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Preparation and Characterization of Supercapacitor Electrodes Utilizing Catkin Plant as an Activated Carbon Source

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ABSTRACT
Because of the sustainability, cost-effectiveness, and simplicity of fabrication of meso/microporous carbon synthesized from various precursors, including plants, the production, and application of biomass-derived carbon in energy storage have piqued the interest of researchers. A chemical activation technique involving KOH and carbonization at 600 °C in inert gas was used to successfully synthesize activated carbons (AC) derived from willow catkin (WC). The AC sample was characterized using X-ray diffraction patterns (XRD), Fourier transform infrared spectroscopy (FTIR), Scanning electron microscopy (SEM), Energy dispersive X-ray spectroscopy (EDX), and Transmission electron microscopy (TEM). In 3M KOH aqueous electrolyte, cyclic voltammetry (CV), galvanostatic charge-discharge (GCD), and electrochemical impedance spectroscopy (EIS) were employed to elucidate the electrochemical performance of the AC electrode. The AC electrode has a specific capacitance of 105 F. g-1 and exhibited good cycling stability with a capacitance retention of 89.23% after 1000 cycles. The prepared sample is used as an electrode material for a supercapacitor.

Keywords: *Supercapacitor Electrodes, Activated Carbon, Catkin, Biomass*

1. Introduction

Researchers have been working on developing sustainable energy resources, such as solar, wind, and waves, in response to the rapid depletion of nonrenewable resources and increased concern over environmental degradation in recent years (Wu et al. 2021). The major section of renewable technologies is dominated by the sun, wind, and wave. Other energy sources include biomass, geothermal, hydropower plants, and so on. The main issue with employing renewable sources is their intermittent nature (R. S. Salama et al. 2020; Mannaa, Altass, and Salama 2021; R. S. Salama, El-Bahy, and Mannaa 2021). On the way to getting beyond these restrictions, effective energy storage systems are needed to use and discharge a large quantity of energy from renewable sources. Through an energy storage system (D. Chen et al. 2015), one kind of energy is transferred to another, and then stored and transmitted to other places. In line with this, energy storage technologies like supercapacitors, batteries, and fuel cells are being developed for societal advancement. The general goal of the research is to create a device with high energy and power densities (Ho et al. 2014). This signifies that the energy storage mechanism can store up a huge amount of energy and distribute it rapidly. Batteries and fuel cells are high energy density devices that can store a lot of energy but have a long energy delivery time. Traditional capacitors can intentionally discharge their charge during the required period, but cannot save a significant amount of energy (Zhong et al. 2015; De et al. 2017). As seen, supercapacitor devices can fill the gap between batteries, fuel cells, and traditional capacitors where closing the gap in terms of specific energy and power density (Cherusseri and Kar 2015; Cherusseri, Sharma, and Kar

