Preparation and characterization of activated carbon derived from the *Borassus flabellifer* flower as an electrode material for supercapacitor applications

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Activated carbon (AC) samples were prepared from the *Borassus flabellifer* flower (BFF) at different activation temperatures (600, 700, 800 and 900 °C) by a chemical activation method using H₃PO₄ as an activating agent. Scanning electron microscopy (HR-SEM) and X-ray analysis confirmed the surface morphology and the formation of graphite, and the amorphous nature of the activated carbon samples respectively. Fourier-infrared spectroscopy analysis provided the surface functional groups of the activated carbons. The BET specific surface area of the AC samples is found to be 633.43 m² g⁻¹ at an activation temperature of 900 °C. The dc conductivity was determined and the conductivity at ambient temperature was found to increase from 0.012 to 9.64 Ohm⁻¹ cm⁻¹. Electrochemical measurements were carried out using cyclic voltammetry (CV), galvanostatic charge–discharge (GCD) and electrochemical impedance spectroscopy (EIS) with a three-electrode system using 1 M KOH as an electrolyte, the active material (as-prepared AC) as a working electrode, Ag/AgCl as a reference electrode and platinum (Pt) as a counter electrode. The results indicate that BFF can potentially be applied as a precursor material for the production of low cost–high performance activated carbon electrode materials for electric double layer capacitors (EDLCs).

1. Introduction

Depletion of fossil fuels and rapid climate change require sustainable and renewable energy resources. In order to solve these problems, a number of clean, sustainable sources, such as, solar, wind, geothermal energy etc., have been developed. However, renewable energies need to store electric power by using efficient energy storage devices, such as batteries and supercapacitors that are essential to store and use the harvested energy shrewdly. In contrast to batteries and conventional dielectric capacitors, supercapacitors have more advantages, such as, a longer cycle life, a faster charging–discharging rate and better operation safety over other secondary batteries. Supercapacitors is also known as electric double layer capacitors (EDLC) and can carry very high power density and lower energy density than the batteries. For this reason, supercapacitors are mainly appropriate for applications where high power is needed within a few seconds. Compared to typical capacitors, the large electrode surface area in supercapacitors can produce enormous energy storage ability or high capacitance values (expressed in Farads), because a large number of double layers are formed.

One of the most important components of a supercapacitor is the electrode. Usually, conducting polymers, metal oxides, and porous materials, such as activated carbon, carbon aerogels and carbon nanotubes, are used for this purpose. Activated carbon (AC) is widely used as an EDLC electrode, because of its high surface area, better porosity, good thermal and electrical conductivity, good anti-causticity, high stability, low cost and commercial-scale availability. Activation methods and the precursor materials are the two major factors, which may manipulate the final structure and the cost of ACs. The precursor materials of ACs can be various inexpensive agricultural by-products,
such as cherry stones, vine shoots, spirit lees, coconut shells, bamboos, corncob, candlenut shell, acorn shell, pecan shells and lotus stalks.\textsuperscript{5–8}

Palmyrah palm or toddy palm (\textit{Borassus flabellifer}) is an ancient plant belonging to the Arecaceae family. It is broadly distributed and cultivated in tropical Asian countries, such as India, Thailand, Bangladesh, Myanmar, Sri Lanka, and Malaysia. \textit{Borassus flabellifer} produces several agricultural by-products like sweet fruit pulp, seed-shoots, flowers and mature nuts.\textsuperscript{9} Nevertheless, in most cases, the \textit{Borassus flabellifer} flower (BFF) was discarded or thrown away into the soil, which undergoes degradation without any effective utilization.\textsuperscript{10} Hence, the BFF is used as a precursor material to prepare the AC that could result in waste minimization and remarkable cost saving.

