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Steam Activated Carbon Material from the Fruit Shells of *Sterculia foetida* for Energy and Environmental Applications

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Abstract

Steam activation of the biochar from *Sterculia foetida* forms a green route for the synthesis of activated carbon material with high specific surface area (1017 m^2/g). The corresponding value with a chemical activating agent (K_2CO_3) is much lower (596 m^2/g). Structural parameters of the activated carbon materials were deduced from XRD studies. The thermal stability of the activated carbon materials from steam activation is nearly 100°C higher than that of the material derived from K_2CO_3 activation. The oxygen enrichment of the resulting carbon material from steam activation is on par with that derived from K_2CO_3 activation as deduced from the FT-IR analysis. Thus a comparative study of the physical (steam) and chemical (K_2CO_3) activation with the specific example of fruit shells of *Sterculia foetida* as feedstock reveal that steam activation is a green strategy that could be a substitute to chemical methods of activation. The high specific surface area microporous carbon from the fruit shells of *Sterculia foetida* find applications for adsorption of dyes, toxic chemicals, metal impurities apart from being used as electrode materials for electric double layer super capacitors (EDLCs). Future directions in the use of the carbon material developed include applications such as the sonochemical deposition of the carbon material on wearable masks for protection against chemical and biological warfare, as support for noble and non-noble metal electrocatalysts for anode (methanol or ethanol or bioethanol electro-oxidation) and cathode (oxygen reduction), and as adsorbent for hazardous contaminants like Hg (II), As (V) and Cr (VI). Moreover, it is surmised that the rich spin density of the material will facilitate the improvement of the electrochemical device (batteries, super capacitors, fuel cells, and mitochondria mimics) performance via enhancement in the electrical conductivity.

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INTRODUCTION

During the world-war I (1914-1918) and world-war II (1939-1945) precautionary measures and break through scientific innovation saved many from perishing. Masks based on the porous carbon materials produced from biomass by the Polish Chemist Rafal von Ostrejko saved many human lives from chemical war-fare [1–3]. As we are in the war time, such innovation, and break-through in materials for safety is needed. Activated carbon materials act as molecular sieves with life-saving applications. They act as adsorbents for toxic materials. The application of activated carbon materials for environmental remediation, energy related applications and materials for personal safety from chemical, biochemical and nuclear warfare is proven. As a result there is a growing need for exploring sustainable bio-based feedstock as well as green synthetic strategies for the production of activated carbon materials.

Ever since Professor B Viswanathan's group reported for the first time on the synthesis and characterization of activated carbon material from natural sources (*Sterculia foetida*) there has been a renewed interest on this material [4, 5]. The carbon material from the fruit shells of *Sterculia foetida* has been exploited for energy and environmental applications [6–12]. Non-edible oil derived from the seeds of *Sterculia foetida was* used for biodiesel production [13–18]. The tree, leaves and the fruit shells of *Sterculia foetida*, from the IIT Madras, India, campus gifted with rich diversity of flora and fauna, were shown in Figure 1. Like, the famous, *Sterculia foetida*, there are several plant species in the IIT Madras campus that were under-utilized for the production of materials. Ramesh L. Gardas' group is actively working in that direction [11].

Figure 1. The tree, leaves and fruit shells of *Sterculia foetida* in the campus of Indian Institute of Technology Madras.

In general, highly porous carbonaceous materials, are referred to as activated carbon materials One of the most successful and commercial applications of activated carbon materials include the gas separation (oxygen and nitrogen) for producing high pure oxygen. In such applications the microporous carbon materials act as molecular sieves. Interestingly as the pore dimensions of these activated carbon materials are more or less close to the magnitude of the adsorbate molecules, they exhibit high adsorption capacity. The ability of the carbon molecular sieves to effect the separation of gas mixtures depend on the ability of the molecule to diffuse into the micropore volumes. Apart from molecular sieving, activated carbon materials find diverse applications like the treatment of potable and effluent water (from refineries, electroplating industry and from the manufacturing of detergents, chemicals, insecticides, herbicides and fungicides). Also activated carbon being an effective decholorinator, it is used for the purification of edible oils, food stuffs, pharmaceutical intermediates and chemicals. For the recovery of solvents such as chloroform, carbon tetrachloride, acetone, pentane, methyl ethyl ketone, tetrahydrofuran, white spirit, benzene, toluene, xylene, petroleum and ether which were used extensively in industries such as paints, adhesives, printing, rubber, plastics, dry cleaning, chemicals and textiles, activated carbon materials have been exploited extensively. Apart from the above, activated carbon materials do influence each sphere of human activity and are used in military, nuclear reactors, air treatment, recovery of precious metals, catalysts, catalyst supports and templates [19].