2016). A supercapacitor includes two electrodes submerged in an electrolyte solution that is separated by a separator. The charge storage mechanism determines the classification of supercapacitors, which can be electric double-layer capacitors (EDLC) or pseudocapacitors (Guan, Yu, and Chen 2016). For electric double-layer capacitors (EDLC), electrical charges are stored by the mechanism of physisorption of the electrolyte ions at the electrode surface (You et al. 2013). Bilayer formation occurs at electrode-electrolyte interactions. Because the only physical activity involved in the charge and discharge mechanism, carbon allotropes such as carbon nanotubes, graphene, carbon fiber, carbon nanofiber, and activated carbon are used as electrode materials in EDLC (Wei and Yushin 2012; Tang et al. 2017). Once a voltage is applied, negative charge ions from the electrolyte are adsorbed by the positive charge at the carbon electrode and vice versa. As a result, a bilayer is formed. Rapid ion movement in the electrolyte results in high power density (Reda S. Salama Salem. E. Samra, Shady M. El-Dafrawy and Awad I. Ahmed 2018; R. Salama et al. 2022; Altass, Ahmed, et al. 2022; R. S. Salama 2019; S A El-Hakam et al. 2013; R. S. Salama et al. 2018). Among all kinds of carbon allotropes, activated carbon (AC) based electrode materials are a vital family of electrode materials owing to the variety of physical and chemical properties such as chemical inertness, electrical conductivity, a range of morphologies, and tunable porosity (Banerjee, Sharma, and Kar 2017). The synthesis of activated carbons (AC) from agricultural waste materials is a novel approach in carbon supercapacitor electrodes that has previously been reported in several journals, including sugar cane bagasse (Konno et al. 2008), discarded coffee beans (Rufford et al. 2008), corn grains (Balathanigaimani et al. 2008), banana fibers (Subramanian et al. 2007), and willow catkins (K. Wang et al. 2015; Xie et al. 2016). These biomasses have tremendous potential for producing advanced engineered carbon materials for use as supercapacitor electrode materials (Singh et al. 2018; J. Deng, Li, and Wang 2016). Additionally, biomass sources are recyclable, sustainable, abundant, and widely accessible across the globe. The chemical activation technique is the most common way to prepare activated carbon (Gao et al. 2017), In which the carbon precursor is combined with an activator (e.g., KOH, $ZnCl_2$, NaOH, K_2CO_3) and then carbonized at a certain temperature in the latter instance (Pezoti et al. 2016; W. Deng et al. 2018; Bedin et al. 2016; W. Chen et al. 2019; Yu, Li, and Wang 2017). In this study, willow catkins (WCs) have been used as a precursor to synthesize unique activated carbon (AC) using a chemical activation method. AC materials can be sonicated to produce a colloidal dispersion that can be used to create a uniform film. AC was characterized by different techniques such as XRD, FTIR, SEM, and TEM. In KOH aqueous electrolytes, cyclic voltammetry (CV), galvanostatic charge-discharge (GCD), electrochemical impedance spectroscopy (EIS), and stability of the activated carbon electrode supported onto graphite sheets are evaluated.

2. Material and Methods

2.1. Materials

Willow catkins WCs were gathered from a river in the Damietta Governorate, Egypt, between March and May 2021. Hydrochloric acid (HCL, 36%) and potassium hydroxide (KOH, 90%) were bought from Alfa Aesar. Polyvinylidene fluoride (PVDF) powder was obtained from Alfa Aesar. Loba Chemie found N-methyl-2-pyrrolidone (NMP, 98%). Ethanol (99.8%) was bought from Fisher Chemical Companies. All chemicals were used with no additional purification. A graphite sheet with a carbon ratio of more than 99.5%, a thickness of 0.3 mm, and a density of 1.1 g.cm-3 was obtained from XRD carbon, China.

2.2. Preparation of activated carbon extracted from willow catkin

The activated carbon from WC was synthesized according to a previously reported approach with some modifications (K. Wang et al. 2015). Firstly, a definite amount of WC is rinsed many times with distilled water to eliminate adhering contaminants and soil. After that, it is dried for 12 hours at 120 °C. The dried WC was completely stirred with a stochiometric ratio of KOH solution (2:1) for 1 hour at room temperature and then overnight dried at 120 °C. The dried material was carbonized for 1 h at a temperature of 600 °C in a nickel crucible under a nitrogen flow of 30 ml min-1 and a heating rate of 5 °C min^{-1}. The resulting material was then washed with distilled water and rinsed frequently with 1 M HCl until the pH of the filtrate was equal to 7. The residue was dried for 5 hours at 80 °C.

2.3. Electrode Fabrication

Firstly, graphite sheets were cut into 1.0 cm*1.0 cm rectangular dimensions. Then, to eliminate the impurities, these sheets were treated with 0.1 M HCl in an ultrasonic bath for 20 minutes and then rinsed with ethanol in an ultrasonic bath for 15 minutes. These sheets were dried at 60 °C for 20 minutes. On the other hand, the nanocomposite working electrode was synthesized by combining 80% of the prepared composites as an active material, 10% acetylene black, and 10% PVDF binder in NMP to form a slurry. Finally, the working electrode was made by depositing the nanocomposite mixture dropwise on the graphite sheet current collector and then drying at 60 °C for 12 hours.