Generally two methods can be employed to produce AC: physical or chemical activation. In physical activation, the precursor material is carbonized and then activated at high temperature using either steam or an inert atmosphere. In chemical activation, the precursor material is treated with activants to help the initial hydration. Generally, chemical activation is a preferred route as it achieves higher yield, larger surface area, low operating temperature, and is cost-effective.\textsuperscript{11} The dehydrating effect of the used active agents inhibits the formation of tar, which helps to enhance the yield of porous carbon and to decrease the activation temperature and activation time compared with the physical activation method. The activating agents are important in tailoring the textural properties of activated carbons.\textsuperscript{12}

Among ZnCl\textsubscript{2}, KOH, NaOH and ortho phosphoric acid (H\textsubscript{3}PO\textsubscript{4}), H\textsubscript{3}PO\textsubscript{4} is the most widely used activating agent. Using H\textsubscript{3}PO\textsubscript{4}, numerous studies have been carried out so far\textsuperscript{13} on the preparation of ACs and they have focused on the influence of the concentration of the impregnation solution and the soaking rate to maintain a neutral pH. The washed activated carbon samples were dried under vacuum at 110 °C for 2 h under vacuum conditions. The resulting samples were then heated in a muffle furnace to prepare ACs at different temperatures 600, 700, 800 and 900 °C at a heating rate of 5 °C min\textsuperscript{-1} for 2 h. After the temperature was raised, the activated carbon was washed several times with cold water to maintain a neutral pH. The washed activated carbon samples were dried under vacuum at 110 °C for 24 h and stored in desiccators. The final samples were labeled as BFF 6, BFF 7, BFF 8 and BFF 9 according to the activation temperatures 600, 700, 800 and 900 °C, respectively.

The objective of this study is to investigate the applicability of activated carbons derived from BFF as supercapacitor electrodes. The activated carbon prepared by H\textsubscript{3}PO\textsubscript{4} activation has better surface characteristics. The prepared activated carbons were characterized to determine their structural, pore texture, surface morphology, and electrochemical characteristics. The results were compared with the earlier published findings for activated carbons prepared from other biomass residues and waste.\textsuperscript{14} To the best of our knowledge, no study has been carried out on the preparation of activated carbon from the \textit{Borassus flabellifer} flower (BFF) by the chemical activation method using H\textsubscript{3}PO\textsubscript{4} for supercapacitor applications.

2. Experiment

2.1 Preparation of chemically treated AC

BFF as the precursor material was collected from Periyathachur village, Villupuram district, Tamil Nadu, India. The activated carbon was prepared from the precursor material (BFF) by a two-stage process. The first stage is the pre-carbonization process and the second stage is the chemical activation process. In the first stage, the BFF was washed with distilled water to remove dust, and other impurities and then dried in an oven for 24 h at 110 °C to reduce the moisture content. The resulting BFF was heated at 400 °C at a rate of 5 °C min\textsuperscript{-1} for about 4 h under vacuum conditions and cooled to room temperature at the same rate. This is labeled as pre-carbonized carbon (PCC).

In the second stage, chemical activation was performed by mixing 50 g of dried PCC and agitated with 250 g of aqueous solution containing 83% H\textsubscript{3}PO\textsubscript{4} by weight. The impregnation ratio of chemical activating agent/PCC was fixed as 4:2 and it was homogeneously mixed at 85 °C for 4 h. The resulting slurry was dried in an oven for about 24 h at 110 °C. The resulting samples were then heated in a muffle furnace to prepare ACs at different temperatures 600, 700, 800 and 900 °C at a heating rate of 5 °C min\textsuperscript{-1} for 2 h under vacuum conditions. After the heat was reduced, the activated carbon was washed several times with cold water to maintain a neutral pH. The washed activated carbon samples were dried under vacuum at 110 °C for 24 h and stored in desiccators. The final samples were labeled as BFF 6, BFF 7, BFF 8 and BFF 9 according to the activation temperatures 600, 700, 800 and 900 °C, respectively.

2.2 Characterization techniques

2.2.1 XRD analysis. The main crystalline compounds in the samples were identified by qualitative X-ray powder diffraction (XRD) using a Siemens D5000 X-ray powder diffractometer with characteristic Cu Kz radiation and a scintillation detector. Scans were recorded at a scanning rate of 0.01° s\textsuperscript{-1}, typically in the angle range between 10° and 90°.