As the applications are many, the demand for activated carbon is high which has necessitated the search for new precursors for activated carbon and also the environmentally friendly methods for the production of activated carbon materials. *Sterculia foetida* is one of the sustainable feedstocks for activated carbon production with steam activation. More information on the general methods of activation of carbon materials with emphasis on *Sterculia foetida* feedstock and steam activation are provided in the subsequent sections.

METHODS OF ACTIVATION OF CARBON MATERIALS

Methods of activation of carbon materials hold the key for tuning the properties (textural, structural, morphological, thermal, and functional) and applications of the activated carbon materials [20–23]. Activation of carbon precursors leads to generation of porosity and hence the improvement in textural properties (BET specific surface area, pore type, pore volume, pore size). Moreover, rich oxygen, nitrogen and sulphur containing surface functional groups can be generated through activation of carbon materials. In general, the methods of activation of carbon material are broadly classified as chemical and physical based on the type of the activating agent used as shown in Figure 2. The XRD features of activated carbon materials from the fruit shells of *Sterculia foetida* were shown in Figure 4. XRD studies provide valuable information not only on the structure of the activity carbon materials but also on the purity of the materials. The sharp peaks in the XRD pattern [Figure 4 (a)] of the steam activated carbon material are due to the various metal oxides present in the plant tissue which can only be removed by the successive treatments with a base and an acid [Figure 4 (b)]. In general, the XRD profiles of either steam activated or K_2CO_3 activated looked almost the same [Figures 4 (b) and (c)] with two broad peaks at 2θ values of 25 and 43° typical of turbostratic carbon materials [24].

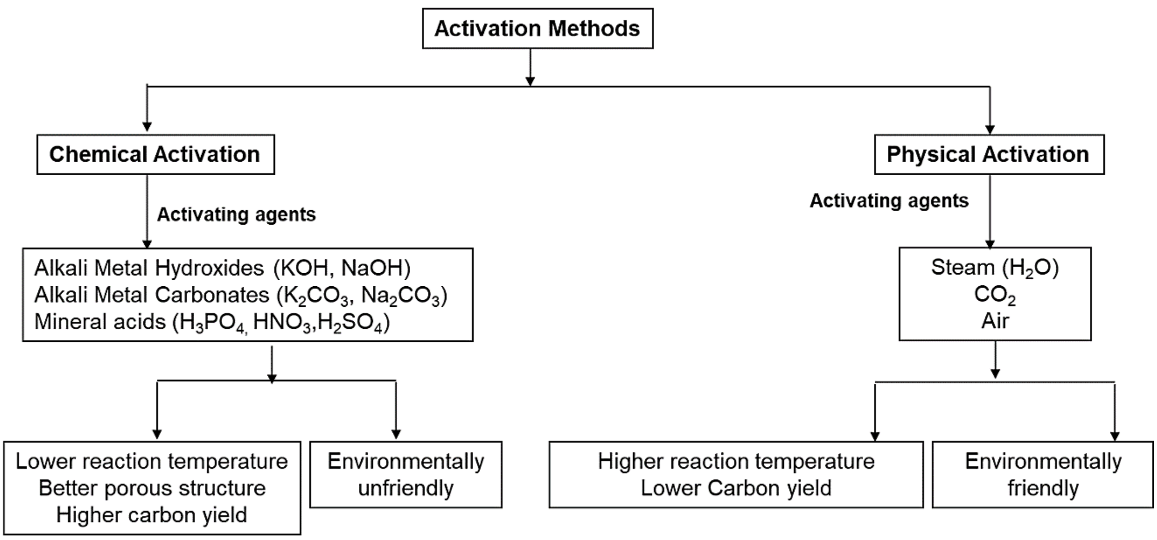

Figure 2. Chemical and physical methods of activation of carbon materials.

However, there is a need for transition to the use of clean and green methods of activation, while achieving the desired features that could be obtained by the use of corrosive chemicals like H_2SO_4, H_3PO_4, HNO_3, HCl, NaOH, KOH, K_2CO_3 and several others as activating agents. The masterpiece on the methods of activation of carbon materials was published from the group of Professor B Viswanathan that serves as a manual for researchers that intend to adopt greener activation methods for the production of activated carbon materials [20]. Steam activation appears to be the promising approach for producing activated carbon materials. However, innovation and breakthrough in the design of experimental step are awaited for the process to be commercially viable. A unique example of the potential of physical (steam) activation method over the chemical (K_2CO_3) activation is shown in the specific case of fruit shells of *Sterculia foetida.*

UNIQUE ADVANTAGE OF PHYSICAL (STEAM) ACTIVATION OVER CHEMICAL (K_2CO_3) ACTIVATION WITH THE FRUIT SHELLS OF *STERCULIA FOETIDA* [4]

Carbonization of fruit shells of *Sterculia foetida*

The shells of *Sterculia foetida* are seen all around the campus of IIT Madras, India. No practical use of these shells was reported prior to the work of Vignesh and coworkers [4]. Transformation of the shells into a value-added product like activated carbon is beneficial from economic and environmental viewpoints. Known weight of the dry shells have been taken and placed in a muffle furnace. The temperature of the furnace has been raised to 300°C and kept for 30 min during which devolatilization and subsequent carbonization of the carbon precursor took place. The carbonized shells were then taken out and ground using a mortar and pestle. The material obtained is sieved through a 200-mesh sieve. The resulting fine carbon powder is designated as carbon material as synthesized.

