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Erman Taer <erman.taer@lecturer.unri.ac.id>

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Cassava peel derived self-doped and hierarchical porous carbon as an optimized electrode for the ultra-high energy density of supercapacitor

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Kepada: Erman Taer <erman.taer@lecturer.unri.ac.id>

Manuscript Number: DIAMOND-D-22-01002

Cassava peel derived self-doped and hierarchical porous carbon as an optimized electrode for the ultra-high energy density of supercapacitor

Dear Dr Erman Taer,

Thank you for submitting your manuscript to Diamond & Related Materials.

I have completed my evaluation of your manuscript. The reviewers recommend reconsideration of your manuscript following minor revision and modification. I invite you to resubmit your manuscript after addressing the comments below. Please resubmit your revised manuscript by 09/15/2022.

When revising your manuscript, please consider all issues mentioned in the reviewers' comments carefully: please outline every change made in response to their comments and provide suitable rebuttals for any comments not addressed. Please note that your revised submission may need to be re-reviewed.

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Diamond & Related Materials values your contribution and I look forward to receiving your revised manuscript.

Kind regards,

Xiliang Luo

Editor

Diamond & Related Materials

Editor and Reviewer comments:

Reviewer #1: Cassava peel derived hierarchical porous carbon was prepared, and its high energy density was reported. However, the manuscript still had a lot of problems, so it should be significantly revised before consideration of its acceptance.
(1) The full name of CIP/Z and CIP should be given when they first appeared, respectively?
(2) //Furthermore, the optimized CIP/Z carbon material exhibits a high content of 94.606% and a self-doping oxygen heteroatom of about 2.672%.// What was the other 2.722%? In addition, //a high content of 94.606%// should be modified to "high carbon content of 94.606%/".
(3) //CIP/Z electrode possess a ultrahigh energy density of 48.15 Wh kg-1.// It was more accurate expression that the symmetric supercapacitors based on CIP/Z electrode possess a ultrahigh energy density of 48.15 Wh kg-1.
(4) The descriptions to be improved are as follows: //hierarchical 3D porous carbon self-doping from cassava peel//. //Carbon N. Y.//. //The 3D pore has a unique sponge-like morphology with dominant micropores of 94%.// //specific power of 173.7 Wh kg-1// //temperature pyrolysis methods//. //It has been reported that cassava peel contains high chemical compounds cellulose of 37.0-66.9% and 11.3-37.9% [32].// //These compounds provide micro and meso structures important for ion diffusion and carbon storage[33]//. //However, the material properties have not been thoroughly optimized due to low relative electrochemical performance.//. //The EIS EIS method was evaluated……//. //during electrochemical//. //carbon SEM results//.
In short, English writing needs to be improved.
(5) What did "although it has resistance" mean in 1. Introduction?
(6) //Cassava peels were harvested from the plantations of the Pekanbaru community activity unit and washed in

