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THE IN-LIQUID MICROWAVE PLASMA IRRADIATION ON NAOH-ACTIVATED CARBON OF THE ENERGY STORAGE ELECTRODES

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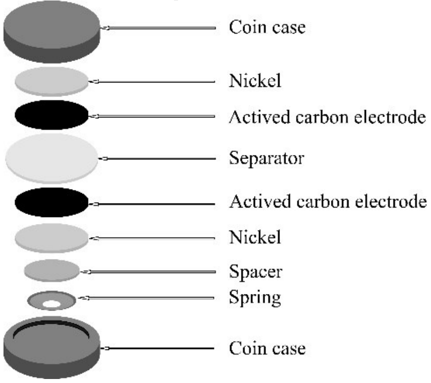
Graphical abstract



Abstract

Evolution of energy storage systems, especially concerning electrodes, always continue In this study. In this work, we use in-liquid MW plasma with to improve the electrochemical properties of energy storage electrodes on the activated carbon. MW plasma irradiation was applied for 1, 2 and 3 minutes in NaOH -activated carbon solution. This was the first time that nickel was used as the current collector, NaOH as electrolyte and PTFE membrane as separator in the electrochemical characteristics of a supercapacitor. For the characterization of the activation of carbon, SEM and XRD analyses were performed and it was shown that when the plasma irradiation time increased the carbon density decreased. The surface topography is characterized here at larger pores and the conversion of potassium oxide phases into potassium sulfide. The galvanostatic charge/discharge and cyclic voltammetry were utilized to study the electrochemical properties of supercapacitors. The highest capacitance obtained was with 1 M NaOH electrolyte with 1 minute microwave plasma treatment and optimal energy and power densities were realized with a 3 M NaOH electrolyte with 1 minute microwave plasma irradiation. where ionic conductors serve as electrolytes between electrodes.

Keywords: Activated carbon, supercapacitor, the in-liquid MW plasma, NaOH electrolyte

1.0 INTRODUCTION

Electrical energy has become a fundamental necessity for global society and has significantly impacted practically all facets of human existence[1]. The dependence on fossil fuels for energy production has led to air pollution, adversely affecting general quality of life and public health [2]. Recent improvements in renewable energy production indicate that it can compete with fossil fuels regarding exhaust emissions [3]. As consequence, rules requiring the utilization of renewable energy for electricity generation have motivated researchers to advance this technology. However, disagreements among researchers persist concerning the generation of electrical energy from renewable sources, especially regarding energy storage. An essential topic of discussion was the use of hybrid systems to charge solar cells via supercapacitors [4, 5, 6]. There has been some debate about the use of supercapacitors in hybrid systems for charging solar cells [5]. Furthermore, the evolution of the electronics sector has caused an uptick in the need for portable power suppliers. Also, many advances in electronics have increased the demand for portable power sources [6]; researchers are encouraged to develop various types of electrochemical energy storage systems by creating high-potential nanomaterials suitable for energy storage applications [7].

One of the most commonly used energy storage devices at present is the battery, but it has disadvantages such as long charging time, limited lifespan and limited power density [8]. Energy storage devices consist of components that are put together to produce an energy storage device including electrodes, current collectors, separators, and electrolytes [9, 10]. The present collector on the electrode is essential for improving the efficiency of energy storage devices like batteries and supercapacitors. Nickel is a widely studied material because of its beneficial characteristics, including great electrical conductivity, high theoretical capacity, fast charge transfer ability, excellent reaction kinetics, and plentiful availability on Earth [11]. The substances employed as electrodes in batteries can create harmful waste, potentially leading to a waste management problem in the future when batteries are used for energy storage [12].

Nonetheless, studies on energy storage are currently advancing quickly with different technologies that researchers are continually improving. One promising technology that could be further developed is microwave plasma (MW plasma) [13]. The idea of plasma was introduced by Langmuir and Tonks in 1928. They described plasma as a gas that becomes ionized through an electric discharge, which means plasma can also be seen as a moving mixture of electrons, radicals, and both positive and negative ions [14]. The interaction between positive ions and negative electrons leads to characteristics that are quite distinct from regular gases, and this state of matter is referred to as the plasma phase. In simple terms, plasma is described as ionized gas and recognized as the fourth state of matter, following solid, liquid, and gas [15]. MW plasma can be used to change materials because it contains charged particles and free radicals that can alter the material's structure [16].

Activated carbon is a material that has a specific structure. It can be employed as an electrode material in supercapacitors, which is more sustainable and readily obtainable [17]. The degree of biomass, particularly coconut shells with their strong fibers, has been found to be able to convert into pores of carbon with finer size. Hence, it might be a good alternative as a material based on energy storage electrodes [18, 19]. Recent research studies have mostly focused on the production of activated carbon using various kinds of biomass [20]. Due to these properties, biomass-derived activated carbon is among the most used and studied materials for Electric Double Layer Capacitor (EDLC) applications [21]. This study shows that biomass serves as promising material for supercapacitors. However, recent studies on supercapacitor electrodes have found a limitation of the carbon biomass activation methods [22, 23]. Essentially, the optimum physical activation process occurs at 1000°C yielding quality activated carbon [24]. A new activation n method is proposed using Chemical Vapor deposition (CVD) plasma radiation and nitrogen gas for Noriaki Sano *et al*. This process created electrodes that could accommodate greater energy-storage capacities. Plasma treatment with nitrogen greatly enhances its electrochemical performance for supercapacitors [25]. The theoretical imbalance and further detail of electroporation is still under much research in order to improve the pore structure and the performance of the electrodes due to some activation processes inducing imperfect pores. Moreover, the caused pores can be tuned to ameliorate their nature and architecture, giving rise to advanced porous carbons with hierarchical porosity and oxygen-enriched chemical compositions [26].

