SUSTAINABLE ROUTE TO MULTIWALLED CARBON NANOTUBE SYNTHESIS USING COCONUT WASTE-DERIVED ACTIVATED CARBON

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ABSTRACT

Coconut wastes was used to create activated charcoal (AC) carbon nanotubes. The activation procedure involved both chemical and heat activation. Both procedures were conducted with N2 gas flowing through them, with H_3PO_4 impregnation being employed for chemical activation and medium temperature heat exposure for heat activation. According to the findings, an excellent output (71.5% on average) was obtained by the activation temperature between 300 and 400°C. High-quality carbon nanomaterials with exceptional performance are produced using impregnated nanoparticles at an activation temperature between 500 and 700°C. A greater total surface area for a better adsorption process is provided by the generated nanomaterials' improved microporous structure and volume, as shown by FTIR and SEM characterization data.

KEYWORDS: Activated charcoal, Nanomaterials, Microporous structure, FTIR, SEM characterization.

INTRODUCTION

The fact that carbon may exist in a number of allotropic forms makes it a wonderful element in addition to being necessary for all living activities [1]. The first step in converting coconut wastes into useful carbon nanotubes is the meticulous processing of raw materials. The fibers, husks, and shells of coconuts make up their trash, which is a rich and convenient source of biomass [2].

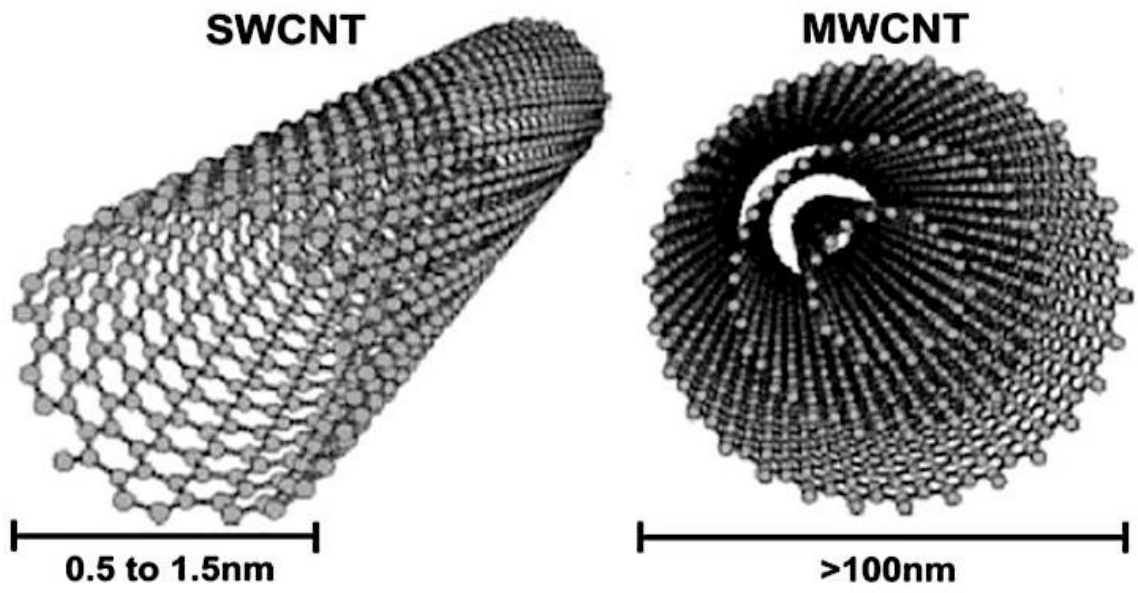
Among the best examples of novel nanostructures created using various chemical synthesis methods are carbon nanotubes. Although they have the most straightforward chemical composition and atomic bonding arrangement among nanomaterials, nanotubes may be the most rich and complex in terms of structure and structure-property interactions.