Physical Activation using steam as an activating agent

Activation essentially involves the generation of pores due to the burn off of the amorphous carbon. If the activating agent were to be in gaseous form like, CO_2, air and steam, the process of activation is called as physical activation. In the present case, steam is used as an activating agent. The process of steam activation involves placing known amount of as synthesized carbon material into the ceramic boat which is located in the tubular (quartz) furnace. Both ends of the quart tube were sealed so that no air enters into the tube. But a provision is made so that steam alone goes through the quartz tube from one end and gaseous products formed as a result of activation were let out of the quartz tube from the other end. One important precaution that needs to be taken during the start of the process of steam activation is that the water taken in the steam generator need to be saturated with high pure nitrogen so that the dissolved oxygen is removed. Otherwise, the dissolved oxygen in the water causes the complete combustion of carbon as synthesized leading only to the formation of ash. The temperature of the furnace is then raised to 800°C. Simultaneously the generation of steam is started and high pure N_2 gas is bubbled through the steam generator so that the steam is carried through the carbon material placed in the quartz tube. The process of activation is carried out for 1 h. Then the temperature of the furnace is lowered to room temperature and the sample is removed from the tube and subsequently treated with base (NaOH) and acid (HCl) for removing the silica and metallic impurities respectively present inherently in the carbon matrix. The carbon material thus obtained is designated at C_{Sf} activated (Physical, steam, activation) where C_{Sf} represents carbon material from *Sterculia foetida* [4].

Chemical activation using K_2CO_3 as an activating agent

The process of activation carried out in presence of activating agents in the form of solid state of liquid state is called as chemical activation. In general, as corrosive chemicals like NaOH and H_2SO_4 are involved in this process, chemical activation is regarded as environmentally unfriendly. But the carbon yields will be higher and also lower temperature of activation is sufficient in the case of chemical activation relative to physical activation. In the current study, K_2CO_3 is used as an activating agent. Typically, the process involves mixing and grinding of equal amounts of carbon material as synthesized and the activating agent (K_2CO_3) and then placing the same in a ceramic boat. The process of activation is carried out in an inert environment in tubular (quartz) furnace wherein the ceramic boat with the carbon material is placed. The temperature and time of activation are 800°C and 1 h respectively. After the activation, the temperature of the furnace is lowered. The activated carbon material is removed and then subsequently treated with base (NaOH) and acid (HCl) for removing the impurities and the decomposition products of the activating agent. The activated carbon material thus obtained is designated as C_{Sf} activated (Chemical, K_2CO_3, activation) where C_{Sf} represents carbon material from *Sterculia foetida* [4].

Base and Acid treatment of activated carbon materials

The carbon material as synthesized, the carbon sample immediately obtained after physical and chemical activations were subjected to the treatment with base and acid to remove impurities like SiO_2

and metallic species (K, Na, Ca, Mg, Fe) which will be inherently present in the plant tissue. Such a treatment with NaOH and HCl is inevitable to get rid of these impurities. Typically, the process comprises of taking the known amount of the carbon sample. To this nearly 10 times by weight, 10 wt. % NaOH solution is added, and the contents are stirred using a magnetic stirring for 1 h followed by filtering and washing the carbon sample. After this treatment with base, the carbon material obtained is weighed and treated with nearly 10 times by weight conc. HCl so as to remove the metallic impurities.

Characterization of carbon materials from the fruit shells of *Sterculia foetida*

Structural features of steam activated carbon material from *Sterculia foetida*: X-ray diffraction is an indispensable tool to study the structural property (crystallinity or amorphous nature) of the carbon materials. XRD patterns of various materials from *Sterculia foetida*, as synthesized, activated with steam, activated with K_2CO_3, were recorded using Rigaku miniflex II desktop X-ray diffractometer operated at 30 kV and 15 mA using CuKα radiation (λ = 1.5418 Å) at a scan rate of 1°/min. The diffraction profiles were obtained in the scan range (2θ) of 5 - 90°. The XRD plots of as synthesized, as well as carbon material after base (NaOH) followed by acid (HCl) treatment obtained from the fruit shells of *Sterculia foetida* were shown in Figure 3. In addition to the two broad peaks at 2θ values of 25 and 41° typical of turbostratic carbon materials, a sharp peak at 2θ value of 28° characteristic of silica are observed in the XRD pattern [Figure 3 (a)]. In addition to silica some tarry material and also metallic impurities like K, Na, Mg, Ca and Fe will be inherently present in carbon material as they constitute an essential component of the plant tissues. In order to remove the afore mentioned impurities, the carbon as synthesis is treated with a base first and then with an acid. The XRD pattern of the base and acid treated sample is depicted in Figure 3 (b). Interestingly, only the diffraction peaks typical of carbon were seen in the XRD pattern and no indication of impurities is observed. This indicate the effectiveness and also the significance of the treatment with NaOH followed by HCl for the removal of metal oxide and metallic impurities that are inherently present in biomass.