2.4. Characterization of Samples

The XRD-7000 Shimadzu-Japan was used to record X- ray diffraction patterns for activated carbon sample and was performed using a Cu K α radiation X-ray source (λ= 1.5406 °A) which run at 30 mA and 40 kV. The scanning was completed at a 2θ angle from 1 °A to 80 °A, with a step size of 0.02 and a step time of 2 sec. The Fourier transform infrared spectra of activated carbon samples were measured using a spectrophotometer (PerkinElmer-Spectrum 2B, USA) by mixing milligrams of the samples with 0.1g KBr in 30nm diameter self-supporting discs. The surface morphology was studied using scanning electron microscopy (SEM) (JEOL, JSM-IT200) analysis and transmission electron microscopy was performed using (TEM-JEOL, JEM-2100 LaB6).

2.5. Electrochemical Performance

The electrochemical performance of supercapacitor electrode material based on activated carbon derived from willow catkin was investigated in a three-electrode configuration to confirm the prospective applications. So, cyclic voltammetry (CV) and galvanostatic charge and discharge (GCD), the three-electrode configuration were used to assess the electrochemical performance of the as-prepared electrode material in the Scientific Research Laboratory at Delta University for Science and Technology in Egypt, using Metrohm Auto Lab Potentiostat/Galvanostat Instruments. The test was carried out in a 3 M KOH aqueous electrolyte solution. The counter electrode and reference electrode were formed of platinum foil and Ag/AgCl, respectively. The potential windows ranged from -0.8V to 0V. The specific capacitance Cs (F. g^{-1}) of the electrodes was calculated using the following equations using curves of cyclic voltammetry (CV) (equation 1) and galvanostatic charge-discharge (GCD) (equation 2) (Al-Thabaiti et al. 2022), respectively:

$$C_s(F/g) = \frac{\int_{Vi}^{Vf} I\,(V)\,dv}{2\,k.\,m.\,\Delta V} \qquad (1)$$

$$C_s(F/g) = \frac{I.\,t}{m.\,\Delta V} \qquad (2)$$

m is the active material mass loading on a working electrode (gm), V is a potential window range in (V), t is the discharge time (sec), and I is the discharge current (mA).

3. Results and Discussion

3.1. Structural investigation of activated carbon using x-ray diffraction

The X-ray diffraction (XRD) patterns illustrated in Fig. 1 are used to study the crystalline nature of activated carbon (Alshorifi, Alswat, and Salama 2022; Tobbala et al. 2022; Alshorifi et al. 2022; Alshorifi, Ali, and Salama 2022; Altass, Khder, et al. 2021). The XRD of activated carbons agrees well with previous literature, which displayed broad peaks around 27.1° and 43.3° which correspond to (002) and (101), indicating an outstanding alignment of disordered graphitic carbon layers (JCPDS file no-41–1487) (Tongpoothorn et al. 2011; Kalagatur et al. 2017).