Recent attempts to employ novel solid-state nanomaterials in biological applications have sparked bioimmobilization and nanotube functionalization. Tiny biological electrical devices, such sensors and probes, could be created by combining the unique physical properties of molecular-scale or nanoscale solids (dots or wires) with the remarkable biomolecular recognition abilities. The contact between nanomaterials and biological molecules is crucial for these kinds of applications. Given the abundance of potential that nanotubes provide, such study is still in its infancy[3].

Researchers are becoming more interested in carbon nanotubes, a fast growing nanomaterial, due to its extensive use in creating new technologies in the domains of chemistry, electronics, health, biotechnology, and the environment. Since Ijima's discovery of carbon nanotubes (CNT) in 1991 [4], research has focused on CNT in a variety of fields due to its unique structural, electrical, optoelectronic, semiconductor, mechanical, chemical, and physical properties [5]. Its surface area is enormous, its surface is inert, and its atomic structure is far more compact and organized than that of ordinary activated carbon.

Among the many applications proposed for carbon nanotubes are field emission displays, supercapacitors, ultrahigh strength materials, and nanometer-sized semiconductor devices, probes, and interconnects. Hydrogen adsorption on very impure carbon single-wall nanotubes was initially measured by Dillon et al. [6]. Based on their findings, they concluded that potential developments in hydrogen storage will follow. Liu et al. [7] eventually looked into the adsorption of hydrogen on 50–60% pure SWNTs made with a semi-continuous arc discharge method. They found that at normal temperature and somewhat high pressure, the materials exhibited a 4.2 percent storage capacity. SWCNTs are produced by rolling a single layer of graphene into a seamless cylinder, typically with a diameter of 0.8–2 nanometers. Their structure is simple yet stable, and it can vary depending on how the graphene sheet is rolled. This results in them becoming metallic or possessing other electrical properties.

Multiwalled nanotubes have been shown to have potential, despite the fact that single-walled nanotubes have gotten the most attention. MWCNTs are shaped like nested loops and are made of several layers of graphene wrapped into cylinders. Their diameter can range from 2 to over 100 nanometers, and their length can reach several micrometers, depending on how many walls there are. MWCNTs' multilayer structure makes them more resilient overall, although they are slightly less flexible than SWCNTs [8] .



Schematic representation of single walled carbon nanotube (SWCNT) and multi walled carbon nanotube (MWCNT) [9]

Due to its vast potential, several synthesis techniques are being developed quickly to generate huge quantities of size-controlled carbon nanotubes for use in industrial applications. Chemical-based processes such laser vaporization, arc discharge, pyrolysis, and thermal chemical vapor deposition (CVD) or plasma-enhanced CVD have proven to be difficult to regulate the wall number, chirality, and nanotube diameter [10]. Nevertheless, a lengthy synthesis time, high reaction temperature, and intricate process control are always necessary for the majority of those tactics [11]. A well-known technique is the catalytic breakdown of hydrocarbons, which also necessitates a difficult purification procedure to remove the metal catalyst particles [12]. The efficiency of the large-scale synthesis of carbon nanotubes had decreased as a result of those tactics.

EXPERIMENTAL SECTION

Materials and methods

The coconut shell waste was taken from a local market of Prayagraj, India. The chemical composition is presented in Table I.

Item	Value
Proximate analysis (wt% db)	
Non-Volatile Carbon	52
Volatile Matter	32
Ash	10
Moisture	7
Elemental analysis (wt% db)	
Carbon	60.5
Oxygen	25.6
Hydrogen	3.2
Nitrogen	0.31

TABLE I : PROPERTY OF COCONUT WASTE ACTIVATED CHARCOAL

PREPARATIONS

Activated charcoal carbon nanotubes (CNT) were prepared through two methods:

a) Heat activated, b) H_3PO_4 impregnation.
Coconut shell wastes were collected and washed thoroughly with fresh water and allowed to tray drying initially in the sun and then in the oven at 120^0C for 24-48 hours. It was then crushed and sieved to a size range of few nanometers [13].

