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Optimization of phosphoric acid activation on palm shell activated carbon for bio-battery

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Abstract. The worsening global energy crisis and the environmental impact of conventional battery waste have prompted us to find ways to store energy that are more sustainable and less harmful to the environment, such as using bio-batteries. The purpose of this research is to examine how different concentrations of phosphoric acid (H_3PO_4) affect the properties of activated carbon made from natural palm kernel shells, and how this affects its performance when used in bio-battery systems. The palm kernel shells were first heated at 550 °C, then activated using H_3PO_4 liquids with different concentrations, namely 1 M, 1.5 M, 2 M, 2.5 M, and 3 M, with a weight and volume ratio of 1:2. The resulting activated carbon was then used as a solid electrolyte in a bio-battery, with aluminum as the anode and copper as the cathode. The performance of the bio-battery was assessed under a constant load using a 2 (two) volt LED, by measuring parameters such as voltage, current, power output, and battery capacity. The results showed that higher H_3PO_4 concentrations resulted in higher iodine adsorption capacity, larger surface pore size according to SEM analysis, and better electrochemical performance of the bio-battery. The best conditions were obtained when the H_3PO_4 concentration was 3 M, which resulted in an iodine absorption capacity of 888.3 mg/g, a maximum voltage of 3.66 V, and an output power of 9.835 mW. Based on these results, it can be concluded that the use of H_3PO_4 at a concentration of 3 M can produce activated carbon with the most optimal physicochemical properties for bio-battery applications, thereby potentially supporting the development of more environmentally friendly renewable energy technologies.

Keywords : Activated carbon, bio-battery, palm shell, phosphoric acid, renewable energy

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Introduction

Bio-batteries are increasingly being researched as a more sustainable energy storage alternative to conventional batteries, mainly due to their more environmentally friendly nature and use of naturally biodegradable materials. Conventional batteries generally contain heavy metals such as cadmium, lead, and mercury, which can have serious impacts on the environment and health if not properly managed after their useful life ends [1]. In contrast, bio-battery systems offer a safer and cleaner method of energy storage, in line with global efforts in the development of environmentally friendly technologies and renewable energy [2][3].

In recent years, biomass-based activated carbon has been reported to have great potential for use as electrode and electrolyte materials in energy storage devices. This is due to its characteristics of high porosity, large surface area, and fairly good electrochemical performance [4]. One interesting source of biomass for development is palm kernel shell waste, which is produced in large quantities by the palm oil industry in Indonesia and is rich in lignocellulose content. The use of palm kernel shells as raw material for activated carbon not only produces value-added materials, but also contributes to waste reduction and improved environmental sustainability [5].

In its application in bio batteries, activated carbon is often chosen as an electrode material due to its good chemical stability, low manufacturing cost, and compatibility with water-based and bio-based electrolytes. The ability of activated carbon to store and transfer charges is greatly influenced by its physicochemical properties. Its high surface area provides many active sites for ion adsorption and facilitates the formation of an electric double layer, while the right balance between micro pores (<2 nm) and meso pores (2–50 nm) allows for efficient ion storage and easy ion diffusion within the electrode structure. In addition, the good electrical conductivity of the carbon framework is crucial for supporting electron transport and reducing internal resistance during operation [6]. This combination of characteristics allows activated carbon to function optimally as an important energy storage element in bio-battery systems.

However, the characteristics of activated carbon are not inherent properties, but are

greatly influenced by the activation strategy and synthesis conditions applied during its manufacture. Chemical activation plays an important role, as it controls pore formation and surface functionalization, which impact electrochemical behavior. Among various chemical activation agents, phosphoric acid (H_3PO_4) has been recognized as a highly effective activator because it can increase pore formation and adsorption capacity more efficiently than physical activation methods. Variations in H_3PO_4 concentration can significantly alter the texture and electrochemical performance of the resulting carbon, both of which are critical for ion transport and charge storage in bio-battery systems [7]. In addition to acting as a physical pore former, phosphoric acid (H_3PO_4) also plays a role in chemical reactions with lignocellulose components in biomass. During the activation stage, H_3PO_4 contributes to the breaking of ester and glycoside bonds found in cellulose and lignin, resulting in a more open and accessible carbon structure. In addition, this process also introduces phosphorus-containing functional groups on the carbon surface, which can enhance the interaction between the electrode and ions in the electrolyte [8].

