Journal of Advances and Scholarly Researches in Allied Education
Vol. XII, Issue No. 23, October-2016, ISSN 2230-7540

Synthesis & Characterization of Caron Nanomaterials from Natural Sorbents

Indrajit N. Yadav*

Dept. of Chemical Engineering, Bharati Vidyapeeth College of Engineering, Navi Mumbai, (MS) India

Abstract – The methods of carbon nanomaterial manufacture prevalent in the society at large, which mainly emphasized on usage of precursors based on fossil fuel or other carbon rich fuels. This motivates to search for an abundant and accessible material that furnishes the same needs. This brings to the basic raw material or precursor utilized i.e. oil seeds and plant fibers. The objective of this work is to analyze various types of oil seeds (e.g., sunflower, neem etc.), and plant based fibers (like palas, wheat husk, etc.) to synthesize carbon nanomaterials. Since these materials are solid, pyrolysis could be done without the use of any catalyst. If we succeed in this exploration, then production of carbon nanomaterial could be easy and economical. Keeping these ideas in mind selection of plant fibers and various oil seeds were made. These materials were pyrolysed at suitable temperature hence Morphology, when analyzed on a microscopic level, gives sharp grain like features which do not collapse in situ.

Keywords: Carbon Nanomaterial, Microwave Technology, Plant Fibers, Oil

- X -

1. INTRODUCTION

The enclosing and burying of hazardous waste is becoming increasingly unacceptable due to growing concern about the effect of pollutants on the environment and more restrictive environmental regulations. Efficient regeneration systems are required to permit a wider application of carbon adsorption processes and to ensure their economic feasibility. This has motivated companies to develop methods for regenerating and reusing saturated activated carbon. Over the years, a wide variety of regeneration techniques have been suggested and applied. These are based either on desorption induced by increasing the temperature or by displacement with solvents, or on decomposition induced by thermal, chemical, catalytic or microbiological processes. By far the most extensively used technique is thermal regeneration under steam or an inert atmosphere. Unfortunately, the regeneration process is time consuming and after successive heating and cooling cycles, the carbon becomes damaged.

The application of microwave heating technology for regenerating industrial waste activated carbon has been investigated, with very promising results. The main difference between microwave devices and conventional heating systems is in the way the heat is generated. Thermal regeneration is conventionally performed in rotary kilns or vertical furnaces, where the carbon bed is indirectly heated by conduction and convection. In the microwave device, the microwaves supply energy directly to the carbon bed. Energy transfer is not by conduction or convection as in conventional heating, but is readily transformed into heat inside the particles by dipole rotation and ionic conduction. When high frequency voltages are applied to a material, the response of the molecules with a permanent dipole moment or induced dipole to the applied potential field is to change their orientation in the direction opposite to that of the applied field. The synchronized agitation of molecules then generates heat. Microwave regeneration offers possible advantages over conventional treatment. These include the rapid and precise control of the carbon bed temperature, a more compact furnace, and energy savings. Some authors have also found that microwave regeneration gives rise to a better performance of the activated carbons in terms of ulterior adsorption capacity and rate of adsorption compared to conventional heating. Consequently, processes based on microwave energy are gaining importance in several fields of application on an industrial scale, and microwave technology is now considered a promising available technology for the regeneration of carbon materials. The aim of this work was to acquire a deeper insight into the thermal regeneration of activated carbons. To this end a comparative study of the regeneration of activated carbons by conventional thermal methods and single mode microwave energy was performed. Changes in the porous texture of the regenerated carbons resulting from the thermal treatment in both devices were studied in relation to the changes in the yield capacity of the adsorbents and surface area.

2. EXPERIMENTAL

Carbon replication was performed by the impregnation of biomass as the carbon precursor like sunflower seeds, neem seeds, wheat husk, palas leaves. For comparison, the carbonization was carried out by two kinds of heating methods, viz. the conventional thermal heating and the microwave assisted heating. The configuration of the pilot-scale experimental setup is shown in the Fig. 1. it consist of simple procedure in which the chosen precursor was weighed by microbalance to the required quantity, the required quantity was subjected to predetermined pyrolysis conditions in either microwave or muffle furnace, hence pyrolysed precursor gave us our activated carbon nanomaterial, which was weighed again for surface area and yield calculation.