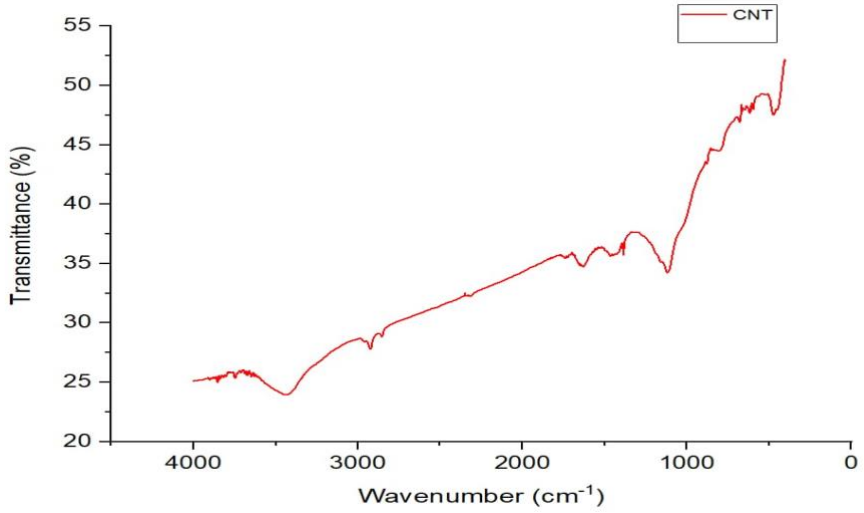
a) 10 g of coconut shell charcoal was placed in the silica boat inside the Muffle Furnace (Thermotech Model PID-91Se). Nitrogen gas was continuously supplied to prevent oxidative condition. Medium temperature heat (300-400 °C) produced from the furnace was provided for heat activation process. Heating rate was set at 10 °C/min.

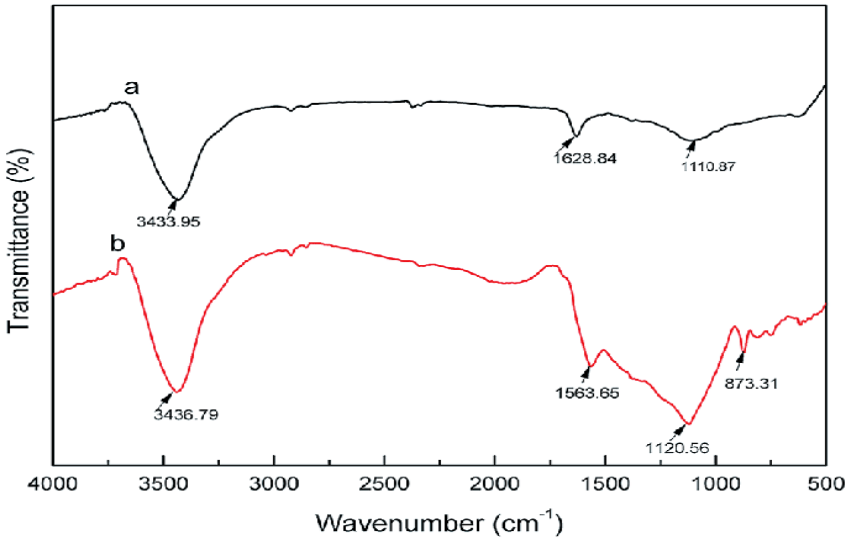
b) A solution of phosphoric acid was prepared for impregnation process. The coconut waste charcoal was impregnated with 50% H_3PO_4 solution for 24 h in the ratio 1:1 and stored in the Digital Hot Air Oven (i-Therm Model AI-7781) at 100 ºC for 24 hrs [14]. Then dried again for 24 hrs in the oven. Then, calcination process of dried product was performed at 550 ºC for 4 hrs in the Muffle Furnace which was purged with high purity nitrogen (N_2) (99.99%) at a flow rate of 3.5 lit/min for 15 min to remove the atmospheric air present inside the tube [15]. Finally, the impregnated CNT was cooled down to the room temperature and stored in for future use. The prepared sample was then washed through distiiled water until the pH reaches 6-7 and then dried in the oven for few more hours.