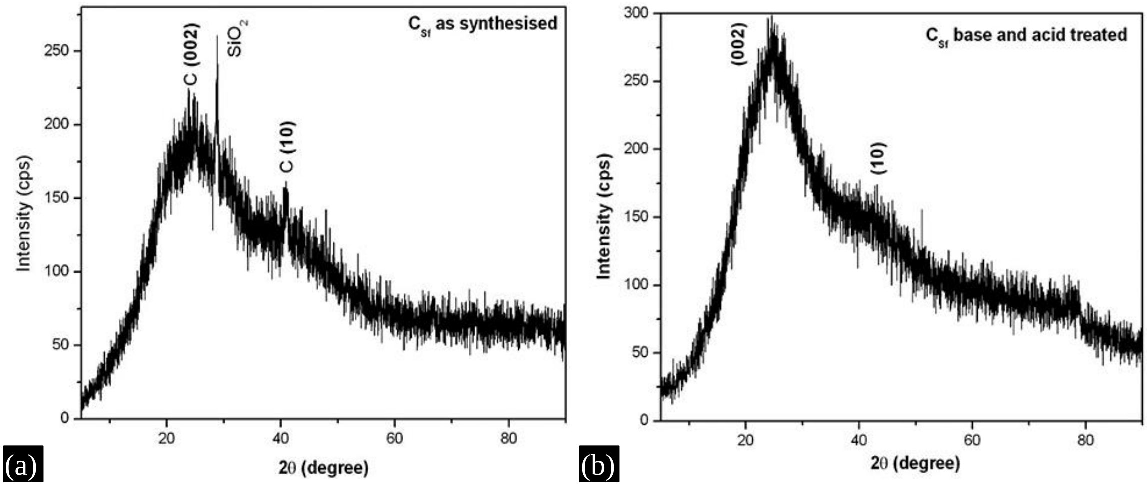

Figure 3. XRD plots of (a) carbon material (a) as synthesiszed and (b) after treatment with base followed by acid treatment to remove metal oxide and metallic impurities [4].

(a) (b)