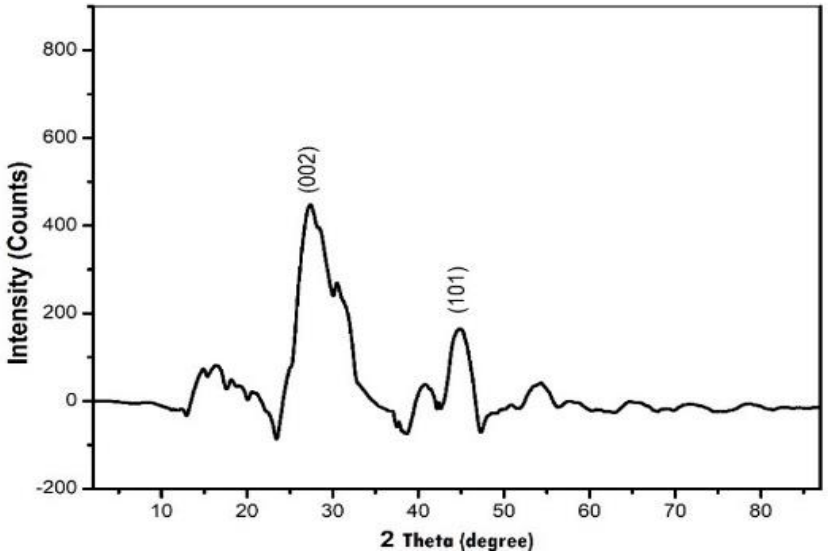

Fig. 1: XRD patterns of activated carbon.

3.2. Structural investigation of activated carbon using Fourier Transform Infrared Spectroscopy

The FTIR spectrum of activated carbon derived from willow catkin is shown in Fig. 2. It contains a broad band at a wavelength of around 3421 cm^{-1} (Bakry et al. 2022; Mannaa et al. 2021; El-Yazeed et al. 2022; Sohier A El-Hakam et al. 2022), which is attributed to the stretching vibrations of adsorbed water molecules on the surface of pure and other prepared nanocomposites (Altass, Morad, et al. 2022, 2021). The peak shown at 1580 cm^{-1} is matched to the C = O stretching of the carboxylic group (Baikousi et al. 2012). The band at 1405 cm^{-1} is assigned to C-H asymmetric and symmetric bending vibrations. The weak peaks in the range between 900 and 1200 cm^{-1} are due to the presence of the C– O group in the sample (Mojoudi et al. 2019). The band found around 870 cm -1 is related to the stretching vibrations of the C−H out of the plane band (Alshorifi et al. 2021; Ibrahim et al. 2021; El-Dafrawy et al. 2020).

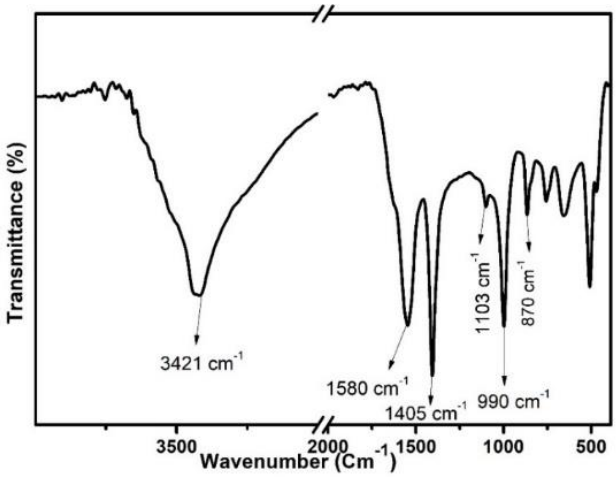

Fig. 2: FTIR spectrum of activated carbon.

3.3. Morphology and elemental analysis of activated carbon

The morphology and microstructural characterization of the prepared AC is shown in Fig. 3. The SEM image of AC derived from WC showed smooth, brittle, and broken sheets of the WC, which matched well with previous work (Xie et al. 2016). As shown in Fig. 4, activated carbon derived from a willow catkin sample contains both carbon (C), oxygen (O), and some traces of sodium (Na), sulfur (S), and silicon (Si) in its structure. These traces are related to metals that exist in willow catkins plants, which also appeared in all the prepared samples.