RESULTS AND DISCUSSIONS

The yield was determined once the manufacture of activated charcoal CNT was complete. It was found that the activation temperature range of 300 to 400°C produced an excellent yield (71.5 percent on average). The yield decreases with increasing temperature, though, as the higher activation temperatures helped to remove volatile elements from the structure of the charcoal. For impregnated charcoal, the yield of AC carbon nanotubes was reduced by higher activation temperatures (over 700°C), which was on average roughly 48%. At activation temperatures between 500 and 700°C, high-quality carbon nanomaterials with exceptional performance are generated. In contrast to the lowest AC output from heat activation, the yield of AC carbon nanotubes from chemical activation was 23 percent lower [16].

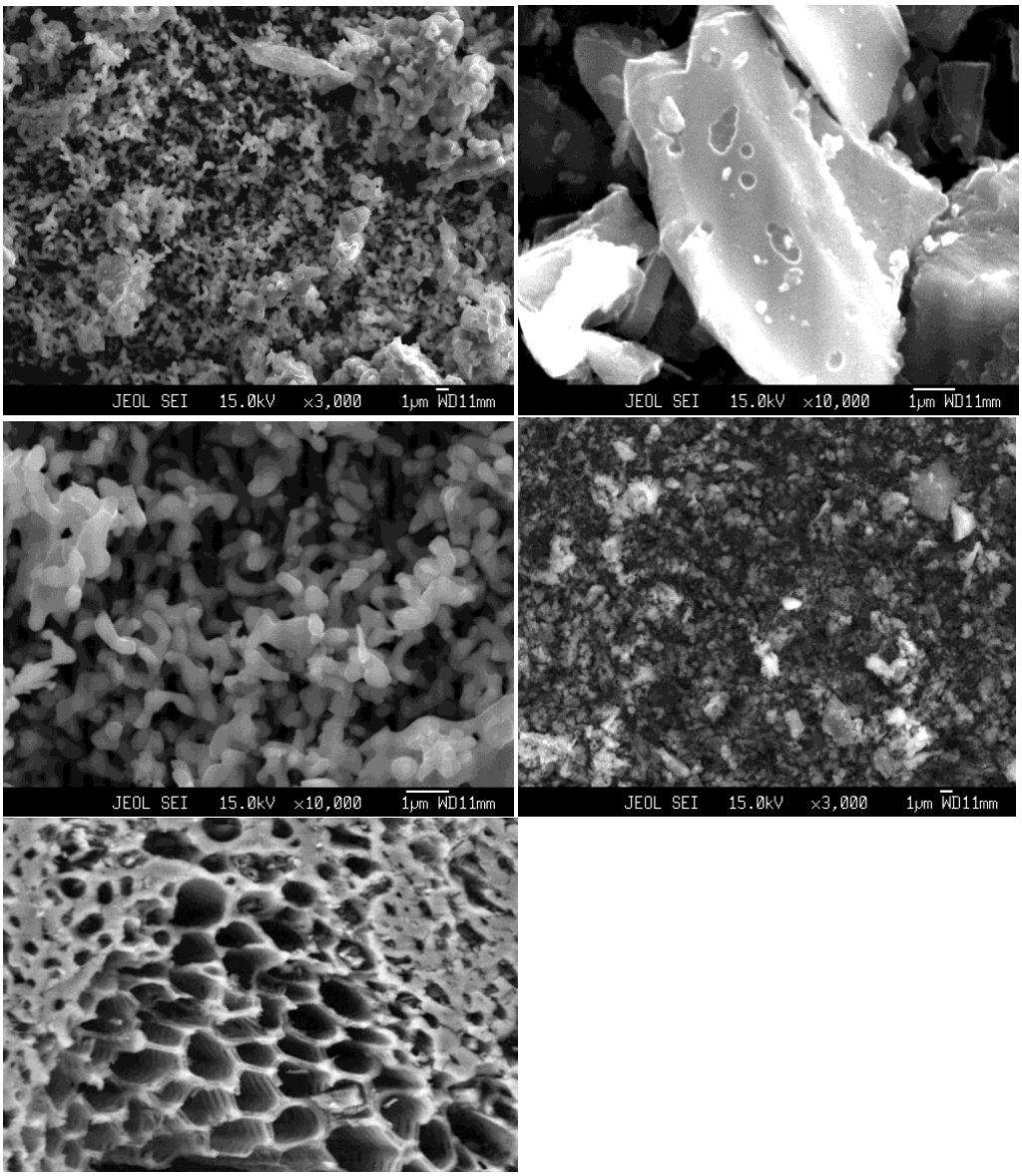
The FTIR and SEM were used to further characterize the above mentioned, prepared samples. The FTIR spectra can be used to detect these functional groups and monitor the chemical changes that take place during carbonization. The CNTs in coconut wastes can vary, but they usually exhibit peaks at approximately 1730-1745 cm^{-1} (ester groups), 1545-1655 cm^{-1} (amide II), 1520-1540 cm^{-1} (amide II), 3420-3430 cm^{-1} (-NH, -OH stretching), and 1030-1035 cm^{-1} (C-O stretching in carbs) [17].