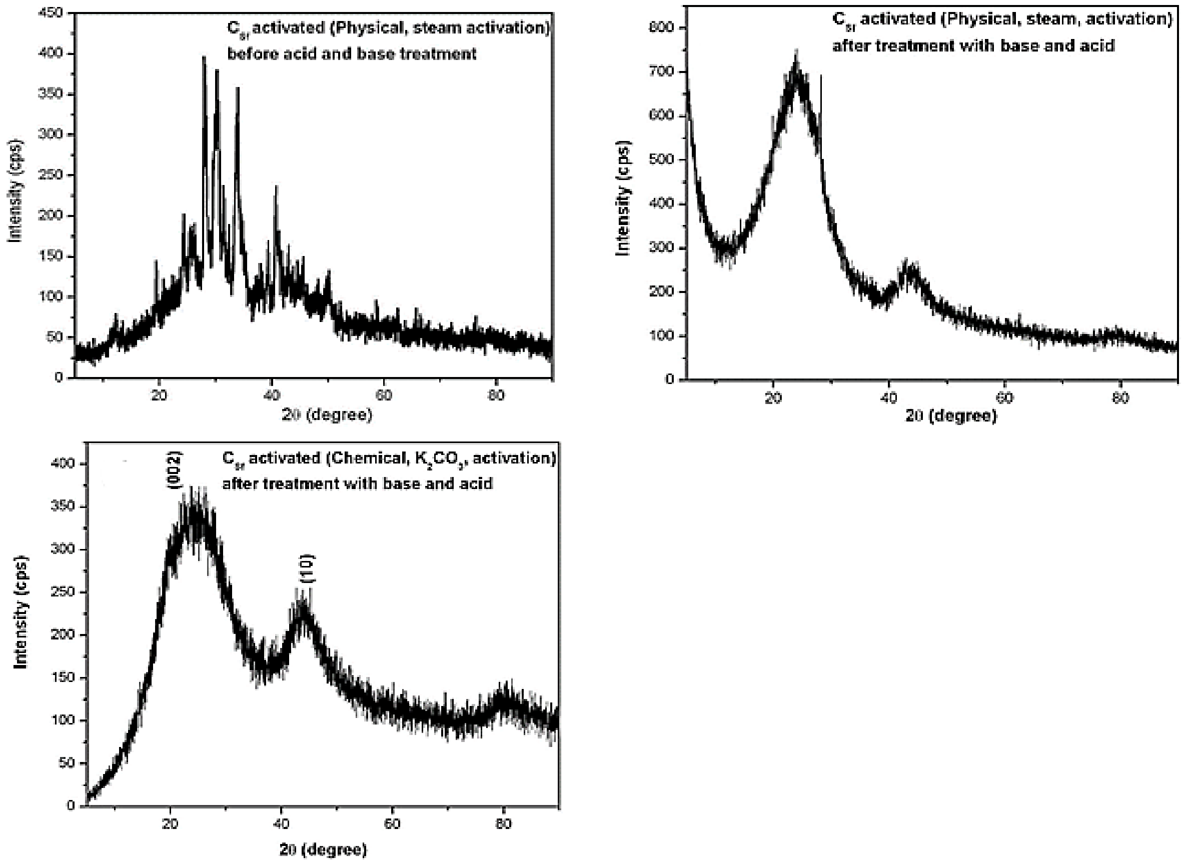

Figure 4. XRD patterns of activated carbon materials from the fruit shells of *Sterculia foetida* (a) after steam activation, (b) steam activation followed by successive treatment with a base (NaOH) and an acid (HCl) and (c) K_2CO_3 activation followed by successive treatment with a base (NaOH and an acid (HCl) [4].

The structural parameters, namely, the inter planar spacing, d_{002} as well as the crystallite sizes along the a- and c- axes, of the carbon materials, as-ynthesized and activated carbon material (physical and chemical activation) were obtained from the XRD patters shown in Figures 3 and 4 using the Bragg's law (1) as well as the Debye Scherrer' equation (2).

The interlayer spacing is calculated from the Bragg equation as shown below:

$$d = n\lambda/2\text{Sin}\theta \quad (1)$$

where λ is the wave length of the radiation (CuKα) used = 0.15405 nm

θ is the diffraction angle for the peak position.

Using Scherrer equation (shown below), the values of average crystallite size along the c-axis (stacking axis), L_c and along the a - axis, L_a, were determined.

$$L = K\lambda/B\text{Cos}\theta \text{ where } L = L_c \text{ or } L_a \quad (2)$$

B is the half-width of the peak in radians and K is the shape factor. The shape factor K depends on the lattice dimension. The K values of 0.9 and 1.84 were used respectively for calculating L_c and L_a values [21].

The crystallographic parameters (d_{002} and L_c, L_a values) deduced from the XRD plots shown in Figures 3 and 4 for steam and K_2CO_3 activated carbon material were summarized in Table 1. The d_{002} values of the activated carbon materials were found to be greater than that of the d_{002} values of graphite indicating that the structure of the activated carbon materials is turbostratic. The carbon material obtained from physical activation was made up of crystallites with dimensions of about 13 cell lengths along the c-direction and about 18-19 cell lengths along the a-direction relative to pure graphite. The carbon material obtained from chemical activation is made up of crystallites with dimensions of about 12-13 cell lengths along c-direction and about 17-18 cell lengths along a-direction relative to pure graphite. This imply that the structural parameters (crystallite sizes) as small as those derived from chemical activation could be obtained from physical action as well. Such small crystallite sizes of the activated carbon particles are of immense value when these materials are used as adsorbents for the removal of chemicals used for bioterrorism as used in Syria by Mr Bashar Al-Assad in the chemical war-fare.

Table 1. Crystallographic parameters of activated carbon materials.

S.N.	Carbon Material	d_{002} (nm)	Crystallite size, L	
			***L_c*, nm**	***L_a*, nm**
1	Steam activated carbon materials from *Sterculia foetida* after treatment with base and acid	0.4186	0.878	4.575
2	K_2CO_3 activated carbon materials from *Sterculia foetida* after treatment with base and acid	0.4011	0.843	4.30
3	Graphite	0.335	0.0671	0.2461

Typical alignment of graphene sheets along the “a” and “c” axes in pure graphite were shown in Figure 5 [24]. Unlike the orderly arrangement of graphene sheets in graphite, the graphene sheets are slightly misaligned in the turbostratic structure as in the case of the activated carbon material from *Sterculia foetida*. Moreover, the crystallite size along the “a” and “c” axis in the activated carbon materials from *Sterculia foetida* are larger as shown in Table 1.

