Abstract: An anode in a lithium ion capacitor, including: a carbon composition comprising: a coconut shell sourced carbon in from 85 to 95 wt%; a conductive carbon in from 1 to 10 wt%; and a binder in from 3 to 8 wt%; and an electrically conductive sub-strate. The invention being based on the high temperature of carbonization of the coconut shell, between 1000 and 1600°C. Further, for low temperature the carbon can be washed with HCl. The carbon obtained shows more amorphous carbon than crystalline carbon.
ANODE FOR LITHIUM ION CAPACITOR BASED ON CARBONIZED COCONUT SHELL

CROSS-REFERENCE TO PRIORITY APPLICATION

[0001] This application claims the benefit of priority under 35 U.S.C. § 120 of U.S. Application Serial No. 14/610782 filed on January 30, 2015 the content of which is relied upon and incorporated herein by reference in its entirety.

CROSS-REFERENCE TO RELATED APPLICATIONS

[0002] The present application is related commonly owned and assigned USSN Application Nos., filed concurrently herewith:

14/610,752 filed on January 30, 2015, entitled "COKE SOURCED ANODE FOR LITHIUM ION CAPACITOR";
14/61081 filed January 30, 2015, entitled "POLY-VINYLIDENE DIFLUORIDE ANODE IN A LITHIUM ION CAPACITOR";
14/610848 filed January 30, 2015, entitled "PHENOLIC RESIN SOURCED CARBON ANODE IN A LITHIUM ION CAPACITOR"; and
14/610868 filed January 30, 2015, entitled "CATHODE FOR LITHIUM ION CAPACITOR," but does not claim priority thereto.

[0003] The entire disclosure of each publication or patent document mentioned herein is incorporated by reference.

Background

[0004] The disclosure relates to a lithium ion capacitor (LIC), to an anode in the LIC, and to a composition in the anode.

Summary

[0005] In embodiments, the disclosure provides a carbon composition for an anode in a lithium ion capacitor.
[0006] In embodiments, the disclosure provides an anode for a lithium ion capacitor having a low surface area carbon obtained from an inexpensive carbon source, such as coconut shell.

**Brief Description of the Drawings**

[0007] In embodiments of the disclosure:

[0008] Fig. 1 shows Raman spectra comparing frequency shifts and peak intensities of different carbons.

[0009] Fig. 2 shows discharge capacities from conditioning cycles for carbon based on different raw materials (discharge capacity normalized based on anode carbon weight). These capacities are measured at C/2 rate.

[0010] Fig. 3 shows volumetric Ragone plot comparing the rate performances of seven carbons from different raw materials.

[0011] Fig 4 shows discharge capacities in mAh/gm (based on anode carbon weight - C/2 rate) from conditioning cycles for coconut shell carbon treated with different conditions.

[0012] Fig. 5 shows a volumetric Ragone plot comparing coconut shell carbon treated with the different conditions.

**Detailed Description**

[0013] Various embodiments of the disclosure will be described in detail with reference to drawings, if any. Reference to various embodiments does not limit the scope of the invention, which is limited only by the scope of the claims attached hereto. Additionally, any examples set forth in this specification are not limiting and merely set forth some of the many possible embodiments of the claimed invention.

[0014] Features or aspects recited in any of the claims are generally applicable to all facets of the invention. Any recited single or multiple feature or aspect in any one claim can be combined or permuted with any other recited feature or aspect in any other claim or claims.
Definitions

[0015] "Anode," "anode electrode," "negative electrode," or like terms refers to an electrode through which positive electric charge flows into a polarized electrical device and electrons flow out of the electrode to an outside circuit.

[0016] "Cathode," "cathode electrode," "positive electrode," or like terms refers to an electrode from which positive electric charge flows out of a polarized electrical device.

[0018] "Include," "includes," or like terms means encompassing but not limited to, that is, inclusive and not exclusive.

[0019] "About" modifying, for example, the quantity of an ingredient in a composition, concentrations, volumes, process temperature, process time, yields, flow rates, pressures, viscosities, and like values, and ranges thereof, or a dimension of a component, and like values, and ranges thereof, employed in describing the embodiments of the disclosure, refers to variation in the numerical quantity that can occur, for example: through typical measuring and handling procedures used for preparing materials, compositions, composites, concentrates, component parts, articles of manufacture, or use formulations; through inadvertent error in these procedures; through differences in the manufacture, source, or purity of starting materials or ingredients used to carry out the methods; and like considerations. The term "about" also encompasses amounts that differ due to aging of a composition or formulation with a particular initial concentration or mixture, and amounts that differ due to mixing or processing a composition or formulation with a particular initial concentration or mixture.

[0020] "Optional" or "optionally" means that the subsequently described event or circumstance can or cannot occur, and that the description includes instances where the event or circumstance occurs and instances where it does not.

[0021] The indefinite article "a" or "an" and its corresponding definite article "the" as used herein means at least one, or one or more, unless specified otherwise.

[0022] Abbreviations, which are well known to one of ordinary skill in the art, may be used (e.g., "h" or "hrs" for hour or hours, "g" or "gm" for gram(s), "mL" for milliliters, and "rt" for room temperature, "nm" for nanometers, and like abbreviations).

[0023] Specific and preferred values disclosed for components, ingredients, additives, dimensions, conditions, times, and like aspects, and ranges thereof, are for illustration
only; they do not exclude other defined values or other values within defined ranges. The
composition and methods of the disclosure can include any value or any combination of
the values, specific values, more specific values, and preferred values described herein,
including explicit or implicit intermediate values and ranges.

[0024] Lithium ion capacitors (LICs) are a new class of innovative hybrid energy storage
devices. Unlike EDLC’s, which store energy based on double layer mechanism on both
electrodes, the hybrid lithium ion capacitors store energy on the cathode via the double
layer mechanism whereas energy storage on the anode is via Faradaic mechanism. As a
result, the energy density in such devices can be, for example, five times greater than an
EDLC, while maintaining the power also at a 3 to 4 times greater than an EDLC’s power.
Despite the Faradaic mechanism of energy storage, these LIC devices still show very
high cycle life, for example, in excess of 200,000 cycles, making the devices attractive
for many applications. LICs utilize a high surface area (typically greater than 1,000 m²/g)
carbon on the positive electrode and an intercalating carbon with low porosity and low
surface area (typically less than 300 m²/g) on the anode, which combination of a high and
a low surface area carbon supports fast intercalation and de-intercalation of lithium ions.
During charge and discharge, lithium intercalation and de-intercalation occurs within the
bulk of the negative electrode (i.e., the anode), whereas anion adsorption and desorption
takes place on the positive electrode (i.e., the cathode). The adsorption and desorption on
the positive electrode is a non-Faradaic reaction, which is relatively faster than the
lithium ion intercalation and de-intercalation on the negative electrode.

[0025] In the lithium ion capacitor, as mentioned above, the negative electrode
(intercalating and de-intercalating) can be pre-doped with lithium metal. The pre-doping
of the lithium ion capacitor allows an increase in the voltage to approximately about 3.8
volts.

[0026] A lithium ion capacitor permits a voltage of about 1.5 times that of an EDLC. The
cell capacitance (C= Q/V) can be stated by the charge-discharge curve. Since the energy
density and power are both proportional to square of the voltage there is a significant
increase in energy and power density of the device. In addition to the voltage related
increase, the Faradaic reaction also has significantly greater energy associated with it and
contributes to an increase in the energy and the power density. The negative electrode
stays at a constant or even potential during the charging and discharging of the cell.

