 hr.

- The dispersions were transferred to tray and heated in an air oven at 110⁰C for 48 hr. for foaming and setting.

- The sucrose resin with AC composite is thus obtained. It was cut into proper size and dried in air oven at 100⁰C to obtain solid foam which is porous carbon.

- The porous carbon was carbonized at 400 °C for 1 hr. in a muffle furnace.

- The as-made porous material was carbon foam.

The block diagram and photographic representation of process steps are given in fig 3 & 4.

3.2.3. Synthesis of carbon foam using sucrose and chitosan gel:

- A solution of 10 g Na₂CO₃ and 50 ml distilled water was thoroughly mixed with 10 g chitosan.

- In this chitosan gel, 10 g sucrose was added which is acidified with concentrated nitric acid.

- The solution was heated to form a dark yellowish viscous resin.

- 10g AC powder prepared from saw dust was added to the viscous resin.

- Mixing of the solution with AC powder is done by using a mechanical stirrer for 2 hr.

- The dispersion was transferred to tray and heated in an air oven at 110⁰C for 24 hr. for foaming and setting.

- The chitosan gel with AC composite thus obtained was cut into proper size and dried in air oven at 100⁰C to get solid foam which is porous carbon.

- The porous carbon was carbonized at 450 °C for 1 hr. in a muffle furnace.

- The as-made porous material was carbon foam.

The block diagram and photographic representation of process steps are given in fig. 5 & 6.

4. RESULT AND DISCUSSION

4.1 Characterization of activated carbon derived from saw dust:

The effectiveness of adsorbent synthesized was studied by performing experiments. For this purpose, known quantity (5g) of activated carbon each was put into 25ml samples of 10ppm, 20ppm & 30ppm methylene blue in water solution. After contacting for 48hrs the solution obtained was subjected to UV spectroscopy for determination of methylene blue remained in the solution after adsorption. Table 1 gives the details of initial & final concentrations of these samples. The % adsorption achieved ranged between 60 to 82%.

The percentage adsorption was calculated by using the formula given below:

$$\% \text{ Adsorption} = [(C_0 - C) / C_0] * 100$$

C₀ = initial concentration of methylene blue in the solution

C = final concentration of methylene blue remained in the solution after adsorption.