Another advantage of using H_3PO_4 is its ability to produce efficient chemical activation at relatively low temperatures, ranging from 400–600 °C. Under these conditions, the synthesis process can take place with more controlled energy use, maintaining the integrity of the carbon structure and offering more optimal carbon results compared to traditional alkali activation methods, which usually require high temperatures [9].

In line with these characteristics, a number of previous studies have shown that biomass-based activated carbon has significant potential for applications in electrochemistry, such as in supercapacitors and bio-batteries [10] [11]. However, research specifically investigating the impact of H_3PO_4 concentration variations on the effectiveness of activated carbon from palm kernel shells in bio-battery systems is still quite limited [12]. Therefore, this study aims to explore the effect of variations in phosphoric acid concentration on the physicochemical properties and electrochemical performance of activated carbon produced from palm kernel shells [13]. The uniqueness of this study lies in its systematic approach to evaluating H_3PO_4 activation concentrations and their relationship to bio-battery effectiveness, which is expected to contribute significantly to the development of more efficient and

eco-friendly energy storage technologies.

Experimental

Materials. For this research, palm kernel shells became what we used as the key biomass source, acting like raw stuff used when making activated carbon [14][15]. The palm shells were obtained from a local palm oil mill located in Perajen, Banyuasin Regency, South Sumatra, Indonesia. Phosphoric acid (H_3PO_4 , ≥ 85 wt%, analytical grade) was purchased from Merck (Germany) and used as the chemical activating agent. The H_3PO_4 solution was diluted with deionized water to the desired concentrations, which were systematically varied as an experimental parameter. All other chemicals were of analytical grade and were used as received without further purification. Distilled water (aquadest) was used throughout the washing and sample preparation processes.

For the assembling the bio-battery cell, polyvinyl chloride (PVC) pipes with a diameter of 1 inch and a length of 10 cm, along with matching end caps, were employed as the battery casing. Aluminum (Al) and copper (Cu) rods, each with a diameter of 10 mm and a length of 4.8 cm, were used as the anode and cathode [16][17], respectively. A commercial vislin fabric was applied as the separator to prevent direct contact between electrodes [18].

For the characterization of activated carbon quality, several analytical-grade chemicals were used, including iodine (I_2) 0.1 N, potassium iodide (KI), sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) 0.1 N, sodium bicarbonate (NaHCO_3), potassium iodate (KIO_3) 0.1 N, concentrated hydrochloric acid (HCl) 2 N, and starch (amylose) as an indicator for titration [19].

Preparation of Activated Carbon. Raw palm shells were thoroughly washed with water to remove adhering dirt and impurities, followed by sun-drying for 1–3 days [20]. The dried shells were carbonized in a muffle furnace at 550°C for 2 h to obtain palm shell charcoal. The carbon produced was first ground manually and then separated using a mechanical sieve shaker to obtain particles in the range of 60–80 mesh. This size fraction was selected to achieve a more consistent material prior to the activation stage.

For chemical activation, the sieved carbon was immersed in phosphoric acid (H_3PO_4)

solutions with concentrations of 1 M, 1.5 M, 2 M, 2.5 M, and 3 M. The impregnation process used a solid-to-liquid ratio of 1:2 (w/v). After mixing, the suspensions were stirred briefly and then left to stand at room temperature for 24 hours to allow sufficient contact between the acid and the carbon structure.

Once the activation process was completed, the samples were filtered and rinsed repeatedly with distilled water until the washing solution reached neutral pH. The activated carbon was subsequently dried in an oven at 105°C for about 1–2 hours. After drying, the samples were cooled in a desiccator and stored in airtight containers before being used for characterization and bio-battery preparation.

Proximate Analysis and Iodine Adsorption.

The proximate analysis of the activated carbon was conducted based on the ASTM D7582-10 standard to determine its moisture content, volatile matter, ash content, and fixed carbon. These parameters were used to assess the material's thermal stability, inorganic residue, and overall carbon quality.

The iodine adsorption capacity was determined following ASTM D4607. Approximately 1 g of activated carbon (60-mesh) was equilibrated with a standardized 0.1 N iodine solution, after which the residual iodine was titrated with 0.1 N sodium thiosulfate using starch as an indicator. A blank titration was conducted for correction. The iodine number (mg g^{-1}) was calculated using Equation (1).

$$\frac{((V_{\text{iod}} \times N_{\text{iod}}) - (V_{\text{thio}} \times N_{\text{thio}})) \times 126,9 \times f_p}{W} \quad (1)$$

The combined proximate and iodine adsorption analyses provided a comprehensive assessment of the structural and adsorption characteristics of the activated carbon.

