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Journal	**Energy Sources, Part A: Recovery, Utilization, and Environmental Effects**

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Energy Sources, Part A: Recovery, Utilization, and Environmental Effects

Dear Professor,

We would like to submit our manuscript entitled *"Mission grass bio-waste functional carbon self-single-doped for ultrahigh energy symmetrical supercapacitor"* to be review and considered as an accepted manuscript in the *Energy Sources, Part A: Recovery, Utilization, and Environmental Effects.*

This work submitted has not been previously published, in whole or in part, and is not under consideration for publication elsewhere and the work described is approved for publication by all co-authors and the responsible authorities where the work was carried out.

To improve the electrochemical performance supercapacitor, the several parameters are required: (1) high surface area with the micro-mesoporous structure for redox reactions, (2) tuning the morphology of the electrode material in 3D hierarchical porous and 2D nanomaterial (nanosheet-nanofiber), (e) doping heteroatoms to increase the conductivity and redox activity, and (3) high charge/discharge rate. Bio-waste has been identified as the most common precursor for porous carbon, based on the a high specific surface area of relatively 3000 m^2g^{-1}, unique and optimized pore structure, extremely conductive, and good thermal and electrical stability. Porous carbon derived from biomass provides a natural structure and pore which could be modified, which is plays a key role in the improvement of specific capacitance. The surface modification is obtainable by chemical activation, suggested as a simple and low-cost route. Also, the hierarchically micro-mesopores nanofiber structured is attainable by thermal treatment including one-stage integrated pyrolysis both carbonization and physical activation, by decomposing the cellulose, hemicellulose and lignin from biomass. Here, we have modified the morphological structure of porous activated carbon in a binder-free coin/monolith solid design derived mission grass bio-waste through activating agent ratio in high-temperature pyrolysis.

We report a facile and eco-friendly technique without hard, salt, soft templates, metal framework for hierarchical porous carbon with enrich self-oxygen doped derived mission grass bio-waste for sustainable electrode materials. This study was conducted to show the great potential of mission grass bio-waste to produce activated carbon with hierarchical structure, abundant nanofiber, enrich oxygen functional groups, beneficial provide active channel and wettability to enhanced electrochemical behavior of supercapacitor. As far as we know, this study is the first reported to reveal the potential of mission grass bio-waste as a high-quality porous carbon source for sustainable electrodes.

Furthermore, porous carbon electrodes were produced without the addition of adhesive materials and they are in a binder-free coin/monolith solid design. Combined with the high carbon content of 93.89% and the specific surface of 639.414m^2 g^{-1} with a high specific capacitance of 208 F g^{-1} in symmetrical system at 1.0 Ag^{-1} in 1 M H_2SO_4 electrolyte. Moreover, the energy density of the assembled symmetric supercapacitor electrodes is as high as 28.31 Wh kg^{-1} and power density of 65.47 W kg^{-1}. Therefore, these properties enable an effectively feasible approach to obtaining self-single-doped of porous carbon based on mission grass bio-waste as an electrode material for energy storage devices. Finally, this

result is worthwhile submitted to the *Energy Sources, Part A: Recovery, Utilization, and Environmental Effects.*

In general, the highlights for this study are summarized as follows

- Bio-waste mission-grass converted to self-O-doped porous carbon
- One-step chemical activation strategy enhanced micro-mesoporosity
- In symmetrical supercapacitor, energy output enhanced as high as 28.31 Wh kg^{-1}

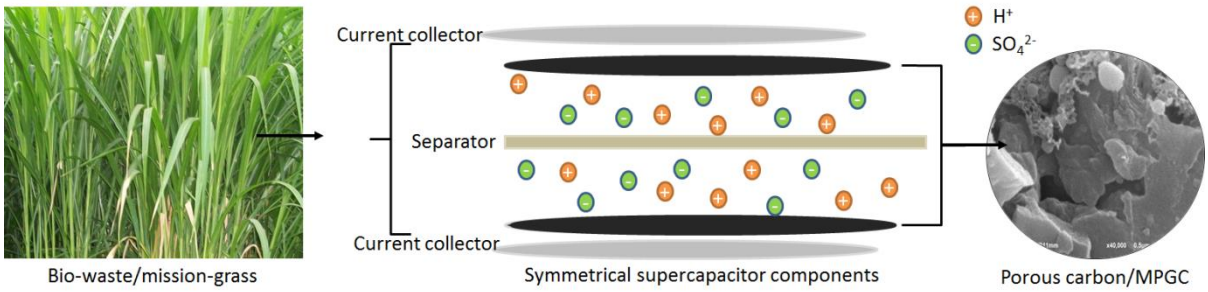

Figure 1. Graphical abstract

We confirm that this manuscript was prepared in accordance with the Author Guidelines of the *Energy Sources, Part A: Recovery, Utilization, and Environmental Effects*, has not been published elsewhere, is not under consideration by another journal, and that all authors have approved the manuscript and agree with its submission. We hope the novelty that we tried to emphasize in this manuscript would give a significant contribution to the readers of the *Energy Sources, Part A: Recovery, Utilization, and Environmental Effects*.

Sincerely yours,

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Mission grass bio-waste functional carbon self-single-doped for ultrahigh energy symmetrical supercapacitor

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Abstract

Several studies have been performed on biomass as an extraordinary natural resource for porous carbon as an electrode material with combined pore structure, self-doping, and nanopores morphology significantly boosted supercapacitor performance. Therefore, this study utilized mission-grass bio-waste as a porous carbon source self-single-doped as electrode material capable of increasing the energy density of supercapacitor. Porous carbon was obtained through the chemical impregnation synthesis route of $ZnCl_2$ and physical activation at temperatures 800, 850, and 900°C. The electrode material was designed in solid form without adding synthetic materials. The optimized mission-grass-based porous carbon (MGPCs) illustrates the diverse pore structure, and wettability properties of the oxygen functional groups. In the supercapacitor symmetrical system, the MGPCs electrode showed the highest electrochemical properties with a specific capacitance of 208 F g^{-1} at 1 A g^{-1} in an aqueous electrolyte. Furthermore, the energy density of the electrodes was increased by 28.31 Wh kg^{-1} with good electrical conductivity at equivalent series resistance of 0.139Ω. The high electrochemical performance of the MGPCs electrodes proved that the insertion of mission grass biomass into porous carbon converted through an environmentally benign strategy obtained high-quality electrode material to boost the energy density of the supercapacitor.

Keywords: porous carbon; biomass; electrode materials; symmetrical supercapacitor

1. Introduction

This decade is considered the highest level of human modernization because it is marked by the development of the global economy, the extraordinary growth of the technology industry, as well as the evolution of biotechnology and cultural engineering, thereby leading to a rapid increase in the level of prosperity globally (Szocik & Braddock, 2022). However, this modernization increase has some determinantal effects, such as higher pollution levels, rapid depletion of fossil fuels, and worsening environmental pollution, which decreases public health and the survival of the next generation (Deng et al., 2022). The world's major institutions have tried to apply a common frame of mind for "green growth technology" to accelerate the transfer of conventional energies to renewable green resource technologies (Guo et al., 2020). Among the numerous intensive researches on increasing renewable energy conversion, the supercapacitor is one of the most widely studied superior energy storage devices because of its extraordinary features (Alem et al., 2022; Ciszewski et al., 2019). These include unlimited life cycle, relatively fast charging-discharge in minute scale, extraordinary power density, system safe operation, long service life, and high component stability (González et al., 2016; Majid et al., 2021). These advantages enable supercapacitors as prime candidates to develop the next generation of energy conversion, particularly in the practical application of electronic components, spare parts, hybrid vehicles, pulse laser components, and crane and lift auxiliary systems (Deraman et al., 2016; Iro et al., 2016). However, supercapacitors are considered to still have obstacles in their practical application, especially at low energy densities lower than conventional fuel cells and batteries. The increase in energy density is heavy because it erodes their high power performance. To overcome this problem, many intensive studies have been dedicated to increasing the energy density and capacity of supercapacitors without reducing their high-power density. The strategies

used to improve the promising, effective and efficient electrochemical performance of supercapacitors' are 2D nanostructures, pore hierarchies, heteroatom doping, selecting the appropriate electrolyte with higher voltage operation, and excellent ionic conductivity (Pal et al., 2019; G. Wang et al., 2012). The combination of these extraordinary properties leads to high energy densities while maintaining their power densities. Furthermore, the design of the electrode material through the incorporation and transition of metal oxides, such as ruthenium, nickel, and vanadium, demonstrates stability during redox reactions in an easily accessible and abundant pore distribution (Dhara & Mahapatra, 2019). Additionally, among various metal oxides, MnO_2 is the leading compound that exhibits superior electrochemical behavior with important applications in many energy conversion and storage processes (Long et al., 2020). On the other hand, electrospinning and the use of hard, soft, and salt template methods based on polymeric materials, such as polypyrrole and polyaniline, allow electrode materials to have rich, regular 2D nanostructures, abundant hierarchical pores that lead to high material conductivity properties, excellent ion accessibility, and good flexibility (Zang et al., 2020). Zhang *et al.* (2017) reported that graphene-polymer-based electrodes with 3D pore features are able to compete with the battery energy density of 28.06 Wh kg^{-1} at a high specific capacitance of 1,182 F g^{-1} (L. Zhang et al., 2017). However, the synthesis routes described are relatively complex, expensive to manufacture, corrosive, and toxic, thereby compromising their service life and hindering the goal of green and environmentally benign energy storage. Biomass is generally known as one of the abundant natural resources with extraordinary potential energy sources and conversion. Several studies suggest that the world's total land biomass is about 1.8 trillion tons each, with a potential energy production capacity of 33,000 EJ (Tursi, 2019). Its conversion value is 80 times greater than the world's annual energy consumption. Interestingly, the high carbon source in biomass has

tremendous potential as a base material for high-quality electrodes. Due to its controllable porous structure, unique nanostructure potential, good physical-chemical stability, high electrical conductivity, and abundant heteroatom doping, biomass-based activated carbon uses approximately 80% of the electrode base material for electrochemical energy storage devices (Abbas et al., 2019; Saini et al., 2021; Y. Zhang et al., 2017). Furthermore, biomass is inexpensive, abundantly available, sustainable, environmentally friendly, easy synthesis route, and non-toxic-corrosive, hence it is very suitable to be developed as a source of green electrode material for sustainable supercapacitor applications (Herou et al., 2018; Z. Li et al., 2020). Several recent studies have reported the potential of waste biomass as electrode material, such as banana leaves (Taer, Taslim, et al., 2022), onion skin (Gopalakrishnan & Badhulika, 2021), eucalyptus wood (Atika & Dutta, 2021), camellia husk (Cui et al., 2022), *Osmanthus fragrans* (Quan et al., 2018), bamboo (Taer et al., 2020), sakura flower (Ma et al., 2019), mangosteen (Yang et al., 2019), and *Terminalia Catappa leaf* (Taer et al., 2018). The *Averrhoa bilimbi* leaves are an extraordinary combination of 3D hierarchical pores and self-doping heteroatoms with impressive energy densities of 26.54 Wh kg^{-1} in a symmetrical supercapacitor system (Taer, Apriwandi, et al., 2022). Li *et al.* (2021) reported the potential of the eggplant as a porous carbon electrode with similar features with an increase in power density reaching 38 Wh kg^{-1} (W. Li et al., 2022). However, the time-consuming synthesis route and real low stability need to be optimized by intensely finding a truly promising approach. In addition, mission grass was confirmed to provide abundant lignocellulose components with the proportion of lignin (15.6%), cellulose (40.02%), and hemicellulose (29.2%). This combination of elementals makes it possible to provide unique nanopores that enhance the electrode material's performance.

This research used the environmentally benign and time-saving approach to obtain biomass-based porous carbon as a quality electrode material for symmetric supercapacitor applications. Biomass precursors of mission grass were defined in all their unique potential, while the chemical impregnation synthesis route at high-temperature pyrolysis obtained porous carbon. The material properties of the porous carbon were controlled through different physical activation temperatures, including 800, 850, and 900°C. The characterization of the material properties results showed that the optimized precursor had a high amorphous carbon structure, with various hierarchically connected pores, and a high purity carbon state. Furthermore, the electrochemical properties exhibit high capacitive properties of 208 F g^{-1} at 1 A g^{-1} in 1 M H_2SO_4 electrolyte with an energy density of 28.31 Wh kg^{-1} in a symmetric supercapacitor system. The proposed environmentally benign approach explicitly explores mission-grass-based porous carbon as an electrode material for sustainable supercapacitor applications.

2. Materials and Methods

2.1. Mission grass-based biomass porous carbon preparation (MGPCs)

One sack of mission grass waste that grows wild in Pekanbaru, Riau Province, is collected, washed with running water, and cut into small pieces about 5 cm in size. The samples were dried in the sun for several days and in an oven at 110°C. Subsequently, the sample was pre-carbonized by about 30 g at 250°C in a vacuum oven. The pre-carbonized samples were also crushed using a milling machine until the samples were in powder form and then sieved through a 250-mesh sieve. The powdered sample was chemically activated in a $ZnCl_2$ solution with a concentration of 0.5 M using a hotplate at 80°C and 300 rpm and dried in an oven at 110°C for 48 hours. Next, carbon powder was printed into monoliths (coins) using a hydraulic press with 15 monoliths for each treatment. The sample was then carbonized by single-stage pyrolysis at room temperature to

a maximum temperature of 600°C in an N_2 gaseous environment, followed by physical activation at different high temperatures such as 800°C, 850°C, and 900°C in a CO_2 gas atmosphere. Finally, the carbon monolith samples were neutralized using distilled water, dried at 110°C, and labeled MGPC-800, MGPC-850, and MGPC-900, according to the varying temperatures.

2.2. Material Characterization

The scanning the electron microscopy (SEM) and energy dispersive spectroscopy (EDS) was used to evaluate morphological structure and elemental status with JEOL-JSM-6510LA instrument with an applied. The N_2 gas adsorption-desorption method was used to reviewed the porosity properties with BET, BJH, and T-plot approaches.

2.3. Electrochemical Measurements

The electrochemical performance of a porous carbon monolith as a supercapacitor electrode was measured in cyclic voltammetry (CV) using a CV UR Rad-Er 5841 instrument calibrated by VersaStat II Princeton Applied Research at an error rate of ±6.0%. The voltage window ranges from 0 to 1 V with scan rates of 1, 2, 5, and 10 mV s^{-1}. The working electrodes are assembled in a symmetric supercapacitor system in a sandwich layer immersed in an aqueous electrolyte of 1 M H_2SO_4. Additionally, the electrochemical properties of the samples were also measured by galvanostatic charge-discharge (GCD). Specific capacitance, energy density, and power density are evaluated using Equations standard. Furthermore, the method of electrochemical impedance spectroscopy (EIS) was also carried out to confirm the in-depth electrochemical properties of the electrode material. Electrochemical impedance spectroscopy (EIS) was performed from 10 mHz to 100 kHz, with the specific capacitance, as well as energy and power densities, determined by standard equations based on a two-electrode system.