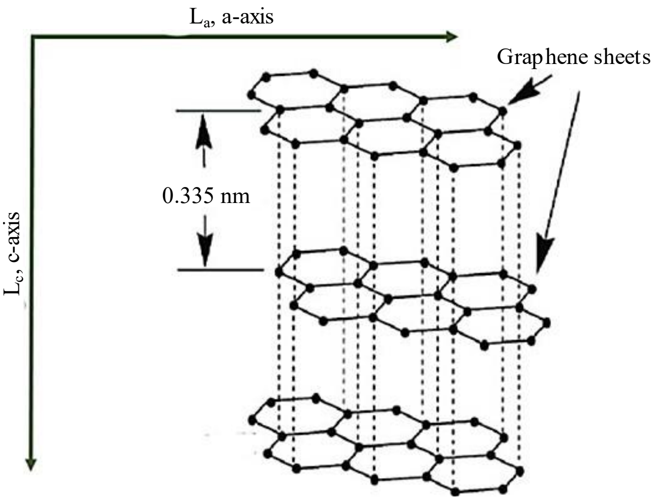

Figure 5. Alignment of graphene sheets along “a” and “c” axis is pure graphite [24].

Textural features of steam activated carbon material from Sterculia foetida [4]: The textural features, namely, Brunauer-Emmett-Teller (BET) specific surface area, pore architecture (type of porosity, pore size and pore volume) of the carbon materials, in general, can be deduced from N_2 sorption studies at liquid nitrogen temperature. The sorptometric analysis on the materials from *Sterculia foetida* was carried out using Sorptometer 1990 Carbo Erba machine using high pure nitrogen (99.99%) as adsorbent at 77 K (-196°C). Prior to the analysis, the carbon samples were out gassed (in an evacuation chamber, Carlo-Erba out gassing unit with a Pirani gauge, at a temperature of 523 K under a vacuum of 10^{-6} Torr for 12 h. The specific surface area values were derived from the isotherm using BET method in the relative pressure range, p/p_0 (p is the adsorption pressure and p_0 is the saturation vapour pressure), of 0.05 and 0.3 where the monolayer coverage is assumed to be complete and also assuming that the surface area occupied by physisorbed nitrogen molecule as 0.162 nm^2. The total pore volume was calculated from the amount adsorbed (liquid volume of nitrogen) at a relative pressure of 0.99. The N_2 adsorption and desorption isotherms recorded on the activated carbon samples (K_2CO_3 Vs steam) from the fruit shells of *Sterculia foetida* were shown in Figure 6.

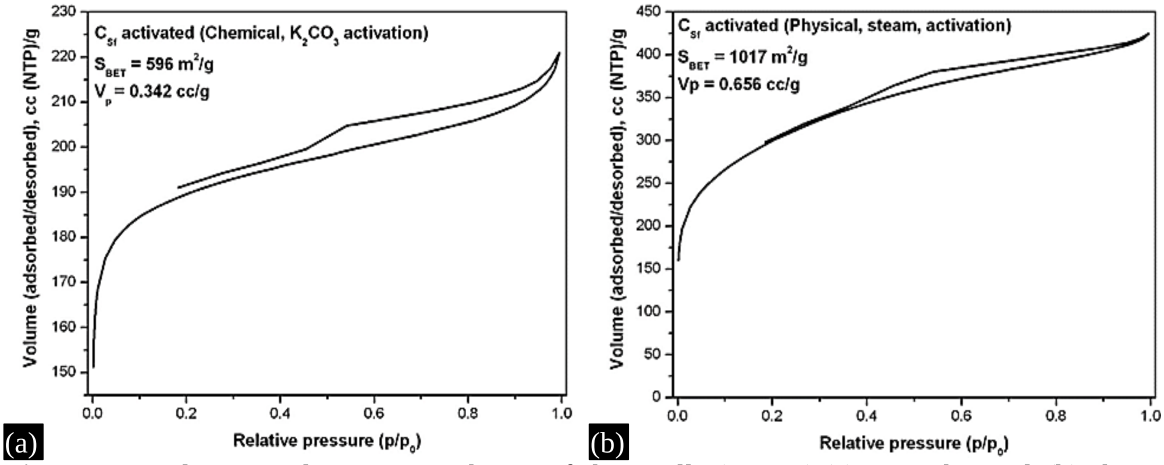

Figure 6. N_2 adsorption-desorption isotherms of chemically (K_2CO_3) (a) as synthesized, (b) physically activated (800°C for 1 h) carbon materials [4].

Fine details of the porous texture can be deduced from the shape of the N_2 adsorption-desorption isotherms. Irrespective of the method of activation (either chemical or physical), both the resulting materials, offered type I isotherm characteristic of microporous carbon materials, implying that the pore size is less than 2 nm. Moreover the specific surface area of physically activated carbon materials is nearly double (1017 m^2/g) compared to the chemically activated carbon material (596 m^2/g) (Figure 6). This shows the promise that physical activation methods can be made as potential as chemical methods, provided smart experimental engineering and optimization is done. These values are three orders of magnitude higher than that of the specific surface area of the as synthesized carbon material (4 m^2g) highlighting the significance of activation in enhancing the textural properties of the carbon materials.