Properties of the negative electrode or anode are significant to the performance of
the device. These properties are derived mainly from the materials comprising the
electrode. The carbon materials comprising the negative electrode are significant
to the performance of the device. The properties required for the Li ion capacitor anode
are different from properties required for Li ion battery anodes, although both involve
insertion of lithium ions in the carbon structure. The Li ion capacitor is a power device
and hence a fast intercalation - deintercalation of ions is essential, whereas for Li ion
batteries a slow intercalation rate is acceptable. In embodiment, the disclosure provides a
carbon for the use in an anode electrode of a lithium ion capacitor. The data provided
was obtained at desired high charge-discharge rates.

Different types of carbons have been evaluated as anode materials of lithium ion
capacitors. Graphite has been used mainly as the material of choice, but hard carbon
materials, i.e., non-graphitic materials having a low surface area are also being used.
Hard carbon materials show superior discharge capacities compared to graphite due to
structural differences between the hard carbon and graphite.

Charge and discharge capacity is a quantitative measure of charge being stored
and discharged, respectively, during the charge-discharge process. Discharge capacity is
an intrinsic property of a carbon material. The higher the discharge capacity, the higher
the energy density of the device. The discharge capacity of the carbon material can
depend on, for example, the structure of the carbon and the impurity levels present in the
carbon material.

The disclosure provides experimental support that shows the superior rate
performance of coconut shell sourced carbon compared to a graphite (i.e., a standard
material used in published studies), a phenolic resin (660 °C) (comparative commercial
material), and coconut shell sourced carbon carbonized at 600 °C, and at 1000 °C.
Coconut shell sourced carbon that was carbonized at 1200 °C, at 1400 °C, and at 1600
°C, shows superior rate performance especially at higher rates (higher currents).

The disclosure provides experimental data for the disclosed carbon composition,
an anode, and an LIC, which data includes a performance comparison as the anode
material in Li ion capacitors of the disclosed anode carbon with anode carbons made from various different raw materials, such as carbon produced from a phenolic resin, and from a graphite.

[0032] The carbon products produced from the coconut shell carbonized at 1200°C, the coconut shell carbonized at 1400°C, and the coconut shell carbonized at 1600°C showed the highest discharge capacities compared to the other carbons and their respective treatments.

[0033] Rate performance is significant as a capacitor device is subjected to high rate charge-discharge cycles unlike batteries where the charge discharge rates are orders of magnitude lower.

[0034] In embodiments, the disclosure includes high performance active materials for lithium ion capacitors, and electrodes fabricated from these materials. The disclosure also includes a method of making the electrode from a carbon composition, which carbon composition is used as an anode in a lithium ion capacitor.

[0035] In embodiments, the disclosure provides a carbon composition for an anode in a lithium ion capacitor.

[0036] In embodiments, the disclosure provides an anode for a lithium ion capacitor having a low surface area carbon obtained from an inexpensive carbon source, such as coconut shell.

[0037] In embodiment, the disclosure provides a hard carbon, which shows superior discharge capacities compared to other hard carbons or graphite. Additionally, the disclosed carbon has superior rate performance, especially at high rates, leading to higher power and superior performance. The material, method of making and use, and the device performance is provided below and is compared with prior art materials.

[0038] In embodiments, the disclosure provides a performance comparison of the disclosed hard carbon prepared from a coconut shell source with a graphite and with carbons produced from different raw materials such as commercial phenolic resin.

[0039] In embodiments, the disclosure provides a method of making a carbon as an anode material in a lithium ion capacitor. The disclosure includes the performance comparison of anodes containing a coconut shell derived hard carbon with carbons produced from different raw materials such as a phenolic resin and a commercial
graphite. This disclosure identifies fundamental aspects of the performance of the lithium ion capacitor with the coconut shell sourced activated carbon as an electrode material on the anode side and compares it to the activated carbons sourced from the phenolic resin and the graphite.

[0040] A carbonaceous material used on an anode electrode in a lithium ion capacitor is known to intercalate and de-intercalate lithium ions (see J. Electrochem. Soc, Vol. 140, No. 4, 922-927, April 1993). Charge and discharge capacity is a quantitative measure of the carbon's ability to store charge or discharge charge. Discharge capacity is an intrinsic property of a carbon. The discharge capacity of the carbon can depend on, for example, the structure of the carbon and the impurity levels in the carbon. The discharge capacities can directly reflect on the performance aspects of an energy cell. A higher discharge capacity is a desirable property in a carbon on the anode side as the carbon will directly and positively affect the packaging volume of a cell, which in turn will positively affect the energy and power density in a lithium ion capacitor device. It is significant to note that the capacity must be measured at high discharge rates since a capacitor is a power device, unlike an energy device such as a battery, where the discharge rates are significantly lower. The capacity of a material at high discharge rates will be different than at low discharge rates and will be a function of the structure of the material, which structure controls the diffusion rate of lithium ions into the structure of the materials.

[0041] In embodiments, the disclosure provides an anode in a lithium ion capacitor, comprising:

a carbon composition comprising: a coconut shell sourced carbon in from 85 to 95 wt%; a conductive carbon in from 1 to 10 wt%; and a binder in from 3 to 8 wt%, based on 100 wt% of the carbon composition; and

an electrically conductive substrate, e.g., a copper or aluminum foil,

wherein the coconut shell sourced carbon composition has a disorder (D) peak to graphitic (G) peak intensity ratio by Raman analysis of from 1.40 to 1.85; a hydrogen content of from 0.01 to 0.25 wt%; a nitrogen content of from 0.01 to 0.55 wt%; and an oxygen content of from 0.01 to 2 wt%.

[0042] In embodiments, the coconut shell sourced carbon can be, for example, present in from 88 to 92 wt%; the conductive carbon can be, for example, present in from 4 to 7
wt%; and the binder is PVDF and can be, for example, present in from 4 to 6 wt% and the PVDF has a molecular weight of from 300,000 to 1,000,000.

[0043] In embodiments, the coconut shell sourced carbon composition can have, for example, a disorder (D) peak to graphitic (G) peak intensity ratio by Raman analysis of from 1.48 to 1.8.

[0044] In embodiments, the coconut shell sourced carbon can have, for example, a hydrogen content of from 0.01 to 0.24 wt%; a nitrogen content of from 0.03 to 0.5 wt%; an and oxygen content of from 0.01 to 1.9 wt%, including intermediate values and ranges.

[0045] In embodiments, the coconut shell sourced carbon can have, for example, a relatively low surface area of from 1 to 100 m²/g, such as from 10 to 75 m²/g, from 20 to 50 m²/g, and like areas, including intermediate values and ranges.

[0046] In embodiments, the coconut shell sourced carbon can have a particle size, for example, from 1 to 30 microns, from 2 to 7 microns, and like particle sizes, including intermediate values and ranges.

[0047] In embodiments, the anode operates at a high charge-discharge rate, for example, of from 1C to 4000 C, including intermediate values and ranges.

[0048] In embodiments, the lithium ion capacitor can further comprise, for example: a cell comprising: the abovementioned anode in combination with a stacked cathode comprising an a heat and KOH activated wheat flour sourced carbon, e.g., 85% Corning carbon (mentioned below), a fluoropolymer, e.g., 10% PTFE DuPont 601 A Teflon, a conductive carbon black, e.g., 5% Cabot Black Pearl 2000, a separator, e.g., NKK-4425, and a lithium composite powder (mentioned below) on the anode.