Surface Morphology Analysis. The surface morphology of the samples was examined using a Scanning Electron Microscope (SEM, TESCAN Vega3). Image acquisition was performed at an accelerating voltage of 15.0 kV. Secondary electron (SE) mode was employed to enhance surface topography contrast. The micrographs were recorded at a magnification of 5.00 kX (5,000 \times), with a corresponding scale bar of 10 μm . This analysis aimed to observe the formation and distribution of pores resulting from different H_3PO_4 concentrations and to establish a correlation between the morphological characteristics and the electrochemical performance of

the material.

Biobattery Assembly and Electrochemical Testing. As illustrated in **Figure 1**, the bio-battery cells were assembled using PVC pipes (1 inch diameter × 10 cm length) as the casing. Aluminum rods (4.8 cm × 10 mm) and copper rods (4.8 cm × 10 mm) served as the anode and cathode, respectively, while vislin cloth functioned as the separator.

The electrolyte paste was prepared by mixing 50 g of palm shell-derived activated carbon with 25 mL of 1 M H₃PO₄. The paste was packed into the PVC casing, sealed with end caps, and the electrodes were fixed and connected to an external circuit for electrochemical testing.

As shown in **Figure 2**, multiple bio-battery cells were connected in series to increase the output voltage for electrochemical performance evaluation. For electrochemical performance evaluation, multiple cells were connected in series to increase voltage output. The open-circuit voltage (OCV) of each configuration was measured using a digital multimeter. Discharge performance was tested by connecting the bio-battery to a 2 V light-emitting diode (LED) as the load. Voltage and current readings were recorded every 20 minutes over a 60-minute discharge period. The electrical power and capacity were calculated from the measured data, with power output determined based on the product of voltage and current values obtained during testing.

$$P = V \times I \quad (2)$$

where P represents the power output (mW), V denotes the measured voltage (V), and I indicates the current (mA). The battery capacity was calcu-

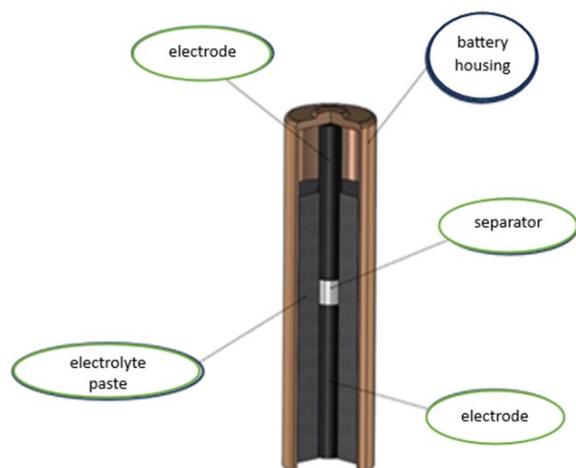


Figure 1. Schematic Illustration of The Main Components of Bio-battery

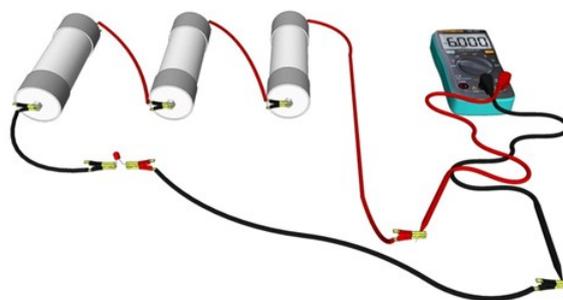


Figure 2. Schematic illustration of The Series configuration of bio-battery cells

lated based on the total discharge duration and the corresponding current flow, with the result expressed in milliampere-hours (mAh).

Data analysis. All experimental data were reported as the mean values obtained from three replicates. The performance evaluation was carried out by examining the influence of H₃PO₄ concentration on key parameters, including iodine adsorption capacity, surface morphology observed through SEM, voltage, current, power output, and battery capacity. The obtained results were subsequently compared with the Indonesian National Standard (SNI) specifications for activated carbon and relevant findings from previous research studies.