Thermal stability of steam activated carbon material from Sterculia foetida [4]: The thermal properties of the activated carbon materials were evaluated on a Perkin Elmer Diamond TG/DTA instrument. A weighed amount of the sample to be analysed was placed in a cylindrical alumina crucible mounted on one of the 2 mm diameter alumina rods. The sample was heated in a dynamic air atmosphere at a rate of 283 K/min.

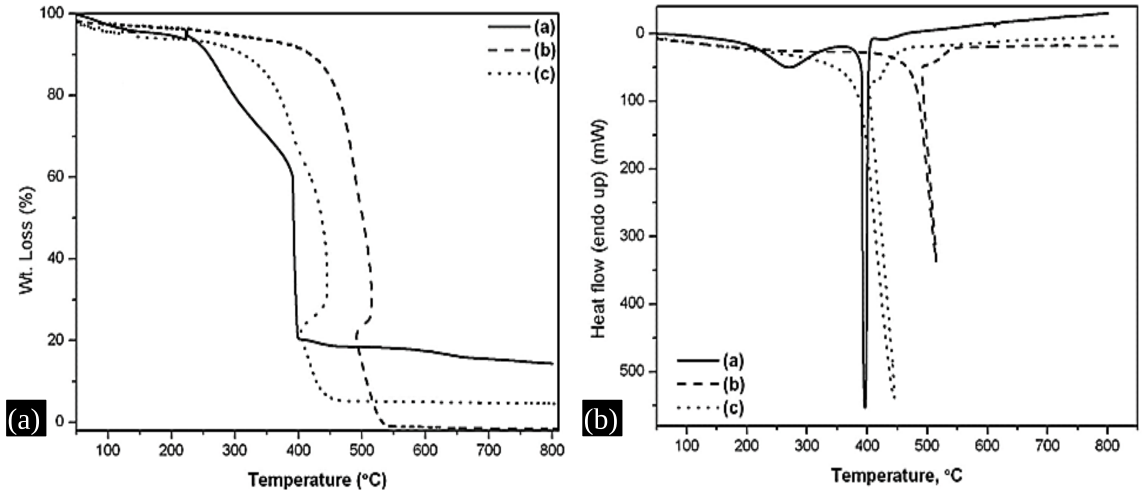

Figure 7. Thermo gravimetry (a) and differential thermal analysis (b) curves of carbon materials (a) physically activated and (c) chemically activated from the fruit shells of *sterculia foetida* [4].

The thermal stability of the carbon materials as synthesized and activated (physical and chemical) were analyzed by TG-DTA and were depicted in Figure 7. Enhancement in the thermal stability is observed upon activation (either physical or chemical). Steam activation resulted in activated carbon material with higher (~ 100°C) thermal stability compared to the K_2CO_3 activated sample. Moreover, in the case of the as synthesized carbon material the weight loss is 85 wt.% indicating the fact that the carbon material contained nearly 15 wt.% impurities. This result is in concurrence with the XRD analysis of the as synthesised as well as the steam activated carbon materials prior to treatment with base and acid [Figure 3 (a) and 4 (a)]. Upon treatment with base and acid the steam activated carbon materials showed, 100% wt. loss implying that there were no metal/metal oxide impurities. In contrast, activated carbon material obtained from chemical activation (K_2CO_3), the observed weight loss is only 95% and the rest of the 5 wt % sample that remained in the machine even after 800°C correspond to ash comprising of metal oxide impurities [Figure 7 A (c)]. Moreover the exothermic peaks observed in the DTA plots corresponding to the weight losses seen in the TG curves imply that the weight loss in the case of carbon materials is because of the oxidation of carbon materials (Figure 7 B). Thus the steam activated carbon material offers better properties in terms of purity and thermal stability compared to K_2CO_3 activated sample.

Surface functional features of steam activated carbon material from Sterculia foetida [4]: FT-IR spectroscopic characterization provides authentic information on the nature of surface functional groups present in carbon materials. The FT-IR spectra of various carbon materials were recorded on Shimadzu spectrophotometer. The spectral range of analysis is 450 - 4000 cm^{-1} with a resolution of 4 cm^{-1}. The spectra were obtained in transmission mode at 20 scans. Pressed KBr pellets were prepared by grinding 200 mg of carbon samples with 0.5 g of KBr. The FT-IR sepectra of the assynthesized and activated carbon materials (steam Vs K_2CO_3) were shown in Figure 8. As expected there are a large number of phenolic, C-O groups (1060 cm^{-1}) in the as-synthesized material which upon activation with either steam or K_2CO_3 converted to carboxylic groups. The increase of carboxyl functional groups in the activated carbon materials, by either steam or K_2CO_3 is evident from the increase in the signal of the band in the region of 1690 to 1760 cm^{-1}. Such oxygen containing functional groups play a vital role for catalytic processes.