While sodium hydroxide (NaOH) is commonly known as a chemical oxidizer in this process, scientists have employed it in both activating and changing activated materials. According to various studies, it was found that NaOH is the best acidic oxidizing agent [27] to increase the oxygen functional groups species on activated carbon modification. The soda NaOH plays a vital role in the modification of activated carbon and increases the adsorption capacity of any material. The surface area, pore volume, and pore size of activated carbon can be increased by NaOH treatment [28].

These changes ensure a strong capacity for energy storage and performance rates, as shown by the developed supercapacitor, which showed a high specific capacitance. The findings emphasize the promise of using microwave and plasma techniques to create affordable porous carbon for effective supercapacitor applications [29]. The selection of electrolyte greatly influences the performance of electrodes in supercapacitors [30]. At present, most studies use liquid electrolytes, which can be divided into acid electrolytes (like H_2SO_4, H_3PO_4), basic electrolytes (such as KOH, LiOH, NaOH), and neutral electrolytes (including LiCl, Li_3PO_4, Na_2SO_4) [31]. Liquid electrolytes are the main option for supercapacitor studies due to their low cost and simple upkeep [32]. The separators play a key role in transferring the charge in the charging and discharging process in energy storage devices [33].

Developing novel electrode materials for energy storage devices, such as supercapacitor (the units with extremely large capacitance specific, power density and durability), has been the target of these studies. The discovery of ultra-thin materials such as graphene, MXenes, transition metal dichalcogenides, and metal oxides as twodimensional (2D) nanomaterials has attracted attention due to their unique physical and chemical properties [34]. Moreover, chalcogenides, carbides, and nitrides including conducting polymers as a host of electrode materials have also been reported to be suitable [35]. However, graphene and polymer materials are relatively expensive, and the complicated production methods lead researchers to look for low-cost, abundant, easy-to-synthesize and green alternatives, including activated carbon [36]. In order for activated carbon to be able to compete with high-performance materials, modifications must be made in order to improve its electrochemical performance. Developing modification technologies could offer new options for energy storage advancements, one of which is MW plasma.

This study aims to evaluate and compare the impact of plasma irradiation on the electrochemical properties of carbon-based electrodes using two electrode configurations. A simple approach is employed to modify and fabricate carbon electrodes, resulting in exceptional electrochemical performance using the same separator, electrolyte, and current collector.

2.0 METHODOLOGY

2.1 Materials

The activated carbon used from coconut shells as an electrode material has the potential to be developed [37], while nickel mesh serves as the current collector [38]. A PTFE membrane was employed as the separator [39]. The electrolyte material is sodium hydroxide (NaOH) [40].

2.2 Preparation of Material

Activated carbon was subjected to in-liquid microwave plasma irradiation to enhance its structural properties as an electrode material as shown in Figure 1. Figure 1 shows some components used in producing plasma and then transmitted to activated carbon. This tool produces plasma by generator used is a microwave magnetron that has been modified to generate microwaves. The microwaves generated by the magnetron are channeled to the reactor using a tungsten electrode through a wave gate. The reactor contains activated carbon material with NaOH solution to carry out irradiation.

Sodium hydroxide (NaOH) was utilized as a solution during the plasma radiation process. Sodium hydroxide (NaOH) plays an important role in modifying activated carbon to improve its capacity to adsorb materials. Using NaOH increases the surface area, pore size, and expands the volume of pores in activated carbon [28] The plasma treatment duration was varied, ranging from 1 to 3 minutes.

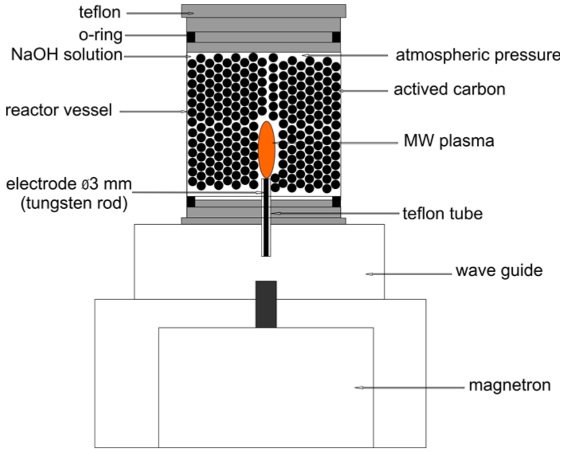


Figure 1 The in-liquid MW plasma method

In this study, nickel was used as a current collector and was subjected to immersion in an H_2O_2 solution to cleanse its surface. The NaOH electrolyte was dissolved in distilled water to achieve the desired molarity. Activated carbon was compressed at hydraulic pressure of 40 kN/cm^2 into a coin-shaped carbon electrode (CSCE) as in Figure 2. Then, the density of 0.5 g of CSCE was evaluated with ASTM D1513 standards.

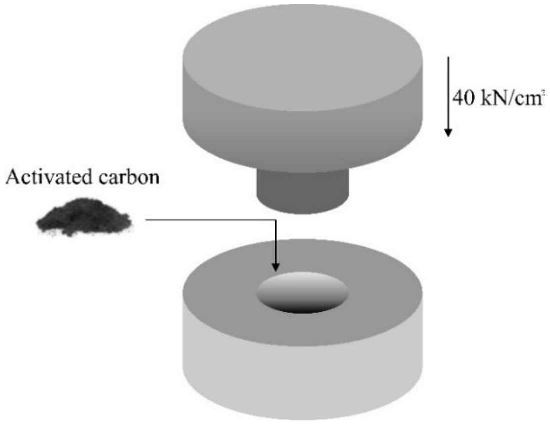


Figure 2 The compaction of activated carbon electrodes

2.3 Characterization

The resulting carbon electrodes were characterized using X-ray diffraction (XRD) on a SHIMADZU Maxima X-7000. The operating parameters were 40 mA current and 40 kV voltage at 3.5 C/min. Then, scanning electron microscopy (SEM) was performed using a Neo Scope Benchtop SEM JCM-7000. The function of SEM is to focus a high-energy electron beam at 5.00 kV on the surface of the electrode material to generate various signals. The aim was to examine the changes in crystallization and morphological structure between plasma irradiated and untreated carbon electrodes.