The FTIR spectra of coconut wastes reveal high quantities of cellulose and lignin, which are necessary for the durability, stiffness, and biocompatibility of the material. According to Tao et al. (2017), these results lend credence to the notion that coconut coir can be a valuable material for prosthetic sockets due to its mechanical properties and sustainability [18]. Additionally, SEM characterisation has been performed on the samples. SEM (Scanning Electron Microscopy) photos provide insight into the microscopic morphology of coconut-based activated charcoal carbon nanotubes (CNTs) and demonstrate the produced carbon materials' bundled, knotted, or uneven structure [19]. To view surface properties, ascertain tube or particle size and distribution, and assess the impact of synthesis parameters such as temperature or chemical modifier use, these materials are typically analyzed using SEM. The sample was inspected under a scanning electron microscope to reveal the porous structure. At a lesser magnification, SEM pictures were taken to see how CNT growth was distributed across the charcoal surface [20]. We also used additional high magnification photos to investigate the CNT dimensions. The CNT's diameter was found to be between 30 and 35 nm, but its length was only a few micrometers. The obtained CNTs' enormous diameter confirms that they are

multiwalled (MWCNT). Below are the SEM pictures of the H3PO4-impregnated and heat-activated CNTs.



CONCLUSION

Because coconut waste has a large diameter (310 μm) and a very variable fiber length, its length-diameter ratio is low. We have successfully proven the production of multi-walled carbon nanotubes (CNTs) from activated charcoal using two different techniques. The manufactured carbon nanotubes had a smaller diameter (30–35 nm) and a larger total surface area, which is one of the important factors, according to the data. Consequently, a high degree of adsorption power is present, which can be applied to different adsorption procedures in the future.