2.2.2 FTIR analysis. FTIR analysis was performed to identify the presence of surface functional groups in the BFF activated and carbonized samples. The analysis was conducted in a Shimadzu FTIR-8400S spectrophotometer based on the KBr disk procedure. The spectral data of the samples were recorded in the wave number range from 4000 cm\textsuperscript{-1} to 400 cm\textsuperscript{-1}.

2.2.3 Surface morphology and EDX analysis. Surface morphology studies and EDX elemental analysis of the BFF pre-carbonized and activated carbon sample were performed mutually by using a Quanta 200 FEG high resolution scanning electron microscope (HRSEM). The AC samples were coated with the carbon tape (1 \times 1 cm in size) and the carbon tape was fixed with the gold grid. After that, the gold grid was placed in a gold sputtering device for obtaining a clear resolution of the surface morphology. Then, the sputtered AC sample of the gold grid was placed in the HRSEM sample holder and finally, the AC samples were ready for the HRSEM analysis.

2.2.4 Specific surface area (BET surface area) measurements. Nitrogen adsorption/desorption isotherms were used to measure the specific surface area, pore width, pore volume and pore diameter of the AC samples. The boiling point of liquid nitrogen (−196 °C) was used in the instrument Micromeritics ASAP 2020 BET analyzer device. The samples were degassed under the conditions of a dynamic vacuum at a temperature of 300 °C for 5 h, prior to the experiments.
2.2.5 Electrical conductivity studies. Two probe method systems were used to measure the dc electrical conductivity ($\sigma$) at room temperature. 2 g of porous activated carbon was dried at 110 °C overnight and the dried AC samples were loaded in a hollow Teflon coated cylinder (HTCC) with an inner diameter of 0.98 mm between the two metal plungers. After the AC is loaded in HTCC, the pressure was applied on the AC sample. The compression pressures range from 0 to 549 kPa and the top and bottom of the metal plungers are made up of silver. This is connected to Ametek PARSTAT 4000 to measure the electrical conductivity of the AC samples. During the compression pressures, the measurements were maintained by placing the AC loaded metal plungers (sample holder) and is compressed by hydraulic press.

2.2.6 Electrochemical measurements. Electrodes are composed of 85 wt% of activated carbon, 10 wt% of poly(vinylidene fluoride) (Alfa Aesar) and 5 wt% of carbon black in $n$-methyl pyrrolidinone (Alfa Aesar) as an organic solvent to form a paste and then the slurry is coated onto the nickel foil, which served as a current collector. The AC coated electrode was dried at 70 °C in a hot air oven for 12 h to remove the organic solvents remaining in the micropores of the electrode and then the electrode weight was measured to determine the amount of AC coated on the nickel foil. The typical mass of the electrode material was found to be 0.004 g. Activated carbon coated on the nickel foil, Ag/AgCl and platinum wire were used as the working, reference and counter electrodes respectively. The electrochemical characterization of BFF 6, BFF 7, BFF 8 and BFF 9 was performed using Ametek model PARSTAT 4000 with the three electrode system by using a 1 M KOH electrolyte.

3. Results and discussion

3.1 XRD analysis

Fig. 2 shows the XRD spectra of the pre-carbonized and activated BFF carbon samples at different temperatures of 600, 700, 800 and 900 °C.

All the AC samples showed broad peaks around 23° and 43° and related to (100/101) Bragg reflections. They signify the partially graphitic structure of the activated carbon samples, which also matches with the JCPDS card number 75-2078.15,16 The sharp peak at 26.3° is related to the (002) graphitic plane16 and it indirectly ensures the presence of in-plane conductivity required for electrochemical applications.17 In addition, AC samples exhibit certain sharp peaks at 26.3°, but the sharp peaks are not present in pre-carbonized carbon and it clearly explains that the graphitized carbon is formed only in the AC samples. On careful observation, the sharp peak at 26.3° will be gradually increased if the temperature increases and it confirms that the activated carbon samples are more graphitic in nature.18

3.2 FTIR studies

The FTIR analysis provides the qualitative information on the characteristic functional groups of the adsorbed materials in
the surface of the porous carbons. Fig. 3 illustrates the FTIR spectra of the PCC and BFF activated carbon samples BFF 6, BFF 7, BFF 8 and BFF 9.

