

Article

Waste-to-Energy Approach: Snail Shell-Derived Electro/Nanocatalysts for Direct Methanol Fuel Cells

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Abstract

In recent years, in the presence of environmental pollution, green materials have emerged as a research hotspot. The value-engineered design of electron nanocatalysts using low-cost biomaterials has demonstrated special electrocatalytic efficiency and performance in methanol oxidation reactions within direct methanol fuel cells (DMFCs). These electro/nanocatalysts were successfully heat-treated in a study involving multiple temperature controls and activation. The research involved studying samples C-400, C-600, and C-800, which were carbonized at temperatures of 400, 600, and 800 °C from snail shells. Next, these samples were activated using potassium hydroxide, resulting in samples AC-400, AC-600, and AC-800. Moreover, the ability of the resulting electron nanocatalysts, as heat-treated catalysts, to enhance the electrocatalytic efficiency of methanol oxidation reactions, along with the significant effects of temperature variations before and after activation, was investigated. The surface area of the sample increased successfully from 4.6691 m²/g to 14.1763 m²/g after activation, while the pore volume increased from 0.02225 m³/g to 0.07233 m³/g. The results clearly show that methanol oxidation reactions were more efficient and active on the surface of the electron nanocatalyst at 800 °C (AC-800). The current density successfully increased from 22.76 mA/cm² to 49.89 mA/cm² after the addition of methanol to C-800, whereas it increased significantly from 25.7 mA/cm² to 65.24 mA/cm² in AC-800 (after activation). Furthermore, the C-400, C-600, C-800, AC-400, AC-600, and AC-800 electro/nanocatalysts exhibited novel power densities of 12.6, 18.2, 29.7, 16.6, 22.86, and 39.56 mW/cm², respectively. This was further confirmed by their morphological, structural, and electrochemical characteristics. The synthesized materials are proven to be sustainable carbon materials for electron nanocatalyst support and methanol electro-oxidation in DMFC systems because of their low cost, eco-friendliness, high performance, and enhanced porous structure. Moreover, this research provides an effective waste-to-energy approach for converting snail shell biomass into valuable functional carbon materials for renewable energy applications.



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Keywords: electro-oxidation; activated carbon; electron nanocatalyst; green material; direct methanol fuel cell; snail shells

1. Introduction

Fuel cells are clean and sustainable energy technologies that offer effective alternatives to traditional fossil fuels. Direct methanol fuel cells (DMFCs) have emerged as a focus of scientific research because of their numerous advantages. These advantages include high energy density, normal operating temperature, efficient fuel storage, and ease of transport and delivery [1]. Moreover, DMFCs convert chemical energy into electrical energy through electrochemical reactions. Therefore, they are strong competitors in the portable electronic and renewable energy application markets [2,3]. On the other hand, DMFCs have limited methanol oxidation kinetics, are toxic to the catalyst, and have high catalyst material costs. Therefore, developing efficient, low-cost, and sustainable materials to support electron nanocatalysts has become important [4,5].

Carbon materials are effective in electrochemical systems because of their high electrical conductivity, large specific surface area, chemical stability, and high porosity [6]. Furthermore, carbon and activated carbon can be excellent electrocatalysts, dispersing the catalyst and enhancing the electrochemical oxidation performance of methanol in DMFCs [7]. The physical and chemical properties can be controlled by selecting the raw material source, temperature, and activation agent [8]. KOH is highly efficient at producing high porosity, a wide surface area, and better electrochemical performance within carbon materials [9,10]. Biomass-derived carbon materials are eco-friendly and sustainable alternatives to traditional carbon materials. Agricultural and biological wastes are suitable raw materials because of their low cost, renewable nature, and availability, as well as the environmental benefits they confer through reuse [11,12]. Snail shell waste is important for biomass production because of its unique composition, which is primarily composed of calcium carbonate and organic compounds. The conversion of snail shells into porous carbon materials activated by electro/nanocatalysis makes them suitable for use as DMFCs [13,14].

Temperature is a key factor in determining the structural and morphological properties of carbon materials. A suitable temperature enhances the carbonization process, improving pore formation and increasing surface area. Furthermore, activation with KOH leads to significant increases in porosity and surface area. In this study, snail shell powder was carbonized at different temperatures (400, 600, and 800 °C) to produce samples C-400, C-600, and C-800. The resulting materials were then activated using KOH to prepare activated carbon samples AC-400-, AC-600-, and AC-800. Thus, electro/nanocatalyst samples for DMFCs were prepared from snail shell waste via carbonization and activation processes, and the effects of temperature on the structural and electrochemical performance of the samples were studied with the aim of developing low-cost, eco-friendly carbon materials for renewable energy applications.