2.4 Supercapacitor

Supercapacitors are composed of a coin-shaped carbon electrode, nickel current collectors, and separator in a case, as in Figure 3. The assembled supercapacitor was tested for capacitance using cyclic voltammetry. The duration of charging and discharging was determined by galvanostatic charge/discharge. All performance evaluations were conducted employing the Corrtest CS350 instrument.

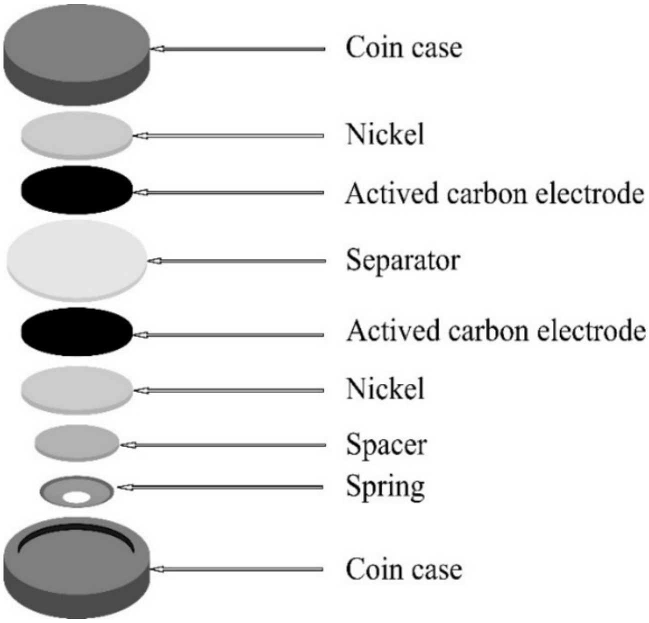


Figure 3 Supercapacitor components

3.0 RESULT AND DISCUSSION

3.1 Activated Carbon Density Analysis

The activated carbon density tends decrease with increasing plasma irradiation time as in Figure 4.

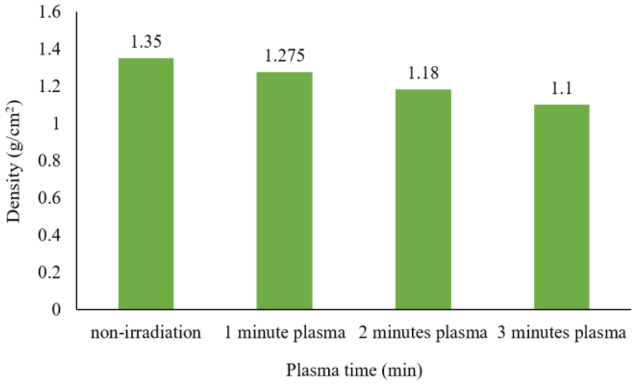


Figure 4 Activated carbon electrode density

In particular, the density of activated carbon declined by as much as 18.52% following treatment with plasma for 3 min. Plasma irradiation process causes the evolution of larger pores in activated carbon due to which, the porosity of activated carbon is increased. Another factor that should be noted, considering that plasma in the form of emissions is a gas that contains various compounds that can change the structure of activated carbon. Plasma treatment can add acidic and basic groups to the surface of activated carbon, altering its catalytic properties [41]; leads the activated carbon to be capable of being decomposed once again [25].

3.2 X-ray Diffraction Analysis

XRD analysis is used to determine changes in compounds contained in activated carbon before and after plasma irradiation. XRD analysis is used to determine changes in compounds, identify the crystal structure and the degree of crystallization contained in activated carbon before and after plasma irradiation by utilizing x-ray diffraction

The diffraction results produced by activated carbon after plasma have a denser amorphous density than before plasma. amorphous density changes indicate the development of porosity of activated carbon [43]. The findings of the current study are similar as reported by J. Sreńscek-Nazzal and K. Kiełbasa [42].

Figure 5 shows the results of XRD testing that has been analyzed using HighScore plus software to determine the miller index of the compounds in activated carbon. HighScore plus software works by analyzing the profile of X-ray diffraction (XRD) measurement results and adjusting the profile or pattern handling with crystallographic database. From which is shown figure 5 the results of the analysis of the HighScore Plus software shows phase changes. including the dominant carbon phase with index (001), potassium sulfide phase with indexes (020) and (110), indicating the presence of Sulphur compounds, and carbon sulfide phase with index (040). and after plasma irradiation shows a carbon phase with indexes (-102), (101), (020), a potassium oxide phase with indexes (110), (200) and a sodium hydroxide phase with indexes (020) and (200), indicating the presence of residual material from the plasma irradiation process using NaOH.

The stated is essentially phase conversion of the potassium from sulfide (K2S) to oxide (K2O) and reduction of the sulfide from the carbon phase (desulphurization process) [44]. These oxides change the surface area, conductivity, and chemical properties of activated carbon [45]; sulfa acid [46]; or sulfide (47) preparation affect the chemistries of activated carbon. It is interesting why they know that plasma can turn sulfides into oxides because in processing metal ores they need a reduction process from sulfide to oxide. It also details about plasma binding oxygen to other compounds such that sulfides are able to be extracted from the compound.