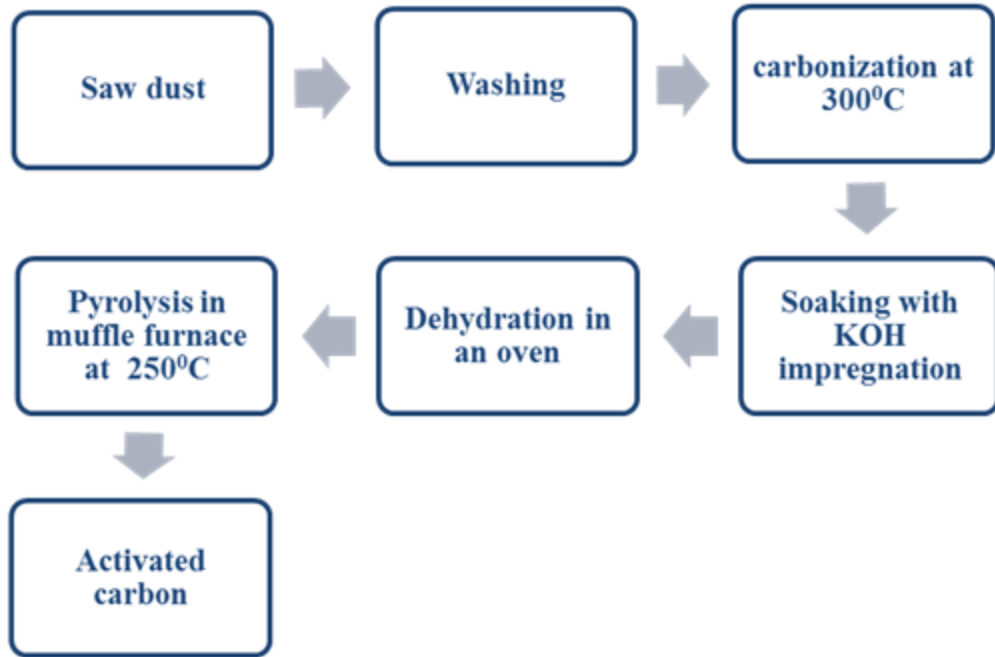


Figure 1: Process flowchart for preparation of Activated carbon from saw dust

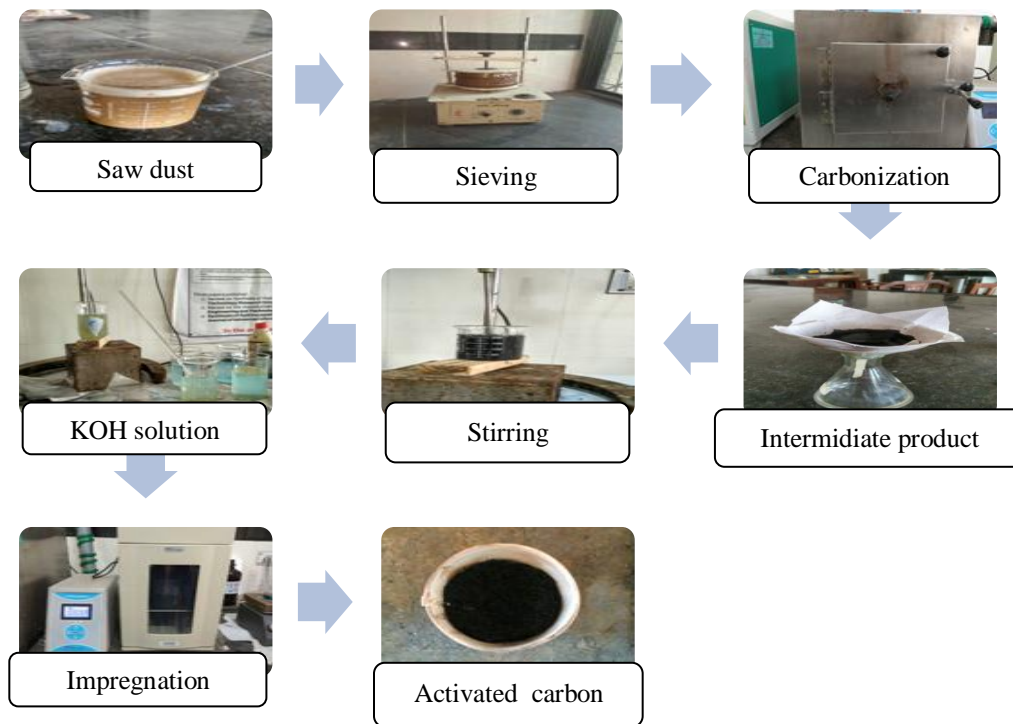


Figure 2: Photographic representation of preparation of Activated carbon from saw dust