Result and Discussion

Proximate analysis. Proximate analysis was conducted to evaluate the moisture content, volatile matter, ash content, and fixed carbon of the activated carbon derived from palm shells. The procedure was carried out in accordance with the ASTM D7582-10 standard [21], and the obtained results are presented in **Table 1**.

Based on the results of proximate analysis, the coconut shell charcoal obtained in this study

Table 1. Results of proximate analysis for activated carbon derived from palm shell biomass

Parameter	Analysis Result (%)	SNI Standard
Moisture Content	6,33	Max 8%
Ash Content	8,93	Max 5%
Volatile Matter	34,07	Max 30%
Fixed Carbon	50,67	Min 65%

does not fully meet the requirements of SNI 01-6235-2000. Of the several parameters tested, the moisture content is still within the permissible range, while the ash and volatile matter content show relatively high values. This condition has a direct impact on the low fixed carbon content produced. The moisture content of 6.33% is still in accordance with the standard, indicating that the drying process has been carried out properly and the charcoal has adequate stability during storage. However, the ash content was recorded at 8.93%, exceeding the maximum limit of 5%, while the volatile matter content reached 34.07%, slightly above the standard threshold of 30%. As a result, the fixed carbon content obtained was only 50.67%, meaning that the charcoal at this early stage did not yet show optimal quality.

This deviation from the standard indicates that the initial carbonization process at a temperature of 550 °C with a heating time of 2 hours is still insufficient to remove all unwanted components. The high ash content indicates the presence of inorganic minerals, such as silica, calcium, and magnesium, which are non-conductive. The presence of these minerals has the potential to clog micro-pores, reduce the effective surface area, and ultimately reduce the adsorption capacity and electrochemical performance of charcoal. Ahiduzzaman [22] reported that excessive ash content can reduce the functional properties of charcoal, requiring further treatment, such as chemical leaching or activation processes, to improve its quality.

In addition to ash content, a sufficiently high volatile matter value also indicates that the pyrolysis process has not been maximized. The presence of organic compounds that have not been completely decomposed is usually related to a relatively low carbonization temperature or insufficient residence time. These results are in line with the findings of Harmiansyah [23], who reported a volatile matter content of 34.09% in palm kernel shell charcoal carbonized at a temperature range of 400–500 °C. These findings reinforce the indication that optimization of carbonization conditions and further activation are still needed to improve the quality of the charcoal produced.

The fixed carbon content obtained, 50.67%, was also below the minimum requirement of the SNI 01-6235-2000 standard, which

specifies a minimum of 65%. The relatively low fixed carbon content further confirms that the aromatic carbon framework was not yet fully developed during carbonization. Since fixed carbon is directly related to thermal stability, electrical conductivity, and electrochemical durability, this condition renders the raw charcoal unsuitable for direct application as an electrochemical material, particularly in energy storage systems such as bio-batteries and supercapacitors.

However, the limitations found in the early stages actually demonstrate the importance of further chemical activation. Activation using phosphoric acid (H_3PO_4) is expected to improve the characteristics of pre-processed charcoal through several mechanisms, such as promoting dehydration reactions, removing residual volatile compounds, dissolving inorganic impurities, and helping to form a more developed pore structure. This activation process involves breaking lignocellulose bonds and forming carbon structures associated with phosphate groups, thereby potentially increasing bound carbon content, expanding surface area, and improving material pore openness [24].

Consequently, although palm kernel shell charcoal prior to activation does not fully meet SNI criteria, this material can still be utilized as a suitable starting material for further activation processes. Through appropriate treatment, the physico-chemical properties of this charcoal can still be significantly improved to meet the requirements of electrochemical energy storage applications.

Surface Morphology (SEM analysis). The surface morphology of the activated carbon was analyzed using Scanning Electron Microscopy (SEM) to observe pore formation and surface texture variations resulting from different phosphoric acid activation concentrations (1 M, 1.5 M, 2 M, 2.5 M, and 3 M). Representative SEM micrographs are presented in **Figure 3**.