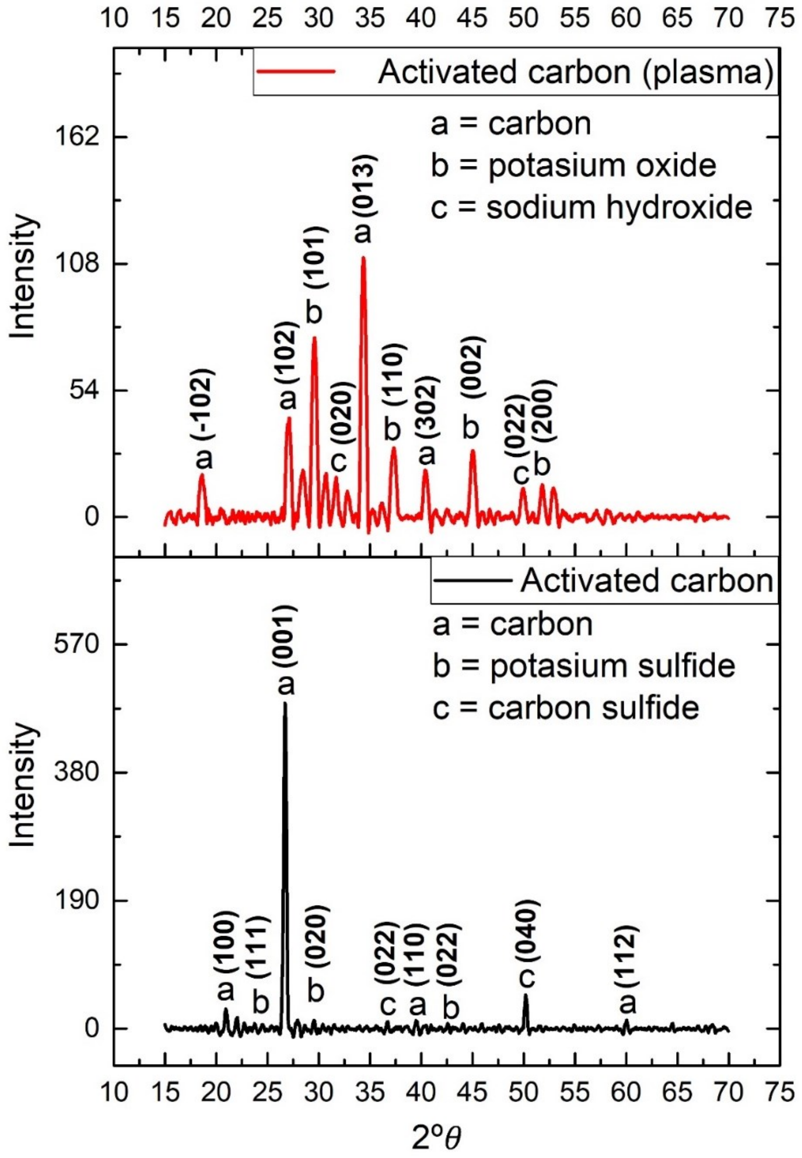


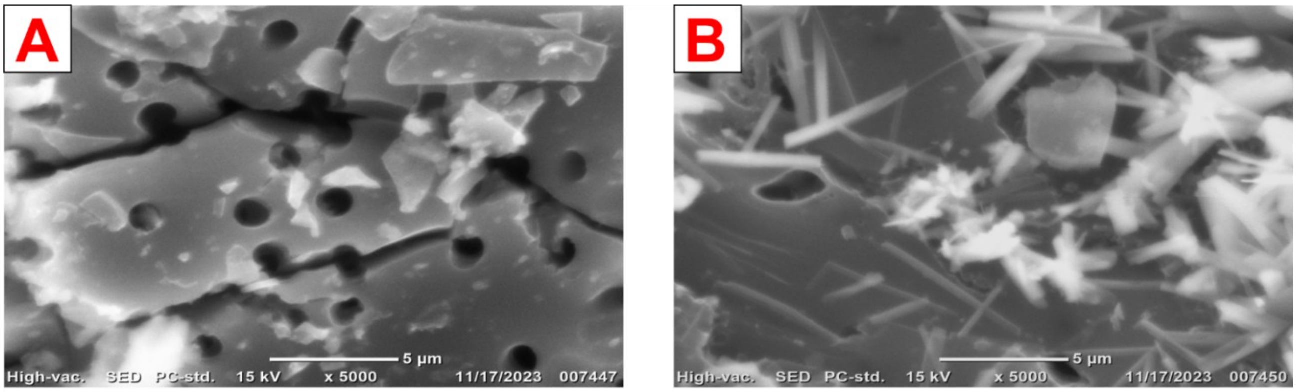
Figure 5 XRD spectrum of activated carbon with plasma (above) and without plasma treatment (below).

3.3 Scanning Electron Microscopy Analysis

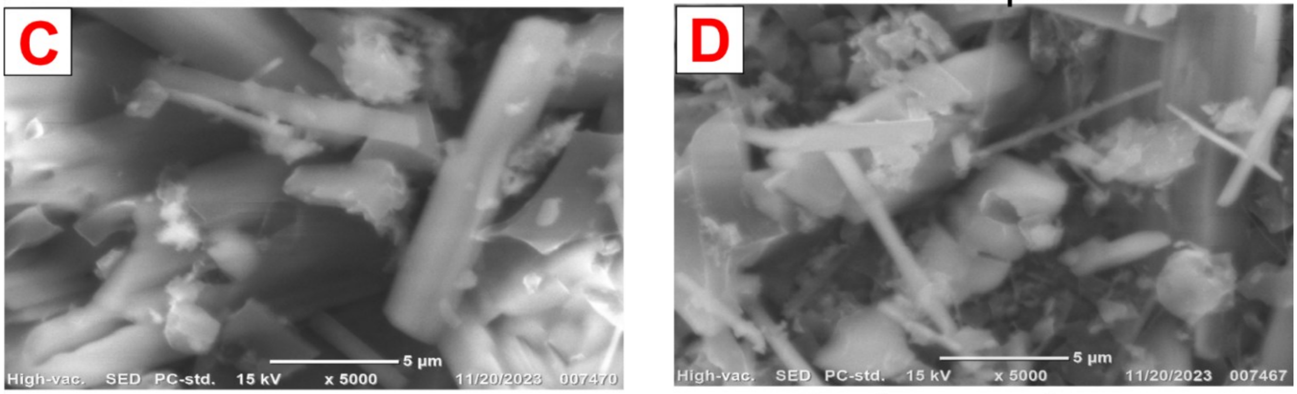
Scanning electron microscopy analysis is basically used to determine the morphology of activated carbon. SEM analysis utilizes an electron beam as a light source to shoot samples. where the wavelength is shorter than the wavelength of light, so that the electron microscope could magnify objects or with higher resolution. This can guide researchers to observe the surface of activated carbon. The plasma irradiation process is inherently unstable, leading to unpredictable irregular morphology in the resulting materials. The scanning electron microscopy (SEM) analysis reveals the surface morphology and fractures of the sample in Figure 6. The morphologies of the activated carbon electrode produced through plasma irradiation show the presence of NaOH particles that act as activators during the irradiation process. As plasma irradiation time increases, the sodium-based NaOH particles also undergo deformation. The surface and fractures of the electrode exhibit varying sizes of sodium crystals, with the smallest observed at a plasma irradiation time of 1 minute and the largest at 2 minutes. Additionally, the increased plasma irradiation time results in larger and more irregular changes in pore size on both the surface and within the fracture of the activated carbon electrode. The dimensions of these pores have a direct impact on the electrochemical characteristics of the electrode, since bigger pore sizes can lower resistance and enhance capacitance in supercapacitors. Larger pores facilitate easier movement of ions within the electrode material [47]. In supercapacitors, the rapid movement of ions in and out of the electrode surface is vital for swift charging and discharging cycles [48]. However, some pores remain invisible due to their coverage by sodium crystals. Optimal pore size is crucial for achieving minimal resistance. Currently, the mechanism behind these effects induced by microwaves remains unexplained [25]. Nonetheless, it is reasonable to hypothesize that the irradiation of microwaves onto conductive materials generates eddy currents, thereby facilitating the modification process in activated carbon [25].