a water container for several cycles until dirt, soil, and silica had been completely removed.// Can the silica in cassava peels be removed by washing with water? The relevant experimental evidence for this should be provided.
(7) What did "20 solid impregnated carbon precursors" and "obtained solid design" mean respectively?
(8) //Specific surface area (S⌣BET) and total volume (Vt) were reviewed using the Brunauer−Emmett−Teller (BET) equation.// The above statement was incorrect. Generally, the specific surface area of the sample is calculated according to the BET equation at the relative pressure of 0.05-0.25 (or 0.30), and the total pore volume is calculated using the Dubinin−Radushkevich curve at relative pressure of 0.995.
(9) What was the "organic membrane" in 2.5. Electrochemical measurements?
(10) //H3PO4 reacts with carbon in high-temperature pyrolysis, leaving P4 byevaporating H2O and CO [40].// What experimental evidence indicated that H3PO4 can react with carbon to form P4 (white phosphorus?) in high-temperature pyrolysis? Generally, the activation of phosphoric acid includes promoting hydrolysis, catalytic dehydration, condensation-crosslinking, promoting aromatization, and pore forming.
(11) //Based on these empirical equations [47, 48], chemically impregnated carbon is predicted to have a surface area of more than 900 m2/g.// //the Debye-Scherrer equation was adapted to produce XRD surface area through empirical equations.// The empirical equations should be provided.
(12) I think that the authors should reference the excellent works on hierarchical porous carbon structure, electrochemical energy storage devices, as far as I know, J. Power Sources, 2022, 536, 231512. Carbon, 2020, 168, 499-507. J. Mater. Chem. A, 2019,7, 1177−1186, and make modifications to the manuscript. In addition, these related works are recommended to be included in the References for balanced citations, providing more valuable information for the broader Readers.
(13) Why is the unit of equivalent series resistance (R⌣s) and charge transfer resistance (R⌣CT) in this manuscript Ω cm-2 ?
(14) //The pore structure found is in the range of 156-342 nm.// How was the specific surface area measured? What experiment / method was used to measure the specific surface area?
(15) //The best EDLC CIPs were found on the CIP/Z electrode, which displayed the highest specific energy of 48.15 Wh kg-1 at a specific power of 173.7 Wh kg-1.// //The best working efficiency of supercapacitor cells can show a jump in energy density of 48.15 Wh kg-1 at a specific power of 173.7 Wh kg-1 in an aqueous electrolyte of 1 M H2SO4//
What data (i.e. specific capacitance and operating voltage) was used to calculate the specific energy of 48.15 wh kg-1?

Reviewer #2: This study reports a hierarchical porous activated carbon derived from cassava peels through an up-to-date, easy, and pollution-free approach. The precursor's hierarchical pore and 3D morphology were optimized by chemical impregnation of ZnCl2, KOH, and H3PO4, followed by high-temperature pyrolysis. The porous carbon is designed as a binder-free coin to maintain the high conductivity of the base material. However, this manuscript has many issues to be addressed before considering the publication in Diamond & Related Materials.
1. In Keywords: "hierarchical porous; activated carbon" can be modified to "hierarchical porous carbon". "Energy density" should be used as one of the Keywords.
2. "specific capacitance 257 F g-1" should be revised to "specific capacitance of 257 F g-1". "The best working efficiency of supercapacitor cells" should be revised to "The best working efficiency supercapacitor cell" or "Supercapacitor cell with best working efficiency". "Figure 2. XRD pattern// should be revised to "Figure 2. XRD patterns". "energy (Esp, Wh kg-1), and power (Psp, W kg-1)" should be revised to "energy density (Esp, Wh kg-1), and power density (Psp, W kg-1)". In a word, written expression needs to be improved.
3. "the SEM micrograph in Figure 3c shows macrospore pores on the inner wall of the hole from CIP/Z with a diameter of 50-89 nm." It should be noted that the SEM image of Fig. 3c showed only a small number of pores, which was not commensurate with the characteristics of porous carbon. It is not an effective method to obtain the pore size of materials from SEM images.
4. "CIP/K confirmed their surface to be rich in mesoporosity and microporosity in the diameter size range of 21-49 nm and 57-189 nm." The above expression is incorrect because the pore size of the micropores is less than 2 nm.
5. It is suggested that the specific surface area data should be kept at most one decimal place. That was, "280.927 m^2 g^−1" should be revised to "280.9 m^2 g^−1". In addition, the pore volume of "0.48788 cm^3 g^-1" should be revised to "0.488 cm^3 g^-1".
6. "The apparent capacitance is indicated by the presence of oxygen in the carbon skeleton." What was the effect of calcium doping in carbon skeleton on its electrochemical capacitive performance?
7. "However, the specific capacitance can be maintained at 56.01-92.43% when the current density is increased from 1 to 10 A g-1." Which porous carbon sample had a capacitance retention rate of 92.43%, and which carbon sample had a capacitance retention rate of 56.01%?
8. Some recent studies on hierarchical pore structure, electrochemical energy storage, e.g., ACS Appl. Mater. Interfaces, 2022, 14, 33328−33339. Carbon, 2021, 180, 135-145. Angew. Chem. Int. Edit., 2022, 61, e202208821, can be included in References.
9. The format of References should be consistent. For example, the first letter of the titles should be in uppercase and the other letters should be in lowercase.
10. The unit of equivalent series resistance and charge transfer resistance should be checked.