3. Results and Discussions

The physical activation reaction of high-temperature CO_2 on the carbon chain affects the surface morphology of MGPC solids, which were reviewed in detail using scanning electron microscopy (SEM) with the images of MGPC-800, MGPC-850, and MGPC900 shown in Fig. 1 at different magnifications. Micrograph of MGPC-800 showed a dominated large particle aggregates, as shown in Fig. 1a. In the enlarged selection area in Fig. 1b, MGPC-800 displays a large pore on the particle block wall, confirming the presence of a micron-sized pore. Furthermore, the increase in the physical activation temperature at 850°C exhibits a relatively dominated surface morphology by small-size carbon blocks with significant aggregate breakdown. According to Fig. 1c, the size of the carbon blocks varies from 0.063µm to 2.498 µm with etching carbon chains at high temperatures, which allows dramatic erosion of the lignocellulosic components, thereby affecting the surface structure. Significant erosion of cellulose and lignin initiates the formation of 2D nanostructures, such as tubular, fiber, and sheet (Kumar et al., 2020). Fig. 1d displays a magnified area of the MGPC-850 SEM image confirming a morphological structure resembling a rod/tube at 30-36nm diameter. The etching of optimal carbon chains occurs at 850^{o}C with morphology that leads to 2D nanostructures. The continuous erosion of the particle aggregate at high temperatures up to 900°C allows the precursor-based material to undergo significant morphological changes, as shown in Fig. 1e. The MGPC-900 displays relatively large pores in the macropores range with aggregates ranging from 0.238µm to 2.276µm and relatively larger than MGPC-850. Furthermore, in the selected area selection, the MGPC-900 displays pores of various mesoporous scales interconnected in 3D. These pore characteristics contribute significantly to ion accessibility and barrier-free charge transfer on the

electrode material to maintain high power density performance in supercapacitor energy storage components (Hao et al., 2017).

The degree of crystallinity and phase change that occurred in the porous carbon of MGPCs at different physical activation temperatures was assessed using an X-ray diffraction (XRD) pattern, as shown in Fig. 2. It showed two significant broad peaks at 2θ approximately 21.53°, 24.15°, and 24.76° indexed to the scattering plane (002) and 42.71°, 44.91°, and 45.44° which are correlated to the scattering plane (100) of MGPC-800, MGPC-850, and MGPC-900, respectively. It further characterizes the amorphous-turbostratic carbon structure rich in pores and attenuated crystallinity (JCPDS No. 41–1487) (Peng et al., 2013; Sodtipinta et al., 2017), which proves that the sample has a confirmed pore structure. The XRD pattern was also obtained on porous carbon from various precursor sources such as potatoes (Chen et al., 2016), cotton stalks (Tian et al., 2021), bamboo (G. Zhang et al., 2018), and *Areca catechu* husk (Taer et al., 2019) to exhibit good results. Increasing the physical activation temperature from MGPC800 to MGPC-850 displays a shift of the indexed width peak (002) towards a smaller one, thereby confirming the abundant increase in the growth of narrow pores. Additionally, the broad peak in the scattering plane (100) shifted to a greater extent from 42.487° to 45.710°, allowing MGPC-850 to have confirmed mesopores on the precursor surface. The combined features of microporous and mesoporous growth in the based material contribute to the electrode's high capacitive properties and low internal resistance. This analysis is further justified through the analysis of electrochemical properties using CV and GCD techniques. However, an increase in the physical activation temperature up to 900°C displays a different pattern and leads to the opposite feature, thereby confirming a good reduction in pore development. This is because excessive carbon chain etching at 900°C damaged the pore framework structure of the precursor.

The status of the elemental components present in MGPCs is revealed through energy dispersive spectroscopy (EDS) measurements, which are summarized in Table 1. Evaporation of water content, volatiles, polysaccharides, and hemicelluloses, as well as degradation of other organic components due to carbonization in inert gases, initiates the production of high carbon fixed. Furthermore, the high physical activation temperature followed by carbon chain removal significantly reduced the lignocellulosic content leading to the strengthening of the porous carbon skeleton. Table 1 show that the main constituent elements of all samples were dominated by high carbon at 93.73%, 93.89%, and 93.13% for MGPC-800, MGPC-850, and MGPC-900, respectively. This high carbon content allows the electrode material to produce high electrical conductivity properties followed by a low equivalent series resistance, enhancing the supercapacitor's outstanding performance. Conversely, oxygen occupies the second highest element in the range of 4.02% to 6.02% because the excessive etching of carbon chains by CO_2 allows the precursor to produce oxidation byproducts that bind to inorganic components. The high oxygen content has a positive side for the electrode material, especially in their contribution to the improvement of wettability, maintained conductivity, self-doping of heteroatoms, and the pseudo-capacitance effect on energy storage devices (Abbas et al., 2019; Zheng et al., 2021). Due to incomplete evaporation of the remaining pyrolysis production, Silicon and zinc are also found in small amounts in MGPC.

The porosity behavior of the MGPCs obtained from high-temperature pyrolysis differ regarding the N_2 sorption isotherm and pore size distribution, as shown in Fig. 2a-b and summarized in Table 1. The N_2 isotherm adsorption-desorption profiles of MGPCs show a prominent combination feature of the type-type I and type IV curves, as showed in Figure 2a. High N_2 absorption in the region of very low relative pressure characterizes strong I-type

features, indicating that more than 90% of micropores dominate MGPCs. This feature is also confirmed in Table 1, which shows that the average pore diameter of MGPCs is around 1 nm. Micropores are abundant on the 1 nm pore size, thereby causing a drastic increase in the ion pool at the electrolyte/electrode interface, initiating the promotion of abnormally high specific capacitance. This assumption is analyzed more deeply in CV and GCD measurements. At the same time, the hysteresis loops displayed over a greater relative pressure region confirm the presence of type-IV, closely related to defined mesoporosity. This feature is useful to increase the accessibility of ionic charges on absorption surfaces for the electrolyte ions to move in all directions, thereby initiating an increase in fast currents and high-power capabilities. However, the hysterical loop on MGPC-800 is imperfect (not closed) and reflects the creation of imperfect mesopores with the assumption that the pore shape resembles a "bottleneck" with a narrow pore surface and relatively large space on the inside. The defect of the mesoporous structure can be corrected by increasing the pyrolysis temperature. The higher porosity temperature in MGPC-850 and MGPC-900 indicates an increase in the mesopores structure, which is illustrated by their better hysteresis loop compared to MGPC-850. Figure 2b shows that the increased pyrolysis temperature increased the pore size distribution on the mesoporous scale with an average size of 3.62nm. This also increases the mesoporous volume from 0.019 cm^3 g^{-1} to 0.040 cm^3 g^{-1}, as shown in Table 1. The increase in pyrolysis temperature can significantly raise the growth of the specific surface area of MGPCs from 800 to 900°C, with a rapid rise in the specific surface area reaching ± 53% of 639.414 m^2 g^{-1}. Moreover, their micropore dominance is also optimized to 94.6% with well-defined mesopores, which simultaneously serve as the main key to boosting the high electrochemical performance of electrode materials. These include increasing the specific capacitance and energy density without compromising their high power density.

The electrochemical performance of the MGPCs electrode was first traced through the cyclic voltammetry (CV) method on a two-electrode configuration in a 1 M H_2SO_4 liquid electrolyte. At a scan rate of 1mV s^{-1}, the CV profiles of the MGPC-800, MGPC850, and MGPC-900 illustrate a distorted/non-ideal rectangular shape (Fig. 3a), thereby revealing normal electrical double-layer properties (Climent & Feliu, 2018). The MGPC-800 electrode displays a relatively small CV profile, which characterizes its low capacitive properties. In the low voltage range V<0.1V, the current density increases suddenly, confirming that the ionic charge is simultaneously transmigrated to the micropores on the electrode surface. Furthermore, their CV profile tends to slope at higher potential ranges, confirming the relatively low ionic charge transfer in the large pores with a low capacitive property of 155 F g^{-1}. On the other hand, the MGPC-850 electrode displayed the largest charge-discharge loop hysteresis, confirming the highest electrochemical properties. At a potential <0.1, the current density increased significantly, indicating that the micropores-rich MGPC-850 material filled with electrolytic ions formed the first electrical double layer. The sudden increase in current density that forms a "camel hump" in the voltage range 0.20<V<0.50 indicates a functional contribution of oxygen as a self-doping heteroatom that initiates the faradaic reaction in the electrode material (Meng et al., 2020), which presents a pseudo-capacitance effect on the electrochemical properties of MGPC-850. Interestingly, in the voltage range V>0.6V, the current density continued to increase drastically until V=1.0V, indicating that the mesoporous MGPC-850 is optimally accessed by the electrolytic ion charge adding to the abundant electrochemical double layer capacitor. The high-performance combination of the MGPC-850 electrode exhibits an increased capacitive value of 191 F g^{-1}. This proves that synthesizing biomass-based porous carbon as electrode material by increasing the physical activation temperature of CO_2 increases the specific capacitance of the supercapacitor,

especially in the two-electrode configuration system. However, the increased physical activation temperature on the MGPC-900 did not display the same characteristic. The CV profile displayed by the MGPC-900 electrode is relatively similar to the MGPC-800, indicating that their capacitive properties are the same. The reduction of amorphous and carbon content of MGPC-900, which XRD and EDS analyzed, had a major impact on the reduction of the electrochemical properties of MGPC-900. The specific capacitance obtained in the symmetric cell MGPC-900 is about 165 F g^{-1}. The electrochemical properties of MGPCs were confirmed through scanning rates from 1 mV s^{-1} to 5 mV s^{-1}, as shown in Fig. 3b-d. The CV profile generally shows an increasingly ideal rectangular shape confirming the potential for MGPCs electrodes to have normal electrical double-layer capacitor behavior based on porous carbon. Moreover, their cyclical performance is also reviewed in terms of specific capacitances at different scanning rates, as shown in Fig. 3e. The specific capacitance of the MGPCs electrodes degraded drastically at a scanning rate of 10 mV s^{-1} because the carbon material has a low meso-macroporosity, therefore, the ionic charge is not optimally induced at the electrode/electrolyte interface (Y. Zhang et al., 2017). The electrochemical behavior of MGPCs was evaluated through a galvanostatic charge-discharge (GCD) which represents the detailed behavior of specific capacitive properties, electrode resistance, energy, and power densities in a symmetric supercapacitor cell system. The GCD profile for the electrodes of MGPCs immersed in 1M H_2SO_4 electrolyte at a current of 1 A g^{-1} is shown in Fig. 4a. In general, all electrodes in a symmetrical cell system displayed disturbed isosceles triangle curves, thereby confirming their normal potential in the electrochemical double layer capacitor type supercapacitor. Furthermore, their relatively long charging time compared to the discharging time indicates ion degradation in the charge transfer process. According to preliminary studies, the wettability of the oxygen

functional group initiates the faradaic reaction (Luo et al., 2021). This leads to an increase in the pseudo-capacitance property, as confirmed in the CV curve. Coulombic efficiency is obtained from the data on the difference between the charging and discharging time of the GCD (η=[td/tc]x100%). Their coulombic efficiency is about 90% close to 100% indicating that their properties are ideal for high EDLC. Furthermore, a coulombic efficiency of around 60-80% indicates a pseudocapacitance effect on the sample due to the ion degradation of heteroatom doped. In this study, coulombic efficiency was found in the range of 50-70% in MGPCs that pseudocapacitance was also found due to ion degradation from doped heteroatoms, especially self-oxygen doped. Furthermore, the relatively dominant distribution of micropores also affects their efficiency.

Additionally, the voltage drop, commonly known as the IR drop, is determined from the GCD profile, thereby revealing the equivalent series resistance of the material electrodes of 0.099, 0.154, and 0.059 Ω for MGPC-800, MGPC-850, and MGPC-900, respectively. In more detail, the relatively higher IR-drop on MGPC-800 is indicated by the effect of pore growth in the material. The rich micropores apparently inhibit the charge transport pathway during ion insertion-deinsertion at the electrode/electrolyte interface. The time required to perform one charge and discharge cycle reflect the specific capacitance of the electrical material. According to the standard equation, the specific capacitance obtained from the MGPC-800, MGPC-850, and MGPC-900 electrodes were 166, 208, and 180 F g^{-1}, respectively. Mission grass-based porous carbon synthesis routes involving chemical impregnation and physical activation of 800°C high-temperature CO_2 significantly improved the high performance of supercapacitor cells in two electrode configurations of 166 F g^{-1} with their relatively low cell resistance of 0.099 Ω. This is because the chosen strategy resulted in the obtained carbon material properties with high

potential in porosity, surface morphology, clear elemental status, and high electrical conductivity (Selvaraj et al., 2021). Furthermore, increasing the physical activation temperature to 850°C drastically raised the specific capacitance by 208 F g^{-1}, indicating that the porosity optimization and abundant active channels can accommodate the high ion migration markedly, thereby improving their electrochemical performance (H. Wang et al., 2021). MGPC-850 has the best amorphous properties with the highest predicted specific surface area, enabling them to form an enriched electrical double layer, thereby enhancing their capacitive properties. The presence of the pseudo-capacitance effect of functional oxygen adds to the high performance of the electrode material. However, increasing the physical activation temperature to 900°C led to a decrease in capacitive properties by 180 F g^{-1} due to the deformation of the pore structure in the MGPC-900 carbon material. The reduced amorphous and empirical surface area affected their capacitive behavior (Deraman et al., 2015). Meanwhile, the energy and power density performances of the MGPCs electrodes were evaluated through the Ragone plot, as shown in Fig. 4b. The energy densities obtained from the MGPC-800, MGPC-850, and MGPC-900 are 22.64, 28.31, and 24.58 Wh kg^{-1}, at optimum power densities of 73.83, 65.47, and 60.62 W kg^{-1}. This value is relatively similar to preliminary studies that have been reported with activated carbon sources biomass-based, such as bacterial celulose (Hao et al., 2017) and European wood (Jain et al., 2021). The highest electrochemical performance on the MGPC-850 electrode was reviewed in detail through electrochemical impedance spectroscopy (EIS) in a symmetrical system on an H_2SO_4 electrolyte with a measurement frequency range of 100 mHz to 100 kHz. The Nyquist plot of the MGPC-850 electrodes, shows in Fig. 5a, shows that their curve consists of several sections. Firstly, the high frequency semicircular curvature confirms the ions' rapid migration at the electrode/electrolyte interface, which affects the charging and discharging processes in the

electrode material (Lämmel et al., 2013). Secondly, the first Z' axis intercept characterizes the selected electrolyte resistance of 0.038Ω, while the second Z' intercept represents the equivalent series resistance (ESR) reflecting the MGPC850 electrode's electronic resistance of 0.139 Ω. This low resistance is due to the contribution of high porosity followed by the wettability properties of the optimized oxygen functional groups in the MGPC-850 material (Yadav et al., 2020). The Warburg curve found at intermediate frequencies indicates fluctuating ion insertions into the internal pore channels of the electrode, as shown in insert Fig. 5a. At low frequencies, the vertical line pattern clearly reflects the hallmarks of an ideal electrical dual layer response, which is also in agreement with the analysis revealed in the CV and GCD measurements. Fig. 5b shows the Bode phase plot in the low to a high-frequency range of the MGPC-850 electrode. At low frequencies, the value of the phase angle is about -78°, which confirms the capacitive behavior of the normal porous carbon-based electrical double layer with low resistance and high charge transfer (Atika & Dutta, 2021). Furthermore, the knee frequency at the phase angle of -45° determines the relaxation time of the electrode material, which is closely related to the discharge and charge time efficiency at the electrode/electrolyte interface. The relaxation time constant of the MGPC-850 found through the $1/f_0$ equation is 25.12 ms, which indicates high-quality electrode materials for practical applications in electrochemical energy storage devices.

4. Conclusions

In conclusion, this research successfully applied an eco-benign and time-saving strategy as a high-quality electrode material for the conversion of mission-grass biomass into porous carbon for renewable energy storage devices. The pore morphological features, amorphism, wettability, and elemental status were fully controlled through the physical activation temperatures of 800, 850, and 900°C. Furthermore, an increase in the physical activation temperature significantly

affects the changes in their pore morphology, leading to the growth of hierarchically connected 3D pores. In the optimized porous carbon, the elemental state of high carbon is 93.89%, with the wettability properties contributed by the oxygen functional group. The amorphousness was significantly confirmed in the two-electrode configuration system to obtain specific capacitances of 166, 208, and 180 F g^{-1} for MGPC-800, MGPC-850, and MGPC-900 electrodes, respectively. The highest energy density of 28.31 Wh kg^{-1} at 1 A g^{-1} was found at the MGPC-850 electrode in an aqueous electrolyte of 1 M H_2SO_4. Therefore, the proposed environmentally benign approach explicitly explores mission-grass-based porous carbon as an electrode material for sustainable supercapacitor applications.

