

Synthesis and Characterisation of Activated Carbon from Tea Waste for Super Capacitor

A.Senthil Kumar*
{ask.apsc@psgtech.ac.in}

^aDepartment of Applied Science (Physics),PSG College of Technology, Coimbatore

Abstract. Supercapacitors are considered as an energy storage device where charge transfer takes place without chemical reactions and it is more environmentally friendly in nature. In this present work, activated carbon was prepared from the tea waste by means of activation process using an activating agent like Sodium Hydroxide (NaOH). The obtained activated carbon powders were systematically studied from X-Ray Diffraction (XRD) to know the structure of activated carbon, Scanning Electron Microscope (SEM) studies to reveal the surface morphology of activated carbon and the existence of pores formed after the activation process. HRTEM analysis shows that how complex is the structure with carbon layers and their atomic packing. FTIR analysis was carried out to know their functional group. Hence activated carbon prepared through chemical activation process from tea waste can be a better choice as an electrode material for supercapacitor.

Keywords: Activated Carbon, Electrodes, Functional group, Micro Structure, Particle size, Supercapacitors.

1 Introduction

The supercapacitors are considered as a special type of capacitor with high capacitance value, combine the properties of capacitors and batteries into a single device. These types of supercapacitors have significantly developed over the last few decades and exhibited their potential properties to provide advanced technology in energy storage system. Considering the environmental issues, world's attention has been turned into focused the research on energy conservation and storage devices. Many countries are replacing their quality fuel with natural resources like solar, wind and electric energies [9]. The electrochemical energy storage systems like lithium-ion capacitors [10], Li-S batteries [11,19], lithium-ion batteries [22] and supercapacitors [23] are extensively studied by different research groups worldwide.

Technology of batteries are well developed and extensively used in the widespread range of applications. The main disadvantages of these battery technologies are lower life cycle, moderate power density, higher charging time, high heating problem and they are not eco-friendly in nature. To rectify this immediate demand for the high energy-power devices to store energy, electrochemical reactions were considered as a main source. Supercapacitors are the only choice with achievable results, because they have the following advantages like quick charging, lesser liberation performance, high-power density and extended cyclability [5]. The capacitors with extra-large capacitance value are called as supercapacitors. These

supercapacitors have the ability to supply the potential power required to meet the energy demand instantly.

Supercapacitors (SC) are the energy storage system that are more ecologically friendly in nature. Supercapacitors are widely used in areas such as digital technology, cell phones and also in electric motors [8]. Supercapacitors have the three significant and important components like electrodes, electrolytes and separators. Electrode materials of supercapacitors are made of activated carbon [3], metal oxides [20], conductive polymers [25] and nanocomposites [31].

The active component of supercapacitor is its electrode coated with activated carbon (AC) and has been studied widely as an adsorbent [26]. The activated carbon with high porosity, high capacitance, rapid adsorption, and thermal stability are considered as their featured properties [12]. Fabrication of these low-cost activated carbon becomes the main challenges in commercializing the low cost electrode materials. The young researchers have adopted various new and innovative techniques for the preparation of activated carbon from inexpensive raw materials including the utilization of waste materials from agricultural products as a precursor with low inorganic content [1] and high carbon content [4, 13]. Activated carbon can be prepared from different natural resources and its byproducts. More than a few affordable natural materials are available in and around the world for producing activated carbon for supercapacitor electrodes. The preparation of activated carbon from the available natural resources like tea waste, water melon rind, pomegranate rind, paulownia flower, coconut shell, cherry stones and bamboo waste [13, 18] has been studied in the recent decades by different research groups worldwide.

In these present research work, an effort has been taken to prepare activated carbon from tea waste for supercapacitor electrodes. Activated carbon was successfully prepared by means of activation process through chemical method using NaOH as an activating agent. The carbonized tea waste was activated in nitrogen atmosphere and their characteristic properties were thoroughly studied in details from XRD, SEM, HRTEM and FTIR analysis and were reported.

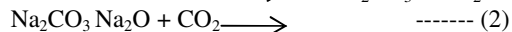
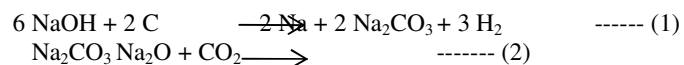
2. Experimental Details

2.1 Carbon Powder Synthesis:

Tea waste were collected from home and tea shops are systematically washed in water for removal of dust and milk content adhered to it. The tea waste was then dried in sunlight for 20 days in natural atmospheric conditions and the samples were named as T₁ (home) and T₂ (Shop) respectively. After drying them in natural atmosphere, it was kept at 110°C for 5 hours in the hot air oven for the removal of moisture and other volatile impurities. Followed by drying process, the tea dust was crushed into fine powder using mortar pestle.