A wide band noticed at 3450 cm$^{-1}$ is due to the O–H stretching mode of hydroxyl functional groups, while the band at 2830 cm$^{-1}$ is attributed to C–H interaction with the surface of the carbon. The band at 1350 cm$^{-1}$ indicates the presence of bonds such as Si–O–C, which is due to the presence of sp$^3$ hybridized carbon. The strong band at 1600–1580 cm$^{-1}$ is due to C–C vibrations in aromatic rings. The shoulder at 1080–1070 cm$^{-1}$ can be ascribed to the ionized linkage P$^+$–O$^-$ in acid phosphate esters and to the symmetrical vibration in a chain of P–O–P (polyphosphate). The bands at 775 cm$^{-1}$ is due to the out-of-plane deformation mode of Si–H. The presence of the surface functional groups in the BFF derived activated carbon may be due to the phosphoric acid activation.

3.3 SEM and EDX studies

Fig. 4a–e shows the SEM morphology of PCC and the activated carbon samples at four distinct temperatures of 600, 700, 800, and 900 °C.

The SEM micrograph of PCC (Fig. 4a) shows a flat surface without pore development. Honeycomb-like morphology and lots of nanopores are found in Fig. 4e. Comparatively, the SEM micrographs of the sample BFF at four different temperatures showed an increase in the pore development as depicted in Fig. 4(b–e). It is revealed that initially the activated carbon has an intensive number of pores and after activation the carbon has an extensive number of pores with uniform size. The nanopore shaped channels have distinct sizes that are helpful for electrolyte exchange. Fig. 4(b–e) illustrates that the activation process had occurred on the surface of the pore, while the activating operator H$_3$PO$_4$ has covered and penetrated through the surface of the pre-carbonized samples. This phenomenon is mainly due to the release of volatile matter and reaction between the activating agent and the carbon atom in the precursor. At lower temperature, the reaction proceeded at the interface of the pre-carbonized carbon and showed the presence of little pores only. With an increase in temperature, the small ones gradually became larger and resulted in the formation of micro and mesopores like morphology. As a result of the creation of pores, there is an increase in both the surface area and the pore volume, which are stably created in the activated carbons. Fig. 4f depicts the energy dispersive X-ray (EDX) spectrum of BFF 9, which shows the presence of elemental composition of carbon and oxygen only. The component gold (Au) crest was seen at 2 keV and it indicates that gold sputtering has been carried out on the sample, when SEM images were taken.

3.4 Nitrogen adsorption–desorption isotherms

The nitrogen adsorption–desorption isotherms of the prepared BFF activated carbons with different temperatures are shown in Fig. 5. Samples BFF 6 and BFF 7 showed type I isotherms, whereas BFF 8 and BFF 9 showed type IV isotherms with an H3 hysteresis loop. Comparatively, sample BFF 9 exhibits a broader hysteresis loop from $P/P_0 = 0.45$ to $P/P_0 = 1$, which is due to the formation of additional mesopores.

These results indicate the mesoporous adsorption of the activated carbon samples and signify the formation and increase of mesoporosity as the temperature increases. The reason is that the action of phosphoric acid on BFF carbon essentially leads to the formation of mesopores, because, a majority of micropores would be burnt off as a result of the high activation temperature i.e. many small pores inside the carbon walls were gradually destroyed with the increasing temperature as observed by Chunzhong Li et al. Details of pore characteristics of BFF activated carbon samples are listed in Table 1.