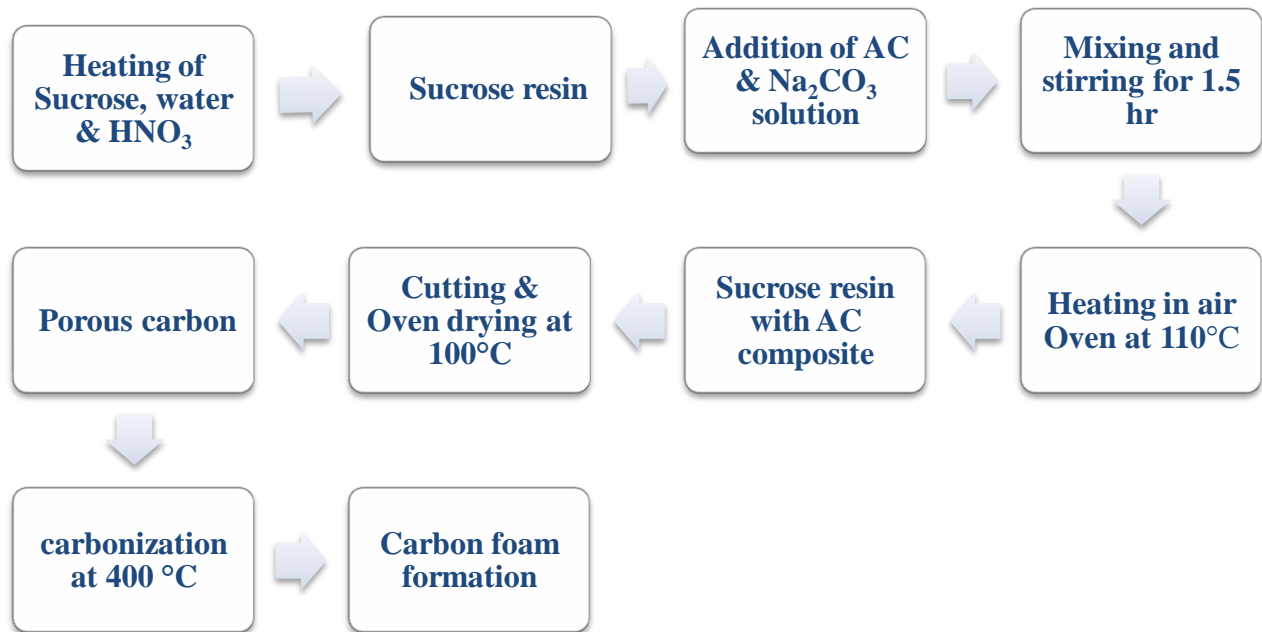


Figure 3: Process flow chart for synthesis of carbon foam using sucrose resin & activated carbon

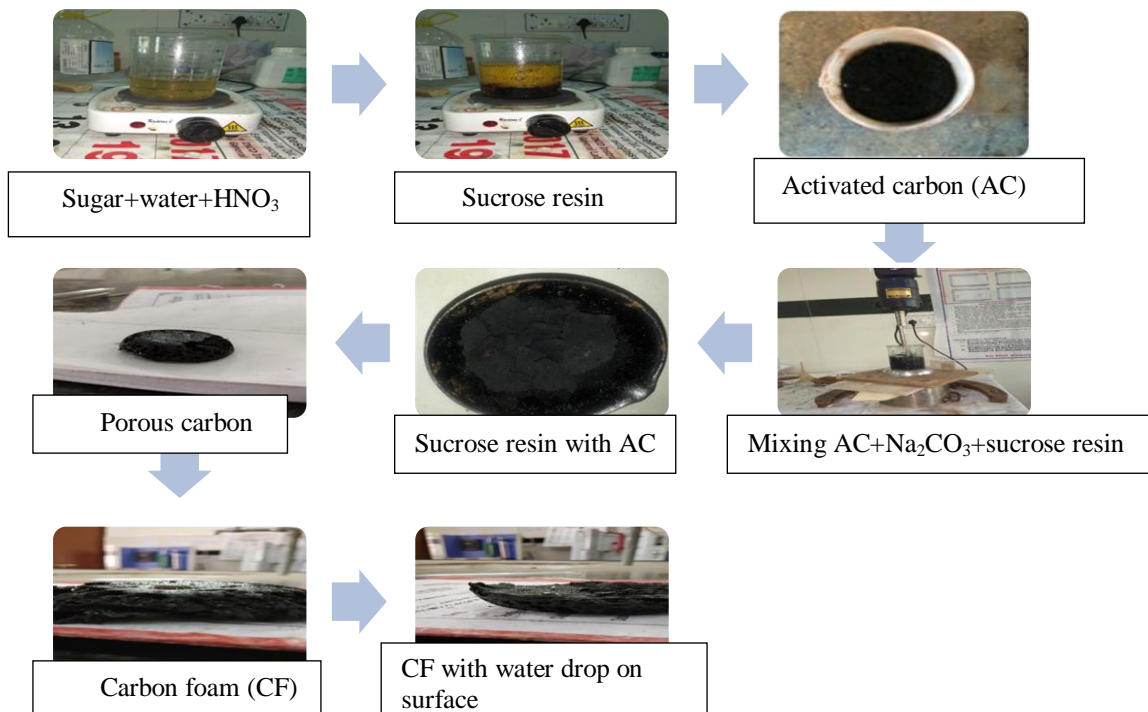


Figure 4: Photographic representation of synthesis of Carbon foam by sucrose resin & activated carbon