Acknowledgements

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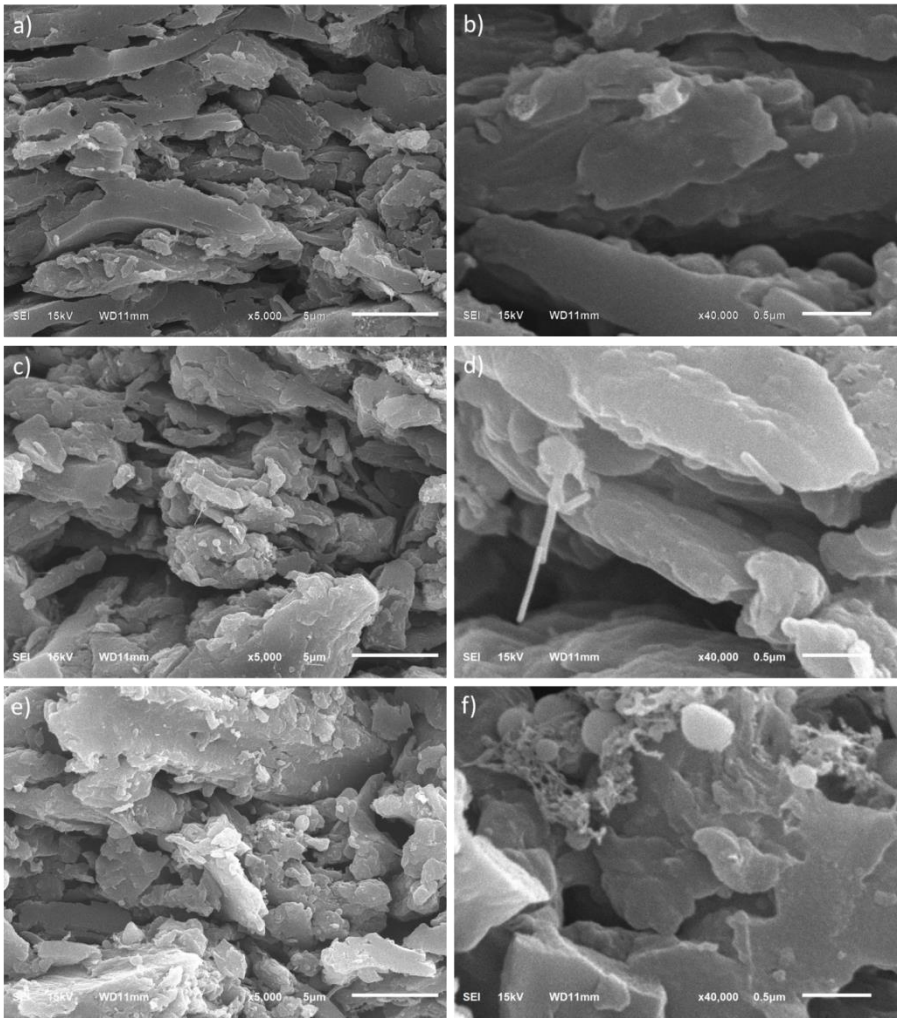

Fig. 1. Image SEM for a)-b) MGPC-800, c)-d) MGPC-850, and e)-f) MGPC-900 in 5000x and 40000x magnification

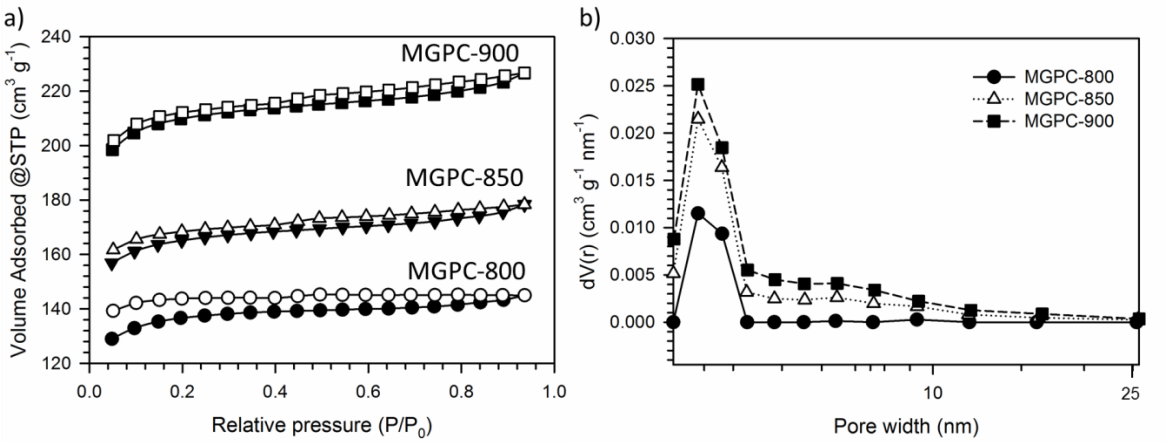

Fig. 2. a) Nitrogen adsorption-desorption profiles and b) The pore size distribution (PSD) of MGPCs

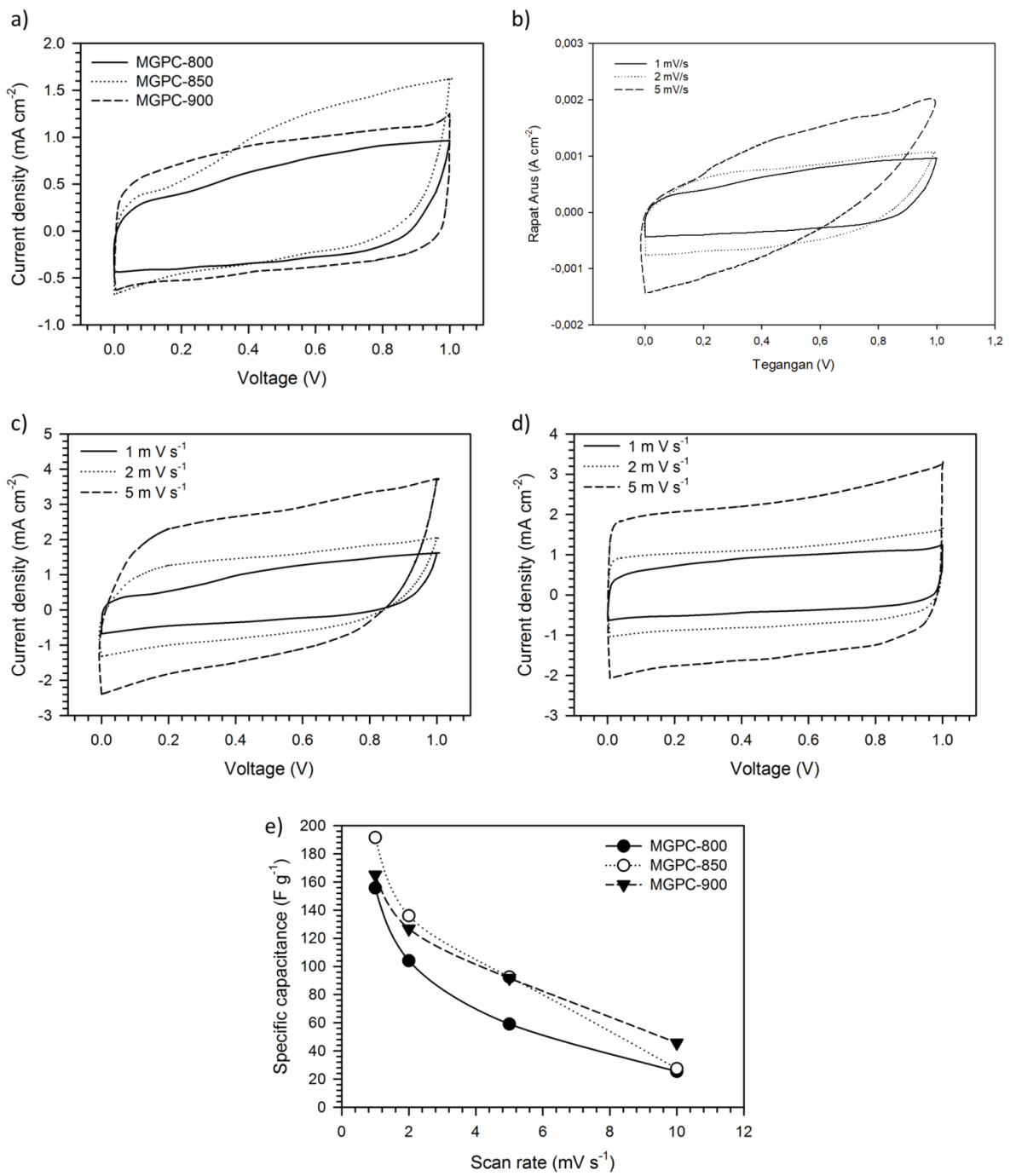

Fig. 3. CV profile of MGPCs samples a) at scan rate 1 mV s^{-1}, b) MGPC-800 at scan rate 1-5 mV s^{-1}, c) MGPC-850 at scan rate 1-5 mV s^{-1}, d) MGPC-900 at scan rate 1-5 mV s^{-1}, and e) Specific capacitance vs. Scan rate

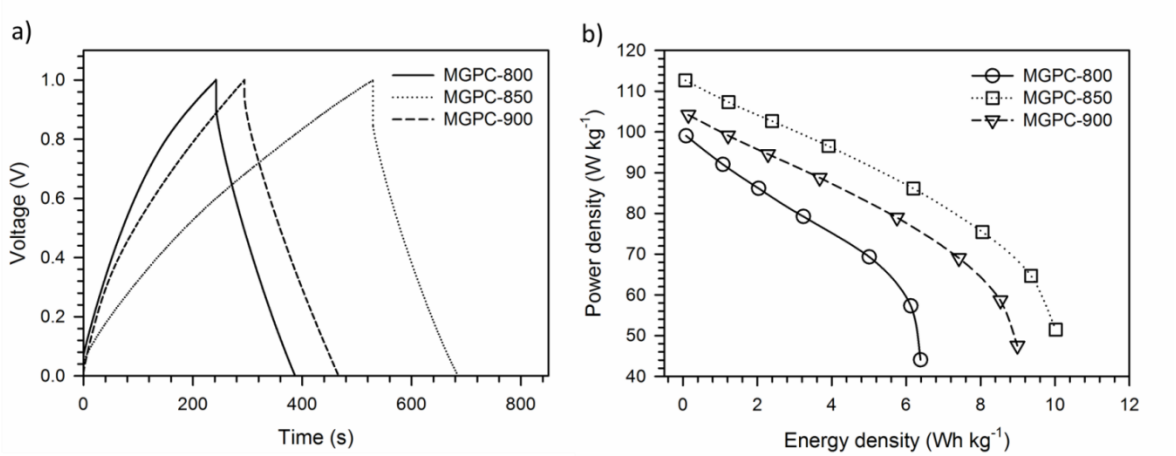

Fig. 4. a) GCD profile of MGPCs samples in 1 M H_2SO_4, and b) Ragone plot of MGPC samples in 1 M H_2SO_4

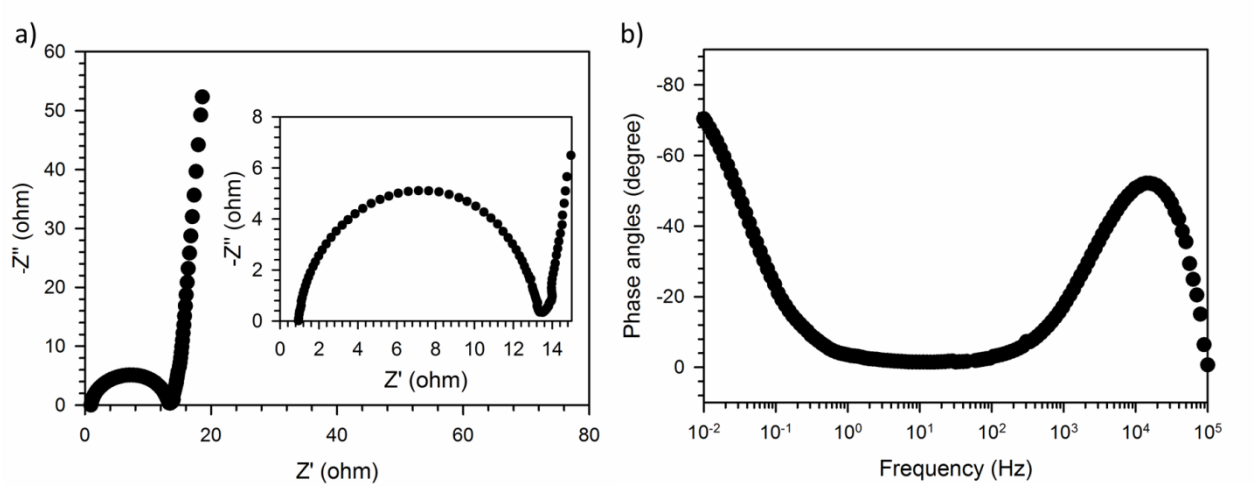

Fig. 5. a) The Nyquist plot of the MGPC-850, and b) the Bode phase plot of the MGPC-850

Table 1. Porosity properties and elemental status of MGPCs

Precursor	S_{BET} ($m^2\ g^{-1}$)	S_{micro} ($m^2\ g^{-1}$)	S_{meso} ($m^2\ g^{-1}$)	V_{tot} ($cm^3\ g^{-1}$)	V_{micro} ($cm^3\ g^{-1}$)	V_{meso} ($cm^3\ g^{-1}$)	D_{ave} (nm)	C (%)	O (%)	Zn (%)	Si (%)
MGPC-800	416.892	400.299	16.593	0.225	0.206	0.019	1.078	93.73	4.51	1.34	0.42
MGPC-850	503.551	476.252	27.299	0.277	0.245	0.032	1.099	93.89	4.02	1.55	0.55
MGPC-900	639.414	605.048	34.366	0.352	0.312	0.040	1.099	93.13	6.02	0.00	0.84

Gmail

Rika Taslim <rikataslim@gmail.com>

234063593 (Energy Sources, Part A: Recovery, Utilization, and Environmental Effects) A revise decision has been made on your submission

1 message

Energy Sources, Part A: Recovery, Utilization, and Environmental Effects <onbehalfof@manuscriptcentral.com>
Reply-To: snizetic@fesb.hr
To: rikataslim@gmail.com

Wed, Jun 21, 2023 at 2:12 PM

21-Jun-2023

Dear Dr Rika Taslim:

Your manuscript entitled "Mission grass bio-waste functional carbon self-single-doped for ultrahigh energy symmetrical supercapacitor" which you submitted to Energy Sources, Part A: Recovery, Utilization, and Environmental Effects, has been reviewed. The reviewer comments are included at the bottom of this letter.

The reviewer(s) would like to see some revisions made to your manuscript before publication. Therefore, I invite you to respond to the reviewer(s)' comments and revise your manuscript.

When you revise your manuscript please highlight the changes you make in the manuscript by using the track changes mode in MS Word or by using bold or colored text.

In accordance with our format-free submission policy, an editable version of the article must be supplied at the revision stage. Please submit your revised manuscript files in an editable file format.

When you submit your revision, please also provide your response to the reviewer comments as a separate "Supplementary Material - for review" source file.