From Table 1, the activated carbons’ pore structure is a combination of micropores and mesopores. From the results, it can be seen that the BET surface area of BFF 9 was 633.43 m$^2$ g$^{-1}$ which is higher than those of the other AC samples. This result
indicates that the H₃PO₄ activation greatly enhances the pore development during the activation process. This evidence is also supported by the pore size distribution of the activated carbons as shown in Fig. 6.

In chemical activation using H₃PO₄ as the activating agent, at a higher temperature of 900 °C, the reaction between H₃PO₄ and carbon occurs. Then, phosphorous will intercalate into the carbon matrix, thus resulting in the widening of the spaces between carbon atomic layers and increases the total pore volume.

<table>
<thead>
<tr>
<th>Sample</th>
<th>S_BET (m² g⁻¹)</th>
<th>S_mic (m² g⁻¹)</th>
<th>S_meso (m² g⁻¹)</th>
<th>V_Total (cm³ g⁻¹)</th>
<th>V_Micro (cm³ g⁻¹)</th>
<th>V_Meso (cm³ g⁻¹)</th>
<th>D_p (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BFF 6</td>
<td>463.06</td>
<td>287.10</td>
<td>176.84</td>
<td>0.329</td>
<td>0.140</td>
<td>0.189</td>
<td>7.3</td>
</tr>
<tr>
<td>BFF 7</td>
<td>555.60</td>
<td>270.39</td>
<td>285.20</td>
<td>0.283</td>
<td>0.122</td>
<td>0.161</td>
<td>6.4</td>
</tr>
<tr>
<td>BFF 8</td>
<td>583.09</td>
<td>245.87</td>
<td>337.22</td>
<td>0.265</td>
<td>0.117</td>
<td>0.148</td>
<td>5.4</td>
</tr>
<tr>
<td>BFF 9</td>
<td>633.43</td>
<td>224.16</td>
<td>409.26</td>
<td>0.202</td>
<td>0.107</td>
<td>0.095</td>
<td>5.2</td>
</tr>
</tbody>
</table>


![Fig. 4](image1.png)

Fig. 4  SEM micrographs of (a) PCC, (b) BFF 6, (c) BFF 7, (d) BFF 8, (e) BFF 9 and (f) EDX analysis of BFF 9.

![Fig. 5](image2.png)

Fig. 5  Nitrogen adsorption–desorption isotherms of BFF activated carbon samples.

![Fig. 6](image3.png)

Fig. 6  Pore size distribution of BFF activated carbon samples.
4. Electrochemical characterization

4.1 Electrical conductivity studies

In the supercapacitor applications, electrical conductivity studies (EC) play a precious role in identification of the electrical conductivity nature of the activated carbon samples. In this EC study, we use a two probe method system. The sample holder HTCC is fitted with the two metal plungers that are made of silver metal. In the HTCC, one metal plunger is movable and another one is immovable and the as-prepared AC samples were loaded in the HTCC. After loading the sample, the pressure was applied to the HTCC in the range of 0 to 549 kPa. Finally, HTCC is connected to the instrument Ametek PARSTST 4000 that was used to measure the electrical conductivity of the AC samples (BFF 6, BFF 7, BFF 8 and BFF 9). The input voltage of 0 to 10 V is applied to obtain the output voltage from the AC sample. The electrical conductivity was calculated by using eqn (1) as shown below.

\[
\sigma = \frac{L}{RA}
\]