Cyclic voltammetric (CV) tests were conducted to assess the electrochemical performance of the modified activated carbon. Cyclic voltammetry analyzes the results of reactions that occur in electrochemical cells. In cyclic voltammetry, changes in current and voltage potential are measured, so that reduction and oxidation information can be measured properly.

Figure 7 (a-c) illustrates the curve of activated carbon result CV measurement under different NaOH electrolyte concentrations: 1 molar (a), 2 molar (b), and 3 molars (c). The tests were carried out at a constant operating voltage of 1.2 V, with varying plasma irradiation durations. The CV curve area is commonly employed as a qualitative indicator of electrode capacitance. Figure 8 presents a comparison of CV loops from symmetric supercapacitors based on four different samples, using a scan speed of 10 mV/s. The CV curve of activated carbon without plasma irradiation exhibits minimal response, while the electrode with plasma irradiation displays larger loops. Electrodes subjected to plasma radiation exhibit higher currents compared to those without plasma radiation, resulting in increased capacitance. Among the various electrolyte molarities, 1 molar NaOH has the greatest impact. This can affect both the molarity of the electrolyte and the electrochemical processes that occur during the charging and discharging of the electrodes in the current collector utilized [54].

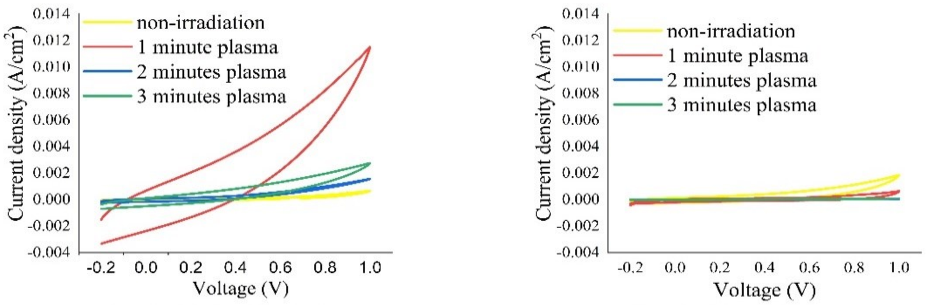


Non-irradiation 1-minute plasma



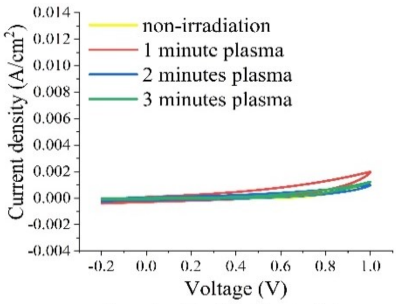
2-minutes plasma 3-minutes plasma

Figure 6 Activated carbon electrode morphology A. Non-irradiation, B. 1-minute plasma, C. 2-minutes plasma, and D. 3-minutes plasma



1 molar NaOH Electrolyte

2 molar NaOH Electrolyte



3 molar NaOH Electrolyte

Figure 7 Voltammogram of activated carbon electrodes 1, 2, and 3 molar NaOH Electrolyte