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Changing the author list for a revision is rare and requires two criteria be met. First, every author being added or removed must provide their agreement for the change. Second, each author who is being added must also explain why they meet the definition of authorship for this paper in detail and elaborate on specific areas of the research they contributed to. This definition is given at https://authorservices.taylorandfrancis.com/defining-authorship/. Any requested changes in the order of the author list also require the agreement of all authors and an explanation of why the changes are necessary. If you need to change your paper's author list, please email all necessary agreements and explanations to the handling editor.

Once again, thank you for submitting your manuscript to Energy Sources, Part A: Recovery, Utilization, and Environmental Effects and I look forward to receiving your revision.

Sincerely,
Professor Nižetić
University of Split Faculty of Electrical Engineering Mechanical Engineering and Naval Architecture
Editor-in-Chief, Energy Sources, Part A: Recovery, Utilization, and Environmental Effects
snizetic@fesb.hr

Comments from the Editor and Reviewers:

Editor remarks:

1) Present similarity index (26%, iThenticate) must be reduced to not more than 20% with not more than 3% from a

source,
2) The novelty of the work must be clearly addressed and discussed, compare your research with existing research findings and highlight novelty, (compare your work with existing research findings and highlight novelty),
3) The main objective of the work must be written on the more clear and more concise way at the end of introduction section,
4) Research gap should be delivered on more clear way with directed necessity for the conducted research work,
5) Conclusion section is missing some perspective related to the future research work, quantify main research findings,
6) English language should be carefully checked and carefully check paper for language typos,
7) Any authorship changes will need to have a specific, valid reason for the update that will be evaluated by the Editor according to journal defining authorship guidelines.

Reviewer: 1

Comments to the Author
In this work, the authors have tried to report a mission grass derived carbon as the supercapacitor electrode material. The morphology, microstructures, and capacitive performance of as-prepared electrode material have been systematically investigated. It shows good capacitive performance. However, the conclusions of the study were not fully supported due to insufficient characterization and electrochemical analysis. Therefore, this manuscript can be considered for publication after the following revisions:

1. The author used mission grass as raw material, and what advantages does it have over common biomass raw materials?
2. The author should provide the Raman spectra of the samples and perform the analysis,
3. The author should provide XPS data of the sample surface to analyze the chemical state of the sample surface.
4. Cyclic stability data of the sample should be provided to assess its service life.

Reviewer: 2

Comments to the Author
1) Author should improve the analysis for the effect of oxygen-containing groups on the capacitance contribution, such as Applied Surface Science 2018, 460, 8-16.; Journal of Energy Storage 2022, 51, 104347.
2. Some language errors should be corrected, such as the Title of Fig.1 and 2.
3. So high IR-drop for the MGPC-850 shown in Fig. 4a, why? please provide necessary analysis.
4. The Coulombic efficiency of MGPC-850 shown in Fig. 4a is low, why? please analyze.
5. Authors should provide the long-term cycle test for the electrode up to 5000 cycles.

Dear Prof. Sandro Nizetic

Thank you for evaluating our manuscript in Journal of *Energy Sources Part A: Recovery, Utilization, and Environmental Effects*.

We greatly appreciate the editor's consideration of this manuscript for publication and valuable review comments. We have made every attempt to respond to all comments and questions raised by the reviewer and have adjusted the manuscript accordingly. All revisions in the manuscript are given a yellow highlight background.

We confirm that this manuscript was revised in accordance with the reviewers' comments of Journal of *Energy Sources Part A: Recovery, Utilization, and Environmental Effects*. We hopeful this manuscript can be accepted and published in Journal of *Energy Sources Part A: Recovery, Utilization, and Environmental Effects*.

Best regards,

Rika Taslim
Corresponding author

Author's comments to the reviewers:

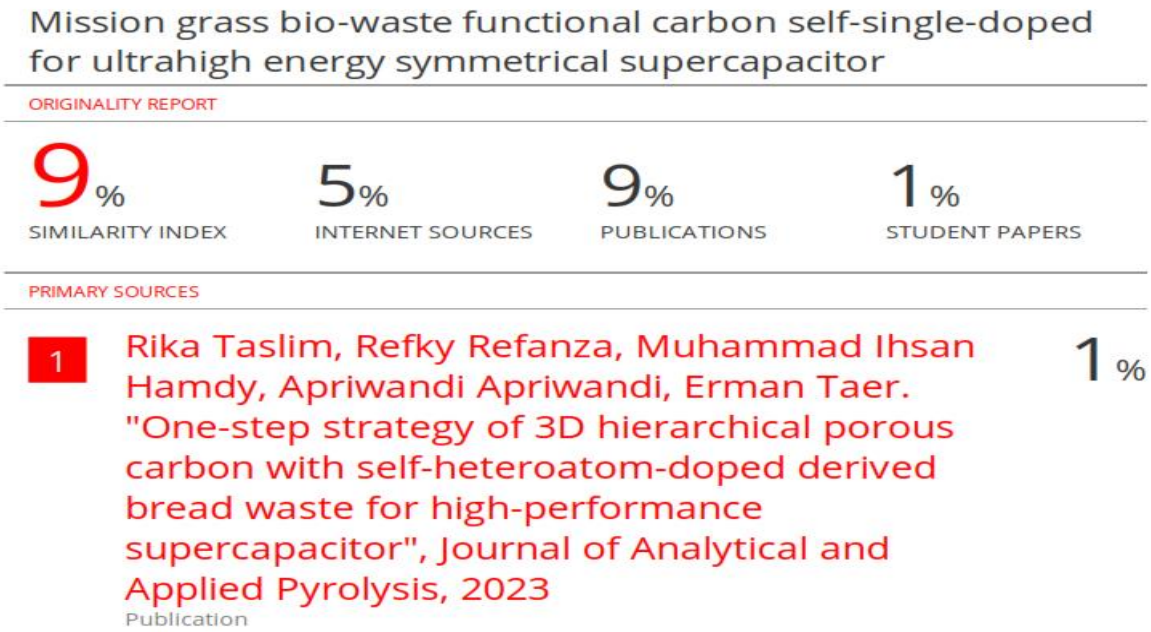
Comments from the Editor and Reviewers:

Editor remarks:

1) Present similarity index (26%, iThenticate) must be reduced to not more than 20% with not more than 3% from a source,

Author's response:

We thank the editor for the very constructive comment. We have rewritten this manuscript and it reduced the similarity. In revised version, our manuscript has 9% similarity index. Hopefully, the revised manuscript is found to be more convenient to read.



2) The novelty of the work must be clearly addressed and discussed, compare your research with existing research findings and highlight novelty, (compare your work with existing research findings and highlight novelty),

Author's response:

We thank the editor for the very constructive comment. We have addressed and discussed the novelty of this research with compere with previous report in the introduction section. We also added more clearly highlight in the introduction part. This modification was added in the manuscript revised, page 2-4, introduction part, remarked with yellow highlight background. Hopefully, the revised manuscript is found to be more convenient to read.

Page 2-4, introduction part, remarked with yellow highlight background

These advantages enable supercapacitors as prime candidates to develop the next generation of energy conversion, particularly in the practical application of electronic components, spare parts, hybrid vehicles, pulse laser components, and crane and lift auxiliary systems (Deraman et al., 2016; Iro et al., 2016). However, supercapacitors are considered to still have obstacles in their practical application, especially at low energy densities lower than conventional fuel cells and batteries. The increase in energy density is heavy because it erodes their high power performance. To overcome this problem, many intensive studies have been dedicated to

increasing the energy density and capacity of supercapacitors without reducing their high-power density. The strategies used to improve the promising, effective and efficient electrochemical performance of supercapacitors' are 2D nanostructures, pore hierarchies, heteroatom doping, selecting the appropriate electrolyte with higher voltage operation, and excellent ionic conductivity (Pal et al., 2019; G. Wang et al., 2012). The combination of these extraordinary properties leads to high energy densities while maintaining their power densities. Furthermore, the design of the electrode material through the incorporation and transition of metal oxides, such as ruthenium, nickel, and vanadium, demonstrates stability during redox reactions in an easily accessible and abundant pore distribution (Dhara & Mahapatra, 2019). Additionally, among various metal oxides, MnO_2 is the leading compound that exhibits superior electrochemical behavior with important applications in many energy conversion and storage processes (Long et al., 2020). On the other hand, electrospinning and the use of hard, soft, and salt template methods based on polymeric materials, such as polypyrrole and polyaniline, allow electrode materials to have rich, regular 2D nanostructures, abundant hierarchical pores that lead to high material conductivity properties, excellent ion accessibility, and good flexibility (Zang et al., 2020). Zhang *et al.* (2017) reported that graphene-polymer-based electrodes with 3D pore features are able to compete with the battery energy density of 28.06 Wh kg^{-1} at a high specific capacitance of 1,182 F g^{-1} (L. Zhang et al., 2017). However, the synthesis routes described are relatively complex, expensive to manufacture, corrosive, and toxic, thereby compromising their service life and hindering the goal of green and environmentally benign energy storage. Biomass is generally known as one of the abundant natural resources with extraordinary potential energy sources and conversion. Several studies suggest that the world's total land biomass is about 1.8 trillion tons each, with a potential energy production capacity of 33,000 EJ (Tursi, 2019). Its conversion value

is 80 times greater than the world's annual energy consumption. Interestingly, the high carbon source in biomass has tremendous potential as a base material for high-quality electrodes. Due to its controllable porous structure, unique nanostructure potential, good physical-chemical stability, high electrical conductivity, and abundant heteroatom doping, biomass-based activated carbon uses approximately 80% of the electrode base material for electrochemical energy storage devices (Abbas et al., 2019; Saini et al., 2021; Y. Zhang et al., 2017). Furthermore, biomass is inexpensive, abundantly available, sustainable, environmentally friendly, easy synthesis route, and non-toxic-corrosive, hence it is very suitable to be developed as a source of green electrode material for sustainable supercapacitor applications (Herou et al., 2018; Z. Li et al., 2020). Several recent studies have reported the potential of waste biomass as electrode material, such as banana leaves (Taer, Taslim, et al., 2022), onion skin (Gopalakrishnan & Badhulika, 2021), eucalyptus wood (Atika & Dutta, 2021), camellia husk (Cui et al., 2022), *Osmanthus fragrans* (Quan et al., 2018), bamboo (Taer et al., 2020), sakura flower (Ma et al., 2019), mangosteen (Yang et al., 2019), and *Terminalia Catappa leaf* (Taer et al., 2018). The *Averrhoa bilimbi* leaves are an extraordinary combination of 3D hierarchical pores and self-doping heteroatoms with impressive energy densities of 26.54 Wh kg^{-1} in a symmetrical supercapacitor system (Taer, Apriwandi, et al., 2022). Li *et al.* (2021) reported the potential of the eggplant as a porous carbon electrode with similar features with an increase in power density reaching 38 Wh kg^{-1} (W. Li et al., 2022). However, the time-consuming synthesis route and real low stability need to be optimized by intensely finding a truly promising approach. In addition, mission grass was confirmed to provide abundant lignocellulose components with the proportion of lignin (15.6%), cellulose (40.02%), and hemicellulose (29.2%). This combination of elementals makes it possible to provide unique nanopores that enhance the electrode material's performance.

3) The main objective of the work must be written on the more clear and more concise way at the end of introduction section,

Author's response:

We thank the editor for the very constructive comment. We have added the main objective of this work in introduction section, page 4-5, remarked with yellow highlight background. Hopefully, the revised manuscript is found to be more convenient to read.

Introduction section, page 4-5, remarked with yellow highlight background

However, the time-consuming synthesis route and real low stability need to be optimized by intensely finding a truly promising approach. In addition, mission grass was confirmed to provide abundant lignocellulose components with the proportion of lignin (15.6%), cellulose (40.02%), and hemicellulose (29.2%). This combination of elementals makes it possible to provide unique nanopores that enhance the electrode material's performance. This research used the environmentally benign and time-saving approach to obtain biomass-based porous carbon as a quality electrode material for symmetric supercapacitor applications. Biomass precursors of mission grass were defined in all their unique potential, while the chemical impregnation synthesis route at high-temperature pyrolysis obtained porous carbon.

4) Research gap should be delivered on more clear way with directed necessity for the conducted research work,

Author's response:

We thank the editor for the very constructive comment. We have added the research gap in this manuscript revised, especially in introduction part, page 4, remarked with yellow highlight background. Hopefully, the revised manuscript is found to be more convenient to read.

Introduction part, page 4, remarked with yellow highlight background

Li *et al.* (2021) reported the potential of the eggplant as a porous carbon electrode with similar features with an increase in power density reaching 38 Wh kg^{-1} (W. Li et al., 2022). However, the time-consuming synthesis route and real low stability need to be optimized by intensely finding a truly promising approach. In addition, mission grass was confirmed to provide abundant lignocellulose components with the proportion of lignin (15.6%), cellulose (40.02%),

and hemicellulose (29.2%). This combination of elementals makes it possible to provide unique nanopores that enhance the electrode material's performance.

5) Conclusion section is missing some perspective related to the future research work, quantify main research findings,

Author's response:

We thank the editor for the very constructive comment. We have added the perspective related to future research work in the end of conclusion part, remarked with yellow highlight background. Hopefully, the revised manuscript is found to be more convenient to read.

Conclusion part, remarked with yellow highlight background

The amorphousness was significantly confirmed in the two-electrode configuration system to obtain specific capacitances of 166, 208, and 180 F g^{-1} for MGPC-800, MGPC-850, and MGPC-900 electrodes, respectively. The highest energy density of 28.31 Wh kg^{-1} at 1 A g^{-1} was found at the MGPC-850 electrode in an aqueous electrolyte of 1 M H_2SO_4. Therefore, the proposed environmentally benign approach explicitly explores mission-grass-based porous carbon as an electrode material for sustainable supercapacitor applications.

6) English language should be carefully checked and carefully check paper for language typos

Author's response:

We thank the editor for the very constructive comment. We have seriously improved and checked the language typos in this manuscript. Furthermore, we have proofread this manuscript to native speakers and professional editors through trusted institutions. In the attached file section, we also attached the proofreading certificates and tracking of the sentences in the manuscript. Hopefully, the revised manuscript is found to be more convenient to read.

7) Any authorship changes will need to have a specific, valid reason for the update that will be evaluated by the Editor according to journal defining authorship guidelines.

Author's response:

We thank the editor for the very constructive comment. We have no authorship changes.

Reviewer: 1

Comments to the Author

In this work, the authors have tried to report a mission grass derived carbon as the supercapacitor electrode material. The morphology, microstructures, and capacitive performance of as-prepared electrode material have been systematically investigated. It shows good capacitive performance. However, the conclusions of the study were not fully supported due to insufficient characterization and electrochemical analysis. Therefore, this manuscript can be considered for publication after the following revisions:

1. The author used mission grass as raw material, and what advantages does it have over common biomass raw materials?

Author's response:

We appreciate the reviewer for very constructive suggestion. Mission grass (*Pennisetum polystachyon*) is one of the weeds commonly found in Indonesia, Philippines, Thailand, and Malaysia. This plant belongs to the Poaceae family and grows wild. In Indonesia, mission grass is widely cultivated for livestock feed purposes, and it is rich in lignocellulose complex compounds and consists of 39.8-40.0% cellulose, 23.3-29.2% hemicellulose, 6.2-14.6% lignin, and 3.3-7.5% ash [23]. Therefore, it is potentially converted as activated carbon to supercapacitor electrodes. This sentence was added in the manuscript, especially in introduction part, page 4, remarked with yellow highlight background.