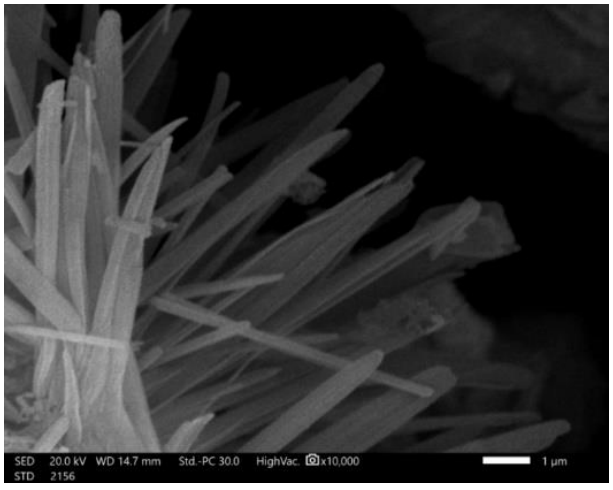

Fig. 3: SEM image of activated carbon.

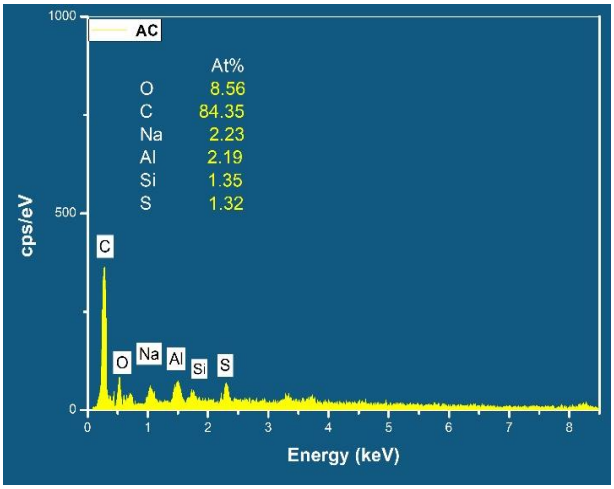

Fig. 4: EDX analysis of activated carbon.

3.4. TEM images of activated carbon

As shown in Fig. 5, the TEM image shows that pure activated carbon derived from willow catkin showed highly porous sheets with rich micropores. This is in good agreement with previous work (K. Wang et al. 2015).

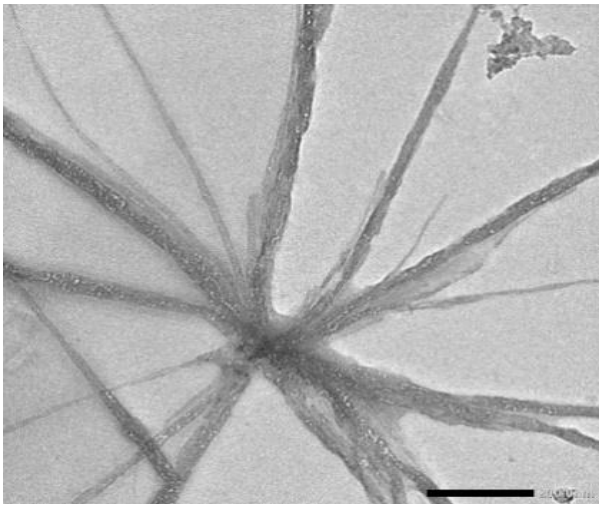

Fig. 5: TEM images of activated carbon

3.5. Electrochemical performance of pure activated carbon electrode

Cyclic voltammetry CV curves are performed with different scan rates: 5, 10, 20, 30, and 50 mV/sec. As shown in Fig. 6, the potential window is chosen to be from -0.8 to 0.0 V. This is consistent with the work performed by Kai Wang (K. Wang et al. 2015). The CV curves of the AC electrode showed a quasi-rectangular shape, which confirms the capacitive behavior of the EDLC (H. Wang and Pilon 2012) and depends only on electrolyte ions which are intercalated into the electrode surface. The specific capacitance of the AC electrode material is calculated from CV curves using equation 1. The results show that specific capacitance increases with a decrease of scan rates due to allowing sufficient time for the electrolyte ions to adsorb and desorb on the electrode surface. It also showed a maximum and a minimum specific capacitance of 129.7 F. g^{-1} at 5 mV/sec and 49.4 F. g^{-1} at 50 mV/sec, respectively.