2. Results and Discussion

Snail shells are a vital, abundant, low-cost, eco-friendly material that is essential for clean energy production, as shown in Figure 1. They contain a large amount of calcium (95% of CaCO_3), along with other organic matter (5%) [14,15]. Moreover, they are sustainable biowaste precursors because of their unique biomineralized composition, consisting of CaCO_3 along with minor organic constituents that generate carbon structures.

During thermal treatment, CaCO_3 and organic matrices decompose according to the following conditions:

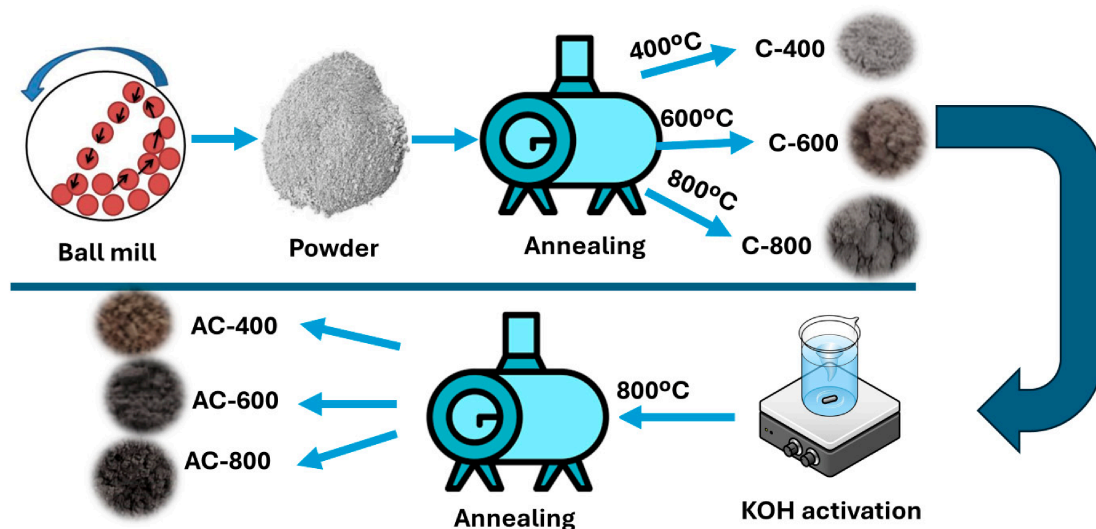


Figure 1. Synthesis of C-400, C-600, C-800, AC-400, AC-600, and AC-800.

At 400°, some of the internal organic matter burned, but the CaCO_3 did not decompose. Thus, the C-400 sample had little amorphous carbon, low porosity, and more CaCO_3 . At 600 °C, some CaCO_3 decomposed with increasing amounts of organic substances. Therefore, the C-600 sample had several amorphous carbons, medium porosities, and some CaCO_3 . At 800°, the C-800 sample had a more carbonized structure, a porous carbon matrix, CaO particles, and semigraphitic carbon. Thus, the release of gaseous products during carbonization contributes to pore generation and surface development. In addition, organic components form carbon structures that increase electrical conductivity. The activation process involves a series of redox and gasification reactions during high-temperature treatment, leading to pore generation and surface development.

The selected 1:3 (sample) ratio ensured sufficient activation and effective pore development while maintaining the structural integrity of the carbon framework. Subsequent chemical activation using KOH further improved the porous structure through reactions. After activation, AC-400, AC-600, and AC-800 had porous carbon, a more porous structure, a larger pore volume, an increased surface area, and an enhanced performance [14,16]. Therefore, the activation processes promote the formation of microporous and mesoporous structures, increase the specific surface area, and increase the accessibility of the electrolyte, namely, the availability of active sites, making snail shell-derived carbon materials promising candidates for use as DMFC electro/nanocatalyst materials.