2.2 Carbonization and activation with NaOH:

Fine carbon powder after drying them at 110°C in hot air oven was subjected to carbonization in muffle furnace at 200°C for 4 hours. About 7.4562g and 6.5124g of T₁ and T₂ were obtained respectively after carbonizing the samples. Chemical activation was carried out with the alkaline like sodium hydroxide. The anticipated reaction during activation of carbon by NaOH is as follows [18]



This oxidation method has the capability to yield the required porous in the activated carbon powder. About 3gm of both the samples were absorbed by Sodium Hydroxide in the ratio 1:5 i.e., 3g of sample T₁ is digested with about 15ml of Sodium Hydroxide and 3g of sample T₂ is digested with about 15ml of Sodium Hydroxide and kept undistributed for about 15 hours.



FIGURE 1 Homogeneous mixing of sample using a magnetic stirrer.

Both the samples were homogeneously mixed using magnetic stirrer maintained at 50°C in moderate rpm for about 2 hours (Fig.1). After homogeneous mixing samples are subjected to the process of filtration using a suction pump (Fig.2).



FIGURE 2 Filtration of carbon using suction pump.

The filtered sample are dried in hot air oven for 36 hours at 110°C, after that they are grinded into a fine powder using a mortar pestle. T₁ and T₂ samples were subjected into an activation process in tubular furnace maintained at 700°C for 1.5 hours each sample under a nitrogen atmosphere with a flow rate of 70 ml/min.



FIGURE 3 Sample T₁ and T₂ subjected to drying.

Activated carbon was successfully prepared using NaOH as an activating agent under nitrogen atmosphere. From this research work, the high porous activated carbon can act as an electrode coating material for high-performance supercapacitors application.

2.3. Activated carbon characterization:

The activated carbon samples are subjected to characterization from X-Ray diffraction (XRD) to analyse the crystalline nature of the activated carbon, SEM studies was carried out for studying the microstructure and percentage of porosity, HRTEM analysis was taken to distinguish the particle size distribution. FTIR spectral analysis was performed to know the functional groups of both T₁ and T₂ activated carbon samples.

3. Result And Discussion

3.1 XRD Analysis for activated carbon:

The Structural investigation was carried out for both T₁ and T₂ activated carbon samples using Rapid powder -PXRD and to ascertain the impact of activation temperature for the samples T₁ and T₂. Figure 4 shows the diffraction pattern of both the samples after carbon activation at 700°C for 1.5 hours using tubular furnace. The existences of the required carbon structure were observed in both the samples and found to be amorphous polycrystalline. The existence of predominant planes (002) and (311) confirms that the carbon phase was effectively retained in the sample of activated carbon under nitrogen atmosphere [2,27].

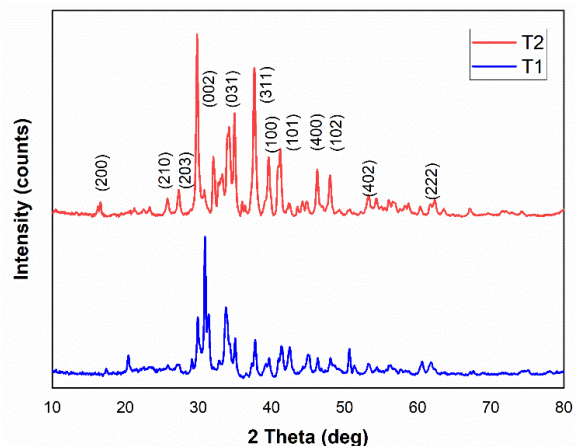


FIGURE4 XRD pattern for T₁ and T₂ samples at 700°C for 1.5 hours.

The spreading peaks for both T₁ and T₂ samples were compared and indexed respectively from JCPDS Card No.(40-1499) of carbon using PCPDF WIN database. More or less there is no identification of any by-product phases, indicating that there is no other elements were present in compound and result in the formation of required carbon phase only[30]. The intensity of peak around 31° with diffraction peaks (002) and at 38° diffraction peaks (311) plane was considered as the predominant peaks for carbon phase [28].

3.2 SCANNING ELECTRON MICROSCOPE (SEM):

The surface morphological studies were carried out for both T₁ and T₂ activated carbon powder samples. Figure 5A and 5B shows the formation of the pores in the carbonized tea waste before activation. Figure 5C and 5D shows the development of the pores in tea waste formed after the activation process carried out at 700°C under nitrogen atmosphere for 1.5 hours. From SEM analysis it is highly discloses that the distribution of the pores size is homogeneous and uniform in nature after activation. The development of pore size will eventually enlarge the surface area of the pores which will be the spot or residence for the accumulation of charge on the surface of activated carbon and also to yield a bulky specific capacitance value [17].