Based on SEM observations, there are significant differences in the surface appearance of activated coconut shell charcoal activated using 1 M and 3 M H_3PO_4 solutions. Samples activated with 3 M H_3PO_4 show a rougher and more uneven surface, with more open and interconnected pores. Meanwhile, the surface structure of the sample activated with 1 M H_3PO_4 tended to be denser and the pores formed were still limited. This difference indicates that the use of higher acid concentrations has a greater effect in opening the carbon structure and expanding the usable surface area, especially in the adsorp-

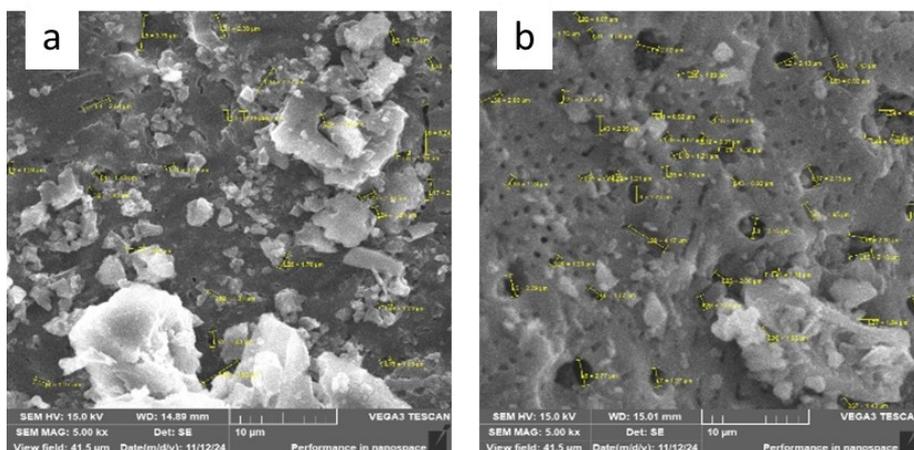


Figure 3. Results of SEM analysis of activated charcoal (a) 1 M concentration and (b) 3 M concentration.

tion process [25]. The presence of open and interconnected pores also facilitates contact between the carbon surface and adsorbate molecules or electrolyte ions.

The improved pore development at a concentration of 3 M H_3PO_4 is thought to be related to the ability of phosphoric acid to trigger dehydration reactions and break structural bonds in biomass components, such as cellulose, hemicellulose, and lignin [26]. At lower concentrations, the interaction between phosphoric acid and the biomass matrix is not yet optimal, so that the pore formation process does not occur maximally and the carbon structure is still relatively closed. These results are in line with the findings of Eso et al. [27], who reported that an increase in H_3PO_4 concentration tends to produce a more developed pore structure due to a more effective removal of residual compounds and contaminants. Considering the observations in this study, the use of 3 M H_3PO_4 can be said to be the most supportive condition for pore development in palm kernel shell activated carbon.

Iodine Adsorption Capacity. The iodine adsorption test was carried out according to ASTM D4607-14, and the results are presented in **Table 2**. The iodine number serves as an indicator of the micropore content and adsorption capacity of the activated carbon.

Based on SNI 06-3730-1995, the minimum iodine adsorption value for activated charcoal is 750 mg/g. In this study, activated charcoal from palm shells activated with an acid solution at a concentration of 1 M had an iodine adsorption capacity of 736.02 mg/g, which is slightly below the standard. This value indicates that the

Table 2. Biobattery Capacity

Activator Concentration (M)	Capacity Bio-batteries (mAh)
1	1,17
1,5	2,37
2	2,59
2,5	2,64
3	2,81

concentration of the activator was not yet able to optimally open the micropores in the charcoal structure.

As shown in **Figure 4**, increasing the activator concentration to 1.5 M and 2 M improved the iodine adsorption capacity to 774.09 mg/g and 786.78 mg/g, respectively, both of which met the SNI 06-3730-1995 standard. This increase indicates that higher activator concentrations can dissolve more volatile compounds and minerals, expand the surface area and create new pores. Phosphoric acid activation has been shown to produce highly porous carbon with acidic surface groups by facilitating dehydration and cleavage of biomass components, leading to improved adsorption capacities. This is reinforced by the results of research reported that chemical activation of mangrove charcoal at elevated temperatures produced iodine adsorption values up to 1,019 mg/g, indicating significant improvement in porosity and micropore quality [28]. Moreover, the iodine adsorption capacity a key indicator of micropore development reflects the accessible micropore volume and is commonly used to evaluate carbon quality. Other research achieved even higher iodine numbers, ranging from 1,172 to 1,238 mg/g, through combined chemical and physical acti-

vation methods, highlighting the strong influence of activation conditions on adsorption capacity [29]. Increased H_3PO_4 concentration and optimized activation conditions have been reported to enhance pore development and adsorption performance in biomass-derived activated carbons, consistent with the trend observed in this study.