Introduction part, page 4, remarked with yellow highlight background

However, the time-consuming synthesis route and real low stability need to be optimized by intensely finding a truly promising approach. In addition, mission grass was confirmed to provide abundant lignocellulose components with the proportion of lignin (15.6%), cellulose (40.02%), and hemicellulose (29.2%). This combination of elementals makes it possible to provide unique nanopores that enhance the electrode material's performance.

2. The author should provide the Raman spectra of the samples and perform the analysis,

Author's response:

We appreciate the reviewer for very constructive suggestion. We sincerely apologize for not providing XPS data. This is due to the limitations of the measurement instruments in our laboratory. Furthermore, we tried to contact several laboratories to measure the XPS data in our country. Moreover, adding data to a significantly revised manuscript can increase the number of pages which results in exceeding the limit set by the publisher. However, we try our best to fulfill all reviewer comments. In our opinion, essentially, the Raman spectra were measured to confirm the degree of gravity of the material that was obtained. In our opinion, these measurements can be replaced by XRD analysis. XRD measurements that have been carried out in this paper show the level of graphitization and amorphousity of the material that has been obtained. This has been discussed in detail in the results and discussion section, page 7, remarked with yellow highlight background.

Results and discussion section, page 7, remarked with yellow highlight background

The degree of crystallinity and phase change that occurred in the porous carbon of MGPCs at different physical activation temperatures was assessed using an X-ray diffraction (XRD) pattern, as shown in Fig. 2. It showed two significant broad peaks at 2θ approximately 21.53°, 24.15°, and 24.76° indexed to the scattering plane (002) and 42.71°, 44.91°, and 45.44° which are correlated to the scattering plane (100) of MGPC-800, MGPC-850, and MGPC-900, respectively. It further characterizes the amorphous-turbostratic carbon structure rich in pores and attenuated crystallinity (JCPDS No. 41–1487) [37,38], which proves that the sample has a confirmed pore structure. The XRD pattern was also obtained on porous carbon from various precursor sources such as potatoes [39], cotton stalks [40], bamboo [41], and *Areca catechu* husk [42] to exhibit

good results. Increasing the physical activation temperature from MGPC800 to MGPC-850 displays a shift of the indexed width peak (002) towards a smaller one, thereby confirming the abundant increase in the growth of narrow pores. Additionally, the broad peak in the scattering plane (100) shifted to a greater extent from 42.487° to 45.710°, allowing MGPC-850 to have confirmed mesopores on the precursor surface. The combined features of microporous and mesoporous growth in the based material contribute to the electrode's high capacitive properties and low internal resistance. This analysis is further justified through the analysis of electrochemical properties using CV and GCD techniques. However, an increase in the physical activation temperature up to 900°C displays a different pattern and leads to the opposite feature, thereby confirming a good reduction in pore development. This is because excessive carbon chain etching at 900°C damaged the pore framework structure of the precursor.

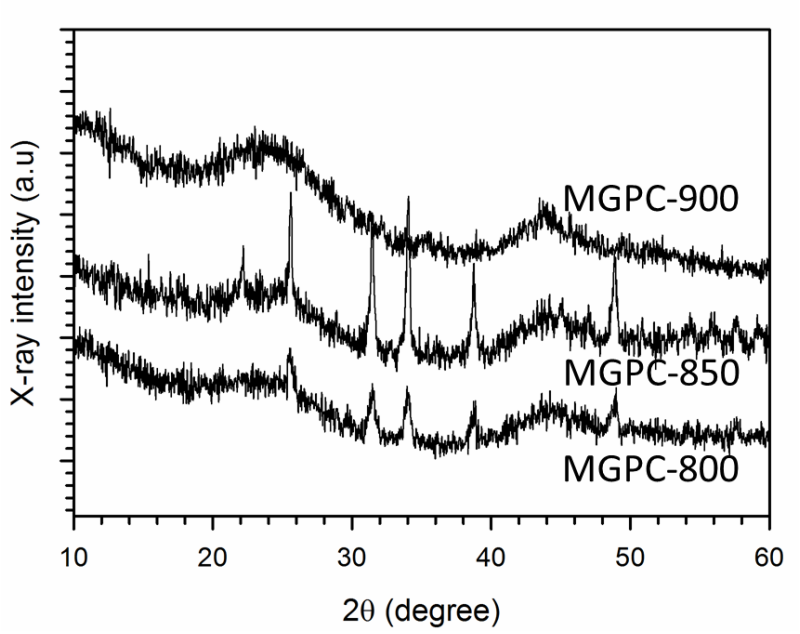


Figure 2. XRD pattern

3. The author should provide XPS data of the sample surface to analyze the chemical state of the sample surface.

Author's response:

We appreciate the reviewer for very constructive suggestion in this manuscript. We sincerely apologize for not providing XPS data. This is due to the limitations of the measurement instruments in our laboratory. Furthermore, we tried to contact several laboratories to measure the XPS data in our country. Moreover, adding data to a significantly revised manuscript can increase the number of pages which results in exceeding the limit set by the publisher. However, we try our best to fulfill all reviewer comments. In our opinion, the analysis of the chemical element ratios in the sample surface has been represented by the EDS measurements. The elements contained in the sample have been represented in Table 1. We hope the reviewers understand our constraints.

Table 1.

Precursor	C (%)	O (%)	Zn (%)	Si (%)
MGPC-800	93.73	4.51	1.34	0.42
MGPC-850	93.89	4.02	1.55	0.55
MGPC-900	93.13	6.02	0.00	0.84

The status of the elemental components present in MGPCs is revealed through energy dispersive spectroscopy (EDS) measurements, which are summarized in Table 1. Evaporation of water content, volatiles, polysaccharides, and hemicelluloses, as well as degradation of other organic components due to carbonization in inert gases, initiates the production of high carbon fixed. Furthermore, the high physical activation temperature followed by carbon chain removal significantly reduced the lignocellulosic content leading to the strengthening of the porous carbon skeleton. Table 1 show that the main constituent elements of all samples were dominated by high carbon at 93.73%, 93.89%, and 93.13% for MGPC-800, MGPC-850, and MGPC-900, respectively. This high carbon content allows the electrode material to produce high electrical conductivity properties followed by a low equivalent series resistance, enhancing the supercapacitor's outstanding performance. Conversely, oxygen occupies the second highest element in the range of 4.02% to 6.02% because the excessive etching of carbon chains by CO_2 allows the precursor to produce oxidation byproducts that bind to inorganic components. The

high oxygen content has a positive side for the electrode material, especially in their contribution to the improvement of wettability, maintained conductivity, self-doping of heteroatoms, and the pseudo-capacitance effect on energy storage devices (Abbas et al., 2019; Zheng et al., 2021). Due to incomplete evaporation of the remaining pyrolysis production, Silicon and zinc are also found in small amounts in MGPC.

4. Cyclic stability data of the sample should be provided to assess its service life.

Author's response:

We appreciate the reviewer for very constructive suggestion. We tried to presenting our best performance to exhibit a good scientific paper. Also we have tried to fulfill all the reviewers' comments. In this paper, we try to obtain something new in this research. In this work, we report the self-single-doped for ultrahigh energy symmetrical supercapacitor derived from bio-waste of mission grass on several points that are different from those mostly reported. Furthermore, their high capacitance has also been obtained significantly through CV, GCD, and EIS analysis in a two-electrode system. These results certainly confirm the previous hypothesis. The results and data in this manuscript have been feasible to confirm the ultimate potential of biomass as a binder-free electrode. However, cyclic stability was not shown in this work due to the design of supercapacitor cells in the development and optimization of their performance. We are very grateful to the reviewer for these very constructive comments. We will present this input in our next work. Nevertheless, the results and data in this manuscript have been feasible to confirm the ultimate potential of biomass mission grass as a self-single-doped carbon source with a design of binder-free for sustainability electrode material. Similar case was also shown in previous scientific articles such as: *Journal of Electronic Materials*, 2021, 50(12), pp. 6910–6919 and *Materials Chemistry and Physics* 246 (2020) 122830. We hope that reviewers understand the intent and purpose of our argument.

However, we have revealed the cyclical performance of the electrodes using CV and GCD through different scanning rates as shown in Figures 3b-d. The specific capacitance can be maintained of 110 F g^{-1} when the scan rate is increased from 1 to 5 mV s^{-1} for MGPC-850. These results indicate that the MGPCs electrode has good EDLC device rate capability, as shown in Figure 3e. In our opinion, this reflects the cyclical performance of the obtained electrode. This is consistent with previously reported studies such as: *Journal of Energy Storage* 52 (2022) 104911, *International Journal of Energy Research* 46 (2022) 1467-1480, and *Journal of Chemical Technology & Biotechnology* 98 (2022) 45-56. The results of this revision are added to the manuscript in the result and discussion part, page 10, remarked with yellow highlight background. Hopefully, the revised manuscript is found to be more convenient to read.

Result and discussion part, page 10, remarked with yellow highlight background

…The specific capacitance obtained in the symmetric cell MGPC-900 is about 165 F g^{-1}. The electrochemical properties of MGPCs were confirmed through scanning rates from 1 mV s^{-1} to 5

mV s^{-1}, as shown in Fig. 3b-d. The CV profile generally shows an increasingly ideal rectangular shape confirming the potential for MGPCs electrodes to have normal electrical double-layer capacitor behavior based on porous carbon. Moreover, their cyclical performance is also reviewed in terms of specific capacitances at different scanning rates, as shown in Fig. 3e. The specific capacitance of the MGPCs electrodes degraded drastically at a scanning rate of 10 mV s^{-1} because the carbon material has a low meso-macroporosity, therefore, the ionic charge is not optimally induced at the electrode/electrolyte interface (Y. Zhang et al., 2017)…

Reviewer: 2

Comments to the Author

1) Author should improve the analysis for the effect of oxygen-containing groups on the capacitance contribution, such as Applied Surface Science 2018, 460, 8-16.; Journal of Energy Storage 2022, 51, 104347.

Author's response:

We thank reviewer for the constructive suggestion. We have added and improved the analysis with some of the references suggested by the reviewer. These references have been cited in the manuscript revised and marked with a yellow highlight background.

2. Some language errors should be corrected, such as the Title of Fig.1 and 2.

Author's response:

We thank reviewer for the constructive suggestion. We have seriously improved the language errors in this manuscript, including title of Fig.1 and Fig.2. Furthermore, we have proofread this manuscript to native speakers and professional editors through trusted institutions. In the attached file section, we also attached the proofreading certificates and tracking of the sentences in the manuscript. Hopefully, the revised manuscript is found to be more convenient to read.

After revision:

Fig. 1. Image SEM for a)-b) MGPC-800, c)-d) MGPC-850, and e)-f) MGPC-900 in 5000x and 40000x magnification

Fig. 2. a) Nitrogen adsorption-desorption profiles and b) The pore size distribution (PSD) of MGPCs

3. So high IR-drop for the MGPC-850 shown in Fig. 4a, why? please provide necessary analysis.

Author's response:

We appreciate the reviewer for very constructive suggestion. In general, the GCD measurements possessed an IR-drop with a resistance of 0.099, 0.154, and 0.059 Ω for MGPC-800, MGPC-850, and MGPC-900, respectively. In more detail, the relatively higher IR-drop on MGPC-800 is indicated by the effect of pore growth in the material. The rich micropores apparently inhibit the charge transport pathway during ion insertion-deinsertion at the electrode/electrolyte interface. This analysis is also presented in previous manuscripts such as: *Appl. Surf. Sci.* 540 (2021) 148352, *Biomass and Bioenergy* 156 (2022) 106301, *and International Journal of Hydrogen Energy* 46 (2021) 31927-31937. These revised statements have been added in the manuscript, result and discussion section, page 13, with yellow highlight background.

Result and discussion section, page 13, with yellow highlight background

Additionally, the voltage drop, commonly known as the IR drop, is determined from the GCD profile, thereby revealing the equivalent series resistance of the material electrodes of 0.099, 0.154, and 0.059 Ω for MGPC-800, MGPC-850, and MGPC-900, respectively. In more detail, the relatively higher IR-drop on MGPC-800 is indicated by the effect of pore growth in the material. The rich micropores apparently inhibit the charge transport pathway during ion insertion-deinsertion at the electrode/electrolyte interface. The time required to perform one charge and discharge cycle reflect the specific capacitance of the electrical material. According

to the standard equation, the specific capacitance obtained from the MGPC-800, MGPC-850, and MGPC-900 electrodes were 166, 208, and 180 F g^{-1}, respectively.

4. The Coulombic efficiency of MGPC-850 shown in Fig. 4a is low, why? please analyze.

Author's response:

We appreciate the reviewer for very constructive suggestion. We really appreciate reviewer comments to get maximum manuscript improvement. In our opinion, the coulombic efficiency shown in this study is rational. This is because coulombic efficiency represents the type of supercapacitor studied. Coulombic efficiency is obtained from the data on the difference between the charging and discharging time of the GCD ($\eta=[t_d/t_c]x100\%$). Their coulombic efficiency is about 90% close to 100% indicating that their properties are ideal for high EDLC. Furthermore, a coulombic efficiency of around 60-80% indicates a pseudocapacitance effect on the sample due to the ion degradation of heteroatom doped. In this study, coulombic efficiency was found in the range of 50-70% in MGPCs that pseudocapacitance was also found due to ion degradation from doped heteroatoms, especially self-oxygen doped. Furthermore, the relatively dominant distribution of micropores also affects their efficiency, as previously reported (*Journal of Colloid and Interface Science* 606 (2022) 817–825). This analysis is clearly stated in the manuscript, results and discussion section. Moreover, this case and analysis is considered normal for heteroatom doping for supercapacitors studies, as has been reported recently such as: *Appl. Surf. Sci.* 540 (2021) 148352, *Biomass and Bioenergy* 156 (2022) 106301, *and International Journal of Hydrogen Energy* 46 (2021) 31927-31937. These revised statements have been added in the manuscript, result and discussion section, page 13, with yellow highlight background.

Result and discussion section, page 13, with yellow highlight background

According to preliminary studies, the wettability of the oxygen functional group initiates the faradaic reaction (Luo et al., 2021). This leads to an increase in the pseudo-capacitance property, as confirmed in the CV curve. Coulombic efficiency is obtained from the data on the difference between the charging and discharging time of the GCD ($\eta=[td/tc]x100\%$). Their coulombic efficiency is about 90% close to 100% indicating that their properties are ideal for high EDLC. Furthermore, a coulombic efficiency of around 60-80% indicates a pseudocapacitance effect on the sample due to the ion degradation of heteroatom doped. In this study, coulombic efficiency was found in the range of 50-70% in MGPCs that pseudocapacitance was also found due to ion degradation from doped heteroatoms, especially self-oxygen doped. Furthermore, the relatively dominant distribution of micropores also affects their efficiency.

5. Authors should provide the long-term cycle test for the electrode up to 5000 cycles

Author's response:

We appreciate the reviewer for very constructive suggestion. We tried to presenting our best performance to exhibit a good scientific paper. Also we have tried to fulfill all the reviewers' comments. In this paper, we try to obtain something new in this research. In this work, we report the self-single-doped for ultrahigh energy symmetrical supercapacitor derived from bio-waste of mission grass on several points that are different from those mostly reported. Furthermore, their high capacitance has also been obtained significantly through CV, GCD, and EIS analysis in a two-electrode system. These results certainly confirm the previous hypothesis. The results and data in this manuscript have been feasible to confirm the ultimate potential of biomass as a binder-free electrode. However, cyclic stability was not shown in this work due to the design of supercapacitor cells in the development and optimization of their performance. We are very grateful to the reviewer for these very constructive comments. We will present this input in our next work. Nevertheless, the results and data in this manuscript have been feasible to confirm the ultimate potential of biomass mission grass as a self-single-doped carbon source with a design of binder-free for sustainability electrode material. Similar case was also shown in previous scientific articles such as: *Journal of Electronic Materials*, 2021, 50(12), pp. 6910–6919 and *Materials Chemistry and Physics* 246 (2020) 122830. We hope that reviewers understand the intent and purpose of our argument.