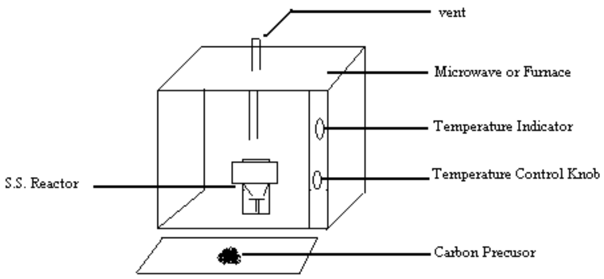


Fig 1. An experimental setup for synthesis of Nanomaterial

Sr. No	Sorbents	Equipment	Pyrolysis Condition		
			Weight of substance (gram)	Time (min.)	Temperature (^{0}C)
1	Neem Seed	Microwave	2	10	400
		Furnace	2	300	400
2	Sunflower Seed	Microwave	2	10	400
		Furnace	2	300	400
3	Palas leaves	Microwave	2	10	400
		Furnace	2	300	400
4	Wheat husk	Microwave	2	10	400
		Furnace	2	300	400

3. RESULT AND DISCUSSION

3.1 Surface Area Measurement

Methylene blue method was used for surface area calculation, which involves following accounting to accomplish surface area measurement, five Standard solutions of Methylene blue were made in concentration range 10 to 100 ppm having same volume. All solutions were made in distilled water. Methylene blue spectrum was obtained with spectrophotometer to find out its λ_{max}, found to be 670 nm; hence a standard calibration graph was plotted between optical density and concentration of Methylene blue. Henceforth, 100 ml variable range (10 to 100ppm) Methylene blue solution + 15 milligrams of pyrolysed Precursor (Kept for 24 hours undisturbed in close Test tube to avoid evaporation) then Ultraviolet Spectrphotometry carried 5 such samples and hence 5 closed test tubes obtained for spectrophotometric analysis. With the help of standard calibration graph, the corresponding value of total concentration of Methylene blue left in the solution was found out. The difference of concentration of Methylene blue was taken as the quantity of Methylene blue adsorbed by 15 mg of carbon material. From these results, surface area of carbon material was calculated as:

Where,

S is the specific surface area (m^2/g) of carbon material,

Ng is the number of molecules of Methylene blue adsorbed by CNM,

A is the surface area of one molecule of Methylene blue (197.2 $^{\circ}A^2$)[1],

N is Avogadro number ($6.02 \times 10-23\ mol^{-1}$) and

M is the molecular weight of Methylene blue (373.9 g)[1]

Surface Area $S = Ng \times A \times N \times 10^{-20} / M$

In case of sunflower the value of total concentration of Methylene blue left in the solution was 103.597ppm. The initial concentration of Methylene blue was 2000ppm. The difference attributes to the quantity of Methylene blue adsorbed by 15 mg of sunflower seeds based carbon nanomaterial.

Using the given formula;

$Ng = (2000 - 103.597) X 10^{-6}$ gram $= 1.896 X 10^{-3}$ gram

The molecular weight of Methylene blue is 373.9 gram,

Moles of Methylene blue $= 1.896 X 10^{-3}/373.9 = 5.071 X 10^{-6}$ moles

1 mole of a pure compound corresponds to $6.023 X 10^{23}$ molecules,

Thus $5.071 X 10^{-6}$ moles correspond to, $5.071 X 10^{-6} X 6.023 X 10^{23} = 3.05 X 10^{18}$ molecules