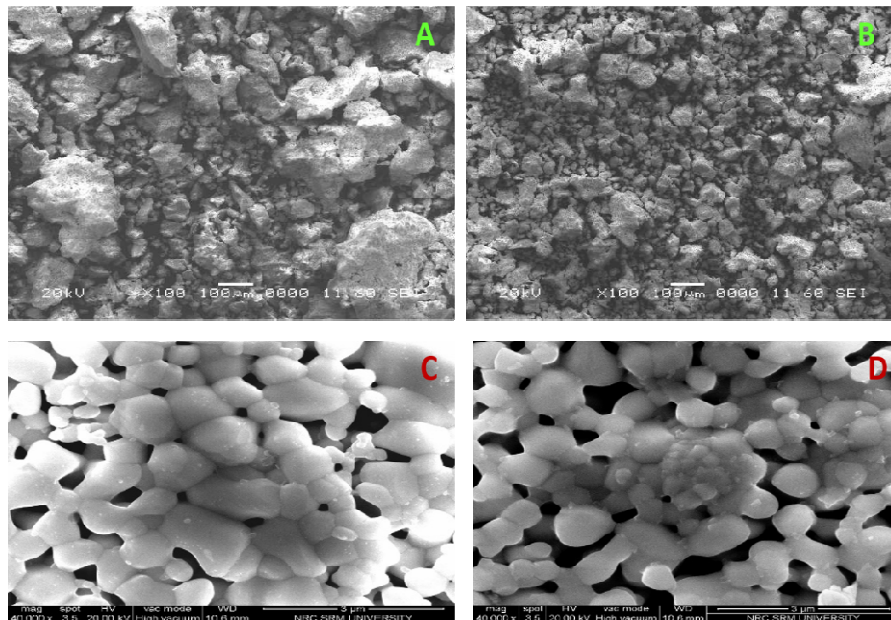


FIGURE 5 SEM image for T_1 and T_2 samples at 700°C for 1.5 hours.

From this SEM results, it was observed that the particles are homogeneously distributed in both T_1 and T_2 carbon activated samples with connected pores. The particles are uniformly interconnected with the small pores as observed in the activated carbon. The small pores with well-connected particle after activation are the essential features for good electrochemical property of a supercapacitor [21].

3.3. HRTEM - PARTICLE SIZE ANALYSIS:

HRTEM analysis was performed for both T_1 and T_2 activated carbon powders under nitrogen atmosphere to know the information about the particle size distribution of chemically activated carbon powder. The JEOL JEM 2100 HRTEM analyzer was used to record the samples. The HRTEM image displayed in Figure. 6a and 6d confirm the particle distribution and the crystalline variations in the carbon powder. The particle size for carbon powder was found to be around 27 nm and are comparable with the values calculated from diffraction results, evidencing the development of carbon particles processed through chemical route. The d-spacing values of both the samples were calculated as 0.31 nm and 0.32 nm shown in Figure 6c and 6f corresponds to the (002) and (311) plane of activated carbon respectively [17,28].

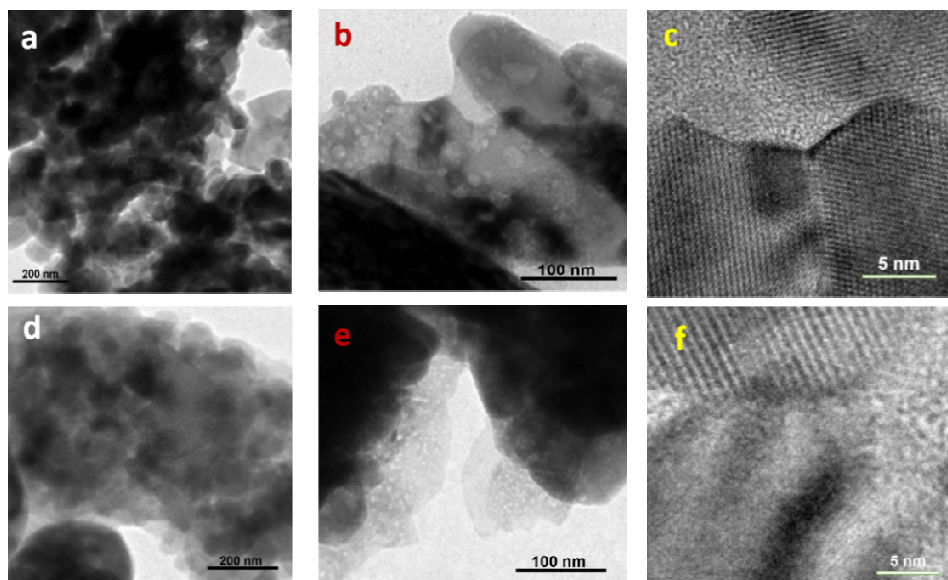


FIGURE 6 High resolution TEM images for T₁(a,b,c) and T₂(d,e,f)samples at 700°C for 1.5 hours.