However, we have revealed the cyclical performance of the electrodes using CV and GCD through different scanning rates as shown in Figures 3b-d. The specific capacitance can be maintained of 110 F g^{-1} when the scan rate is increased from 1 to 5 mV s^{-1} for MGPC-850. These results indicate that the MGPCs electrode has good EDLC device rate capability, as shown in Figure 3e. In our opinion, this reflects the cyclical performance of the obtained electrode. This is consistent with previously reported studies such as: *Journal of Energy Storage* 52 (2022) 104911, *International Journal of Energy Research* 46 (2022) 1467-1480, and *Journal of Chemical Technology & Biotechnology* 98 (2022) 45-56. The results of this revision are added to the manuscript in the result and discussion part, page 10, remarked with yellow highlight background. Hopefully, the revised manuscript is found to be more convenient to read.

Result and discussion part, page 10, remarked with yellow highlight background

…The specific capacitance obtained in the symmetric cell MGPC-900 is about 165 F g^{-1}. The electrochemical properties of MGPCs were confirmed through scanning rates from 1 mV s^{-1} to 5 mV s^{-1}, as shown in Fig. 3b-d. The CV profile generally shows an increasingly ideal rectangular shape confirming the potential for MGPCs electrodes to have normal electrical double-layer capacitor behavior based on porous carbon. Moreover, their cyclical performance is also reviewed in terms of specific capacitances at different scanning rates, as shown in Fig. 3e. The specific capacitance of the MGPCs electrodes degraded drastically at a scanning rate of 10 mV s^{-}

[1] because the carbon material has a low meso-macroporosity, therefore, the ionic charge is not optimally induced at the electrode/electrolyte interface (Y. Zhang et al., 2017)…

Mission grass bio-waste functional carbon self-single-doped for ultrahigh energy symmetrical supercapacitor

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Abstract

Several studies have been performed on biomass as an extraordinary natural carbon resource for functional electrode with combined pore structure, self-doping, and nanopores morphology significantly boosted supercapacitor performance. Therefore, this study utilized mission-grass bio-waste as a porous carbon source self-single-doped as electrode material capable of increasing the energy density of supercapacitor. Porous carbon was obtained through the chemical impregnation synthesis route of $ZnCl_2$ and physical activation at temperatures 800, 850, and 900°C. The precursor materials were designed solid shape-like without adding synthetic materials. The optimized mission-grass-based porous carbon (MGPCs) illustrates the diverse pore structure, and wettability properties of the oxygen (O) internal doped. With supercapacitor symmetrical system, the MGPCs electrode showed the highest electrochemical properties at capacitive behavior as high as 208 F g^{-1} in 1 A g^{-1} with H_2SO_4 electrolyte. Furthermore, the energy density of the electrodes was increased by 28.31 Wh kg^{-1} with good electrical conductivity at internal resistance at 0.139Ω. The high electrochemical performance of the MGPCs electrodes proved that the insertion of mission grass biomass into porous carbon converted through an environmentally benign strategy obtained high-quality electrode material to boost the energy density of the supercapacitor.

Keywords: porous carbon; biomass; electrode materials; symmetrical supercapacitor

1. Introduction

This decade is considered the highest level of human modernization because it is marked by the development of the global economy, the extraordinary growth of the technology industry, as well as the evolution of biotechnology and cultural engineering, thereby leading to a rapid increase in the level of prosperity globally (Szocik & Braddock, 2022). However, this modernization increase has some determinantal effects, such as higher pollution levels, quick consumption of non-renewable energy sources, and demolishing ecological contamination, which decreases public health and the survival of the next generation (Deng et al., 2022). The world's major institutions have tried to apply a common frame of mind for "green growth technology" to accelerate the transfer of conventional energies to renewable green resource technologies (Guo et al., 2020). Among the numerous intensive researches on increasing renewable energy conversion, the supercapacitor is considering broadly concentrated prevalent energy storage in view of its uncommon elements (Alem et al., 2022; Ciszewski et al., 2019). These include unlimited life cycle, relatively fast charging-discharge in minute scale, extraordinary power density, system safe operation, long service life, and high component stability (González et al., 2016; Majid et al., 2021). These advantages enable supercapacitors as prime candidates to develop the next generation of energy conversion, particularly in the practical application of electronic components, spare parts, hybrid vehicles, pulse laser components, and crane and lift auxiliary systems (Deraman et al., 2016; Iro et al., 2016). However, supercapacitors are considered to still have obstacles in their practical application, especially at low energy densities lower than conventional fuel cells and batteries. The increase in energy density is heavy because it erodes their high power performance. To overcome this problem, numerous serious investigations have been committed to expanding the energy output and limit of supercapacitors without lessening

their power output. The strategies used to improve the promising, effective and efficient electrochemical performance of supercapacitors' are 2D nanostructures, pore hierarchies, heteroatom doping, selecting the appropriate electrolyte with higher voltage operation, and excellent ionic conductivity (Pal et al., 2019; G. Wang et al., 2012). The combination of these extraordinary properties leads to high energy densities while maintaining their power densities. Furthermore, the design of the electrode material through the incorporation and transition of metal oxides, such as ruthenium, nickel, and vanadium, demonstrates stability during redox reactions in an easily accessible and abundant pore distribution (Dhara & Mahapatra, 2019). Additionally, among various metal oxides, MnO_2 is the leading compound that exhibits superior electrochemical behavior with important applications in many energy conversion and storage processes (Long et al., 2020). Meanwhile, electrospinning and hard, soft, and salt template methods based on polymeric materials, such as polypyrrole and polyaniline, allow electrode materials to have rich, regular 2D nanostructures, abundant hierarchical pores that lead to high material conductivity properties, excellent ion accessibility, and good flexibility (Zang et al., 2020). Zhang *et al.* (2017) reported that graphene-polymer-based electrodes with 3D pore features are able to compete with the battery energy output as 28.06 Wh kg^{-1} in high specific capacitance of 1,182 F g^{-1} (L. Zhang et al., 2017). However, the synthesis routes described are relatively complex, expensive to manufacture, corrosive, and toxic, thereby compromising their service life and hindering the goal of green and environmentally benign energy storage. Biomass is generally known as one of the abundant natural resources with extraordinary potential energy sources and conversion. Several studies suggest that the world's total land biomass is about 1.8 trillion tons each, with a potential energy production capacity of 33,000 EJ (Tursi, 2019). Its conversion value is 80 times greater than the world's annual energy consumption. Interestingly,

the high carbon source in biomass has tremendous potential as a base material for high-quality electrodes. Because of its controllable permeable construction, novel nanostructure potential, great physical-compound dependability, high electrical conductivity, and bountiful heteroatom doping, biomass-based actuated carbon utilizes roughly 80% of the terminal base material for electrochemical supercapacitor (Abbas et al., 2019; Saini et al., 2021; Y. Zhang et al., 2017). Moreover, biomass is cheap, bounteously accessible, practical, harmless to the ecosystem, simple combination course, and non-poisonous destructive, consequently, it is entirely reasonable to be created as a wellspring of green terminal material for maintainable supercapacitor applications (Herou et al., 2018; Z. Li et al., 2020). Several recent studies have reported the potential of waste biomass as electrode material, such as yellow mangosteen (Taer, Apriwandi, Chow, et al., 2023), onion skin (Gopalakrishnan & Badhulika, 2021), eucalyptus wood (Atika & Dutta, 2021), camellia husk (Cui et al., 2022), *Osmanthus fragrans* (Quan et al., 2018), *Laurus nobilis* (Taer, Apriwandi, Windasari, et al., 2023), sakura flower (Ma et al., 2019), mangosteen (Yang et al., 2019), and Solanum torvum Fruit (Taer, Nursyafni, Apriwandi, et al., 2023). The *Averrhoa bilimbi* leaves are an extraordinary features of 3D pores structured and internal doped heteroatom with impressive energy densities of 26.54 Wh kg^{-1} in a symmetrical supercapacitor system (Taer et al., 2022). Li *et al.* (2021) reported the potential of the eggplant as a porous carbon electrode with similar features with an increase in power density reaching 38 Wh kg^{-1} (W. Li et al., 2022). However, the time-consuming synthesis route and real low stability need to be optimized by intensely finding a truly promising approach. In addition, mission grass was confirmed to provide abundant lignocellulose components with the proportion of lignin (15.6%), cellulose (40.02%), and hemicellulose (29.2%). This combination of elementals makes it possible to provide unique nanopores that enhance the precursor material features.

This study utilized the ecologically harmless and efficient way to deal with getting biomass-based permeable carbon as a quality cathode material for symmetric supercapacitor applications. Biomass antecedents of mission grass were characterized in the entirety of their novel potential, while the compound impregnation blend course at high-temperature pyrolysis got permeable carbon. The material properties of the permeable carbon were controlled through various actual initiation temperatures, including 800, 850, and 900°C. The characterization of the material properties results showed that the optimized precursor had a high amorphous carbon structure, with various hierarchically connected pores, and a high purity carbon state. Furthermore, the electrochemical properties exhibit high capacitive properties of 208 F g^{-1} in 1 A g^{-1} with H_2SO_4 electrolyte at an energy output of 28.31 Wh kg^{-1} in a symmetric supercapacitor system. The proposed earth-harmless methodology expressly investigates mission-grass-based permeable carbon as a forerunner terminal hotspot for reasonable electrochemical supercapacitor.

2. Methodology

Mission grass-based biomass porous carbon preparation (MGPCs)

One sack of mission grass waste that grows wild in Pekanbaru, Riau Province, is collected, washed with running water, and cut into small pieces about 5 cm in size. The examples were dried in the sun for a few days and continued with electrical dried at 101°C. Consequently, the precursors were pre-carbonization by around 30 g at 250°C in a vacuum stove. The pre-carbonized samples were also crushed using a milling machine until the samples were in powder form and then sieved in 60μm sieve. The powdered precursor was synthetically enacted in a $ZnCl_2$ arrangement with a convergence of 0.5 mmol/L utilizing a hotplate at 79-81°C and 300 rpm and dried in a broiler at 110°C for 48 hours. Then, carbon synthetically enacted was printed to stone monuments (coins) involving a water driven press with 15 stone monuments for every

treatment. The example was then carbonized by pyrolysis at room temperature to a greatest temperature of 600°C in a N_2 vaporous gase, trailed by actual enactment at various high temperatures, for example, 800°C, 850°C, and 900°C in a CO_2 gas. At last, the carbon stone monument tests were neutralized utilizing refined water, dried at 110°C, and named MGPC-800, MGPC-850, and MGPC-900, as per the shifting temperatures.

Material Characterization

The examining the electron microscopy (SEM) and energy dispersive spectroscopy (EDS) was utilized to assess morphological construction and basic status with JEOL-JSM-6510LA instrument. The graphitization confirmation was identify with X-ray diffraction (XRD) in 10-100° degree. The N_2 gas adsorption-desorption strategy was utilized to evaluated the porosity properties with BET, BJH, and T-plot techniques.

Electrochemical Measurements

The electrochemical presentation of a permeable carbon stone monument as a supercapacitor terminal was estimated in cyclic voltammetry (CV) utilizing a CV UR Rad-trama center 5841 instrument adjusted by VersaStat II Princeton Applied Exploration at a blunder pace of ±6.0%. The voltage window goes from 0 to 1 V with examine paces of 1-10 mV s^{-1}. The functioning cathodes are collected in a symmetric supercapacitor framework in a sandwich layer drenched in a fluid electrolyte of 1 M H_2SO_4. Furthermore, the electrochemical properties of the examples were likewise estimated by galvanostatic charge-discharge (GCD). Explicit capacitance, energy, and power output are assessed utilizing conditions standard. Furthermore, the strategy for electrochemical impedance spectroscopy (EIS) was additionally done to affirm the in-depth electrochemical properties of the electrode material. EIS was performed from 0.01 Hz to 100,000

Hz, with the particular capacitance, as well as energy and, not entirely settled by standard conditions in view of a symmetrical framework.

3. Results and Discussions

The physical activation reaction of high-temperature CO_2 on the carbon chain affects the surface morphology of MGPC solids, which were reviewed in detail using scanning electron microscopy (SEM) with the images of MGPC-800, MGPC-850, and MGPC900 shown in Fig. 1 at different magnifications. Micrograph of MGPC-800 showed a dominated large particle aggregates, as shown in Fig. 1a. In the enlarged selection area in Fig. 1b, MGPC-800 displays a large pore on the particle block wall, confirming the presence of a micron-sized pore. Furthermore, the expansion in the actual enactment temperature at 850°C exhibits a relatively dominated surface morphology by small-size carbon blocks with significant aggregate breakdown. According to Fig. 1c, the size of the carbon blocks varies from 0.063µm to 2.498 µm with etching carbon chains at high temperatures, which allows dramatic erosion of the lignocellulosic components, thereby affecting the surface structure. Significant erosion of cellulose and lignin initiates the formation of 2D nanostructures, such as tubular, fiber, and sheet (Kumar et al., 2020). Fig. 1d displays a magnified area of the MGPC-850 SEM image confirming a morphological structure resembling a rod/tube at 30-36nm diameter. The etching of optimal carbon chains occurs at 850^{o}C with morphology that leads to 2D nanostructures. The continuous erosion of the particle aggregate at high temperatures up to 900°C allows the precursor-based material to undergo significant morphological changes, as shown in Fig. 1e. The MGPC-900 displays relatively large pores in the macropores range with aggregates ranging from 0.238µm to 2.276µm and relatively larger than MGPC-850. Furthermore, in the selected area selection, the MGPC-900 displays pores of various mesoporous scales interconnected in 3D. These pore

characteristics contribute significantly to ion accessibility and barrier-free charge transfer on the electrode material to maintain high power density performance in supercapacitor energy storage components (Hao et al., 2017). The degree of crystallinity and phase change that occurred in the porous carbon of MGPCs was assessed using an X-ray diffraction (XRD) pattern, as shown in Fig. 2. It showed two significant broad peaks at 2θ approximately 21.53°, 24.15°, and 24.76° indexed to the scattering plane (002) and 42.71°, 44.91°, and 45.44° which are correlated to the scattering plane (100) of MGPC-800, MGPC-850, and MGPC-900, respectively. It further characterizes the amorphous-turbostratic carbon structure rich in pores and attenuated crystallinity (JCPDS No. 41–1487) (Peng et al., 2013; Sodtipinta et al., 2017), which proves that the sample has a confirmed pore structure. Expansion in the actual enactment temperature at MGPC800 to MGPC-850 displays a shift of the indexed width peak (002) towards a smaller one, thereby confirming the abundant increase in the growth of narrow pores. Additionally, the broad peak in the scattering plane (100) shifted to a greater extent from 42.487° to 45.710°, allowing MGPC-850 to have confirmed mesopores on the precursor surface. The combined features of microporous and mesoporous growth in the based material contribute to the electrode's high capacitive properties and low internal resistance. This analysis is further justified through the analysis of electrochemical properties using CV and GCD techniques. However, an expansion in the actual enactment temperature up to 900°C displays a different pattern and leads to the opposite feature, thereby confirming a good reduction in pore development. This is because excessive carbon chain etching at 900°C harmed the pore system construction of the antecedent. The ratio of basic parts present in MGPCs is uncovered through energy dispersive spectroscopy (EDS) estimations, and it summarized in Table 1. Evaporation of water content, volatiles, polysaccharides, and hemicelluloses, as well as degradation of other organic components due to