Hence, for calculating surface area we revert to the equation given back,

$S = 3.05 X 10^{18} X 197.2 X 6.023 X 10^{23} / 373.9 X 10^{20}$

$S = 96.9\ m^2$/gram

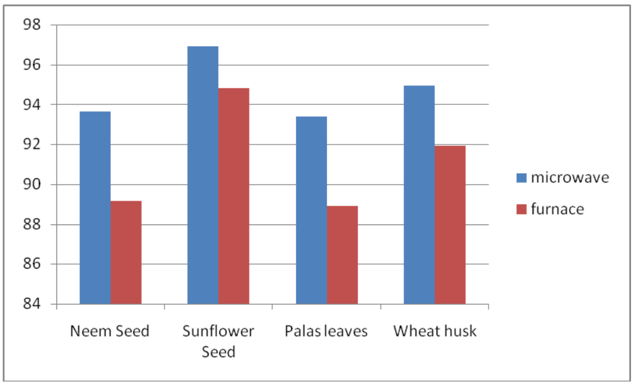


Fig 2. Comparison of Surface area of sorbents (m^2/gm)

3.2 Percentage Yield Measurement

The % yield calculated for sorbent material based on following formula;

(Initial quantity of precursor- finally obtained quantity of pyrolysed precursor)

$$X \frac{100}{\text{Initial quantity of precursor}}$$

Hence, the comparative results of microwave and furnace materials which clearly give better performance of microwave irradiated material which is shown in figure 3

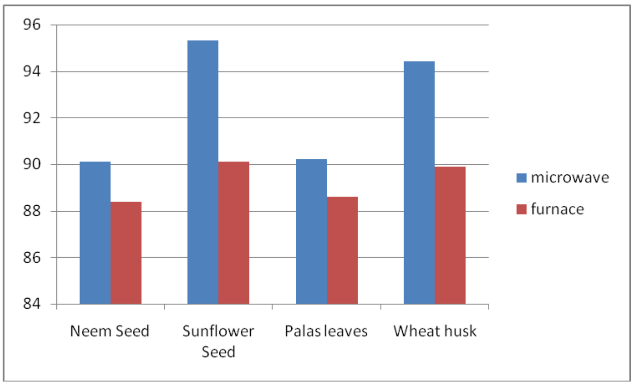
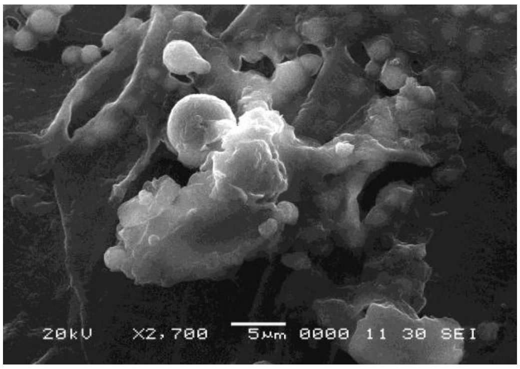


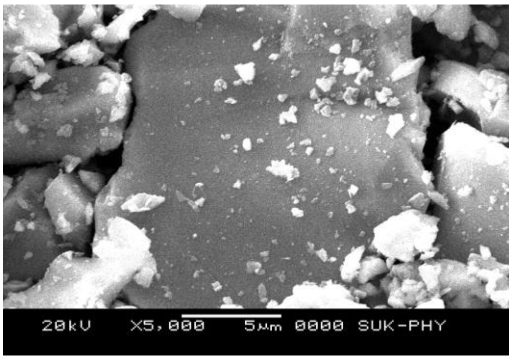
Fig 3. Comparison of % yield of sorbents