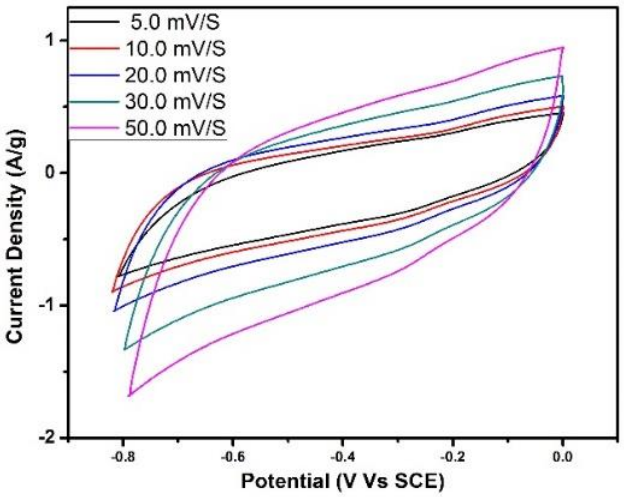

Fig. 6: CV curves of activated carbon at different scan rates.

Galvanostatic charge and discharge GCD curves of AC at different current densities, 1, 1.5, 2, and 2 A. g^{-1} show symmetrical triangles and linearity with a constant slope and high voltage drops due to high internal resistance as shown in Fig. 7. The voltage decreases from its initial value relatively fast at the beginning of the process of discharge and slows down with time, which is characteristic of an EDLC capacitor with good electrochemical reversibility. The specific capacitance of the AC electrode material is calculated from GCD curves using equation 2. It is seen that the AC electrode at a lower discharge current takes a longer time to discharge due to sufficient time for the electrolyte ions to diffuse out of the inner pores of the AC electrode, showing higher charge storage capacity. Specific capacitance is increased from 65.0 F. g^{-1} to 173.3 F. g^{-1}, as the current density is decreased from 2.0 A. g^{-1} to 0.7 A. g^{-1}, respectively.

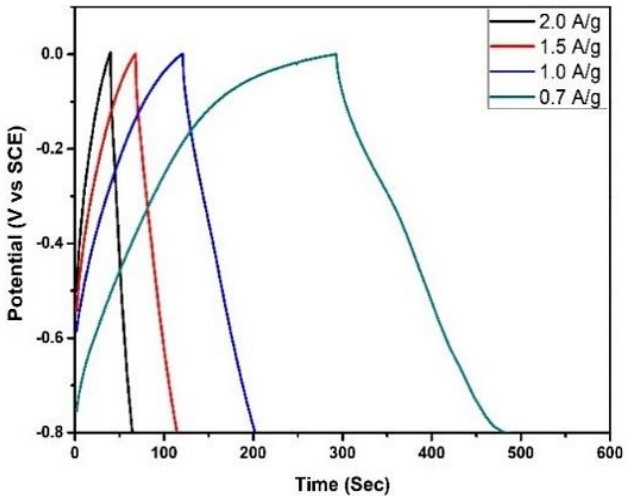

Fig. 7: GCD of activated carbon at different current densities.

EIS is used to study the kinetic properties of the electrode materials. The Nyquist plots shown in Fig. 8 for the AC sample is measured in the frequency range of 100 mHz to 100 kHz under open circuit potential. The result shows a depressed semi-circle at middle frequency due to the electrode's porosity and a steep linear curve at the low-frequency region, facilitating the ions' diffusion. According to the curve, charge transfer resistance (Rct) related to the interface between electrolytes and electrodes is calculated from the semicircle diameter with the capacitive component of the prepared electrodes (Magar, Hassan, and Mulchandani 2021; Sobhani-Nasab et al. 2017). In addition, the ohmic series resistance (Rs) is calculated from the intercept of (Z'-axis), which is related to a combination of the electrolyte's ionic resistance, the intrinsic resistance of the active materials, and contact resistance with the current collector. The values of Rs and Rct are measured by using Z-View software, and the results are equal to 1.39 and 8.18 Ω, respectively. These results confirm the high electrochemical performance of the AC electrode.