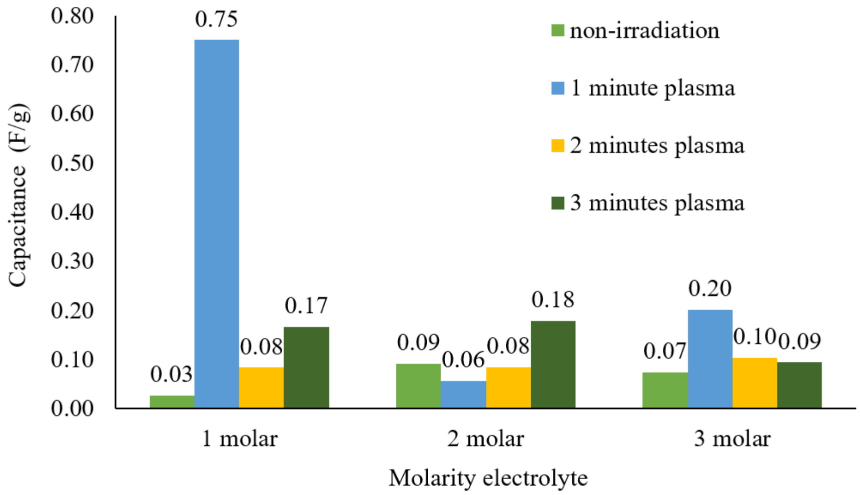


Figure 8 Activated carbon electrode specific capacitance

3.4 Voltametric Cyclic of Activated Carbon

The capacitance generated by plasma irradiation on the activated carbon electrode exhibited an increase, with the most significant improvement observed after one minute of plasma treatment as in Figure 8. However, the 2 and 3 minutes of plasma irradiation did not produce a significant increase, where the electrode without irradiation produced a capacitance of 0.03 F/g and with an irradiation time of 1 minute produced 0.75 F/g while 2 and 3 minutes produced 0.08 and 0.17 F/g respectively. This phenomenon is attributed to the structural alterations undergone by the activated carbon, which enhance the electrochemical processes at the electrode. Nevertheless, it is worth noting that the capacitance obtained in this study remains relatively minimal. This can be attributed to the irregular plasma ignition process, leading to an inconsistent electrode structure, as well as electrolyte factors that do not align with the nature and composition of the electrode. While the molarity of the electrolyte affects the capacitance produced by the electrode, high molarity electrolytes are not able to produce high capacitance because they do not match the properties of the electrode. Additionally, the current nickel collector used in this study fails to optimize the capacitance to a reasonable degree.

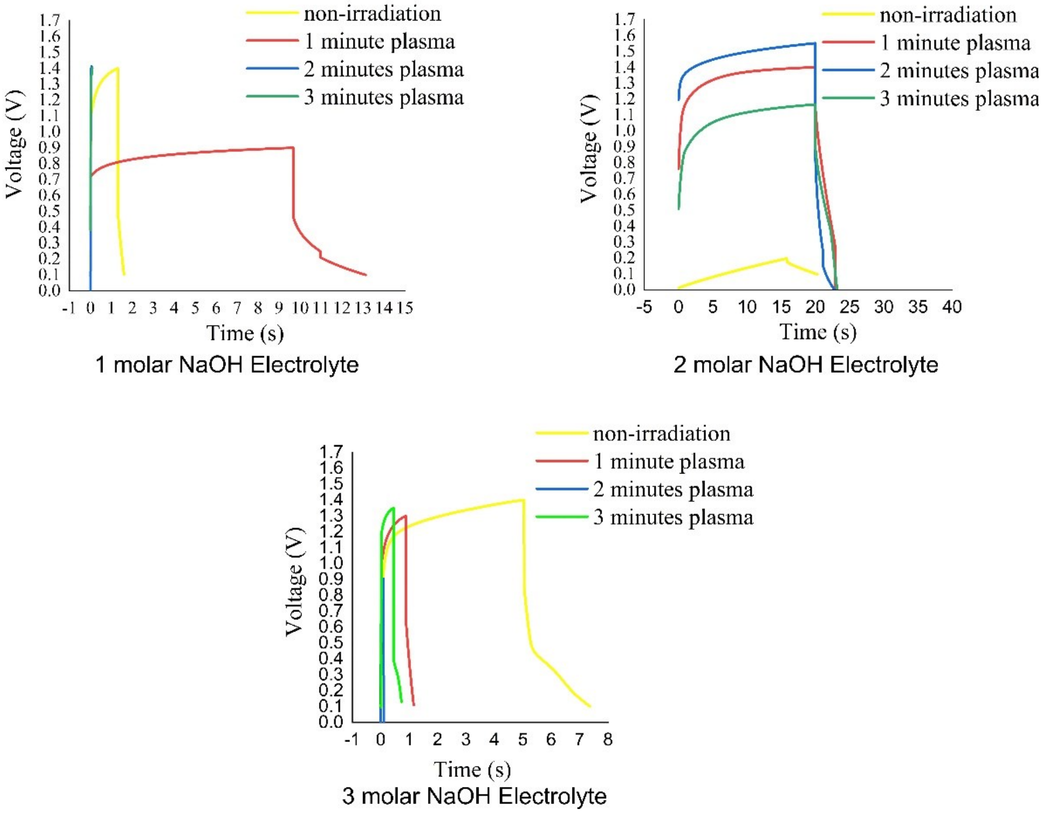


Figure 9 Galvanostatic charge/discharge activated carbon electrode 1, 2, and 3 molar NaOH Electrolyte

3.5 Galvanostatic Charge/discharge of Activated Carbon

Galvanostatic charge/discharge allows to measure the energy storage capacity, the charging and discharging efficiency, and to evaluate the response to load consistently. By monitoring the current and voltage during charge and discharge cycles, galvanostatic analysis helps to understand the actual capacity, energy storage capability, and stability during use. The GCD (Galvanostatic Charge-Discharge) measurements were conducted within a consistent voltage range, as shown in Figure 9. The charge-discharge curve exhibits a non-linear pattern characterized by a broad peak, indicating pseudocapacitive behavior. The energy storage mechanism in pseudocapacitive materials involves electrochemical reactions that are not fully reversible, in contrast to the electrostatic capacitance mechanism in conventional capacitors. During charging, ions from the electrolyte must migrate towards the electrode surface to form an electric double layer. If the electrode material is pseudocapacitive, a redox reaction or ion intercalation also occurs, which takes longer as it involves more complex kinetic processes. whereas during discharge, previously adsorbed or intercalated ions are released back into the electrolyte in a more spontaneous manner, as the stored electrostatic energy naturally tends to seek a balance. By utilizing GCD data, it is possible to calculate the energy density and power density generated by carbon electrodes in supercapacitors. On using 3 molar electrolytes, plasma-exempt electrodes show lesser charging times at identical current density. Meanwhile, when plasma irradiation is used in activated carbon, it can noticeably shorten the discharge time and increase the power density compared with that without plasma irradiation. Electrode novel features are highlighted by the exceptionally short charge and discharge times due to the uniquely the electrode's structure~~.~~

Figures 10 and 11 that the energy density and power density generated by carbon electrodes with plasma irradiation surpass those, of electrodes without plasma irradiation.

The enhancement in energy density and power density is a result of altering the structure through plasma irradiation technology, which causes changes in phases and pore size, ultimately boosting the efficiency of carbon electrodes [49].