11. The authors have miscalculated the gravimetric specific energy density of the device. For a symmetric supercapacitor system, the energy density metrics should be calculated based on the total mass of the active materials in the two electrodes. The following equations are provided for the authors to calculate energy density accurately:
Csingle electrode (F g−1) = 4Cdevice = 4 × I × Δt/(mtotal × ΔV).
E (Wh kg−1) = 1/2 × Cdevice × ΔV2 = 1/7.2 Cdevice × ΔV2
where Csingle electrode denotes the capacitance of one electrode, Cdevice is the device capacitance, mtotal represents the total mass of activate material on two electrodes, Im is the current density (A g−1).
According to my assessment, the reported value of 257 F g−1 should be the capacitance of a single electrode, the device capacitance accordingly should be 64.25 F g−1. Then, the energy density of the device should be 8.9 Wh kg−1. Obviously, the claimed energy density of 48.15 Wh kg−1 by these authors is wrong. The authors should recalculate the electrochemical values in the full work in the right way.

Reviewer #3: 1.The these chemicals activation of biomass carbon has been extensively studied, could you please pointed out the novelty of this work ?
2.The properties of porous carbon obtained are quite different. What is the main reason of this? Further activation mechanisms and thier effects should be explained here.
3.It would be better to add the testing of cycling performance, which is considerably important to supercapacitors.

Reviewer #4: The manuscript titled "Cassava peel derived self-doped and hierarchical porous carbon as an optimized electrode for the ultra-high energy density of supercapacitor" is not novel enough to publish in this journal. There are a lot of technical corrections that need attention. This paper just reports another biomass-derived carbon without any novelty in its synthesis or results. Therefore, it is rejected. Following are the major comments that need to be addressed before the next submission.
1- In the last part of the introduction, the authors have stated that there are papers on cassava peel-based supercapacitor application. In what way this activated carbon differs from the previously published paper? I do not see any novel method of activation. Authors have just used different activating agents and two different atmospheres during pyrolysis, which makes the experimental process high cost and time-consuming. Cite and refer: Carbon 2020, 168, 209-219
2- Author's did not mention, how the working electrodes are prepared without a binder.
3- The SEM images don't confirm the porosity of the samples. But authors have stated mesoporosity. There is no visible pore structure present in any of the activated samples.
4- Moreover, the specific surface area is not extremely high when compared to the hierarchical porous carbon reported in the literature. Cite and refer: Journal of Energy Storage 2021, 43, 103261.
5- From the GCD plot of Fig. 7(b), the value of discharge time and the current density does not match the values of specific capacitance. For example, at 2 A/g, the discharge time is approx. 500. If so the capacitance should be 1000 F/g. It's contradictory. Moreover, the charge-discharge curves are not linear at 1A/g.
6- The ESR value for each sample should be given from the EIS spectrum, which further confirms the overall resistance.
7- The manuscript states it has heteroatom doping of oxygen. Oxygen element is found along with carbon in almost all cases, by which author's cannot claim it be an additional heteroatom doping.
8- The stability measurement are missing. Authors should report cyclic stability of symmetric device up to 10000 charge-discharge cycles in both the electrolytes. Cite and refer: Journal of Energy Storage 38, 102533.
9- Authors should indicate the comparison values of other reported materials in the form of table with all capacitance parameters with recent publications such as: Energy & Fuels, 2022, 36, 1, 638-654; Renewable Energy 161, 173-183.

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Kepada: Erman Taer <erman.taer@lecturer.unri.ac.id>

Manuscript Number: DIAMOND-D-22-01002R1

Cassava peel derived self-doped and hierarchical porous carbon as an optimized electrode for the ultra-high energy density of supercapacitor

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Thank you for submitting your manuscript to Diamond & Related Materials.

I have completed my evaluation of your manuscript. The reviewers recommend reconsideration of your manuscript following minor revision and modification. I invite you to resubmit your manuscript after addressing the comments below. Please resubmit your revised manuscript by 10/03/2022.