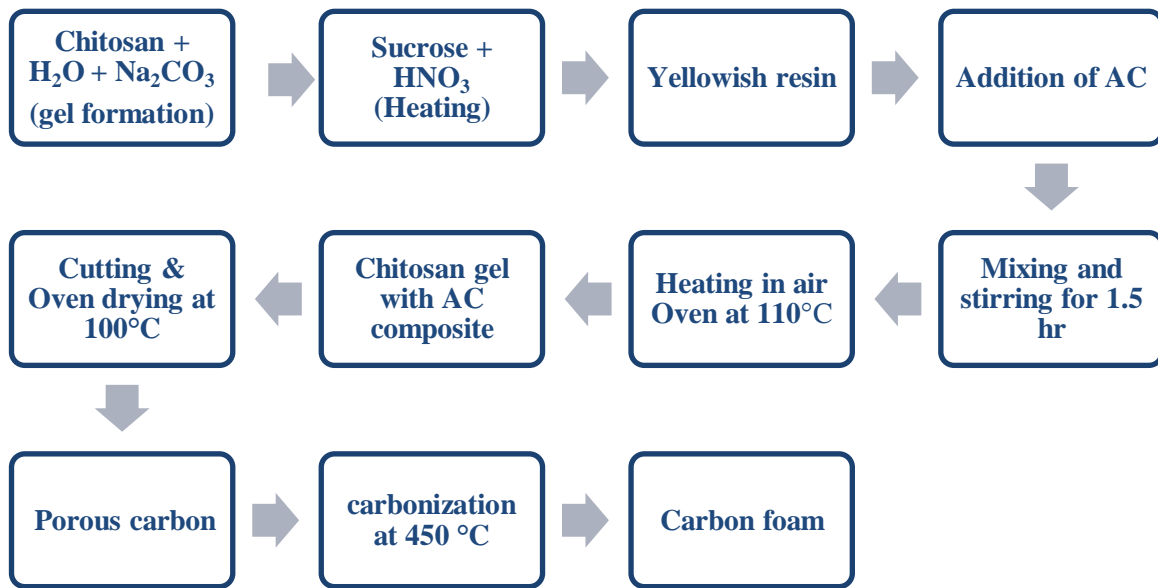


Figure 5: Process flow chart for synthesis of carbon foam using chitosan gel, sucrose & activated carbon