where \(\sigma\) is the conductivity, \(R\) is the resistance (\(\Omega\)), \(A\) is the area of the sample (\(\text{cm}^2\)) and \(L\) is the sample height (cm). The calculated conductivity is ohmic in nature, the ohmic conduction of the AC samples, BFF 6, BFF 7, BFF 8 and BFF 9, at a compression pressure of 0 kPa were \(8.02 \times 10^{-5}\), \(1.5 \times 10^{-4}\), \(3.28 \times 10^{-4}\) and \(5.28 \times 10^{-3}\) Ohm\(^{-1}\) m\(^{-1}\), respectively. And also at a compression pressure of 549 (kPa), it was \(10.02 \times 10^{-2}\), \(2.2 \times 10^{-1}\), \(3.28 \times 10^{1}\) and \(9.64 \times 10^{3}\) Ohm\(^{-1}\) m\(^{-1}\) respectively. The above data indicate that the BFF 9 activated carbon samples have high electrical conductivity, because BFF 9 has a higher specific surface area than the other AC samples. The high specific surface area enhances the mobility of ions into the pores faster to achieve larger electrical conductivity.33 Fig. 7 shows the electrical conductivity of the samples, BFF 6, BFF 7, BFF 8 and BFF 9, which indicates that the BFF 9 sample has higher electrical conductivity than the other activated carbon samples.

4.2 Cyclic voltammetry studies

Cyclic voltammetry (CV) is a viable electrochemical technique and it is normally embraced to measure the specific capacitance \(C_s\) of electrode materials for supercapacitors.34 The CV curves of all activated carbon samples (BFF 6, BFF 7, BFF 8 and BFF 9) are displayed in Fig. 8a–d and it is recorded at different scan rates (5 to 100 mV s\(^{-1}\)). The CV measurement studies are carried out in the three-electrode cell framework system and the specific capacitance, \(C_s\), can be calculated from eqn (2),

\[
C_s = \frac{\Delta I}{2Vm(V_2 - V_1)} \quad \therefore \Delta I = I_a - I_c
\]

Herein, \(\Delta I/2\) means the half of the integration area of the CV curve, \(I_a\) and \(I_c\) are the anodic and cathodic current, \(V\) stands for the scan rate, \(m\) represents the mass of the active material present in the electrode, \(V_2 - V_1\) is the given potential difference and \(C_s\) is the specific capacitance. In Fig. 8a–d, the CV curves are almost similar and quasi rectangular in shape. It indicates that the capacitive behaviour of the AC samples observed from the formation of an electrical double-layer capacitor (EDLC) is like an ideal supercapacitor. But Fig. 8d (BFF 9) shows that the CV curve area is larger and also the potential window would be very low compared to the other AC samples. It can be concluded that BFF 9 displays the highest \(C_s\) value (234.4 F g\(^{-1}\)) at low scan rates and also withstand up to higher scan rates. The good capacitive response of BFF 9 in a 1 M KOH aqueous electrolyte originates from the blend of electrical double layer capacitance (EDLC) and pseudo-capacitance35 caused by its well-developed pores and a large surface area as shown in Table 1. If the temperature increases, the specific capacitance also increases, because at a higher temperature, the BFF 9 AC sample shows a high BET surface area with better mesoporosity.
KOH electrolyte ions with high mesoporous nature mobility move faster into the AC electrode and it is easy to form an electric double layer more statically that in turn representing the higher specific capacitance.

### 4.3 Galvanostatic charge–discharge studies

A galvanostatic charge–discharge (GCD) study is the most efficient measurement to measure the specific capacitance of AC samples. It is also well known that the capacitance of electrode materials can be calculated on the basis of eqn (3),

$$C_s = \frac{I \times \Delta t}{m \times \Delta V}$$

where $C_s$ is the capacitance (F g$^{-1}$), $I$ is the current density (A g$^{-1}$), $\Delta t$ is the charging/discharging time (s), $m$ is the mass of the active material (g) and $\Delta V$ is the working potential window of the electrode (V s$^{-1}$). The galvanostatic charge–discharge curves of the AC electrode samples (BFF 6, BFF 7, BFF 8 and BFF 9) at different current densities (1 to 5 A g$^{-1}$) are shown in Fig. 9a–d.