[0049] In embodiments, the cell can have a discharge capacity, for example, of from 60 to 120 mAh/gm, such as 96.32 mAh/gm based on the anode carbon weight, and a maximum energy density, for example, of from 20 to 60 Wh/l, such as 44.77 Wh/l.

[0050] In embodiments, the disclosure provides a method of making the above mentioned carbon composition comprising:

- a first heating (e.g., carbonizing) of a coconut shell sourced carbon at from 1000°C to 1700°C for 0.5 hrs to 5 hrs in an inert atmosphere; and
- grinding the resulting carbon to a powder.
In embodiments, the method of making can further comprise, for example, washing the resulting powder with an aqueous acid (e.g., HCl). In embodiments, the method of making can further comprise, for example, a second heating (i.e., re-heating) of the aqueous acid washed powder at from 1000 °C to 1700 °C.

In embodiments, the first and second heating can be accomplished, for example, at from 1000°C to 1600°C for 1 to 3 hrs.

In embodiments, at least one of the first heating and second can be accomplished at, for example, 1100 to 1300°C, 1150 to 1250°C, including intermediate values and ranges.

In embodiments, at least one of the first heating and second can be accomplished at, for example, 1300 to 1500°C, 1350 to 1450°C, including intermediate values and ranges.

In embodiments, at least one of the first heating and second can be accomplished at, for example, 1500 to 1700°C, 1550 to 1650°C, including intermediate values and ranges.

In embodiments, the disclosure provides a carbon composition prepared by the above mentioned method. The disclosed carbon composition is particularly useful in, for example, lithium ion capacitor applications.

The present disclosure is advantaged is several aspects, including for example: high discharge capacity carbons, high rate performance for improved power performance, and reduced production costs.

Coconut shell sourced hard carbon was determined to have high discharge capacities and superior rate performance. Referring to the figures, Fig. 2 shows the discharge capacities (normalized on the anode carbon weight, measured at C/2 rate) during the conditioning cycles for a phenolic resin sourced carbon (where the resin was carbonized at 660°C), a coconut shell carbon (carbonized at 1600°C, 1400°C, 1200°C, 1000°C, and 600°C), and graphite.

The discharge capacities (Fig. 2) for coconut shell sourced carbon that was carbonized at 1200°C, 1400°C, and 1600°C, were higher than the phenolic resin sourced carbon that was carbonized at 660°C, the coconut shell sourced carbon that was carbonized at 600°C, the coconut shell carbon that was carbonized at 1000°C, and
graphite. The coconut shell sourced carbon that was carbonized at 1000°C, washed with 
HC1, and retreated at 1000°C, showed a higher discharge capacity of 70.59 mAh/gm 
compared to the phenolic resin sourced carbon that was carbonized at 660°C (39.37 
mAh/gm), the coconut shell sourced carbon that was carbonized at 600°C (18.20 
mAh/gm), and the graphite (48.46 mAh/gm), after three conditioning charge-discharge 
cycles.

[0060] Coconut shell carbons carbonized at 1200°C, 1400°C, and 1600°C, showed a 
higher discharge capacity of 96.32 mAh/gm, 88.64 mAh/gm, and 105.39 mAh/gm, 
respectively, when compared to: the coconut shell carbon that was carbonized at 1000°C, 
washed with HC1 and retreated at 1000 °C, which had a discharge capacity of 70.59 
mAh/gm; a phenolic resin carbonized at 660°C had a discharge capacity of 39.37 
mAh/gm; a coconut shell carbon carbonized at 600°C had a discharge capacity of 18.20 
mAh/gm; and graphite had a discharge capacity of 48.46 mAh/gm, each after three 
conditioning charge discharge cycles.

[0061] This desirable trait of the coconut shell sourced carbon translates into a positive 
effect in the energy density and power density (rate performance) for a lithium ion 
capacitor constructed with the coconut shell sourced carbon that was carbonized at 
1200°C, 1400°C and 1600°C, respectively.

[0062] Fig. 3 shows a rate performance comparison for seven different carbons, i.e., 
carbon produced from a phenolic resin source, a coconut shell source, and a graphite. The 
carbon made from (i.e., sourced) the coconut shell carbon and that was carbonized at 
1000 °C, washed with HC1, and retreated at 1200°C, 1400°C, or 1600°C, respectively, 
showed the best rate performance at higher C-rates. The carbon made from coconut shell 
carbon and that was carbonized at 1000°C, washed with HC1, and retreated at 1200°C, 
1400°C, or 1600°C, showed higher energy densities and power densities compared to the 
other four carbons at low, medium, and high rates. However, the coconut shell carbon 
that was carbonized at 1000°C, washed with HC1, and retreated at 1200°C, 1400°C, or 
1600°C, showed superior performance at higher discharge rates. The carbonization 
temperature can have a significant impact on the discharge capacity. Although not 
limited by theory, the impact of carbonization temperature on the discharge capacity
might be explained by the changing structure of the carbon with the changing carbonization temperature as seen from Raman data given above.

[0063] In embodiments, the disclosure provides a coconut shell hard carbon having high discharge capacities and superior rate performance. Fig. 2 shows the discharge capacities (normalized on the anode carbon weight, measured at C/2 rate) during the conditioning cycles for a phenolic resin sourced carbon (that was carbonized at 660°C), a coconut shell sourced carbon (that was carbonized at 1600°C, 1400°C, 1200°C, 1000°C, or 600°C), and graphite.

[0064] Data is also provided for the performance of coconut shell sourced carbons produced by different treatment conditions. Three different conditions were evaluated: a coconut shell sourced carbon that was carbonized at 1000°C; a coconut shell sourced carbon that was carbonized at 1000°C followed by wash with HCl; a coconut shell sourced carbon that was washed with HCl, and was then carbonized to 1000°C, and compared to the coconut shell sourced or based carbon that was carbonized at 1000°C, washed with HCl, and retreated at 1000°C.

[0065] Fig. 4 shows the discharge capacity for three conditioning cycles for three different coconut shell carbons produced by different treatment conditions compared to the coconut shell based carbon carbonized at 1000°C, washed with HCl, and retreated at 1000°C. A higher discharge capacity (70.59 mAh/gm) was observed for coconut shell carbon carbonized at 1000 °C, washed with HCl, and retreated at 1000°C, when compared to the other two conditions, i.e., coconut shell carbon carbonized at 1000°C (65.97 mAh/gm), coconut shell carbon carbonized at 1000°C followed by wash with HCl (50.32 mAh/gm). The coconut shell carbon made by washing raw coconut shell flour/granules followed by carbonization at 1000°C showed comparable discharge capacity (69.61 mAh/gm) when compared to coconut shell carbon carbonized at 1000°C, washed with HCl and retreated at 1000°C.

[0066] Fig. 5 shows the rate performance for three different coconut shell carbons treated under different conditions and compared to a coconut shell carbon carbonized at 1000°C, washed with HCl, and retreated at 1000°C. The coconut shell carbon carbonized at 1000°C followed by HCl washing, and second treatment at 1000°C showed the best rate performance (volumetric). The coconut shell carbon that was carbonized at 1000°C
followed by HCl washing, and a second treatment at 1000°C showed higher energy and power density at higher current rates. The coconut shell carbon made by washing raw coconut shell flour/granules followed by carbonization at 1000°C showed comparable rate performance to the coconut shell carbon made by carbonization at 1000°C followed by HCl washing, and a second treatment at 1000°C and had lower current rates but slightly lower performances at higher current rates. Overall, the disclosed coconut shell carbon made by carbonization at 1000°C followed by HCl washing, and a second treatment at 1000°C had higher discharge capacities and superior rate performance of all the different carbons evaluated. The HCl washing and 1000°C treatment plays a significant role in removing trace metallic and organic impurities from the carbon. The added HCl washing and the second 1000°C treatment assists in providing better performance (i.e., capacity and rate performance) of the carbon.