3.3 SEM Characterization

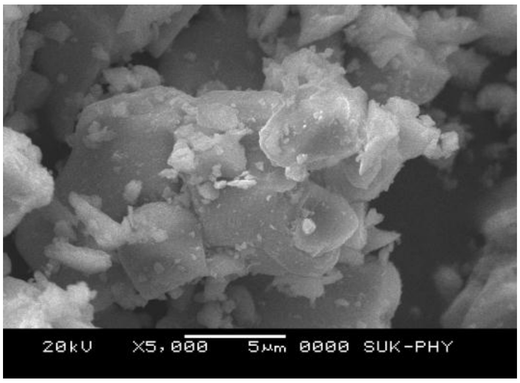
The SEM images of the microwave material shown in Fig. 4,5 clearly demonstrate that the material exhibits uniform particle morphologies as well as a highly ordered mesostructure, even though it is obtained using microwave irradiation for only 10 min at 400 ^{0}C. The SEM image shows the overall particle morphology of the microwave material to be very similar to that of the mesoporous. Compared with the approximately 5 h required for obtaining the furnace materials, it takes only 10 min for the formation of the microwave materials, respectively. Even though the carbonization period under microwave irradiation is much shorter than that of the conventional thermal process, the surface areas of the microwave materials are comparable or better than those of the furnace materials. More interestingly, the preparation of microwave material is possible even at extremely low temperatures of around 400^{0}c under microwave irradiation, whereas the conventional heating does not develop any ordered mesostructure at the same temperature. After the carbonization, the colors of the furnace materials and microwave thus obtained are obviously different: the former is dark brown and the latter is black, indicating that the carbon precursor for the microwave material is relatively more carbonized by the microwave irradiation than the furnace material is by the conventional thermal treatment. Therefore, the composite material, containing a kind of carbon intermediate which may be active to microwaves, seems to be self-heated to a higher temperature than the detectable temperature during the process. It should be mentioned here that due to the design of the microwave device, the thermocouple measures the temperature of the atmosphere surrounding the sample, but not the sample's temperature directly.



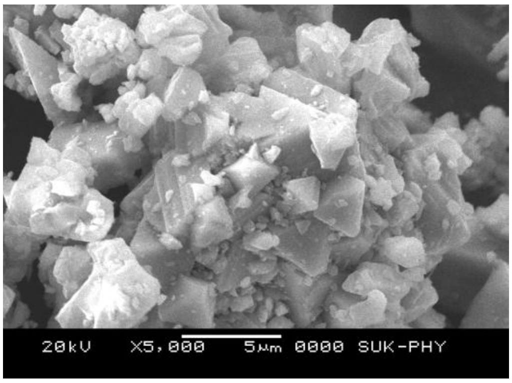
(a)



(b)

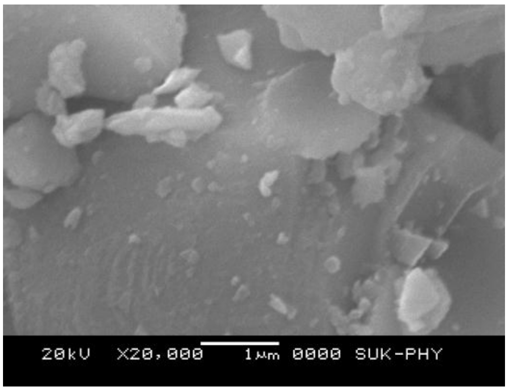


(c)

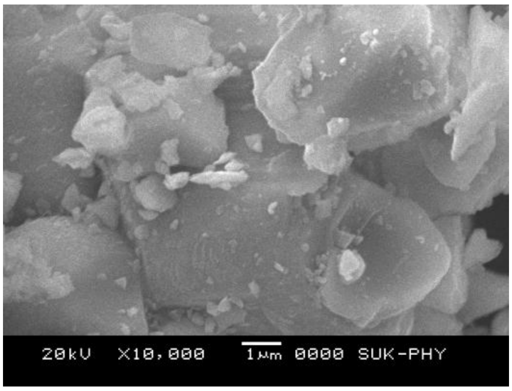


(d)

