REGULAR ARTICLE

Biomass derived graphene-like activated and non-activated porous carbon for advanced supercapacitors

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MS received 19 October 2016; revised 15 January 2017; accepted 31 January 2017

Abstract. Graphene-like activated and non-activated carbon nanostructures were synthesized from various natural sources like sugar, rice husk and jute. These carbon nanostructures were characterized using SEM, FTIR and Raman spectroscopy, surface area and thermogravimetric analysis. The electrochemical studies of these carbon materials confirm their promising characteristics for supercapacitor applications. Activated carbon nanostructures exhibit higher specific capacitance compared to that of non-activated carbons (non-Ac sugar). The activated carbon (Ac-jute) exhibits maximum specific capacitance of 476 F/g at an applied current density of 0.2 A/g which is much higher than that of graphene oxide (GO).

Keywords. Graphene; biomass; supercapacitor; porous carbon; energy storage.

1. Introduction

Energy storage is the primary component of sustainable energy systems. Renewable energy sources like photovoltaics, wind turbines and hydroelectricity are not yet adopted as primary energy sources. Energy storage technologies have the potential to store these renewable energy and then provide the access to the energy on demand. Energy storage systems also play a very crucial role in energy transportation. Currently, the main energy storage systems are batteries where energy can be stored electrochemically and the chemical reactions can release the energy in terms of electrical carriers which can be extracted to the external circuit. However, batteries have low power density and are unable to retain its storage ability over time. Electrochemical capacitors, mainly supercapacitors or sometimes Ultracapacitors, are similar to batteries but store energy electrostatically rather than in chemical form, and do not involve any chemical reactions as in batteries.^{1,2} The supercapacitors store electrical charge on conducting materials having very high surface area. The main features of supercapacitors are, (a) high power density and can be charged very quickly, and (b) they do not lose their energy storage ability over time as no chemical degradation takes place. Unlike batteries, the low energy density of the supercapacitors is the main limitation.

Supercapacitors are divided into two categories: electrochemical double layer capacitors (EDLC) and pseudocapacitors. In EDLCs, charge accumulation takes place at the interface between electrolyte and electrode and in pseudocapacitors, conducting materials *e.g.*, metals, metal oxides and organic polymers are involved which follow Faradaic mechanism to accumulate charge.³ Mainly, carbon based materials with high specific surface area are involved in EDLCs. Carbon based supercapacitors have been investigated and used most widely because of their high surface area, ease of processability, electrochemical inertness, controllable porosity and low cost.⁴ Different carbon materials, like activated carbon sheets, carbon nanotubes, porous templated carbons, carbon fibers, graphene and graphene oxides, have been investigated extensively as EDLCs.⁵ The maximum specific capacitance of 550 F/g can be obtained from graphene. Single layer graphene having specific surface area of ~ 2630 m²/g exhibits the gravimetric capacitance of 355 F/g.⁶ Rouff *et al.*, have demonstrated that KOH activation of microwave exfoliated GO exhibits a specific surface area of 3100 m²/g which is beyond the theoretical limit.⁷ A simple DVD LightScribe writer was used for Laser reduction of graphite oxide to graphene having specific surface area of 1520 m²/g and conductivity of 1738 S/m.⁸ Activated carbon with high surface area can be derived from organic precursors having high carbon content by carbonization followed by oxidation in CO₂, water vapor and KOH. Various porous carbon materials with

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varying pore size and pore structure can be synthesized using templates. For example, Zeolite has been used for synthesis of ordered microporous carbon materials and mesoporous silica nanoparticles (MCM-48, HMS, SBA-15, MCF, and MSU-X) were used for the synthesis of mesoporous carbons having ordered pore structure.⁹ Li *et al.*, have discussed the properties of different types of carbons with their dimensionality and cost.¹⁰ The double layer capacitance of activated carbon is in the range of 100–120 F/g in organic electrolyte and in the range of 150–300 F/g in aqueous electrolyte.¹¹ Graphene-like porous hollow carbon materials having high specific surface area and pores have been shown to exhibit high specific capacitance than that of solid microspheres.¹² Multilayer carbon nanotubes¹³ have been shown to be a possible supercapacitor material with specific capacitance of ~ 135 F/g. Nitrogen doped carbon nanostructures have been shown to exhibit specific capacitance of 300 F/g in acidic electrolyte.¹⁴ Beguin *et al.*, have reported the specific capacitance of various activated carbon by varying specific surface area from 800 m²/g to 3000 m²/g, a maximum specific capacitance of 357 F/g was obtained.¹⁵