Open Circuit Voltage (OCV). The open circuit voltage (OCV) represents the initial electrochemical potential difference of the bio-battery prior to applying an external load. Measurements indicated that increasing the concentration of H_3PO_4 activator led to a consistent rise in OCV values (Figure 5).

At 1 M, the OCV averaged 2.47 V, which progressively increased to 3.50 V at 3 M. These findings demonstrate the critical role of H_3PO_4 in

enhancing pore development and reducing internal resistance, thereby facilitating better ionic conductivity.

The improvement in OCV can be attributed to the expanded micro- and mesopore structures that develop during activation, enabling more efficient proton (H^+) transport across the electrode-electrolyte interface. A study emphasized that high specific surface area carbons enhance electrode/electrolyte interactions[30], while the other confirmed that high porosity provides efficient ionic pathways, both of which directly improve voltage generation in biomass-based devices[31].

Discharge Performance. Discharge testing was performed by connecting the bio-battery cells in series to power a 2 V LED for 60 minutes. Voltage and current were recorded at 20-minute intervals. Results showed that bio-batteries with higher H_3PO_4

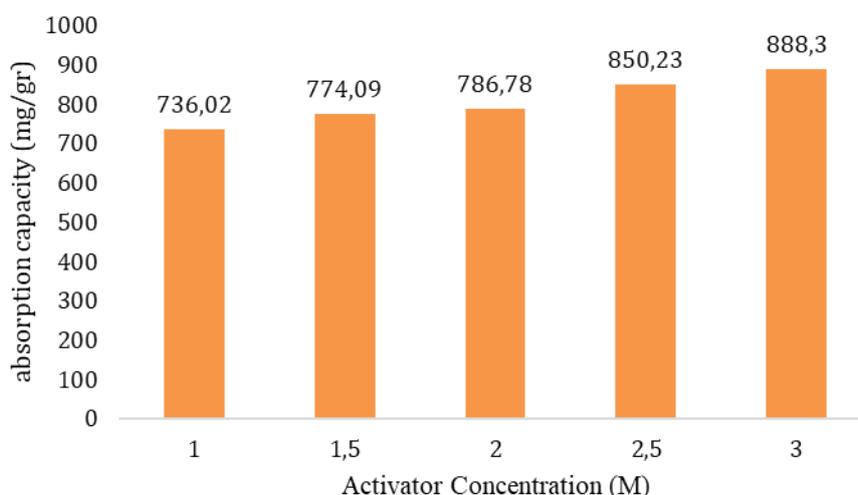


Figure 4. Effect of activator concentration on the absorption capacity of activated charcoal