Figure 6 clearly shows a high-resolution TEM micrograph of the carbon particles processed at 700°C under nitrogen atmosphere for 1.5 hours. From these HRTEM analyses, it was identified that the average distribution of particle in the tea waste powder was determined using the imaging software. The variation of particle size was found to be around 20 nm to 27 nm for T₁ and T₂carbon powder samples respectively studied from TEM image[21].

3.4 FTIR analysis:

FTIR spectral analysis for T₁and T₂carbon activated powders were recorded to study their surface chemistry and the functional groups. The effect of modification in the carbon powder was also analyzed from its FTIR spectra and shown in Figure 4. FTIR spectra was recorded between the wavenumber range 400 - 4000cm⁻¹ using Thermo Nicolet Nexus 670 spectrometer. The activated carbon powders were grounded well using agate mortar pestle and assorted with KBr solution at a mass ration ratio of 1:500. The powders were made into disc shaped pellets of 10mm diameter by pressing them at 10 MPa for 10 min using hydraulic pellet maker. The obtained spectrum was carried out for the activated carbon (T₁) and (T₂) adsorption.

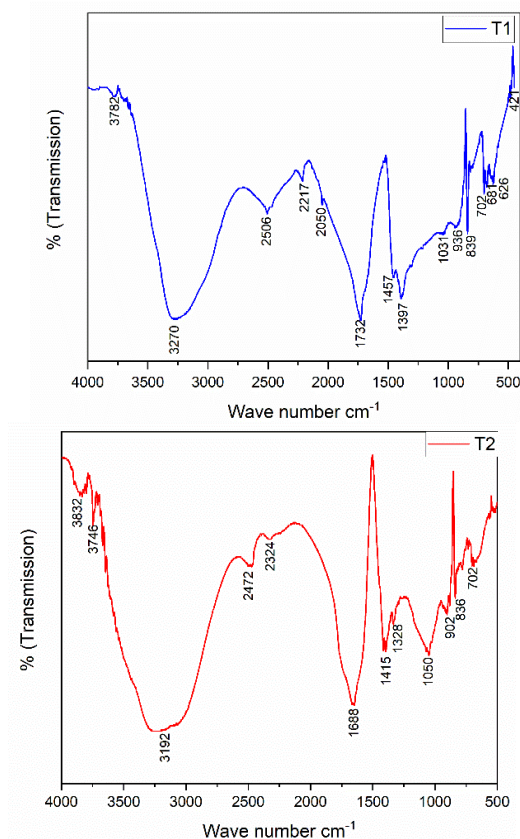


FIGURE 7 FTIR image for T_1 and T_2 samples at 700°C for 1.5 hours.

The modification of the spectrum, drop and loss of some peaks were identified to understand the effect of NaOH adsorption from different functional groups existence. The raise in bands at $3192 - 3270 \text{ cm}^{-1}$ indicates that surface modification of activated carbon has developed some of the surface oxygen functional groups [7,29]. Bands between the range of $2472 - 2506 \text{ cm}^{-1}$ are ascribed as the creation of C-H stretching due to presence of $\text{CH}_2\text{-CO}$ -group. Bands around $2217 - 2324 \text{ cm}^{-1}$ are only due to various C-H stretching modes [16].

The small humps found at 1732 and 1688 cm^{-1} indicate C=C groups present in carbon. A distinct peak at 1397 cm^{-1} was observed for the presence of carbon only. This is due to C=C stretching vibrations in hybridized carbons found in the literature [6,24]. Comparable results were observed using activated maize tassel and mimosa Tannin gel respectively in Pb(II) and Cu(II) [14,15].

4. Conclusions

High absorbent activated carbon (ACT) powder was successfully prepared from tea waste through NaOH activation under nitrogen atmosphere at 700°C for 1.5 hours. Activation using

sodium hydroxide will ultimately increase the size of pore in activated carbon. X-Ray diffraction confirms the amorphous structure of activated carbon, SEM results confirms themorphological variation in the activated carbon powder and shows the pores spots formed only after the activation process carried outin the sample and the pore size distribution is homogeneous distributed for charge storage. HRTEM analysis reveals that how complex is the structure with its carbon layers and about the atomic packing. T₁ and T₂ samples were found to have nearly similar texture but have dissimilar functional groups and zero-point charges. Hence this present investigation providesa novelty easy, cost effective and consistent methods to produce activated carbon from tea waste for high-performance supercapacitor electrode.

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