Biomass derived carbon materials have been investigated for supercapacitor applications.^{16–18} N-rich carbon derived from proteins exhibit supercapacitor properties with specific capacitance of 390 F/g.¹⁶ Microporous carbon nanoplates were synthesized from regenerated silk and it exhibited specific capacitance of 264 F/g in aqueous electrolyte.¹⁹ 3D dendritic-like hierarchical porous carbon aerogels exhibit good specific capacitance of 368 F/g at 0.05 A/g in KOH.²⁰ Lotus seedpods-derived porous carbon shows outstanding capacitive behavior with 402 F/g at 0.5 A/g due to high surface area, optimum pore size and high oxygen containing functionalities.²¹ Activated porous carbon synthesized from biomass Paulownia flower shows specific capacitance of 297 F/g at 1 A/g current density in H₂SO₄ electrolyte.²² Gelatin was mixed with boric acid and annealed to get B/N co doped carbon.²³ which exhibits specific capacitance of 358 F/g at current density of 0.1 A/g. Wan *et al.*, have shown higher specific capacitance of 306 F/g of corn husk derived carbon at 10 A/g current density compared to other biomass derived carbons.²⁴ Recently, rice husk-derived activated carbon having surface area of 2696 m²/g exhibited specific capacitance of 147 F/g whereas another report on activated carbon (surface area = 2681 m²/g) from rice husk using NaOH showed specific capacitance of 198.4 F/g.^{25,26} Carbon material synthesized from natural cellulose at 1000°C have been shown to exhibit supercapacitor properties with specific capacitance of 107 F/g.²⁷

Here, we have synthesized graphene-like activated and non-activated carbon sheets from starch. Similarly, we have also synthesized activated carbon nanostructures from natural sources like jute and rice husk. The supercapacitor properties of these carbon materials have been investigated and the specific capacitance was compared with that of graphene oxide and reduced graphene oxide.

2. Experimental

2.1 Materials and synthesis procedure

Activated and non-activated carbon nanomaterials have been synthesized from different natural sources *e.g.*, sugar, jute and rice husk.

Interconnected activated and non-activated carbon sheets have been synthesized from starch. Briefly, starch (2 g) was ground well with KOH (1 g) and heated at 200°C for 1 h followed by heating at 750°C for 3 h in N₂ atmosphere. Later, the product was washed with dil. HCl solution. To get non-activated carbon sheets, 2 g of starch and 1 g of pluronic F127 were mixed together in 50 mL water and then dried to powder. The powder was ground well and heated at 200°C for 1 h followed by heating at 750°C for 3 h in N₂ atmosphere.

Jute fibers (3 g) were added to a mixture of conc. H₂SO₄ (40 mL) and water (10 mL) resulting in a black slurry which was then diluted with 500 mL water. The product was filtered and washed with water several times. The dry powder was ground well with KOH (1 g) and heated at 750°C for 3 h in N₂ atmosphere. The resulted activated carbon from jute was washed with dil. HCl solution.

Rice husk was mixed with 10 g of NaOH in 300 mL water and stirred for overnight. The material was filtered and dried. Dried husk (4 g) was mixed with 1 g of KOH and heated at 200°C for 1 h followed by heating at 750°C for 3 h in N₂ atmosphere. The product was washed with dil. HCl solution followed by HF to etch out SiO₂ from the product.

Graphene oxide and reduced graphene oxide have been synthesized from graphite oxide as reported earlier.²⁸ Briefly, graphite has been oxidized to get graphite oxide and then it was exfoliated using sonication to get graphene oxide. Again, the graphite oxide was reduced and exfoliated at high temperature to get reduced graphene oxide.

2.2 Characterization techniques

A field-emission scanning electron microscope (FESEM, FEI 3G Quanta) operated at 20 kV was used for the surface morphology analysis. FTIR spectrum was recorded in a Cary 660 FTIR spectrometer (Agilent). The samples were mixed with KBr and pelletized for the measurements. Raman spectra were recorded in a Confocal Raman spectrometer (model: Xplora, Horiba JobinYvon) using a Laser excitation at 785 nm. Thermogravimetric analysis (TGA) were carried out in the temperature range of 50–900°C on PerkinElmer Pyris

Diamond TGA/DTA system in N_2 atmosphere at a heating rate of $10^\circ\text{C}/\text{min}$. Surface area of the samples were calculated from the N_2 gas adsorption profile measured in a Quantachrome 2000e instrument.