In general, the GCD curves are expected to be in a triangular shape. But, we have observed in Fig. 9a that it is not perfectly triangle in shape at a high current density, instead a slight linear plane is formed between the charge–discharge curves due to the small internal resistance$^{36}$ in BFF 6. All activated carbon samples show the slight linear plane, but decreases from the high current density (5 A g$^{-1}$) to low current density (1 A g$^{-1}$). Furthermore, when the activated temperature increases the slight linear plane formation is decreased and finally, the BFF 9 sample showed the perfect triangular shapes of GCD curves at all current densities (1 to 5 A g$^{-1}$) and it is revealed that there is no internal resistance which in turn helps in increasing the specific capacitance value than the other AC samples. Evidently, the calculated $C_s$ values of GCD and EIS using eqn (3) and (4) were successfully matched with the calculated $C_s$ values of cyclic voltammetry as listed in Table 2.

Also, the results showed that the BFF 9 sample has a higher $C_s$ value at low current densities than at higher current densities. On the other hand, BFF 6, BFF 7 and BFF 8 AC samples have low $C_s$ values. This may be due to the higher surface area of BFF 9 that allows a large amount of electrical charge to accumulate on the electrode and electrolyte interface. The small pore size provides more EDLC formation,$^{37}$ because it shortens the ion transport length and makes the ion diffusion easier to the porous activated carbon and thereby enhances the specific capacitance. We attribute this high specific capacitance value to the hierarchically porous structure of BFF 9, which facilitates the electrolyte ions accessing the interior surfaces of the electrode and leads to an increased charge storage system.$^{38}$

### 4.4 Electrochemical impedance spectroscopy

Electrochemical impedance spectroscopy (EIS) of the porous activated carbon electrode in 1 M KOH was employed within the frequency range of 1 mHz to 100 kHz with a 5 mV AC voltage. Electronic resistance should be related to the semicircle as present at the high frequency region as shown in the Nyquist plot (Fig. 10).
Hence, the electronic resistance includes the equivalent series resistance of the electrode, charge transfer resistance of the electrode and solution resistance of the electrolyte. The slope of the line is close to 45° of the middle frequency and was ascribed to the diffusion of the electrolyte ions in the electrode pores, while the oblique line at a low frequency corresponds to the capacitive behavior.\textsuperscript{39} The fitted equivalent circuit impedance spectra are shown in Fig. 10 (inset). The charge transfer mechanism surprises the solution resistance ($R_s$, internal resistance of the electrode materials and ohmic resistance of the electrolyte) in series with parallel coupling of the double layer capacitance ($C_{dl}$), interfacial charge-transfer resistance ($R_{ct}$) and the Warburg impedance ($W$). The $R_s$ values for all the samples were obtained in the range of 0.35–1.11 $\Omega$ which is attributed to the resistance of the KOH electrolyte. The second factor $R_{ct}$ for BFF 6 is 7.65 $\Omega$ and for all the other activated carbon samples they are below 5 $\Omega$. A lower value of $R_{ct}$ leads to the shortening of the ion diffusion path which reflects the higher charge discharge performance and also the third factor $C_{dl}$ provides the tidings of the electrode capacitance behaviour. The specific

### Table 2  Specific capacitance from cyclic voltammetry (CV) and galvanostatic charge–discharge of BFF AC samples

<table>
<thead>
<tr>
<th>BFF AC samples</th>
<th>Specific capacitance (CV) (F g$^{-1}$)</th>
<th>Specific capacitance (GCD) (F g$^{-1}$)</th>
<th>Specific capacitance (EIS) (F g$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BFF 6</td>
<td>32.8</td>
<td>35.4</td>
<td>38.5</td>
</tr>
<tr>
<td>BFF 7</td>
<td>88.4</td>
<td>90.7</td>
<td>92.3</td>
</tr>
<tr>
<td>BFF 8</td>
<td>125.8</td>
<td>124.7</td>
<td>122.4</td>
</tr>
<tr>
<td>BFF 9</td>
<td>234.4</td>
<td>238.2</td>
<td>236.5</td>
</tr>
</tbody>
</table>