When revising your manuscript, please consider all issues mentioned in the reviewers' comments carefully: please outline every change made in response to their comments and provide suitable rebuttals for any comments not addressed. Please note that your revised submission may need to be re-reviewed.

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Diamond & Related Materials values your contribution and I look forward to receiving your revised manuscript.

Kind regards,

Xiliang Luo

Editor

Diamond & Related Materials

Editor and Reviewer comments:

Reviewer #1: The manuscript has been extensively and well revised. Accepted after minor modification.
Minor questions:
(1) //with large organic elements of calcium and phosphorus at 7% and 1%.// It should be noted that calcium is not an organic element, so "organic elements" can be changed into organic components (including organic components containing calcium).
(2) "isotherm" should be revised to "isotherms" because the isotherms include the two parts of adsorption and desorption.

Reviewer #2: Accept.

Reviewer #4: The authors have improved up to some extent but failed to address the main comments as mentioned below. This manuscript cannot be accepted in this format. Following are the main comments to be addressed for a supercapacitor device application.

1- The calculation of specific capacitance from GCD curves doesn't match the values provided in Fig. 7(c). From the GCD plot of Fig. 7(b), at 1 A/g, the discharge time is approx. 350. If so the capacitance should be 350 F/g. It's contradictory in Fig. 7(c) at 1 A/g (shows below 300 F/g). Also, the charge-discharge cures show low coulombic efficiency with very low discharge time.

2- Moreover, the Ragone plot was plotted using specific capacitance from CV curves, which is not the right way to do it.

3- Authors have considered the mass of a single electrode for calculation, which leads to very high energy density, which is wrong. As stated by other reviewers, authors should reconsider the calculation for a device on the whole and report the energy density accordingly. "According to the assessment, the reported value of 257 F g−1 should be the capacitance of a single electrode, the device capacitance accordingly should be 64.25 F g−1. Then, the energy density of the device should be 8.9 Wh kg−1. Obviously, the claimed energy density of 48.15 Wh kg−1 by these authors is wrong. The authors should recalculate the electrochemical values in the full work in the right way"

4- The stability measurement is an important parameter for supercapacitor device applications. Authors have reported symmetric cell assembly, which is more than enough to report the stability of the constructed device. They should the report cyclic stability of symmetric devices up to 10000 charge-discharge cycles.

5- Authors failed to provide a comparison table with all capacitance parameters with recent publications.

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Kepada: Erman Taer <erman.taer@lecturer.unri.ac.id>

Manuscript Number: DIAMOND-D-22-01002R2

Cassava peel derived self-doped and hierarchical porous carbon as an optimized electrode for the ultra-high energy density of supercapacitor

Dear Dr Erman Taer,

Thank you for submitting your manuscript to Diamond & Related Materials.

I have completed my evaluation of your manuscript. The reviewers recommend reconsideration of your manuscript following minor revision and modification. I invite you to resubmit your manuscript after addressing the comments below. Please resubmit your revised manuscript by 10/14/2022.

When revising your manuscript, please consider all issues mentioned in the reviewers' comments carefully: please outline every change made in response to their comments and provide suitable rebuttals for any comments not addressed. Please note that your revised submission may need to be re-reviewed.

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Diamond & Related Materials values your contribution and I look forward to receiving your revised manuscript.

Kind regards,

Xiliang Luo

Editor

Diamond & Related Materials

Editor and Reviewer comments:

Reviewer #1: The manuscript had been revised as per the comments and suggestions (Original Submission and Revision Number 1) made by this reviewer. Accept.
It should be noted that the energy density of CIP/Z based symmetrical supercapacitor assembled by 1M Hv2SOv4 electrolyte was 35.69 Wh kg^-1, which was indeed very much higher than other reported values. Check this.

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Manuscript Number: DIAMOND-D-22-01002R3

Cassava peel derived self-doped and hierarchical porous carbon as an optimized electrode for the ultra-high energy density of supercapacitor

Dear Dr Erman Taer,

Thank you for submitting your manuscript to Diamond & Related Materials.

I am pleased to inform you that your manuscript has been accepted for publication.

My comments, and any reviewer comments, are below.
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