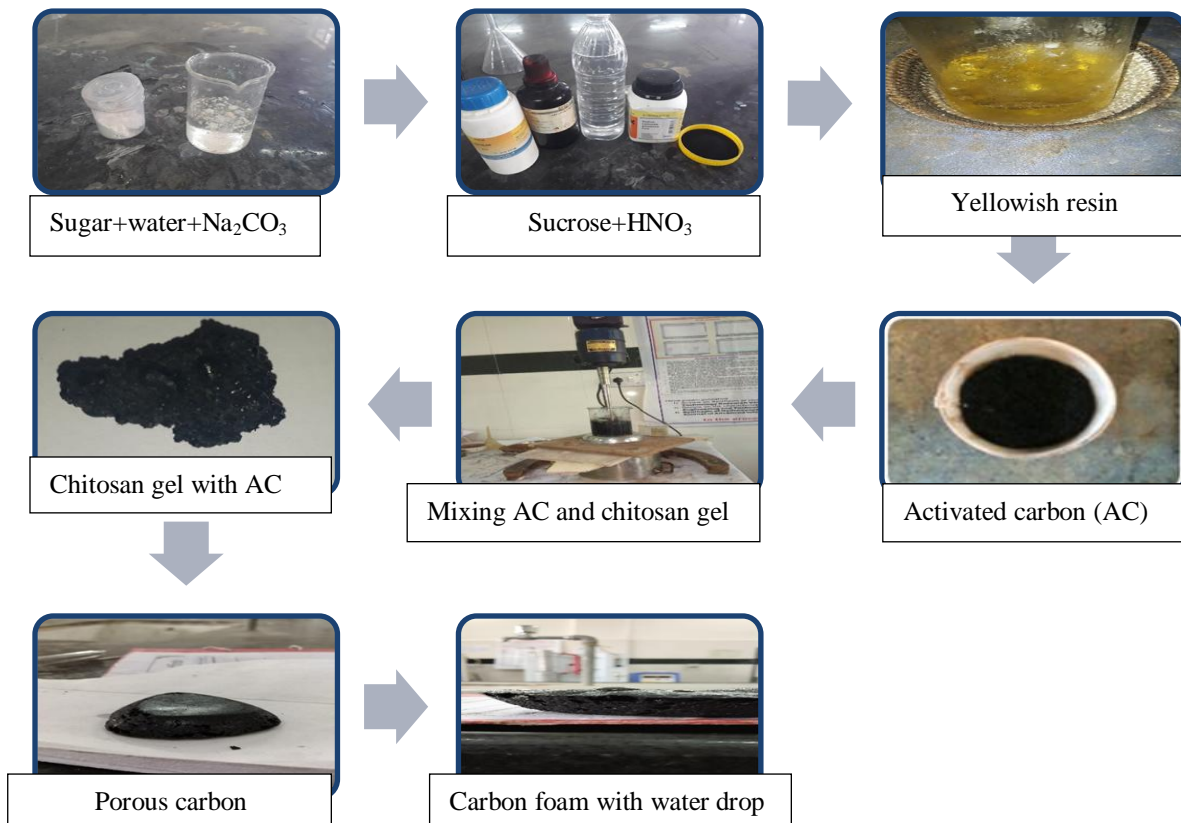


Figure 6: Photographic representation of synthesis of Carbon foam by chitosan gel, sucrose & activated carbon.

Sr. No.	Initial Concentration (ppm)	Final Concentration (ppm)	%Adsorption
1.	30	5.3963	82.1033
2.	20	5.4688	72.65
3.	10	4.55	60

Table 1: Adsorption study of methylene blue using porous carbon

4.2 Characterization of carbon foam and porous carbon from sucrose resin, chitosan gel and activated carbon from saw dust:

4.2.1 FTIR analysis:-

4.2.1.1. Sample C1- carbon foam by sucrose resin, Chitosan gel and activated carbon:

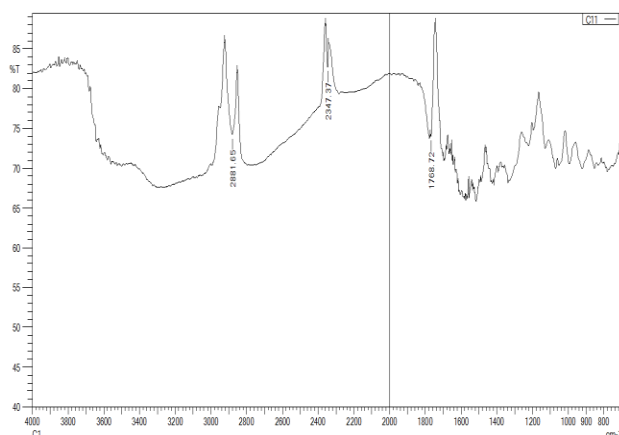


Figure 7: C1- FTIR analysis of carbon foam by sucrose resin, Chitosan gel and activated carbon

The detailed interpretation of FTIR spectrogram of sample C1 is tabulated in table 2 & depicted in fig 7, shows that strong stretch band is obtained at the wavelength range of 1700-1725 cm^{-1} . Extensively broad OH stretching is obtained at the wavelength lying between 2500-3300 cm^{-1} . However, for the wavelength falling between 1400-1600 cm^{-1} , medium weak multiple bands are obtained. Furthermore, two bands of C=O stretching is obtained at wavelengths belonging to the range of 1740-1775 cm^{-1} ; at 1420 bonding is C-H; at the wavelength of 1320 cm^{-1} , C-O strong stretch is obtained; at 1000-1300 cm^{-1} C-O, two stretch or more bands are seen and within the

wavelength range of 2850-3000, strong C-H bonding is obtained.

Sr. No	Peaks value(cm^{-1})	Functional group	Description
1	2850-3000	C-H	Stretch & strong
	3500-3700	O-H	Stretch, strong & sharp
2	1400-1600	-C=C-	Stretch, medium, weak & multiple bands
3	1735-1750	C=O	Stretch & Strong, two bands
4	675-1000	=C-H	Bending & Strong
5	1000-1300	C-O	Strong & stretch
	1350-1480	-C-H	Variable bending
6	1800-1830 & 1740-1775	C=O	Stretch, two bands
7	2250-2300	-C=C-	variable, not present in symmetrical alkynes

Table 2: Interpretation of spectrogram of C1

It can be observed that the wave numbers fairly agree with the values reported in literature, which supports the presence of sucrose and chitosan resin in the carbon foam. The stretching patterns indicate the presence of compounds like sucrose, activated carbon, chitosan and sodium bicarbonate in the prepared carbon foam.