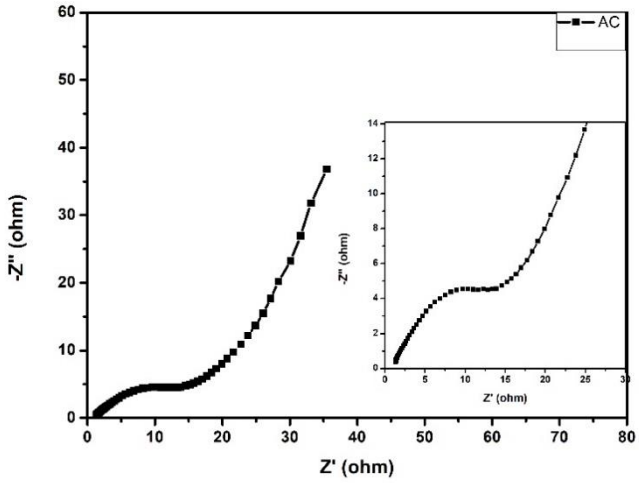

Fig. 8: Nyquist plots as a function of frequency of activated carbon, Inset shows the results in the high-frequency region.

The cycling stability of the electrode is tested by repeating charge-discharge measurements at a constant current density of 2.0 A. g^{-1} for 1000 cycles as displayed in Fig. 9. As the cycle numbers increase for an electrode, the specific capacitance values decrease. The initial specific capacitance is measured at 65 F. g^{-1}, then this value declines to 58.1 F. g^{-1} after 1000 charge-discharge cycles with capacitance retention of 89.23%.

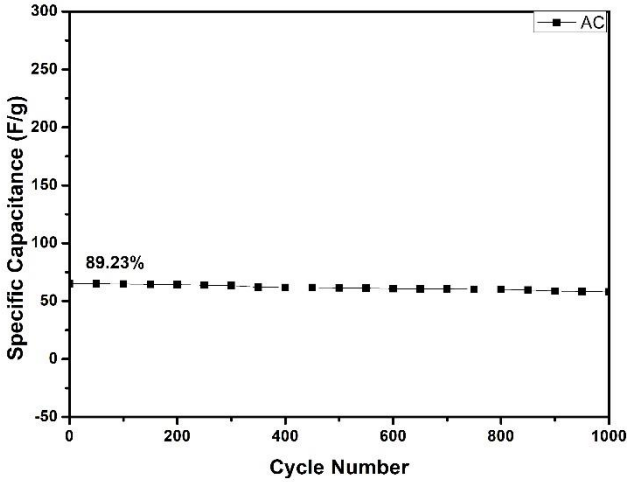

Fig. 9: Cycling stability of activated carbon electrode at a current density of 2 A. g^{-1}.

4. Conclusion

In summary, sustainable and environmentally abundant WC are employed as raw materials to prepare novel porous AC by a simple KOH activation process for high performance supercapacitor electrodes. The AC sample has a high porosity structure. The higher activation temperature leads to carbon materials with a lower moisture content. The resultant activated carbon material, which is carbonized at 600 °C, possesses rich surface heteroatoms doped with functional groups (1.32 wt.% S and 8.56 wt.% O species), a high graphitization degree of 84.35 wt.% C, as well as good electrical conductivity, which results in excellent electrochemical performance due to the contribution of EDLCs and pseudocapacitance. The specific capacitance of the AC electrode is 173.3 F. g^{-1} at a current density of 0.7 A. g^{-1} and 129.7 F. g^{-1} even at a scan rate of 5 mV/sec with capacitance retention of 89.23% over 1000 cycles. This work presents a successful way of preparing high added value of AC from biomass WC for energy storage applications.

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