EXAMPLES

[0067] The following Examples demonstrate making, use, and analysis of the disclosed activated carbon, LIC anode, LIC, and methods in accordance with the above general procedures.

Comparative Example 1

[0068] Phenolic resin source carbon at 660°C (mentioned in US 2013/0201606A1) A phenolic resin, GP® 510D50 RESI-SET® Phenolic Impregnating Resin, was acquired from Georgia Pacific and cured at 100 to 125°C. A phenolic resin plate was also prepared by curing the phenolic resin at 100 to 120 °C and then ground to a fine powder. The resin was then placed in a retort furnace and heated at 50°C/hr to 500°C. The furnace temperature was then held at 500°C for 1 hr. The furnace was then ramped at 10°C/hr to 660°C. The furnace was then held at 660°C for 1 hr. The furnace was switched off and passively cooled. The resulting carbon was ground to a 5 micron particle size. The ground carbon was subjected to Raman spectroscopy for structure analysis. The stacked Raman spectra are shown in Fig. 1. The phenolic resin sourced carbon treated at 660°C shows an I_D peak (disorder peak) at 1308.83 cm⁻¹ with an intensity of 2339.745 a.u., and an I_G peak at about 1601.01 cm⁻¹ with an intensity of 1952.962 a.u. (Table 1). The peak
The ratio of ID/IG for the phenolic resin sourced carbon treated at 660°C was 1.1980. The phenolic resin sourced carbon was also subjected to relative percentage carbon, hydrogen, nitrogen, and oxygen elemental analysis. The samples were dried under vacuum at 125°C for 6 hrs prior to the analysis. All the elemental results are reported on a dried basis and are summarized in Table 2. Found: C: 95.27%, H: 1.76%, N: 0.1%, and O: 2.11%. The phenolic resin sourced carbon was also subjected to BET analysis to probe the surface area of the carbon, which was 426.8621 m²/gm.

The resulting carbon was used in casting anode electrodes for a lithium ion capacitor. The anode consisted of 90 wt% of the ground phenolic resin sourced carbon, 5 wt% Timcal Super C-45 conductive carbon, and 5 wt% of KYNAR HSV 900 grade PVDF (molecular weight: 1,000,000) as a binder.

The anode was prepared as follows. 3.6 grams of the ground phenolic resin sourced carbon and 0.2 grams of Timcal Super C-45 were balled milled in a Retsch PM-100 ball mill for 15 mins at 350 rpm. 0.2 gram of PVDF was added to the milled mixture of phenolic resin sourced carbon and the Timcal Super C-45, and the mixture was ball-milled for 15 mins at 350 rpm. Several drops of N-methyl pyrrolidinone (NMP) were added to the mixture to form a paste of the mixture. The paste was then coated on a copper foil (Product number -Oak Mitsui TLB-DS), and passed through a rolling mill to produce the 4 mil thickness. Then 5 mg of lithium composite powder (LCP; is used for pre-doping the anode) was coated on an anode made from the ground phenolic resin sourced carbon 660°C. The calendared anode electrodes were punched to make 14 mm diameter circular electrodes. The punched anode electrodes were the dried 16 hrs at 120°C under vacuum.

A Li ion capacitor was built in a CR2032 format cell. The cathode consisted of 85 wt % of the activated Corning carbon mentioned below, 10 wt% PTFE (DuPont 601A Teflon PTFE), and 5 wt% Cabot Black Pearl 2000 (see for example, US Patent Nos.: 8,318,356, 8,784,764, 8,524,632, and 8,541,338). The separator was NKK-4425 separator.

The lithium composite powder (LCP) is an encapsulated lithium particle comprising: a core comprised of at least one of: lithium; a lithium metal alloy; or a combination thereof; and a shell comprised of a lithium salt, and an oil, the shell
encapsulates the core, and the particle has a diameter of from 1 to 500 microns (see commonly owned and assigned USSN 13/673019, filed Nov. 9, 2012, entitled "LITHIUM COMPOSITE PARTICLES," and USSN 14/493886, filed Sept. 23, 2014, entitled "ENCAPSULATED LITHIUM PARTICLES AND METHODS OF MAKING AND USE THEREOF").

[0073] The Corning carbon mentioned above was made from a wheat flour precursor. The wheat flour was carbonized at from 650 to 700°C. The carbonized carbon was ground to a particle size of approximately 5 microns. The ground carbonized carbon was then activated at 750°C with KOH (alkali) in a weight ratio of 2.2: 1 KOH:carbon for 2 hrs. The carbon was further washed with water to remove any remaining KOH. The resulting activated carbon was then treated with HCl to neutralize any trace of KOH and then washed with water to neutralize the carbon to a pH of 7. The activated carbon was then heat-treated under nitrogen and hydrogen forming gas at 900 °C for 2 hrs.

[0074] The cell was then crimped on an MTI coin cell crimper and conditioned on an Arbin BT 2000 and conditioned at constant current charge/discharge at 0.5 mA current from 3.8V to 2.2V. The ground phenolic resin source carbon had a discharge capacity of 39.374 mAh/gm (based on the anode carbon weight) after the third conditioning cycle (Fig. 2). The cell was subjected to C-Rate performance where the cell was charged at a constant current of 1 mA and discharged at different rates. Fig. 3 shows the C-rate performance of the phenolic resin source carbon on volume basis. The cell showed a maximum energy density of 29.44 Wh/l.

Comparative Example 2

[0075] Graphite The Timcal TB-17 graphite powder specially synthesized for Li ion electrode applications was acquired from MTI Corp., and used as received. The received carbon was subjected to Raman spectroscopy for structure analysis. The stacked Raman spectra are shown in Fig. 1. The graphite had an Ip peak (disorder peak) at 1316.33 cm⁻¹ with an intensity of 3019.06 a.u., and an Ig peak at approximately 1599.91 cm⁻¹ with an intensity of 2000.583 a.u. (Table 1). The peak ratio of Ip/Ig was 1.51. The Timcal TB-17 graphite was subjected to elemental analysis as in Comparative Example 1; found: C: 99.4%; H: 0.27%; N: 0.02%; and O: less than 0.1%.

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The graphite was used in casting anode electrodes for a lithium ion capacitor. The electrodes consisted of 90 wt% Timcal TB-17 graphite powder, 5 wt% Timcal Super C-45 conductive carbon from MTI Corp., and 5 wt% of KYNAR HSV 900 grade PVDF (molecular weight: 1,000,000) as a binder.

The anode was prepared as follows. 3.6 grams of the Timcal TB-17 graphite powder and 0.2 grams of the Timcal Super C-45 were balled milled in a Retsch PM-100 ball mill for 15 mins at 350 rpm. 0.2 grams of PVDF was added to the graphite and Timcal Super C-45 mixture and was ball-milled for 15 mins at 350 rpm. A few drops of N-methyl pyrrolidinone (NMP) were added to the mixture to form a paste of the mixture. The paste was then coated on a copper foil (Product number -Oak Mitsui TLB-DS), and passed through a rolling mill to achieve a 4 mil thickness. The calendared electrodes were punched to make 14 mm diameter circular electrodes. The punched electrodes were dried 16 hrs at 120 °C under vacuum.