BFF 6 is 7.65 $\Omega$ and for all the other activated carbon samples they are below 5 $\Omega$. A lower value of $R_{ct}$ leads to the shortening of the ion diffusion path which reflects the higher charge discharge performance and also the third factor $C_{dl}$ provides the tidings of the electrode capacitance behaviour. The specific
The capacitance of the electrodes can be evaluated by using the low frequency data of the Nyquist plot and it is calculated by eqn (4)

$$C_{dl} = \frac{-1}{2\pi f Z_{im}}$$  \hspace{1cm} (4)

where ‘f’ is the frequency and ‘Z_{im}’ is the imaginary part of the impedance which is obtained from the y axis of the Nyquist plot.\textsuperscript{39,40} Warburg impedance values are obtained in the range of 0.033–1.07 Ω corresponding to the electrolyte ion diffusion into the porous network of the activated carbon electrode material.\textsuperscript{41,42} The Warburg impedance contributes little to the total impedance signifying the good contact for the electrolyte ions into the activated carbon material even at higher frequencies. In conclusion, the calculated ohmic resistance values of EIS are shown in Table 3 and it is better evidence for the calculated specific capacitance of CV, GCD and EIS spectra.

4.5 Cyclic stability and scan rate capability

For supercapacitor applications, the long-term cycling stability is also one of the important aspects. The cycling stability of the three composite electrodes was examined by consecutive charge–discharge cycles at a current density of 1 A g\textsuperscript{-1} (Fig. 11).

The total charge and discharge processes of BFF 9 took 400 s for each cycle, which still shows a fast charge–discharge rate. The specific capacitance of BFF 6 remains at 76.9% after 1000 charge–discharge cycles. However, the BFF 9 AC sample shows a slight drop during the first 300 cycles and subsequently remains stable. The final capacitance of the BFF 9 AC sample is still much higher than those of the other AC samples, and it

![Graph](image-url)
maintains the capacitance retention up to 90.2% after 1000 charge–discharge cycles as shown in Fig. 12.

The enhanced cycling stability may come from the synergistic effect between the active material (working electrode) and electrolyte interfaces. High micro and mesoporous nature of the BFF 9 AC samples prevents continuous degradation, during the charge–discharge process, which withstands strain/stress relaxation during the electrochemical reaction but also provides a stable channel for electron transportation.43

The most important function in cyclic voltammetry is the scan rate capability and the specific capacitance details are provided in Fig. 13.

Samples BFF 6, BFF 7, BFF 8 and BFF 9 activated at different temperatures show various results in the specific capacitance, due to their different specific surface areas, but the samples without H3PO4 activation have negligible specific capacitance because of their very small surface areas. When the scan rate is increased, the specific capacitance gradually dropped and the specific capacitance of BFF 9 became higher than those of the other AC samples (BFF 6, BFF 7 and BFF 8) after a scan rate of 50 mV s⁻¹, due to its slightly larger pore size. A larger pore size has the advantage of easy access to electrolyte ions at a higher scan rate.44 Also, for sample BFF 9, the microporous nature is decreased, and mesoporosity is increased, when the activation temperature is raised.

5. Conclusion

In the present work, activated carbons were produced from the *Borassus flabellifer* flower (BFF) by chemical activation with H3PO4 in an atmosphere. With the increase of the activation temperature from 600 to 900 °C, BET surface areas and the specific capacitance of produced activated carbon were gradually increased. The optimal conditions for the activation of the *Borassus flabellifer* flower (BFF) AC are estimated at an activation temperature of 900 °C 2 h, giving a BET surface area of 633.3 m² g⁻¹ with a high micro-mesoporous area and this porous nature leads to the formation of an efficient electric double layer over a large surface. More EDLC layer formation shows a good capacitive behavior and the specific capacitance (Cₛ) value is 238.2 F g⁻¹ at a current density of 1 A g⁻¹. This hierarchical nature of activated carbon with a very high specific surface area could find use in supercapacitors, batteries, and fuel cells. In future, biodegradable waste will be used for the industrial scale preparation of activated carbons and will play a significant role in supercapacitor applications.

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Notes and references