The energy density produced by the electrode increases after plasma irradiation whereas the electrode without plasma irradiation produces an energy density of 0.34 Wh/kg and after plasma for 1 minute produces 0.93 Wh/kg with 3 molar electrolyte molarity.

The power density produced by the electrode also increases after plasma irradiation whereas the electrode without plasma irradiation produces a power density of 519 W/kg and after plasma for 2 minutes produces 17,166 W/kg with 3 molar electrolyte molarity.

Energy density in supercapacitors is influenced by the capacitance of the electrode and the voltage achieved by the electrode during charging. The activated carbon electrode achieved both after and before the plasma is different for each electrode, it shows the quality of the resistance of different electrodes. The resulting capacitance is also different so that the resulting energy density is low because the resulting capacitance is also low. but the electrode with the highest capacitance uses a 1 molar electrolyte but produces low energy density because the electrode's ability is not able to produce the working voltage produced by the electrode with a 3 molar electrolyte. while power density is influenced by energy density and the time it takes for the supercapacitor to release the stored energy. The advantages of activated carbon electrodes specialized in after plasma is that the pore size produced is larger so that the resulting resistance is small so that ions can move faster to produce a short discharge time so that the resulting power density is larger.

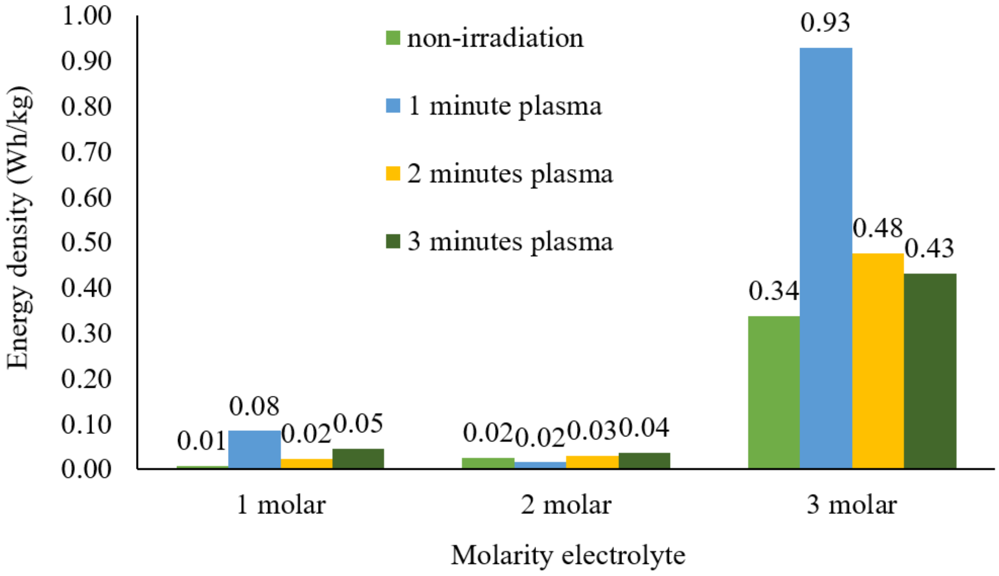


Figure 10 Energy density of activated carbon electrodes

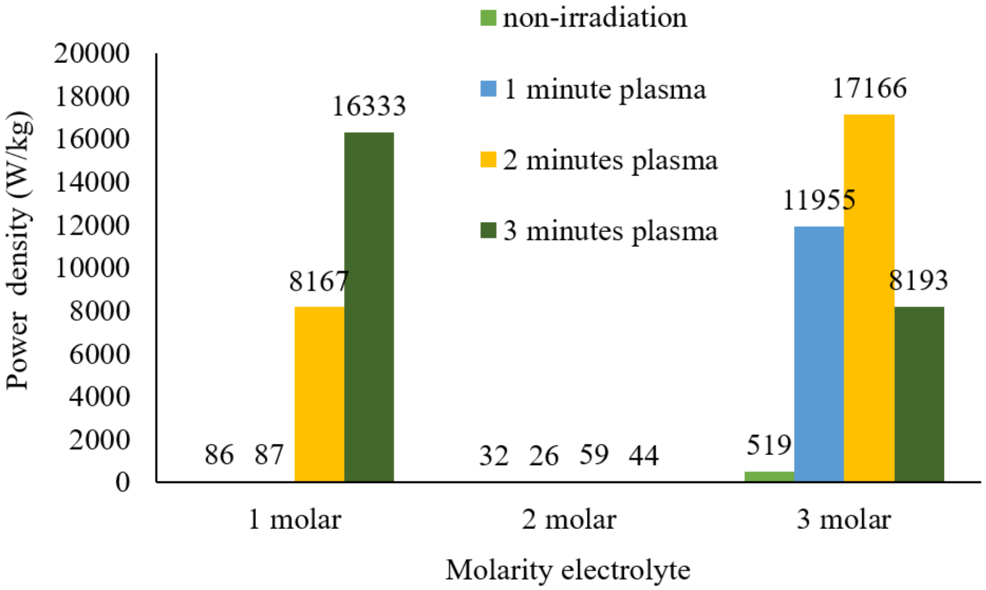


Figure 11 Power density of activated carbon electrodes

Testing was carried out using the GCD method for 500 cycles with the same working current and voltage to evaluate the performance of the supercapacitor. Figure 12 shows the characteristics of electrodes that have the potential to be used as supercapacitor electrodes. For long-term behavior evaluation, carbon electrodes were cycled within a potential range of 01 – 14 V for 500 cycles. In the first cycle, a capacitance of 027 F/g−1 was observed and maintained for the initial 200 cycles; subsequently, it decreased to 0267 F/g−1 after 500 cycles Based on the charge-discharge curve in Figure 12, the charge and discharge behavior is nearly linear.