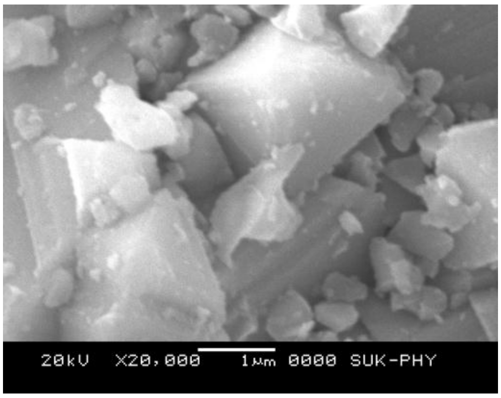
Fig 4. SEM images of Microwave Material of a) Neem Seed b) Sunflower Seed c) Palas leaves d) Wheat husk



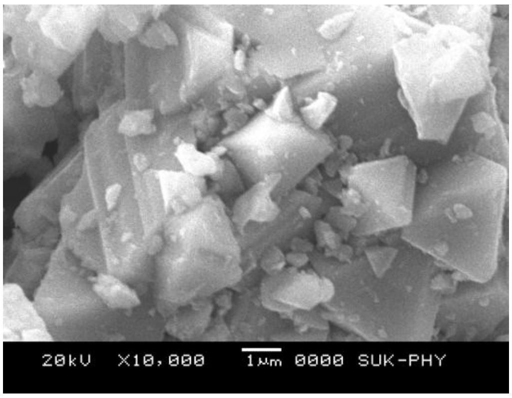
(a)



(b)



(c)



(d)

Fig 5. SEM images of Furnace Material of a) Neem Seed b) Sunflower Seed c) Palas leaves d) Wheat husk

4. CONCLUSION

The experimental findings reveal that the sunflower seeds gave the highest surface area per unit weight of adsorbent (96.9m^2/gm), while the highest yield (90.10%) obtained by instant carbonization within 10 min using the energy efficient microwave assisted carbonization process as determined by the Methylene Blue method. Thus as far as capacity for hydrogen storage is concerned, sunflower seeds are the best available option due to high specific surface area, while, the most economical to use also. Although the above analysis was a great simplification of what may be actual conditions encountered, but to an extent lay the footstone for the further research on usage of plant fibers and oilseeds as potent precursors for nanomaterial preparation and their usage in hydrogen storage, should have great potential for use in applications such as selective adsorbents, catalyst supports and so on.

REFERENCES

C.O. Ania et. al., (2005). Effect of microwave and conventional regeneration on the microporous and mesoporous network and on the adsorptive capacity of activated carbons, microporous and mesoporous, *Materials*, vol. 85, pp. 7–15.

Hyung Ik Lee et. al., (2007). Ultrafast production of ordered mesoporous carbons via microwave irradiation, *Carbon*, vol. 45, pp. 2843–2854.

Iva´n Cabria et. al., (2007). The optimum average nanopore size for hydrogen storage in carbon nanoporous materials, *Carbon*, Vol. 45, pp. 2649–2658.

L.L. Vasiliev et. al., (2007). New sorbent materials for the hydrogen storage and transportation, *International Journal of Hydrogen Energy*, Vol.32, pp. 5015 – 5025.

Maheshwar Sharon et. al., (2007). Hydrogen storage by carbon materials synthesized from oil seeds and fibrous plant materials, *International journal of hydrogen energy,* Vol. 32, pp. 4238 – 4249.

Nor Hasridah Abu Hassan et. al., (2007). Study of hydrogen storage by carbonaceous material at room temperature, *Diamond & Related Materials*, Vol. 16, pp. 1517–1523.

P. Ndungu et. al., (2008). Carbon nanomaterials synthesized using liquid petroleum gas: Analysis toward applications in hydrogen storage and production, *International journal of hydrogen energy*doi:10.1016/j.ijhydene.2008.02.007

Velmurugan P. et. al., (2011). Dye removal from aqueous solution using low cost adsorbent, *International Journal Of Environmental Sciences*, vol. 1,pp. 7.

Corresponding Author

Indrajit N. Yadav*

Dept. of Chemical Engineering, Bharati Vidyapeeth College of Engineering, Navi Mumbai, (MS) India

E-Mail – indrajityadavpatil@gmail.com