A Li ion capacitor was built in a CR2032 format cell. The cathode comprised 85% of the above mentioned Corning carbon, 10% PTFE (DuPont 601A Teflon PTFE), and 5% Cabot Black Pearl 2000. The separator was NKK-4425 separator. The 5 mg of lithium composite powder (LCP is used for pre-doping the anode) was coated on an anode made from Timcal TB-17 graphite powder.

The cell was then crimped on a MTI coin cell crimpler and conditioned on an Arbin BT 2000 at a constant current charge/discharge at 0.5 mA current from 3.8V to 2.2V. The cell with Timcal TB-17 graphite powder gave a discharge capacity of 48.46 mAh/gm (based on the anode carbon weight) after the third conditioning cycle (Fig. 2). The cell was subjected to C-Rate performance where the cell was charged at a constant current of 1 mA and discharged at different rates. Fig. 3 shows the C-rate performance of the Timcal TB-17 graphite powder on a volume basis. The cell had a maximum energy density of 36.69 Wh/l.

**Comparative Example 3**

Coconut Shell Sourced Carbon (600 °C carbonization; HC1 treatment; and second 600°C treatment) Coconut shell granules were acquired from Reade Materials and carbonized to 600°C for 2 hrs under nitrogen conditions. The resulting carbon was
then ground to 5 micron particle size. The ground carbon was then washed with 1N HC1 16 hrs followed by washes with distilled water to pH 5. The washed carbon was then heat treated at 600°C for 2 hrs under nitrogen. The resulting heat treated carbon was subjected to Raman spectroscopy for structure analysis. The stacked Raman spectra are shown in Fig. 1. The heat treated carbon had an $I_D$ peak (disorder peak) at 1334.48 cm⁻¹ with an intensity of 2328.612 a.u., and an $I_G$ peak at approximately 1587.88 cm⁻¹ with an intensity of 2002.222 a.u. (Table 1). The peak ratio of $I_D/I_G$ was 1.16. The heat treated carbon was subjected elemental analysis as in Comparative Example 1: found: C: 94.10%; H: 2.12%; N: 0.15%; and O: 2.88%.

[0081] The heat treated carbon was used in casting anode electrodes for a lithium ion capacitor. The anode electrode consisted of 90 wt.% of the heat treated coconut shell sourced carbon, 5 wt.% Timcal Super C-45 conductive carbon, and 5 wt.% of KYNAR 761 grade PVDF (molecular weight: 300,000 to 400,000) as a binder.

[0082] The anode was prepared as follows. 3.6 grams of the heat treated coconut shell sourced carbon and 0.2 grams of Timcal Super C-45 were balled milled in a Retsch PM-100 ball mill for 15 mins at 350 rpm. 0.2 grams of PVDF was added to the heat treated coconut shell sourced carbon and Timcal Super C-45 mixture and ball-milled for 15 mins at 350 rpm. Next 6 mL of N-methyl pyrrolidinone (NMP) was added to the mixture. The mixture was again ball-milled for 20 minutes at 350 rpm. The slurry after ball-milling was rod coated on a copper foil (Product number -Oak Mitsui TLB-DS). The coated electrodes were dried under vacuum at 25 °C for 16 hrs. The dried electrodes were then calendared to the desired thickness of 4 mil. (the current collector thickness was not included in 4 mils). The calendared electrodes were punched to make 14 mm diameter circular electrodes. The punched electrodes were then dried for 16 hrs at 120°C under vacuum.

[0083] A Li ion capacitor was built in a CR2032 format cell. The cathode comprised 85% of the abovementioned Corning carbon, 10% PTFE (DuPont 601 A Teflon PTFE), and 5% Cabot Black Pearl 2000. The separator was NKK-4425 separator. The 5 mg of the abovementioned lithium composite powder (LCP is used for pre-doping the anode) was coated on an anode made from coconut shell sourced carbon treated at 600 °C.
The cell was then crimped on a MTI coin cell crimper and conditioned on an Arbin BT 2000, at constant current charge/discharge at 0.5mA from 3.8V to 2.2V. The coconut shell sourced carbon gave a discharge capacity of 18.208 mAh/gm (based on the anode carbon weight) after the third conditioning cycle (Fig. 2). The cell was subjected to C-Rate performance where the cell was charged at a constant current of 1mA and discharged at different rates. Fig. 3 shows the C-rate performance of the comparative coconut shell sourced carbon (600°C; HC1; 600°C) on volume basis. The cell showed a maximum energy density of 16.31 Wh/l.

Example 4

Coconut Shell Sourced Carbon (with HC1 wash and 2nd 1000°C treatment).

Coconut flour 200 mesh was acquired from Reade Materials. The flour was then carbonized to 1000°C for 2 hrs under nitrogen. The resulting carbon was then ground to 5 micron particle size. The ground carbon was then washed with IN HC1 16 hrs followed by washes with distilled water to pH 5. The carbon was then heat treated to 1000 °C for 2 hrs under nitrogen. The resulting heat treated carbon was subjected to Raman spectroscopy for structure analysis. The stacked Raman spectra are shown in Fig.1. The heat treated carbon had an I_D peak (disorder peak) at 1305.47 cm⁻¹ with an intensity of 3445.148 a.u., and an I_G peak at approximately 1599.91 cm⁻¹ with an intensity of 1928.368 a.u. (Table 1). The peak ratio of I_D/I_G was 1.79. The heat treated carbon was subjected to elemental analysis as in Comparative Example 1; found: C: 94.48%; H: 0.14%; N: 0.45%; and O: 1.42%. The heat treated carbon was also subjected to BET analysis to probe the surface area of the carbon, which was 27,5834 m²/gm.

The resulting heat treated carbon was used in casting anode electrodes for lithium ion capacitor. The anode consisted of 90 wt% of the heated treated coconut shell carbon, 5 wt% Timcal Super C-45 conductive carbon, and 5 wt% of KYNARHSV 900 grade PVDF (molecular weight: 1,000,000) as a binder.

The anode was prepared as follows. 3.6 grams of the heated treated coconut shell carbon and 0.2 grams of Timcal Super C-45 were balled milled in a Retsch PM-100 ball mill for 15 mins at 350 rpm. 0.2 grams of PVDF was added to the mixture and ball-milled for an additional 15 mins at 350 rpm. A few drops of N-methyl pyrrolidinone...
(NMP) were added to the mixture to form a paste. The paste was then coated on a copper foil (Product number -Oak Mitsui TLB-DS) and passed through a rolling mill to achieve a thickness of 2.75 mil.

[0088] A Li ion capacitor was built in a CR2032 format cell. The cathode comprised 85% of the abovementioned Corning carbon, 10% PTFE (DuPont 601A Teflon PTFE), and 5% Cabot Black Pearl 2000. The separator was NKK-4425 separator. The 5 mg of the above mentioned lithium composite powder (LCP is used for pre-doping the anode) was coated on an anode made from coconut shell sourced carbon treated at 1000 °C.