4.2.1.2. Sample C2 - Carbon foam by sucrose resin and activated carbon:

The detailed interpretation of FTIR spectrogram of sample C2 is tabulated in table 3 & depicted in fig 7 depicted in fig 8, shows that at the wavelength lying between 3850-3590 cm^{-1} strong sharp OH stretching is obtained; for 3560 cm^{-1} and 3540 cm^{-1} strong and broad O-H (alcohol) group is detected ; for the wavelength falling within 1700-1725 cm^{-1} , strong stretch band is obtained ; for the wavelength between 1400-1600 cm^{-1} medium, weak multiple bands are obtained ; for 1420 bonding is C-H; for the wavelength of 1320 cm^{-1} , C-O strong stretch is obtained; for 1000-1300 cm^{-1} , two stretch or more bands are seen; for the wavelength 675-1000 cm^{-1} , strong C-H bonding is obtained. It can be observed that the wave numbers fairly agree with the values

reported in literature, substantiating the presence of sucrose in the carbon foam. The stretching patterns indicate the presence of compounds like sucrose, activated carbon, sodium bicarbonate in the prepared carbon foam. [7]

Sr. No	Peaks value(cm^{-1})	Functional group	Description
1	2850-3000	C-H	Stretch & strong
	3500-3700	O-H	Stretch, strong & sharp
2	1400-1600	-C=C-	Stretch, medium, weak & multiple bands
3	1735-1750	C=O	Stretch & Strong, two bands
4	675-1000	=C-H	Bending & Strong
5	1000-1300	C-O	Strong & stretch
	1350-1480	-C-H	Variable bending
6	1800-1830 & 1740-1775	C=O	Stretch, two bands

Table 3: Interpretation of spectrogram of C2

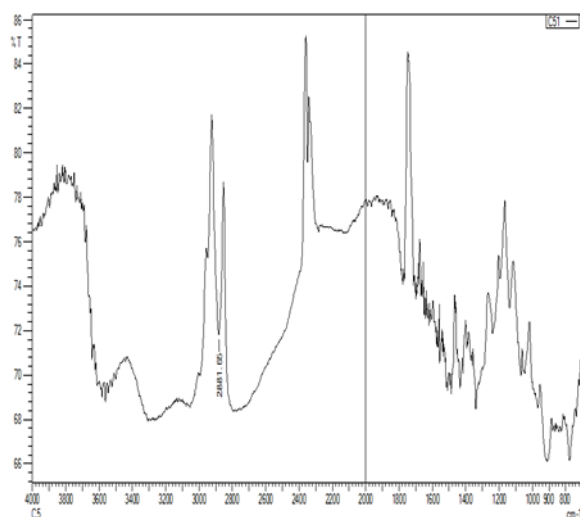


Figure 8: C2- FTIR analysis of carbon foam by sucrose resin and activated carbon

4.2.2 SEM analysis

Figures 9 & 10 show typical photographs of the studies of surface morphology of carbon foam synthesized from sucrose resin, chitosan gel and activated carbon with Na_2CO_3 as foaming agent respectively using SEM. As can be seen from fig 9& 10, the surface clearly shows several small structures of the size of few microns in width of 2-10 of microns in length. This may be sucrose resin, chitosan film and activated carbon deposited. It also shows the changed morphology of the same surface after carbon foam formation. It is more homogenized & is indicative of uniform material that could be carbon. [8]

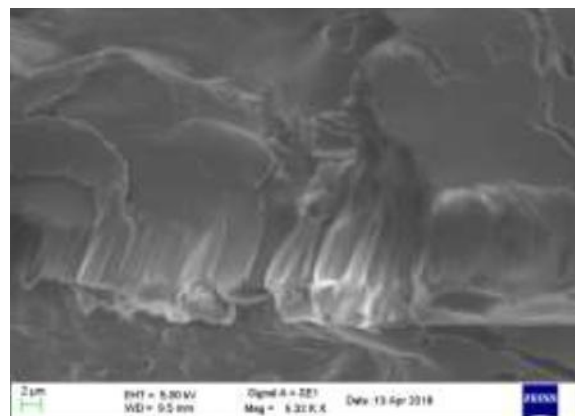


Figure 9: SEM image of carbon foam by sucrose resin, chitosan gel and activated carbon