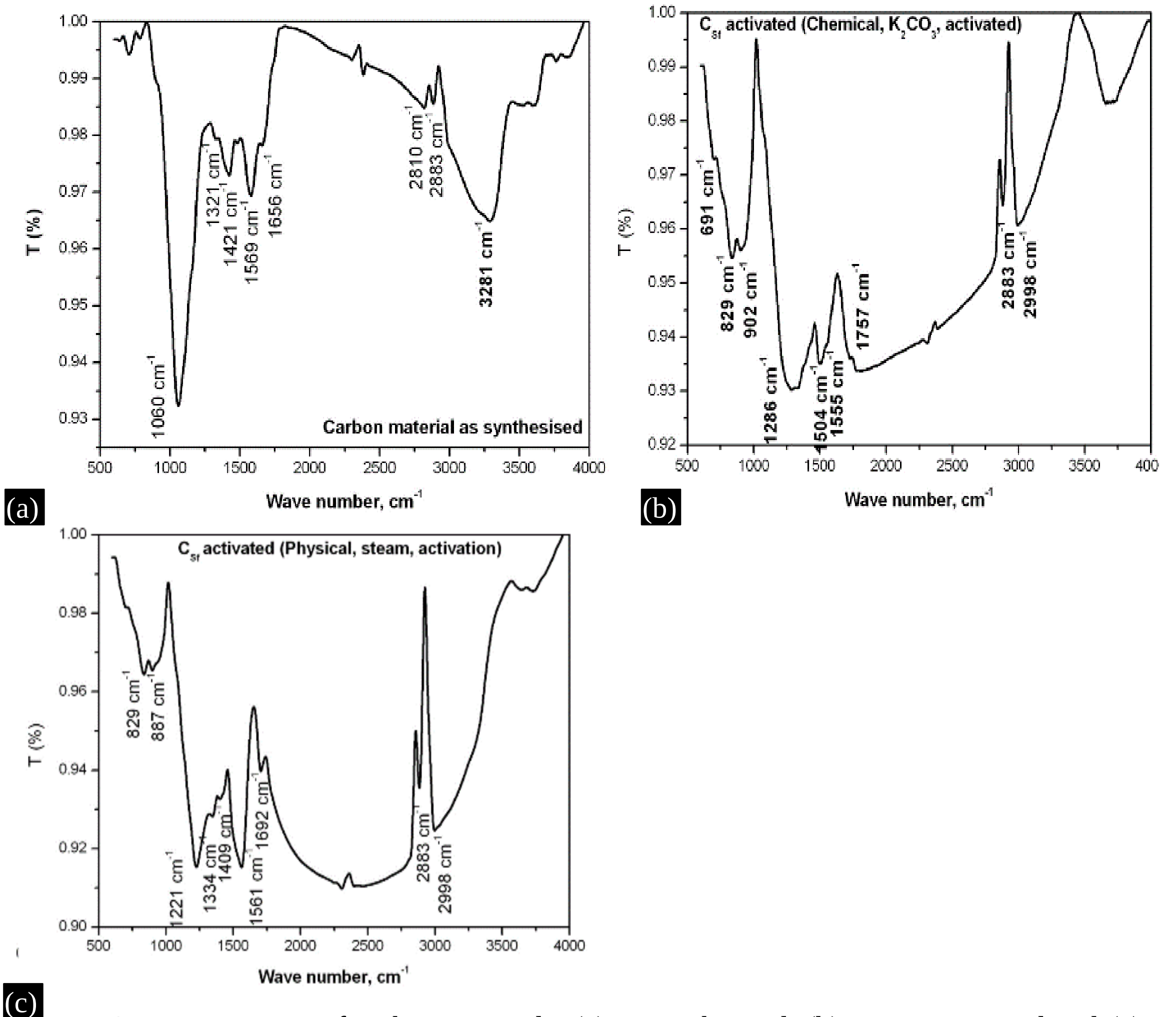

Figure 8. FT-IR spectra of carbon materials, (a) as synthesised, (b) K_2CO_3 activated and (c) steam activated, from the fruit shells of *Sterculia foetida* [4].

CONCLUSION

From the characterization of carbon materials from the fruit shells of *Sterculia foetida*, using XRD, BET sorptometry, TG-DTA and FT-IR analysis, it appears that the properties of the steam activated carbon material out performed those of the activated carbon material from chemical (K_2CO_3) activation. So, why murder, when cards can do? Let's go for greener and clearner activation processes of carbon materials, like the steam activation as shown with the representative feedstock of the fruit shells of *Sterculia foetida*. A web of Science search crossing the keywords, namely, activated carbon materials and steam activation shows 1532 results in total, signifying that the process of steam activation is under-utilized. The new insights provided in this review should act as a user manual for the researchers working on developing activated carbon materials for energy and environmental applications. The biomedical applications of the material should not be overlooked. It is hypothesized that the steam activated carbon material owing to its purity serves as an ideal decolorating agent for the *Gymnema sylvestre* extract, that is widely used as an antidote for diabetes.

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