[0089] The cell was then crimped on a MTI coin cell crimper and conditioned on an Arbin BT 2000 at a constant current charge/discharge at 0.5mA current from 3.8V to 2.2V. The heat treated coconut shell based carbon gave a discharge capacity of 70.60 mAh/gm (based on the anode carbon weight) after the third conditioning cycle (Fig 2). The cell was subjected to C-Rate performance where the cell was charged at a constant current of 1 mA and discharged at different rates. Fig. 3 shows the C-rate performance of the heat treated (1000 °C) coconut shell sourced carbon on a volume basis. The cell showed maximum energy density of 47.66 Wh/l.

Example 5

[0090] Coconut Shell Sourced Carbon (with HC1 wash and second 1200°C treatment) Example 4 was repeated with the exception that the acid washed carbon was heat treated to 1200 °C for 2 hrs under nitrogen. The resulting carbon had an ID peak (disorder peak) at 1308.47 cm⁻¹ with an intensity of 4675.030 a.u. and an IQ peak at approximately 1595.84 cm⁻¹ with an intensity of 3150.750 a.u. (Table 1). The peak ratio of ID/IG was 1.48. The heat treated carbon (1200°C) was subjected to elemental analysis as in Comparative Example 1: found: C: 98.44%; H: 0.13%; N: 0.44%; and O: 0.75%.

[0091] The resulting carbon was used in casting anode electrodes for a lithium ion capacitor as in Example 4 with the following particulars being noted: 90 wt% of coconut shell sourced carbon, 5 wt% Timcal Super C-45 conductive carbon, and 5 wt% KYNAR 761 grade PVDF (molecular weight: 1,000,000) as a binder. The coated slurry on copper foil was passed through a rolling mill to achieve a 2.75 mil thickness.
[0092] A Li ion capacitor was built in a CR2032 format cell. The cathode comprised 85% of the above mentioned Corning carbon, 10% PTFE (DuPont 601 A Teflon PTFE), and 5% Cabot Black Pearl 2000. The separator was NKK-4425 separator. The 3.5 mg of the above mentioned lithium composite powder (LCP is used for pre-doping the anode) was coated on an anode made from coconut shell sourced carbon treated at 1200 °C.

[0093] The cell was then crimped on a MTI coin cell crimper and conditioned on an Arbin BT 2000 at constant current charge/discharge at 0.4 mA current from 3.8V to 2.2V. The cell had a discharge capacity of 96.32 mAh/gm (based on the anode carbon weight) after the third conditioning cycle (Fig. 2). The cell was subjected to C-Rate performance where the cell was charged at a constant current of 1 mA and discharged at different rates. Fig. 3 shows the C-rate performance of the heat treated (1200 °C) coconut shell sourced carbon on a volume basis. The cell showed maximum energy density of 44.77 Wh/l.

Example 6

[0094] Coconut Shell Based Carbon (with HC1 wash and 2nd 1400°C treatment)

Example 4 was repeated with the exception that the acid washed carbon was heat treated to 1400 °C for 2 hrs under nitrogen. The resulting carbon had an Ip peak (disorder peak) at 1310.26 cm⁻¹ with an intensity of 6678.350 a.u., and an IQ peak at about 1604.15 cm⁻¹ with an intensity of 4126.940 a.u. (Table 1). The peak ratio of Ip/IQ was 1.62. The heat treated carbon (1400°C) was subjected to elemental analysis as in Comparative Example 1; found: C : 99.66%; H : 0.09%; N : 0.16%; and O : 0.09%. The coconut shell sourced carbon treated at 1400°C was also subjected to BET analysis to probe the surface area of the carbon, which was 10.9606 m²/gm.

[0095] The resulting carbon was used in casting anode electrodes for a lithium ion capacitor as in Example 4 with the following particulars being noted: 90 wt% of coconut shell carbon, 5 wt% Timcal Super C-45 conductive carbon, and 5 wt% KYNAR 761 grade PVDF (molecular weight: 1,000,000) as a binder. The coated slurry on copper foil was passed through a rolling mill to achieve a 2.75 mil thickness.

[0096] A Li ion capacitor was built in a CR2032 format cell. The cathode comprised 85% of the abovementioned Corning carbon, 10% PTFE (DuPont 601 A Teflon PTFE),
and 5% Cabot Black Pearl 2000. The separator was NKK-4425 separator. 3.5 mg of the abovementioned lithium composite powder (LCP is used for pre-doping the anode) was coated on an anode made from coconut shell sourced carbon treated at 1400°C.

[0097] The cell was then crimped on a MTI coin cell crimer and conditioned on an Arbin BT 2000 at constant current charge/discharge at 0.5mA current from 3.8V to 2.2V. The cell had a discharge capacity of 88.64 mAh/gm (based on the anode carbon weight) after the third conditioning cycle (Fig. 2). The cell was subjected to C-Rate performance where the cell is charged at a constant current of 1 mA and discharged at different rates. Fig 3 shows the C-rate performance of the heat treated (1400 °C) coconut shell sourced carbon on a volume basis. The cell had a maximum energy density of 41.56 Wh/1.

Example 7

[0098] Coconut Shell Based Carbon (with HCl wash and 2nd 1600°C treatment)

Example 4 was repeated with the exception that the acid washed carbon was heat treated to 1600 °C for 2 hrs under nitrogen. The resulting carbon had an I_D peak (disorder peak) at 1306.63 cm⁻¹ with an intensity of 7450.380 a.u., and an I_Q peak at about 1601.58 cm⁻¹ with an intensity of 4139.710 a.u. (Table 1). The peak ratio of I_D/I_G was 1.80. The heat treated carbon (1500°C) was subjected to elemental analysis as in Comparative Example 1; found: C: 99.66%; H: 0.08%; N: 0.08%; and O: less than 0.1%. The coconut shell sourced carbon treated at 1400 °C was also subjected to BET analysis to probe the surface area of the carbon, which was 8.5450 m²/gm.

[0099] The resulting carbon was used in casting anode electrodes for a lithium ion capacitor as in Example 4 with the following particulars being noted: 90 wt% of heat treated (1600 °C) coconut shell carbon, 5 wt% Timcal Super C-45 conductive carbon, and 5 wt% KYNAR 761 grade PVDF (molecular weight: 1,000,000) as a binder. The coated slurry on copper foil was passed through a rolling mill to achieve a 2.75 mil thickness.

[0100] A Li ion capacitor was built in a CR2032 format cell. The cathode comprised 85% of the abovementioned Corning carbon, 10% PTFE (DuPont 601 A Teflon PTFE), and 5% Cabot Black Pearl 2000. The separator was NKK-4425 separator. 3.5 mg of the
The abovementioned lithium composite powder (LCP is used for pre-doping the anode) was coated on an anode made from coconut shell sourced carbon treated at 1600°C.

[00101] The cell was then crimped on a MTI coin cell crimper and conditioned on Arbin BT 2000 at constant current charge/discharge at 0.5mA current from 3.8V to 2.2V. The cell gave a discharge capacity of 105.394 mAh/gm (based on the anode carbon weight) after the third conditioning cycle (Fig. 2). The cell was subjected to C-Rate performance where the cell is charged at a constant current of 1 mA and discharged at different rates. Fig. 3 shows the C-rate performance of the heat treated (1600 °C) coconut shell based carbon on a volume basis. The cell had a maximum energy density of 48.032 Wh/1.