carbonization in inert gases, initiates the production of high carbon fixed. Furthermore, the high physical activation temperature followed by carbon chain removal significantly reduced the lignocellulosic content leading to the strengthening of the porous carbon skeleton. Table 1 show that the main constituent elements of all samples were dominated by high carbon at 93.73%, 93.89%, and 93.13% for MGPC-800, MGPC-850, and MGPC-900, respectively. This high carbon content permits the terminal material to deliver high electrical conductivity properties followed by a low comparable series opposition, improving the supercapacitor's extraordinary execution. Alternately, oxygen possesses the second most elevated component in the scope of 4.02% to 6.02% in light of the fact that the exorbitant carving of carbon chains by CO_2 permits the antecedent to create oxidation results that tight spot to inorganic parts. The high oxygen content has a positive side for the electrode material, especially in their contribution to the improvement of wettability, maintained conductivity, self-doping of heteroatoms, and the pseudo-capacitance effect on supercapacitor (Abbas et al., 2019; Zheng et al., 2021). Due to incomplete evaporation of the remaining pyrolysis production, Silicon and zinc are also found in small amounts in MGPC. The porosity conduct of the MGPCs acquired from high-temperature pyrolysis contrast in regards to the N_2 sorption isotherm and pore size conveyance, as displayed in Fig. 3a-b and summed up in Table 1. The N_2 isotherm adsorption-desorption profiles of MGPCs show a noticeable mix of elements of the kind sort I and type IV bends, as displayed in Fig. 3a. High N_2 absorption in the region of very low relative pressure characterizes strong I-type features, indicating that more than 90% of micropores dominate MGPCs. This component is likewise affirmed in Table 1, which shows that the typical pore width of MGPCs is around 1 nm. Micropores are bountiful on the 1 nm pore size, subsequently causing an extreme expansion in the particle pool at the electrolyte/terminal connection point, starting the advancement of

strangely high unambiguous capacitance. This assumption is analyzed more deeply in CV and GCD measurements. At the same time, the hysteresis loops displayed over a greater relative pressure region confirm the presence of type-IV, closely related to defined mesoporosity. This feature is useful to increase the accessibility of ionic charges on absorption surfaces for the electrolyte ions to move in all directions, thereby initiating an increase in fast currents and high-power capabilities. However, the hysterical loop on MGPC-800 is imperfect (not closed) and reflects the creation of imperfect mesopores with the assumption that the pore shape resembles a "bottleneck" with a narrow pore surface and relatively large space on the inside. The defect of the mesoporous structure can be corrected by increasing the pyrolysis temperature. The higher porosity temperature in MGPC-850 and MGPC-900 indicates an increase in the mesopores structure, which is illustrated by their better hysteresis loop compared to MGPC-850. Figure 2b shows that the expanded pyrolysis temperature expanded the pore size circulation on the mesoporous scale with a typical size of 3.62nm. This additionally builds the mesoporous volume from 0.019 cm^3 g^{-1} to 0.040 cm^3 g^{-1}, as displayed in Table 1. The expansion in pyrolysis temperature can essentially raise the development of the particular surface area of MGPCs from 800 to 900°C, with a fast ascent in the particular surface region coming to ± 53% of 639.414 m^2 g^{-1}. In addition, their micropore predominance is likewise advanced to 94.6% with clear-cut mesopores, which all the while act as the principal key to helping the high electrochemical presentation of cathode materials. These incorporate expanding the particular capacitance and energy output without undermining their power output.

The electrochemical performance of the MGPCs electrode was first traced through the cyclic voltammetry (CV) strategy in a symmetrical configuration at M H_2SO_4 liquid electrolyte. The CV profiles at 1 mV s^{-1} of the MGPC-800, MGPC850, and MGPC-900 illustrate a distorted/non-

ideal rectangular shape (Fig. 4a), thereby revealing normal electrical double-layer properties (Climent & Feliu, 2018). The MGPC-800 electrode displays a relatively small CV profile, which characterizes its low capacitive properties. In the low voltage range V<0.1V, the current density increases suddenly, confirming that the ionic charge is simultaneously transmigrated to the micropores on the electrode surface. Furthermore, their CV profile tends to slope at higher potential ranges, confirming the relatively low ionic charge transfer in the large pores with a low capacitance of 155 F g^{-1}. On the other hand, the MGPC-850 electrode displayed the largest charge-discharge loop hysteresis, confirming the highest electrochemical properties. At a potential <0.1, the current density increased significantly, indicating that the micropores-rich MGPC-850 material filled with electrolytic ions formed the first electrical double layer. The unexpected expansion in current thickness that frames a "camel bump" in the voltage range 0.20<V<0.50 shows a useful commitment of oxygen as a self-doping heteroatom that initiates the faradaic reaction in the electrode material (Meng et al., 2020), which presents a pseudo-capacitance effect on the electrochemical properties of MGPC-850. Interestingly, in the voltage range V>0.6V, the current density continued to increase drastically until V=1.0V, indicating that the mesoporous MGPC-850 is optimally accessed by the electrolytic ion charge adding to the abundant electrochemical double layer capacitor. The elite presentation mix of the MGPC-850 cathode shows an expanded capacitance worth of 191 F g^{-1}. This demonstrates that blending activated carbon derived organic precursor as cathode sources by expanding the actual actuation temperature of CO_2 builds the particular capacitance of the supercapacitor, particularly in the symmetrical arrangement framework. However, the increased physical activation temperature on the MGPC-900 did not display the same characteristic. The CV profile displayed by the MGPC-900 electrode is relatively similar to the MGPC-800, indicating that their capacitive properties

are the same. The reduction of amorphous and carbon content of MGPC-900, which XRD and EDS analyzed, had a major impact on the reduction of the electro-analytical behavior of MGPC-900. The capacitance feature obtained in the symmetric cell MGPC-900 is about 165 F g^{-1}. The electrochemical features of MGPCs were confirmed through scan rates at 1-5 mV s^{-1}, as illustrated in Fig. 4b-d. The CV shape generally performed an increasingly ideal rectangular form confirming the potential for MGPCs electrodes to have normal electrical multi-layer (EDLC) behavior based on activated carbon sources. Moreover, their cyclical performance is also reviewed in terms of capacitances feature in high scan rate, as shown in Fig. 3e. The capacitance behavior of the MGPCs precursor degraded drastically at a scan rate of 10 mV s^{-1} because the carbon material has a low meso-macroporosity, therefore, the ionic charge is not optimally induced at electrolyte/terminal connection point (Y. Zhang et al., 2017). The electrochemical features of MGPCs was identify through a galvanostatic charge-discharge (GCD) which represents the detailed behavior of specific capacitive properties, electrode resistance, energy output, and power output in a symmetric cell system. The GCD profile for the terminals of MGPCs submerged at 1 A g^{-1} in H_2SO_4 electrolyte is displayed in Fig. 5a. In general, all electrodes in a symmetrical cell system displayed disturbed isosceles triangle curves, thereby confirming their normal potential in the electrical layer (EDLC) type. Furthermore, their relatively long charging time compared to the discharging time indicates ion degradation in the charge transfer process. As indicated by primer investigations, the wettability of the oxygen utilitarian gathering starts the faradaic response (Luo et al., 2021). This prompts an expansion in the pseudo-capacitance property, as affirmed in the CV shape. Coulombic productivity is gotten from the information on the distinction between the charging and releasing time of the GCD (η=[td/tc]x100%). Coulombic proficiency was found in the scope of 50-70% in MGPCs

pseudocapacitance was additionally viewed because of particle corruption from doped heteroatoms, particularly self-oxygen doped. Besides, the somewhat prevailing dispersion of micropores additionally influences their proficiency. Additionally, the voltage drop, commonly known as the IR drop, is determined from the GCD profile, thereby revealing the electrode resistances of the material electrodes of 0.099, 0.154, and 0.059 Ω for MGPC-800, MGPC-850, and MGPC-900, respectively. In more detail, the relatively higher IR-drop on MGPC-800 is indicated by the effect of pore growth in the material. The rich micropores apparently inhibit the charge transport pathway during ion insertion-deinsertion at the electrolyte/terminal connection point. The time required to perform one charge and discharge cycle reflect the specific capacitance of the electrical material. According to the standard equation, the specific capacitance obtained from the MGPC-800, MGPC-850, and MGPC-900 electrodes were 166, 208, and 180 F g^{-1}, respectively. Mission grass-based porous carbon synthesis routes involving chemical impregnation and physical activation of 800°C high-temperature CO_2 significantly improved the high performance of supercapacitor cells in two electrode configurations of 166 F g^{-1} with their relatively low cell resistance of 0.099 Ω. This is because the chosen strategy resulted in the obtained carbon material properties with high potential in porosity, surface morphology, clear elemental status, and high electrical conductivity (Selvaraj et al., 2021). Furthermore, increasing the physical activation temperature to 850°C drastically raised the capacitance feature of 208 F g^{-1}, indicating that the porosity optimization and abundant active channels can accommodate the high ion migration markedly, thereby improving their electrochemical performance (H. Wang et al., 2021). MGPC-850 has the best amorphous properties with the highest predicted specific surface area, enabling them to form an enriched electrical double layer, thereby enhancing their capacitive properties. The presence of the

pseudo-capacitance impact of practical oxygen adds to the superior exhibition of the terminal material. Notwithstanding, expanding the actual enactment temperature to 900°C prompted a reduction in capacitive properties by 180 F g^{-1} because of the deformity of the pore structure in the MGPC-900 carbon material. The reduced amorphous and empirical surface area affected their capacitive behavior (Deraman et al., 2015). Meanwhile, the energy and power outputs performances of the MGPCs electrodes were possessed through the Ragone plot, as illustrated at Fig. 5b. The energy output obtained from the MGPC-800, MGPC-850, and MGPC-900 are 22.64, 28.31, and 24.58 Wh kg^{-1}, at optimum power output of 73.83, 65.47, and 60.62 W kg^{-1}. This value is relatively similar to preliminary studies that have been reported with activated carbon sources biomass-based, such as bacterial celulose (Hao et al., 2017) and European wood (Jain et al., 2021). The highest electrochemical performance on the MGPC-850 electrode was reviewed in detail through impedance spectroscopy on an H_2SO_4 electrolyte with a measurement frequency from 0.01 Hz to 100,000 Hz. The Nyquist plot of the MGPC-850 electrodes, shows in Fig. 6a, shows that their curve consists of several sections. Firstly, the high frequency semicircular curvature confirms the ions' rapid migration at electrolyte/terminal connection point, which affects the insersi-deinsersi in the electrode material (Lämmel et al., 2013). Secondly, the first Z' axis intercept characterizes the selected electrolyte resistance of 0.038Ω, while the second Z' intercept represents the equivalent series resistance (ESR) reflecting the MGPC850 electrode's electronic resistance of 0.139 Ω. This low resistance is due to the contribution of high porosity followed by the wettability properties of the optimized oxygen internal doped in the MGPC-850 material (Yadav et al., 2020). The Warburg curve found at intermediate frequencies indicates fluctuating ion insertions into the framework structure carbon, as shown in insert Fig. 5a. At low frequencies, the vertical line pattern clearly reflects the hallmarks of an ideal electrical dual layer

response, which is also in agreement with the analysis revealed in the CV and GCD measurements. Fig. 6b shows the Bode phase plot in the low to a high-frequency range of the MGPC-850 electrode. At low frequencies, the value of the phase angle is about -78°, which affirms the capacitive way of behaving of the typical permeable carbon-based electrical twofold layer with low opposition and high charge move (Atika & Dutta, 2021). Furthermore, the knee recurrence at the staging point of -45° decides the unwinding season of the anode material, which is firmly connected with the release and charge time proficiency at the cathode/electrolyte interface. The unwinding time consistent of the MGPC-850 viewed as through the $1/f_0$ condition is 25.12 ms, which shows great terminal materials for down to earth applications in electrochemical supercapacitor.

4. Conclusions

In conclusion, this research effectively applied an eco-harmless and efficient methodology as a top-notch terminal material for the change of mission-grass biomass into permeable carbon for sustainable power stockpiling gadgets. The pore morphological highlights, amorphism, wettability, and natural status were completely controlled through the actual enactment temperatures of 800, 850, and 900°C. Moreover, an expansion in the actual enactment temperature fundamentally influences the progressions in their pore morphology, prompting the development of progressively associated 3D pores. In the improved permeable carbon, the natural condition of high carbon is 93.89%, with the wettability properties contributed by the oxygen practical gathering. The nebulousness was essentially affirmed in the symmetrical-terminal arrangement framework to acquire explicit capacitances of 166, 208, and 180 F g^{-1} for MGPC-800, MGPC-850, and MGPC-900 cathodes, separately. The most noteworthy energy output of 28.31 Wh kg^{-1} was found at the MGPC-850 in a H_2SO_4 fluid electrolyte at 1 A g^{-1}.

Consequently, the proposed ecologically harmless methodology expressly investigates mission-grass-based activated carbon as a electrode sources for supportable electrochemical supercapacitor.

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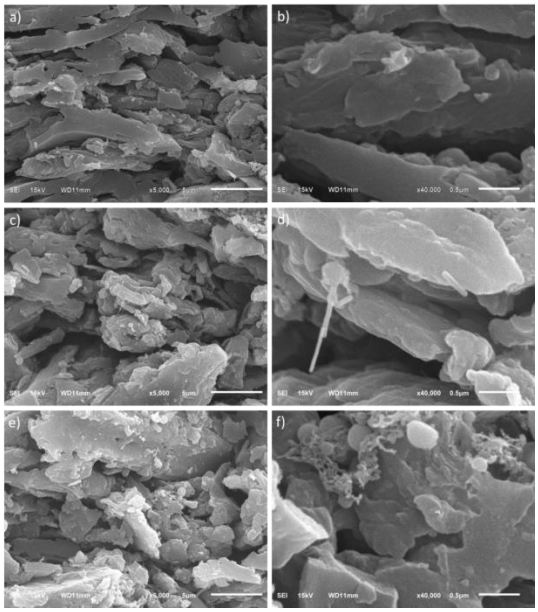

Fig. 1. Image SEM for a)-b) MGPC-800, c)-d) MGPC-850, and e)-f) MGPC-900 in 5000x and 40000x magnification

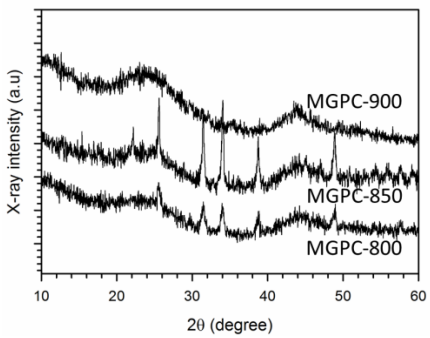

Fig. 2. XRD pattern of MGPCs

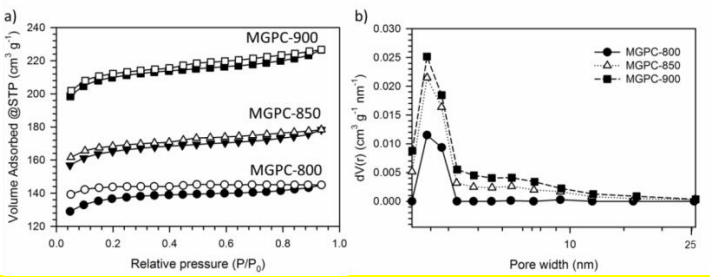

Fig. 3. a) Nitrogen adsorption-desorption profiles and b) The pore size distribution (PSD) of MGPCs