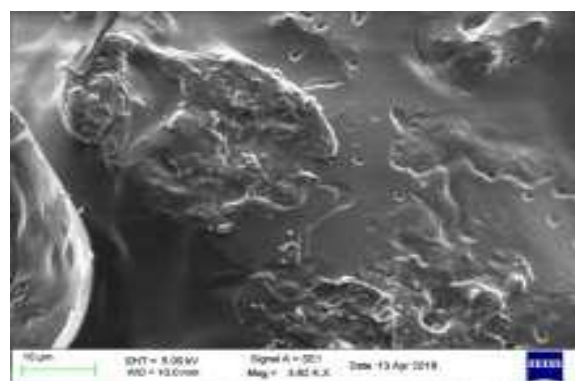


Figure 10: SEM image of carbon foam by sucrose resin and activated carbon

5. Conclusion

The present work addresses to the development of carbon foam using sucrose, chitosan & activated 60% to 82% of methylene blue from its solution. Sucrose resin is made & carbon foam was obtained by carbonising sucrose resin, chitosan gel & activated carbon. The synthesised carbon foam is light in weight & analytical tests such as FTIR & SEM have supported the presence of $-C=C-$ & $-C-OH$ groups & extended surface morphology.

ACKNOWLEDGMENT



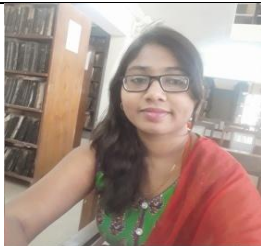

- Authors are thankful to Director of L.I.T. Nagpur for facilities & encouragement provided throughout this work.
- The Authors are thankful to HOD, Physics & Chemistry Dept. of RTMNU and H.O.D. Chemical Engineering department, VNIT, Nagpur for FTIR and SEM analysis support.

REFERENCES

- [1] Q. Lina, L. Xionga, Q. Lüa and C. Fangb, "Preparation and characterization of carbon foams from cyanate ester mixture," *Journal of Analytical and Applied Pyrolysis*, pp. 1-6, 2015.
- [2] H. Zhang¹, Y. Yan and L. Yang, "Preparation of Activated Carbons from Sawdust by Chemical Activation," *H. Zhang et al./Adsorption Science & Technology*, vol. 26, no. 7, pp. 533-543, 19 December 2008.
- [3] A. Sliwak, N. Diez, E. Miniach and G. Gryglewicz, "Nitrogen-containing chitosan-based carbon as an electrode material for high-performance supercapacitors," *J Appl Electrochem*, vol. 46, p. 667-677, 2016.
- [4] R. Narasimman and K. Prabhakaran, "Preparation of carbon foams by thermo-foaming of activated carbon powder dispersions in an aqueous sucrose resin," *Carbon 50*, p. 5583-5593, 2012.
- [5] R. Narasimman, S. Vijayan and K. Prabhakaran, "Effect of activated carbon particle size on the thermo-foaming of aqueous sucrose resin and properties of the carbon foams," *Materials Research Society*, vol. 30, no. 1, pp. 46-55, 2015.
- [6] K. Prabhakaran, P. K. Singh, N. M. Gokhale and S. C. Sharma, "Processing of sucrose to low density carbon foams," *J Mater Sci*, vol. 42, p. 3894-3900, 2007.
- [7] J. Coates, "Interpretation of Infrared Spectra, A Practical Approach," *Encyclopedia of Analytical Chemistry* 12,10815-10837,2000pp. 1-23.
- [8] M. Puchalski, P. Dabrowski, W. Olejniczak, P. Krukowski, P.Kowalczyk and K. Polanski, "The

carbon. Experimental runs have been conducted for synthesis of activated carbon from sawdust. Adsorption studies indicated the effective removal of

study of silver nanoparticles by scanning electron microscopy, energy dispersive X-ray analysis and scanning tunnelling microscopy," *Materials Science-Poland*, vol. 25, no. 2, pp. 473-478, 2007.

	<p>Shekhar L. Pandharipande is an Associate Professor in Chemical Engineering Department of Laxminarayan Institute of Technology, Rashtrasant Tukadoji Maharaj University, Nagpur. He has co-authored three books titled 'Process Calculations', 'Principles of Distillation' & 'Artificial Neural Network'. He has two copyrights 'elite-ANN' & 'elite-GA' to his credit and two patents are filed as coworker and has more than 60 papers published in journals of repute.</p>
	<p>Dr. Rajendra P. Ugwekar is an associate professor & HOD in chemical engineering department of RTMNU, Nagpur. He is author of two edited books & has 8 papers published in journals of repute.</p>
	<p>Akshata Ramteke has completed M.Tech. (CE) from Laxminarayan Institute of Technology, Nagpur & pursuing PhD from IIT delhi.</p>
	<p>Shivank Sharma is pursuing 7th semester B. Tech. (Chemical Engineering) from Laxminarayan Institute of Technology, Nagpur.</p>