Example 8

[00102] Coconut Shell Sourced Carbon (with no wash and no second 1000 °C heat treatment) Example 4 was repeated with the exception that the carbon was not acid washed, did not receive a second heat treatment, there was no Raman analysis, and there was no elemental analysis. The resulting carbon was used in casting anode electrodes for a lithium ion capacitor as in Example 4 with the following particulars being noted: 90 wt% of the coconut shell sourced carbon of this Example, 5 wt% Timcal Super C-45 conductive carbon, and 5 wt% of KYNAR 761 grade PVDF (molecular weight: 300,000 to 400,000) as a binder. The coated slurry on copper foil was passed through a rolling mill to achieve a 4 mil thickness. The current collector is not included in the 4 mils thickness.

[00103] A Li ion capacitor was built in a CR2032 format cell. The cathode comprised 85% of the above mentioned Corning carbon, 10% PTFE (DuPont 601A Teflon PTFE), and 5% Cabot Black Pearl 2000. The separator was NKK-4425 separator. The 5 mg of lithium composite powder (LCP is used for pre-doping the anode) was coated on an anode made from coconut shell sourced carbon (carbonized at 1000°C).

[00104] The cell was then crimped on a MTI coin cell crimper and conditioned on an Arbin BT 2000 at constant current charge/discharge at 0.4 mA current from 3.8V to 2.2V. The cell had a discharge capacity of 65.971 mAh/gm (based on the anode carbon weight) after the third conditioning cycle (Fig. 4). The cell was subjected to C-Rate performance
where the cell was charged at a constant current of 1 mA and discharged at different rates. Fig. 5 shows the C-rate performance of the coconut shell sourced carbon on a volume basis. The cell showed maximum energy density of 40.35 Wh/l.

Example 9

[00105] Coconut Shell Based Carbon (with HC1 wash and no second 1000 °C treatment) Example 4 was repeated with the exception that the carbon did not receive a second heat treatment, there was no Raman analysis, and there was no elemental analysis. The resulting carbon was used in casting anode electrodes for a lithium ion capacitor as in Example 4 with the following particulars being noted: 90 wt% of the coconut shell sourced carbon of this Example, 5 wt% Timcal Super C-45 conductive carbon, and 5 wt% KYNAR 761 grade PVDF (molecular weight: 300,000 to 400,000) as a binder. The coated slurry on copper foil was passed through a rolling mill to achieve a 4 mil thickness (the current collector thickness is not included in the 4 mils).

[00106] A Li ion capacitor was built in a CR2032 format cell. The cathode comprised 85% the above mentioned Corning carbon, 10% PTFE (DuPont 601A Teflon PTFE), and 5% Cabot Black Pearl 2000. The separator was a NKK-4425 separator. 5mg of the above mentioned lithium composite powder (LCP is used for pre-doping the anode) was coated on an anode made from coconut shell sourced carbon (carbonized at 1000°C and washed with HC1).

[00107] The cell was then crimped on a MTI coin cell crimper and conditioned on an Arbin BT 2000 at constant current charge/discharge at 0.4 mA current from 3.8V to 2.2V. The cell had a discharge capacity of 50.31 mAh (based on the anode carbon weight) after the third conditioning cycle (Fig. 4). The cell was subjected to C-Rate performance where the cell is charged at a constant current of 1 mA and discharged at different rates. Fig. 5 shows the C-rate performance of the coconut shell sourced carbon (1000 °C) on volume basis. The cell showed maximum energy density of 45.58 Wh/l.

Example 10

[00108] Coconut Shell Sourced Carbon (Treatment of Raw Coconut Granules with HC1 followed by carbonization at 1000°C) Example 4 was repeated with the
exception that the coconut shell sourced carbon was raw coconut granules that were
treated with aqueous IN HCl followed by carbonization at 1000°C. Specifically, coconut
shell granules were washed with IN HCl for 16 hrs and rinsed with distilled water until a
pH of 5. The resulting HCl treated powder was then carbonized to 1000°C for 2 hrs under
nitrogen.

[00109] The resulting carbon was used in casting anode electrodes for a lithium ion
capacitor as in Example 4 with the following particulars being noted: 90 wt% of the
coconut shell sourced carbon of this Example, 5 wt% Timcal Super C-45 conductive
carbon, and 5 wt% KYNAR 761 grade PVDF (molecular weight: 300,000 to 400,000) as
a binder. The coated slurry on copper foil was passed through a rolling mill to achieve a 4
mil thickness (the current collector thickness is not included in the 4 mils).

[00110] A Li ion capacitor was built in a CR2032 format cell. The cathode comprised
85% of the abovementioned Corning carbon, 10% PTFE (DuPont 601 A Teflon PTFE),
and 5% Cabot Black Pearl 2000. The separator was NKK-4425 separator. 5 mg of the
abovementioned lithium composite powder (LCP is used for pre-doping the anode) was
coated on an anode made from coconut shell sourced carbon (i.e., raw coconut shell
washed with HCl and then carbonized at 1000°C).

[00111] The cell was then crimped on a MTI coin cell crimper and conditioned on an
Arbin BT 2000 at constant current charge/discharge at 0.4 mA current from 3.8V to 2.2V.
The cell had a discharge capacity of 69.61 mAh (based on the anode carbon weight) after
the third conditioning cycle (Fig. 4). The cell was subjected to C-Rate performance where
the cell is charged at a constant current of 1 mA and discharged at different rates. Fig. 5
shows the C-rate performance of the coconut shell sourced carbon of this example on a
volume basis. The cell had a maximum energy density of 44.1 l Wh/l.

[00112] The inventive carbon materials were characterized by: Raman analysis to have
a peak intensity ratio of $I_D/I_G$ from 1.40 to 1.85; and elemental analysis to have a
hydrogen content of from 0.01 to 0.25 wt%; a nitrogen content of from 0.01 to 0.55 wt%;
and an oxygen content of from 0.01 to 2 wt%.
[00113] The disclosure has been described with reference to various specific embodiments and techniques. However, it should be understood that many variations and modifications are possible while remaining within the scope of the disclosure.

Table 1. Comparison of Raman Intensities (I) for the Disorder (I_D) peak and Graphitic peak (I_G) and peak ratios (I_D/I_G) for different carbons.

<table>
<thead>
<tr>
<th>Carbon</th>
<th>I_D (a.u)</th>
<th>I_G (a.u)</th>
<th>I_D/I_G</th>
</tr>
</thead>
<tbody>
<tr>
<td>Comp. Ex. 1 (Phenolic Resin 660 °C)</td>
<td>2339.745</td>
<td>1952.962</td>
<td>1.20</td>
</tr>
<tr>
<td>Comp. Ex. 2 (Timcal Graphite-)</td>
<td>3019.060</td>
<td>2000.583</td>
<td>1.51</td>
</tr>
<tr>
<td>Comp. Ex. 3 (Coconut Char 600 °C)</td>
<td>2328.612</td>
<td>2002.222</td>
<td>1.16</td>
</tr>
<tr>
<td>Ex. 4 (Coconut Char 1000 °C)</td>
<td>3445.148</td>
<td>1928.368</td>
<td>1.79</td>
</tr>
<tr>
<td>Ex. 5 (Coconut Char 1200°C)</td>
<td>4675.030</td>
<td>3150.750</td>
<td>1.48</td>
</tr>
<tr>
<td>Ex. 6 (Coconut Char 1400°C)</td>
<td>6678.350</td>
<td>4126.940</td>
<td>1.62</td>
</tr>
<tr>
<td>Ex. 7 (Coconut Char 1600 °C)</td>
<td>7450.380</td>
<td>4139.710</td>
<td>1.80</td>
</tr>
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</table>

Table 2. Comparison of relative carbon, hydrogen, nitrogen, and oxygen weight percentages in different carbons.