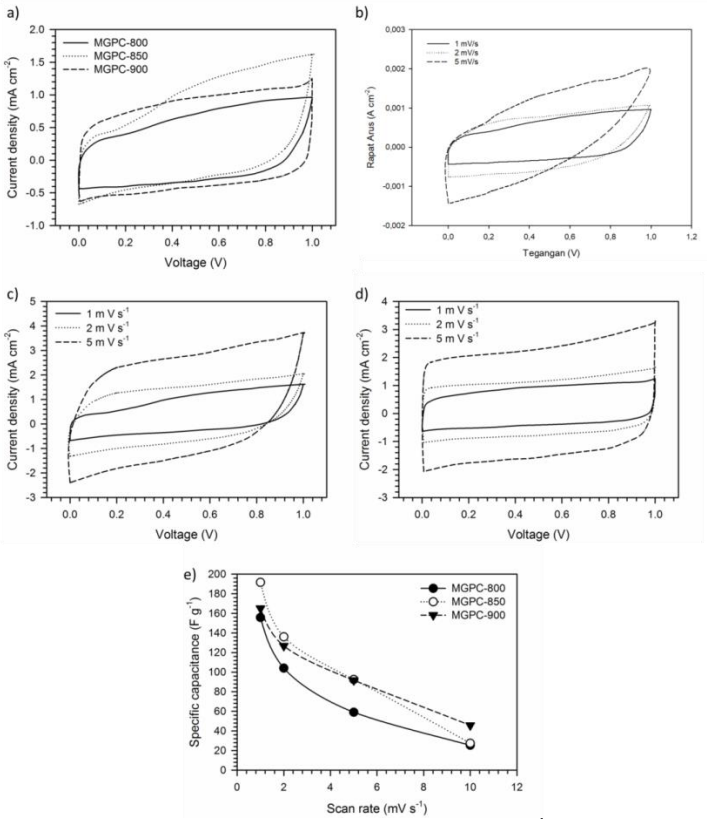

Fig. 3. CV profile of MGPCs samples a) at scan rate 1 mV s^{-1}, b) MGPC-800 at scan rate 1-5 mV s^{-1}, c) MGPC-850 at scan rate 1-5 mV s^{-1}, d) MGPC-900 at scan rate 1-5 mV s^{-1}, and e) Specific capacitance vs. Scan rate

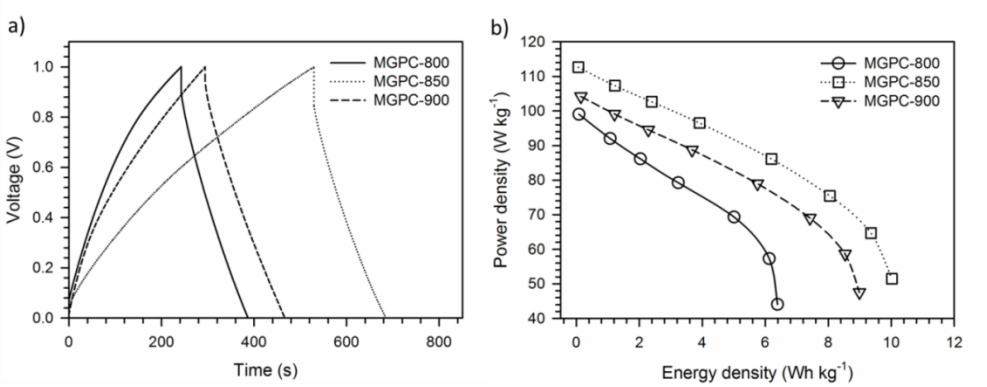

Fig. 5. a) GCD profile of MGPCs samples in 1 M H_2SO_4, and b) Ragone plot of MGPC samples in 1 M H_2SO_4

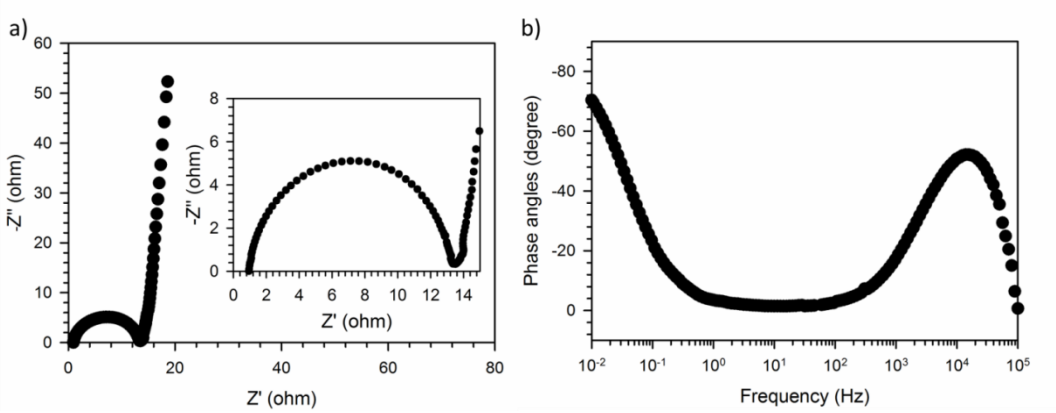

Fig. 6. a) The Nyquist plot of the MGPC-850, and b) the Bode phase plot of the MGPC-850

Table 1. Porosity properties and elemental status of MGPCs

Precursor	S_{BET} ($m^2\ g^{-1}$)	S_{micro} ($m^2\ g^{-1}$)	S_{meso} ($m^2\ g^{-1}$)	V_{tot} ($cm^3\ g^{-1}$)	V_{micro} ($cm^3\ g^{-1}$)	V_{meso} ($cm^3\ g^{-1}$)	D_{ave} (nm)	C (%)	O (%)	Zn (%)	Si (%)
MGPC-800	416.892	400.299	16.593	0.225	0.206	0.019	1.078	93.73	4.51	1.34	0.42
MGPC-850	503.551	476.252	27.299	0.277	0.245	0.032	1.099	93.89	4.02	1.55	0.55
MGPC-900	639.414	605.048	34.366	0.352	0.312	0.040	1.099	93.13	6.02	0.00	0.84

Gmail

Rika Taslim <rikataslim@gmail.com>

Revised submission received for Energy Sources, Part A: Recovery, Utilization, and Environmental Effects (Submission ID: 234063593.R1)

1 message

ueso-peerreview@journals.tandf.co.uk <ueso-peerreview@journals.tandf.co.uk> Thu, Jul 13, 2023 at 10:50 AM
To: rikataslim@gmail.com

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Dear Rika Taslim,

Thank you for submitting your revised manuscript.

Submission ID	**234063593**
Manuscript Title	**Mission grass bio-waste functional carbon self-single-doped for ultrahigh energy symmetrical supercapacitor**
Journal	**Energy Sources, Part A: Recovery, Utilization, and Environmental Effects**

You can check the progress of your submission, and make any requested revisions, on the Author Portal.

Thank you for submitting your work to our journal.
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Kind Regards,
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Gmail **Rika Taslim <rikataslim@gmail.com>**

Energy Sources, Part A: Recovery, Utilization, and Environmental Effects - Decision on Manuscript ID UESO-2023-1274.R1

1 message

Energy Sources, Part A: Recovery, Utilization, and Environmental Effects <onbehalfof@manuscriptcentral.com>
Reply-To: snizetic@fesb.hr
To: rikataslim@gmail.com

Mon, Jul 24, 2023 at 7:29 PM

24-Jul-2023

Dear Dr Taslim:

Ref: Mission grass bio-waste functional carbon self-single-doped for ultrahigh energy symmetrical supercapacitor

Our referees have now considered your paper and have recommended publication in Energy Sources, Part A: Recovery, Utilization, and Environmental Effects. We are pleased to accept your paper in its current form which will now be forwarded to the publisher for copy editing and typesetting. The reviewer comments are included at the bottom of this letter.

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Thank you for your contribution to Energy Sources, Part A: Recovery, Utilization, and Environmental Effects and we look forward to receiving further submissions from you.

Sincerely,
Professor Nižetić
University of Split Faculty of Electrical Engineering Mechanical Engineering and Naval Architecture
Editor-in-Chief, Energy Sources, Part A: Recovery, Utilization, and Environmental Effects
snizetic@fesb.hr

Reviewer(s)' Comments to Author:

Reviewer: 1

Comments to the Author
The revision is good.

Reviewer: 2

Comments to the Author
no other suggestion

Gmail **Rika Taslim <rikataslim@gmail.com>**

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Energy Sources, Part A: Recovery, Utilization, and Environmental Effects Tue, Jul 25, 2023 at 3:36 AM
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Dear Dr Rika Taslim:

Congratulations on your accepted article. Before I can proceed to sending it to production, I need these issues resolved:

1. Please send me a clean version of your manuscript without any markup of revision changes, such as colored text, highlighting, Track Changes, or bold/underlined text used only to indicate changes.

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Please reply to me with any appropriate new file(s) as an attachment. I only need the files that involve the changes requested above, I do not need additional copies of files you have already submitted and that will remain the same. I can edit the system and/or upload files on your behalf. Thanks and let me know if you have any questions

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Ms Melissa Wilkinson
Energy Sources, Part A: Recovery, Utilization, and Environmental Effects

Rika Taslim <rikataslim@gmail.com> Tue, Jul 25, 2023 at 2:39 PM
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Thank you for accepting our manuscript in the Journal of Energy Sources Part A: Recovery, Utilization, and Environmental Effects.

We greatly appreciate the editor's consideration of this manuscript for publication. We have adjusted the manuscript accordingly in a clean version. In addition, all figures in the manuscript are given .tiff file type with 300 dpi. We confirm that any figure in this manuscript was revised in accordance with the reviewers' comments in Journal of Energy Sources Part A: Recovery, Utilization, and Environmental Effects.

Best regards,

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Corresponding author
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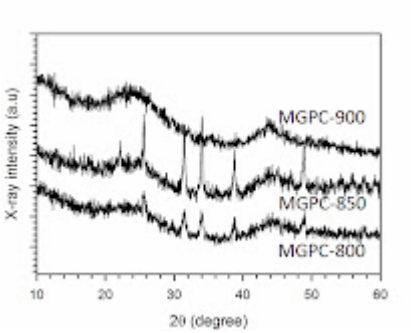
7 attachments



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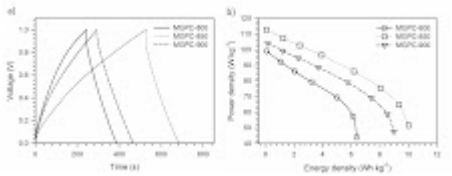

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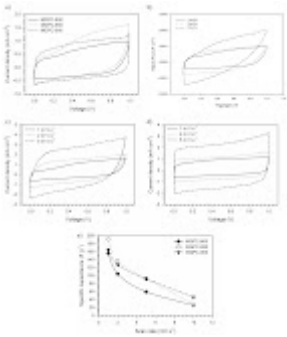

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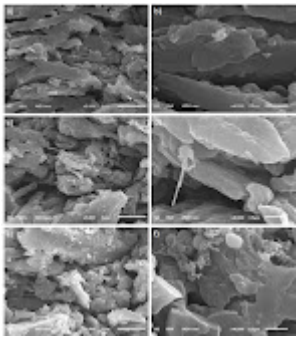

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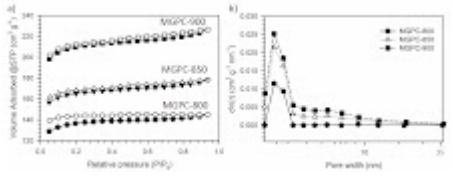

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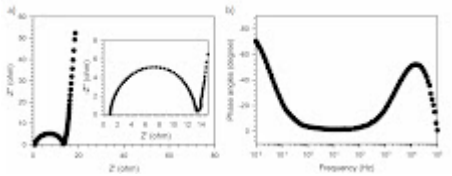

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Hello Dr. Taslim,

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Energy Sources, Part A: Recovery, Utilization, and Environmental Effects

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Cc:
Subject: Re: Re: NOTICE: Outstanding information required for your accepted manuscript

Dear Ms. Melissa Wilkinson

Thank you for accepting our manuscript in the Journal of Energy Sources Part A: Recovery, Utilization, and Environmental Effects.

We greatly appreciate the editor's consideration of this manuscript for publication. We have adjusted the manuscript accordingly in a clean version. In addition, all figures in the manuscript are given .tiff file type with 300

dpi. We confirm that any figure in this manuscript was revised in accordance with the reviewers' comments in Journal of Energy Sources Part A: Recovery, Utilization, and Environmental Effects.

Best regards,

Rika Taslim
Corresponding author

On Tue, Jul 25, 2023 at 3:36 AM Energy Sources, Part A: Recovery, Utilization, and Environmental Effects <onbehalfof@manuscriptcentral.com> wrote:

> 24-Jul-2023
>
> UESO-2023-1274.R1 - Mission grass bio-waste functional carbon self-single-doped for ultrahigh energy symmetrical supercapacitor
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> Dear Dr Rika Taslim:
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> Congratulations on your accepted article. Before I can proceed to sending it to production, I need these issues resolved:
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>
> Sincerely,
> Ms Melissa Wilkinson
> Energy Sources, Part A: Recovery, Utilization, and Environmental Effects

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Rika Taslim <rikataslim@gmail.com> Wed, Jul 26, 2023 at 5:09 AM
To: Apri wandi <apriwandi95@gmail.com>

FYA
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Rika Taslim <rikataslim@gmail.com> Wed, Jul 26, 2023 at 7:52 AM
To: ueso-peerreview@journals.tandf.co.uk

Dear Ms. Melissa Wilkinson

Thank you for the information. we apologize for this omission. we have re-prepared the figures in 300 dpi format. In the attachment file, we attach the revised 300 dpi figures quality.

best regards

Rika Taslim
Corresponding author
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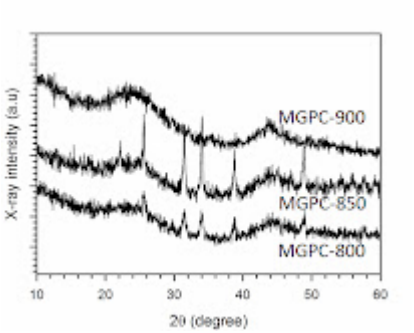
6 attachments



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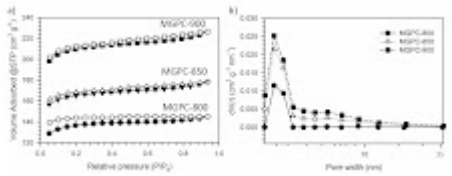

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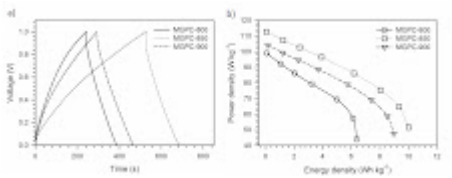

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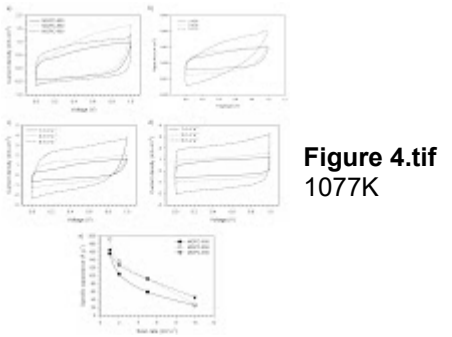

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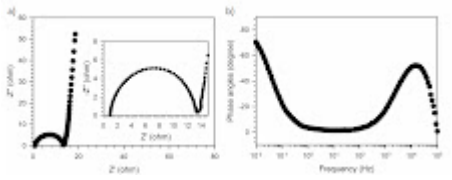

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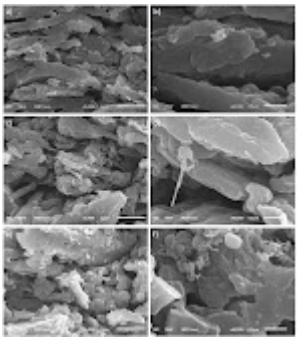

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