2.3 Electrochemical measurements

Electrochemical measurements have been carried out using a three electrode system in Autolab (PGSTAT302N) electrochemical setup in 0.5 M H_2SO_4 electrolyte. Ag/AgCl (sat. KCl) and Pt wire were used as reference and counter electrodes, respectively. The working electrode was prepared on glassy carbon electrode (0.03 cm^2). 5 mg of catalyst was dispersed in a mixture of isopropanol ($300\text{ }\mu\text{L}$), water ($180\text{ }\mu\text{L}$) and Nafion ($20\text{ }\mu\text{L}$). A drop of the mixture ($5\text{ }\mu\text{L}$) was placed on a glassy carbon electrode ($\sim 0.03\text{ cm}^2$) and air-dried for 10 min. Mass loading of the catalyst on the electrode was $1.6\text{ mg}\cdot\text{cm}^{-2}$. Cyclic voltammetry at different scan rates and galvanostatic charge-discharge at different current densities were carried out.

3. Results and Discussion

Carbon nanostructures were synthesized from starch by carbonization at 200°C followed by heating at 750°C in N_2 atmosphere. Here, the non-activated carbon refers to carbon synthesized in the absence of any chemical activation. The oxygen containing functional groups in biomass will be removed during carbonization process. Overall, the non-activated carbon will have less functionalities and random defects leading to low conductivity. KOH was mixed with starch before carbonization to get activated carbon nanostructures. Chemical activation of carbon materials leads to generation of porous nanostructures with high specific surface area, defined distributions of microstructures and pore size. There are several chemicals like KOH, H_3PO_4 , $ZnCl_2$, etc., which are used for chemical activation of carbon materials. These activation processes proceed *via* solid-solid reactions followed by solid-liquid reactions and they produce metallic potassium in the mixture which oxidizes carbon into carbon oxides and carbonates. There are other mechanisms like KOH transforming into K_2O at 400°C which then reacts with CO_2 to produce K_2CO_3 . This chemical activation leads to porous carbon materials. KOH activation of carbon based materials have been reviewed earlier in detail.²⁹ Similarly, jute fibers and rice husk were treated with H_2SO_4 and NaOH, respectively, followed by carbonization in presence of KOH (Figure 1).

All these different carbon nanostructures were characterized after washing with dil. HCl solution. Rice husk contains silicates and during carbonization SiO_2

formation takes place. The carbon product from rice husk was finally treated with HF to remove SiO_2 impurity. The electrochemical properties of these carbon nanostructures were compared with that of graphene oxide and reduced graphene oxides which were synthesized from graphite.²⁸ The SEM images show sheet-like morphology of the carbon materials (Figure 2). Figures 2a and 2b show the sheet-like morphology of graphene oxide (GO) and reduced graphene oxide (rGO) obtained from thermal exfoliation/reduction of graphite oxide. The SEM image of the non-activated carbon from starch shows sheet morphology similar to rGO (Figure 2c). In case of chemical activation of the carbon nanostructures during synthesis, highly porous interconnected carbon sheets were observed.

Activated carbon from starch exhibits highly interconnected sheet-like morphology (Figure 2d). Similarly, carbon from jute also shows interconnected sheets (Figure 2e) whereas carbon from rice husk exhibits porous carbon matrix (Figure 2f). FTIR spectra show graphitic skeletal vibrational bands (Figure S1a–b in Supplementary Information). FTIR spectra suggests that GO having huge functionalities, get reduced to rGO with negligible functionalities. Non-activated carbon from sugar shows FTIR spectrum similar to that of rGO indicating lower functionalities (Figure S1a). Activated carbon nanostructures also exhibit lower functionalities similar to rGO (Figure S1b). Raman spectra show that these are defected carbon as D band is very

Figure 1. Schematic of the reaction steps for the synthesis of biomass derived carbon.