Figure 13 depicts the efficiency change of the active carbon electrode over 500 cycles According to these results, the capacitance decreased by 6% from the first to the last cycle, yet the supercapacitor still maintains a high coulombic efficiency greater than 90 % indicating exceptional cycle stability. Meanwhile, cobalt materials [51] [52] and 2D MXenes materials which have greater capacitance and higher cycle life [53].

The energy density and power density are plotted on a Ragone graph in Figure 14. In analyzing all the data presented, it was agreed that the samples irradiated with plasma for 2 minutes had the highest energy density. The results indicate that the behavior of the electrodes is more like a supercapacitor where the electrodes tend to have a higher power density. The findings of this study demonstrate the viability of MW plasma as a potential alternative due to its practicality in the development of electrode materials and energy storage.

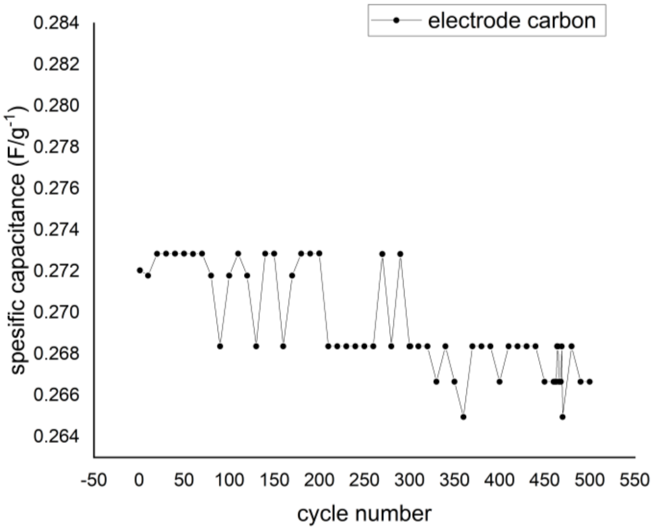


Figure 12 specific Capacitance vs cycle number

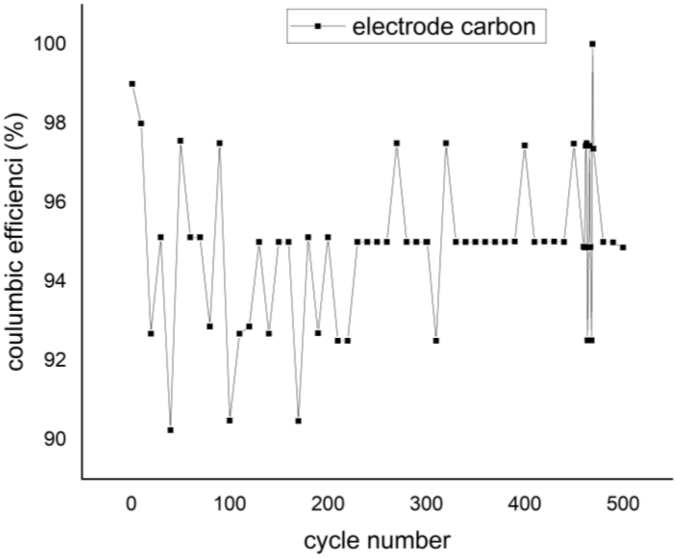


Figure 13 Coulombic efficiency vs cycle number

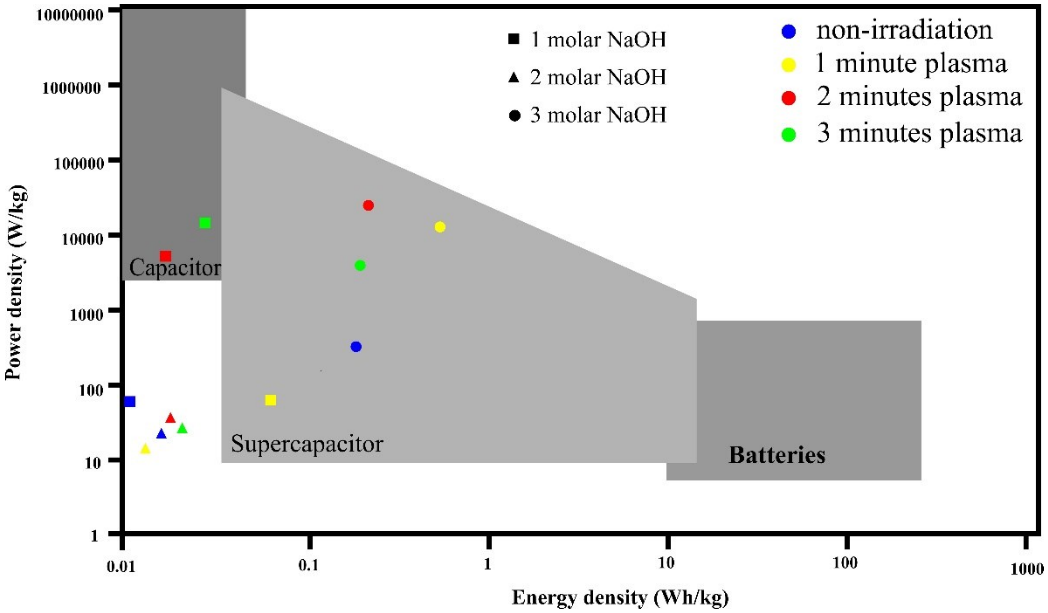


Figure 14 Plot diagram Ragone of a supercapacitor cell

4.0 CONCLUSION

This study aims to analyze the potential of microwave plasma to change the structure of activated carbon. In addition, the results of the study showed a decrease in the density of activated carbon by 18.52%. The highest capacitance and energy density occurred at 1 minute of plasma irradiation of 0.75 F/g with a molarity of 1 molar and 0.93 Wh/kg with 3 molar electrolyte, respectively. Meanwhile, the highest power density was 17,166 W/kg at 2 minutes of plasma irradiation with 3 molar electrolyte. Therefore, plasma irradiation can change the structure of activated carbon electrodes by utilizing the resulting compounds, pressure, and temperature, which can be applied to modify the electrochemical properties of electrodes in a short and simple.

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Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

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