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REFERENCES

1. Coville, N. J., Mhlanga, S. D., Nxumalo, E. N., & Shaikjee, A. (2011). A review of shaped carbon nanomaterials. *South African Journal of Science, 107*(5–6), 1–15. https://doi.org/10.4102/sajs.v107i5/6.490
2. Bilba, K., Arsène, M. A., & Ouensanga, A. (2007). Study of banana and coconut fibers: Botanical composition, thermal degradation and textural observations. *Bioresource Technology, 98*(1), 58–68. https://doi.org/10.1016/j.biortech.2005.11.030
3. Dai, H. (2002). Carbon nanotubes: Synthesis, integration, and properties. *Accounts of Chemical Research, 35*(12), 1035–1044. https://doi.org/10.1021/ar0101640
4. Iijima, S. (1991). Helical microtubules of graphitic carbon. *Nature, 354*(6348), 56–58. https://doi.org/10.1038/354056a0
5. Haddon, R. C. (2002). Chemistry of single-walled carbon nanotubes. *Accounts of Chemical Research, 35*(12), 977–1113. https://doi.org/10.1021/ar010155r
6. Dillon, A. C., Jones, K. M., Bekkedahl, T. A., Kiang, C. H., Bethune, D. S., & Heben, M. J. (1997). Storage of hydrogen in single-walled carbon nanotubes. *Nature, 386*(6623), 377–379. https://doi.org/10.1038/386377a0
7. Liu, C., Fan, Y. Y., Liu, M., Cong, H. T., Cheng, H. M., & Dresselhaus, M. S. (1999). Hydrogen storage in single-walled carbon nanotubes at room temperature. *Science, 286*(5442), 1127–1129. https://doi.org/10.1126/science.286.5442.1127
8. Zeng, Q., Li, Z., & Zhou, Y. (2006). Synthesis and application of carbon nanotubes. *Journal of Natural Gas Chemistry, 15*(4), 235–246. https://doi.org/10.1016/S1003-9953(07)60006-6
9. Ribeiro, B., Botelho, E. C., Costa, M. L., & Bandeira, C. F. (2017). Carbon nanotube buckypaper reinforced polymer composites: A review. *Polímeros, 27*(3), 247–255. https://doi.org/10.1590/0104-1428.03016
10. Rao, G. P., Lu, C., & Su, F. (2007). Sorption of divalent metal ions from aqueous solution by carbon nanotubes: A review. *Separation and Purification Technology, 58*(1), 224–231. https://doi.org/10.1016/j.seppur.2006.12.006
11. Kang, Z., Wang, E., Gao, L., Luo, S., Jiang, M., Hu, C., & Xu, L. (2003). One-step water-assisted synthesis of high-quality carbon nanotubes directly from graphite. *Journal of the American Chemical Society, 125*(45), 13652–13653. https://doi.org/10.1021/ja037626r

12. Zhu, H. W., Xu, C. L., Wu, D. H., Wei, B. Q., Vajtai, R., & Ajayan, P. M. (2002). Direct synthesis of long single-walled carbon nanotube strands. *Science, 296*(5569), 884–886. https://doi.org/10.1126/science.1060928
13. Ketwong, T., & Sitthiseree, S. (2019, October 22–24). Activated carbon production from coconut shell charcoal employing steam and chemical activation for ammonia adsorption application. *Proceedings of the World Congress on Engineering and Computer Science (WCECS 2019)*, San Francisco, USA.
14. Kang, Z., Wang, E., Gao, L., Luo, S., Jiang, M., Hu, C., & Xu, L. (2003). One-step water-assisted synthesis of high-quality carbon nanotubes directly from graphite. *Journal of the American Chemical Society, 125*(45), 13652–13653. https://doi.org/10.1021/ja037626r
15. Ishigami, M., Cumings, J., Zettl, A., & Chen, S. (2000). A simple method for the continuous production of carbon nanotubes. *Chemical Physics Letters, 319*(5–6), 457–459. https://doi.org/10.1016/S0009-2614(00)00185-2
16. Tucureanu, V., Matei, A., & Avram, A. M. (2016). FTIR spectroscopy for carbon family study. *Critical Reviews in Analytical Chemistry, 46*(6), 502–520. https://doi.org/10.1080/10408347.2016.1157013
17. Tao, W., et al. (2017). Coconut coir-based biodegradable composite materials: Preparation and characterization. *Materials Science and Engineering: A, 699,* 138–146. https://doi.org/10.1016/j.msea.2017.05.017
18. Branca, C., Corsaro, C., Frusteri, F., Magazù, S., Mangione, F., Migliardo, U., & Wanderlingh, U. (2004). Structural and vibrational properties of carbon nanotubes by TEM and infrared spectroscopy. *Diamond and Related Materials, 13*(7–10), 1249–1253. https://doi.org/10.1016/j.diamond.2003.11.054
19. Araga, R., & Sharma, C. S. (2016). One step direct synthesis of multiwalled carbon nanotubes from coconut shell derived charcoal. *Materials Letters, 185,* 527–530. https://doi.org/10.1016/j.matlet.2016.09.071
20. Mishra, L., & Basu, G. (2020). Coconut fibre: Its structure, properties and applications. In *ResearchGate* (Chapter 10). https://www.researchgate.net/publication/344993524