<table>
<thead>
<tr>
<th>Example Number</th>
<th>Carbon %</th>
<th>Hydrogen %</th>
<th>Nitrogen %</th>
<th>Oxygen %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Comparative Ex. 1 (Phenolic Resin 660 °C)</td>
<td>95.27</td>
<td>1.76</td>
<td>0.1</td>
<td>2.11</td>
</tr>
<tr>
<td>Comparative Ex. 2 (Timcal-Graphite)</td>
<td>99.44</td>
<td>0.27</td>
<td>0.02</td>
<td>&lt; 0.1</td>
</tr>
<tr>
<td>Comparative Ex. 3 (Coconut Char 600°C)</td>
<td>94.10</td>
<td>2.12</td>
<td>0.15</td>
<td>2.88</td>
</tr>
<tr>
<td>Ex. 4 (Coconut Char 1000°C)</td>
<td>94.48</td>
<td>0.14</td>
<td>0.45</td>
<td>1.42</td>
</tr>
<tr>
<td>Ex. 5 (Coconut Char 1200°C)</td>
<td>98.44</td>
<td>0.13</td>
<td>0.44</td>
<td>0.75</td>
</tr>
<tr>
<td>Ex. 6 (Coconut Char 1400°C)</td>
<td>99.66</td>
<td>0.09</td>
<td>0.16</td>
<td>0.09</td>
</tr>
<tr>
<td>Ex. 7 (Coconut Char 1600°C)</td>
<td>99.66</td>
<td>0.08</td>
<td>0.08</td>
<td>&lt; 0.1</td>
</tr>
</tbody>
</table>
What is claimed is:

1. An anode in a lithium ion capacitor, comprising:
   a carbon composition comprising: a coconut shell sourced carbon in from 85 to 95 wt%; a conductive carbon in from 1 to 10 wt%; and a binder in from 3 to 8 wt%; and an electrically conductive substrate,
   wherein the coconut shell sourced carbon composition has a disorder peak to graphitic peak intensity ratio by Raman analysis of from 1.40 to 1.85; a hydrogen content of from 0.01 to 0.25 wt%; a nitrogen content of from 0.03 to 0.55 wt%; and an oxygen content of from 0.01 to 2 wt% by elemental analysis.

2. The anode of claim 1 wherein:
   the coconut shell sourced carbon in from 88 to 92 wt%;
   the conductive carbon in from 4 to 7 wt%; and
   the binder is PVDF in from 4 to 6 wt% and has a molecular weight of from 300,000 to 1,000,000.

3. The anode of any of claims 1 to 2, wherein the coconut shell sourced carbon composition has a disorder peak to graphitic peak intensity ratio by Raman analysis of from 1.48 to 1.8.

4. The anode of any of claims 1 to 3, wherein the coconut shell sourced carbon has a hydrogen content of from 0.01 to 0.24 wt%; a nitrogen content of from 0.04 to 0.5 wt%; an and oxygen content of from 0.01 to 1.9 wt%.

5. The anode of any of claims 1 to 4, wherein the coconut shell sourced carbon has a low surface area of from 1 to 100 m²/g.

6. The anode of any of claims 1 to 5, wherein the coconut shell sourced carbon has a particle size from 1 to 30 microns.
7. The anode of any of claims 1 to 6, wherein the coconut shell sourced carbon has a particle size from 2 to 7 microns.

8. The anode of any of claims 1 to 7, further comprising lithium composite powder coated on at least a portion of the anode.

9. A lithium ion capacitor, comprising:
   the anode of any of claims 1 to 8.

10. The lithium ion capacitor of claim 9 wherein the anode operates at a high charge-discharge rate of from 1C to 4000 C.

11. The lithium ion capacitor of any of claims 9 to 10, further comprising:
   a cell comprising a stack of:
   the anode having a lithium composite powder on at least a portion of the anode;
   a separator; and
   a cathode comprising a heat and KOH activated wheat flour sourced carbon, a fluoropolymer, and a conductive carbon black.

12. The lithium ion capacitor of any of claims 9 to 11, wherein the cell has a discharge capacity of from 60 to 120 mAh/gm and a maximum energy density of from 20 to 60 Wh/l.

13. A method of making a carbon composition comprising:
   a first heating of a coconut shell sourced carbon at from 1000°C to 1700°C for from 0.5 hrs to 5 hrs in an inert atmosphere; and
   grinding the resulting carbon to a powder.

14. The method of claim 13 further comprising washing the resulting powder with an aqueous acid and a second heating of the acid washed powder at from 1000 to 1700 °C.
15. The method of claim 14 wherein the first heating and the second heating are accomplished at from 1000 to 1600°C for 1 to 3 hrs.

16. The method of claim 14 wherein at least one of the first heating and the second heating is accomplished at 1100 to 1300°C.

17. The method of claim 13 wherein at least one of the first heating and the second heating is accomplished at 1300 to 1500°C.

18. The method of claim 13 wherein at least one of the first heating and the second heating is accomplished at from 1500 to 1700°C.

19. A carbon composition prepared by the method of claim 12.
FIG. 2

Discharge Capacity (mAh/gm)

Cycle Number

- Comparative Ex. 1
- Example 4
- Example 7
- Comparative Ex. 2
- Example 5
- Example 6
FIG. 3

The graph in Figure 3 illustrates the relationship between energy density (Wh/l) and power density (W/l) for various examples and comparative experiments. The graph includes lines and markers for:

- Comparative Ex. 1
- Example 4
- Example 7
- Comparative Ex. 2
- Example 5
- Comparative Ex. 3
- Example 6

The x-axis represents power density (W/l) ranging from 10 to 10,000, while the y-axis represents energy density (Wh/l) ranging from 1 to 100.
FIG. 4
FIG. 5

![Graph showing energy density vs. power density with different examples marked.

- Example 4
- Example 8
- Example 9
- Example 10]
INTERNATIONAL SEARCH REPORT

International application No PCT/US2016/015244

A. CLASSIFICATION OF SUBJECT MATTER
INV. H01G11/06 H01G11/34 H01G11/38 H01G11/42 H01G11/44
ADD. H01G11/50

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
H01M C01B H01G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

<table>
<thead>
<tr>
<th>Category</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
<th>Relevant to claim No.</th>
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<td>X</td>
<td>EP 1 211 702 AI (MITSUBISHI CHEM CORP [JP]; TOYOTA MOTOR co LTD [JP]) 5 June 2002 (2002-06-05) paragraphs [0014], [0015], [0017], [0019], [0024] - paragraph [0027]; cl aims 1-3 paragraphs [0029], [0032], [0033] the whole document</td>
<td>1-19</td>
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<tr>
<td>X</td>
<td>EP 0 757 505 AI (KUREHA CHEMICAL IND CO LTD [JP]) 9 Apr 1 1997 (1997-04-09) cl aims 1, 10, 12; examples 1, 2, 5 the whole document</td>
<td>1-19</td>
</tr>
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</table>

Further documents are listed in the continuation of Box C. See patent family annex.

* Special categories of cited documents:

- "X" document defining the general state of the art which is not considered to be of particular relevance
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Date of the actual completion of the international search: 20 May 2015
Date of mailing of the international search report: 02/06/2016

Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016

Authorized officer: Dessaux, Christopher
<table>
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<th>Patent document cited in search report</th>
<th>Publication date</th>
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