Figure 2. FESEM images of (a) GO, (b) rGO, (c) non-Ac-sugar, (d) Ac-sugar, (e) Ac-hemp and (f) Ac-rice husk.

high in intensity (Figure S1c–d in Supplementary Information). These are mostly amorphous, porous and multilayer carbon sheets. The thermogravimetric analysis (TGA) suggests that GO shows maximum weight loss due to its huge functionalities whereas rGO exhibits minimum weight loss and other carbon nanostructures exhibit similar weight loss (Figure S2). These non-activated carbon flakes synthesized from starch have

high BET surface area of 787 m²/g with large inter-layer space, as observed type-IV hysteresis in N₂ gas adsorption (Figure 3). This is due to fine flake-like morphology having porous structure. When we synthesized activated carbon from sugar in the presence of KOH, uniform interconnected carbon sheet was observed. These are activated carbon material showing similar surface area (655 m²/g). However, the activated carbon

Figure 3. N₂ gas adsorption profile of different carbon materials at different relative pressure (P/P_0) showing BET surface area.

from jute exhibits maximum BET surface area of $825 \text{ m}^2/\text{g}$ whereas carbon from rice husk shows surface area of $547 \text{ m}^2/\text{g}$ (Figure 3).

The key components of crude biomass are cellulose, hemicellulose and lignin.³⁰ Lignin decomposes at higher temperature ($>900^\circ\text{C}$) whereas cellulose and hemicellulose decompose at lower temperature ($\sim 400\text{--}500^\circ\text{C}$).³⁰ Therefore, during carbonization at 750°C , the decomposition of cellulose and hemicellulose results in pores in the carbon materials as observed in SEM images. However, lignin, having aromatic units, creates interconnection between the aromatic rings which enhances the electrical conductivity of the materials. Rice husk contains $\sim 28\%$ cellulose, 28% hemicellulose and $\sim 26\%$ lignin.³¹ whereas jute fibers contains $\sim 65\%$ cellulose, $\sim 17\%$ hemicellulose and $\sim 16\%$ lignin.^{32,33} Therefore, the total amount of cellulose and hemicellulose is higher in jute fibers which is responsible for its high surface area ($\sim 825 \text{ m}^2/\text{g}$) and pores.

3.1 Electrochemical studies

Electrochemical investigations were carried out using a three electrode setup in $0.5 \text{ M H}_2\text{SO}_4$ (electrolyte) where these carbon nanostructures were deposited on GCE (0.03 cm^2) and Pt, Ag/AgCl (sat. KCl) were used as counter and reference electrodes, respectively. Mass loading of the samples were $1.6 \text{ mg}/\text{cm}^2$. In

Figure 4, it is observed that the cyclic voltammograms of these carbon nanostructures exhibit large area under the curve indicating high total charge accumulations and their potential for electrochemical double layer capacitors (EDLCs). The activated carbon from sugar, rice husk and jute shows much higher area under the curve compared to that of non-activated carbon from sugar. Therefore, activated carbon materials show higher charge compared to non-activated carbon due to their higher amount of oxygen-containing functionalities in activated carbon materials as observed in the TGA studies (Figure S2 in Supplementary Information). The area under the cyclic voltammogram at a particular scan rate *i.e.*, 100 mV/s increases in the order of non-ac-sugar $<$ ac-sugar $<$ ac-rice husk $<$ ac-jute, which suggests that the specific capacitance also increases in the same order. This suggests that activated carbon nanostructures from jute have small resistance and short diffusional pathways for ion transportation. However, the cyclic voltammogram of activated carbon from rice-husk and jute exhibit reversible redox peaks corresponding to Quinone-hydroquinone species.^{34,35} This suggests that AC-jute and AC-rice husk contained quinone type functionalities which contribute as pseudo-capacitance to the overall specific capacitance of the materials.

Galvanostatic charge-discharge cycles of the samples at different applied current density show that

Figure 4. Cyclic voltammogram of (a) Ac-sugar, (b) Ac-rice husk, (c) Ac-jute and (d) non-Ac-sugar at various scan rate (figure legends are scan rate in mV/s).