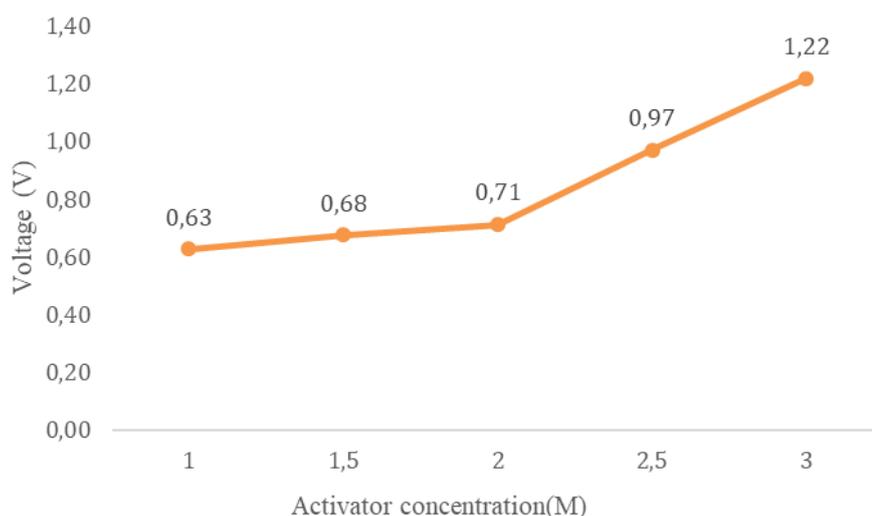


Figure 5. The Effect of Activator Concentration on Biobattery Voltage

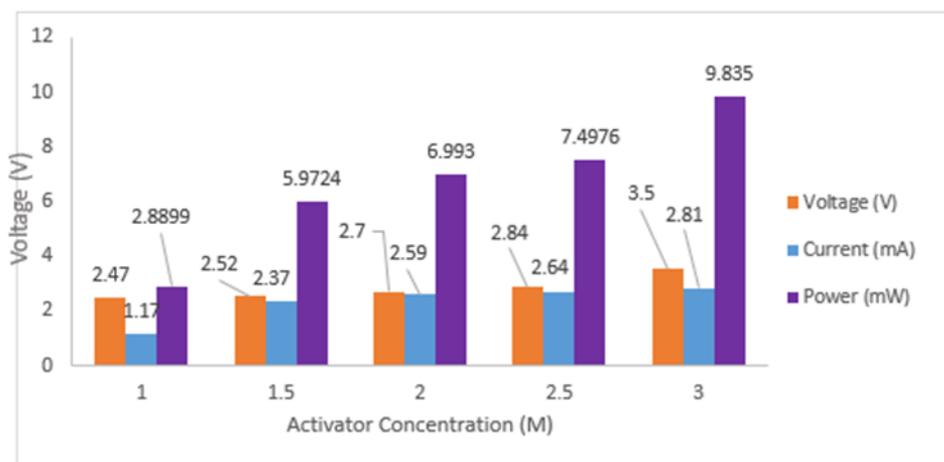


Figure 6. Voltage and Power current discharge curves of bio-batteries at different H_3PO_4 concentrations.

concentrations sustained higher discharge voltages and currents throughout the test (**Figure 6**).

At 1 M, the bio-battery delivered a current of 1.17 mA, but at 3 M this increased to 2.81 mA, accompanied by a stable operating voltage near 3 V under load. This indicates reduced polarization losses and improved electrolyte conductivity. Next research observed similar trends in bio-batteries from pineapple waste, where long-term discharge stability was linked to electrolyte stability and ion transport efficiency[32].

Chemical activation with H_3PO_4 removes volatile compounds, enlarges surface area, and promotes micropore development, all of which increase ionic adsorption and redox activity. These structural enhancements enable the bio-battery to maintain consistent current delivery under load.

The power output and storage capacity of the bio-batteries were also strongly influenced by H_3PO_4 concentration. Power rose from 2.88 mW at 1 M to 9.83 mW at 3 M, while capacity increased from 1.17 mAh to 2.81 mAh.

Capacity reflects the effective charge the bio-battery can store and deliver. The increase with higher H_3PO_4 concentrations is consistent with the development of interconnected pores that enhance ionic transport and reduce internal resistance. Last experiment reported that phosphoric acid activation of cocoa shells yielded carbons with surface areas exceeding $1200 \text{ m}^2/\text{g}$, significantly boosting capacitance and energy storage potential [33].

Conclusion

The results of this study show that the performance of palm kernel shell-based biobatteries is greatly influenced by the concentration of phosphoric acid (H_3PO_4) used in the activation stage. When the activator concentration was increased from 1 M to 3 M, there was a significant improvement in electrochemical performance. The open circuit voltage increased from 2.47 V to 3.50 V, while the discharge current rose from 1.17 mA to 2.81 mA and showed better stability when the battery was operating under load. This increase was also followed by an increase in power output from 2.88 mW to 9.83 mW and battery capacity from 1.17 mAh to 2.81 mAh.

This improvement in performance indicates that the activation process using phosphoric acid is capable of forming a carbon structure with more developed porosity and a more accessible surface area. These conditions facilitate the movement of ions within the material and increase electrochemical activity during the discharge and charging processes. This finding is in line with previous reports stating that proper activation conditions play an important role in improving the electrochemical performance of biomass-based carbon.

In general, activated carbon derived from palm kernel shell waste and activated using H_3PO_4 shows promising potential as a sustainable and efficient bio-battery electrolyte material. To support further application in renewable energy storage systems, further research is needed that focuses on optimizing activation conditions, evaluating long-term stability, and studying the possibility of larger-scale production.

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Author Contributions

Lety Trisnaliani and Ida Febriana designed the study, supervised the research, and secured the funding. Safril Kartika Wardana, Adha Triwibowo, and Arief Fauzan performed the experiments and conducted the data acquisition. Berlian Tiara and Tri Larasati analyzed the results and drafted the manuscript.

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