Figure 5. Galvanostatic charge-discharge cycles at different current density. (a) Ac-rice husk, (b) Ac-jute, (c) Ac-sugar and (d) non-Ac-sugar at various scan rates (figure legends are applied current density in A/g).

the charging or the discharging time decreases as we increase the current density (Figure 5). The discharging time at a particular applied current density *i.e.*, 0.2 A/g was observed maximum in case of activated carbon from jute (Figure 5b). The specific capacitance was calculated from the equation given below.

$$C = \frac{I \Delta t}{m \Delta V} \quad (1)$$

where, C is the specific capacitance (F/g), I is the current (A), Δt is the discharge time (s), ΔV is the potential window (V) and m is mass (g) of the material

loaded on the electrode. The calculated specific capacitance at current density of 0.2 A/g and BET surface area are tabulated for various carbons nanostructures (Table S1 in Supplementary Information). The specific capacitance decreases with increasing applied current density (Figure 6a). Activated carbon from jute (Ac-jute) exhibits maximum specific capacitance of 476 F/g and 200 F/g at an applied current density of 0.2 A/g and 10 A/g, respectively.

Earlier, graphene-like interconnected carbon sheet with high surface area (2287 m²/g) were synthesized from hemp bast fibres which have been shown to exhibit

Figure 6. (a) Specific capacitance as a function of applied current density; (b) bar diagram of specific capacitance of various carbon materials at current density of 0.2 A/g.

Figure 7. Variation of specific capacitance of Ac-jute during Charge-discharge cycles at an applied current density of 10 A/g showing the stability upto 350 cycles. Inset shows the charge-discharge cycles in the potential range of 0–1 V.

maximum specific capacitance of 144 F/g in ionic liquid electrolyte.¹⁷ Activated carbon from rice husk exhibit specific capacitance of 369 F/g and 258 F/g at current density of 0.2 A/g and 1 A/g, respectively, which is much higher than that of recent reports on activated carbon from rice husk.^{25,26} The activated carbon nanostructures show higher specific capacitance than that of non-activated carbon (Figure 6a). Even these activated carbons (Ac-jute and Ac-rice husk) exhibit much higher specific capacitance compared to that of graphene oxide (Figure 6b). However, TGA analysis suggests that activated carbons have low functionalities than that of graphene oxide (Figure S2 in Supplementary Information). In spite of having higher amount of functionalities on GO surface, the specific capacitance is much lower than that of activated carbon. However, carbon nanostructures from starch exhibit lower specific capacitance *i.e.*, 140 F/g and 104 F/g for activated and non-activated, respectively (Figure 6a). In starch, absence of aromatic groups due to lignin results in highly porous carbon nanostructures with low electrical conductivity which in turn decreases the specific capacitance. Therefore synergistic effect of lignin and cellulose in the biomass is the key factor for high specific capacitance.

We have observed the specific capacitance of 255 F/g and 14 F/g for GO and rGO, respectively, at an applied current density of 0.2 A/g. We have compared the specific capacitance of all these carbon nanostructures at an applied current density of 0.2 A/g (Figure 6b) which suggests that rGO exhibits lowest specific capacitance of 14 F/g due to lowest functionalities on rGO surface, as observed in TGA studies (Figure S2 in Supplementary Information).

We have carried out charge-discharge cycles for one of the materials *i.e.*, Ac-jute upto 350 cycles at an applied current density of 10 A/g to look at the stability of the material (Figure 7). Initially, upto 100 cycles, the specific capacitance increased from 220 F/g to 340 F/g and then it became constant at 340 F/g which is quite high for supercapacitor applications. This suggests that the graphene-like activated carbon nanostructures derived from biomass can be a promising candidate for supercapacitors.

4. Conclusions

Various carbon nanostructures (activated and non-activated) having sheet morphology have been synthesized from biomass. Amorphous carbon sheets were synthesized from starch and highly interconnected carbon sheets were observed when the reaction was carried out in presence of KOH. Detailed electrochemical analysis suggests that activated carbon nanostructures exhibit higher specific capacitance compared to non-activated carbons. Activated carbon (Ac-jute) shows higher specific capacitance of 476 F/g at 0.2 A/g which is much higher than that of graphene oxide (255 F/g) and reduced graphene oxide (14 F/g). Quinone type functionalities in Ac-jute and Ac-rice husk contribute pseudo-capacitance to the overall specific capacitance. It has been observed that instead of higher amount of functionalities in graphene oxide compared to activated carbon nanostructures, activated carbon shows much higher specific capacitance than graphene oxide.

Supplementary Information (SI)

FTIR, Raman spectrum and TGA data are provided in the supplementary information which is available at www.ias.ac.in/chemsci.

Acknowledgements

AKG wants to thank DST and DeitY, Govt. of India for financial support. KO thanks UGC, Govt. of India for fellowship.

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