The results of the XRD analysis of the C-400, C-600, and C-800 samples are shown in Figure 2a. The indices of 104 (29.62°), 110 (36.34°), 113 (39.78°), 018 (47.86°), 116 (48.8°), 122 (57.78°), and 214 (60.94°) indicate the presence of CaCO_3 (JCPDS card no. 41-1475 and 29-0306), whereas 111 (29.74°), 200 (34.3°), 220 (54.62°), 311 (62.9°), and 221 (64.74°) indicate the presence of CaO (JCPDS card no. 77-9574) [17–20]. The results of the experiments reveal that C-400 contained a majority of CaCO_3 with prominent peaks and no signs of CaO. The presence of carbon at peaks 24 and 45 is indicated by a blue asterisk. In contrast, C-600 decreased relatively strongly at 29.78° from 686 to 407 a.u., suggesting a small conversion of CaCO_3 to CaO. Furthermore, the CaO peaks are clearly visible for C-800, with a decrease

in the intensity of the CaCO_3 peaks, indicating a significant increase in the presence of CaO . Next, the appearance of several sharp peaks in sample AC-400 compared to C-400 indicates that KOH reacts more strongly on the surface (Figure 2a). However, this differs between samples AC-600 and AC-800, where the peaks are less pronounced than those of C-600 and C-800, suggesting greater KOH penetration into the sample and increased porosity. The crystallite size distribution for each sample is shown in Figure 2c, indicating that temperature and activation reduce the crystallite size and increase its number with increasing surface area. Moreover, the degree of crystallinity was calculated for a more comprehensive study than for the XRD analysis (Figure 2d). The C-400 sample contained a crystalline and an amorphous area. Therefore, the transition from ~62% to ~91% for AC-800 indicates a shift from a less low degree of crystallinity to a more crystalline material during KOH activation and temperature changes. This directly affects performance, resulting in improved hardness, consistent stability, and excellent thermal behavior.

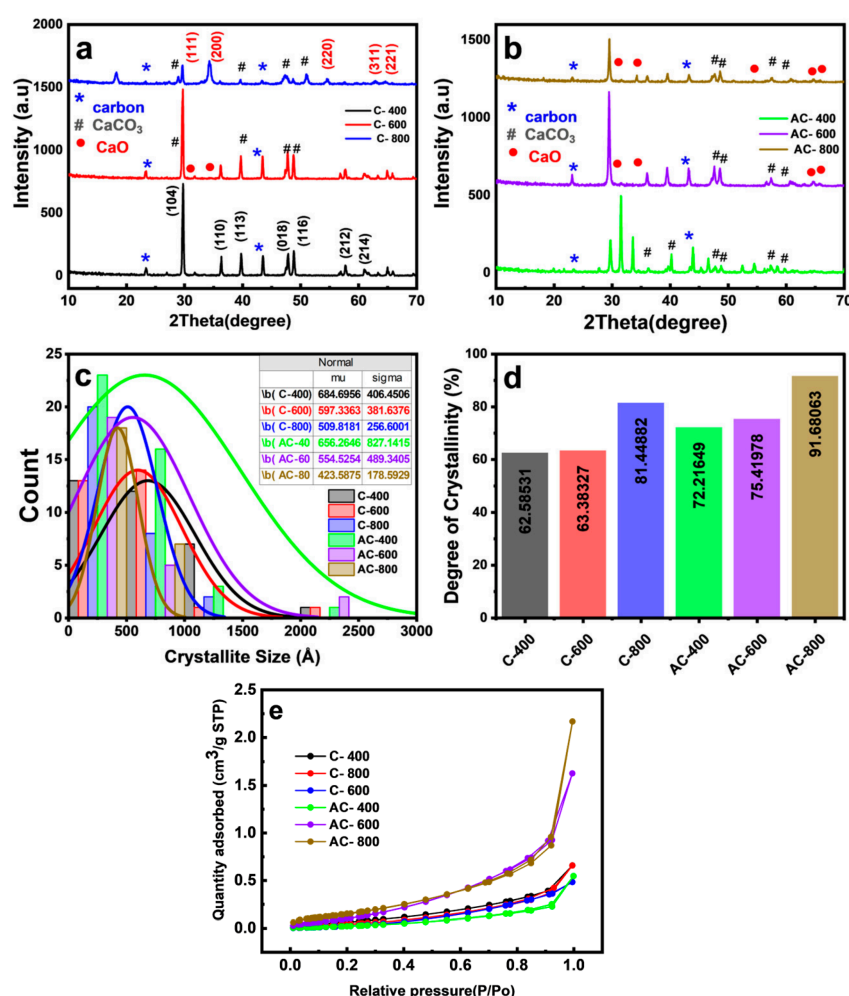


Figure 2. XRD analysis of (a) C-400, C-600, and C-800 and (b) AC-400, AC-600, and AC-800. (c) Distribution of crystallite size, (d) degree of crystallinity, and (e) BET N_2 adsorption-desorption isotherm plot.

The BET N_2 adsorption-desorption isotherms are classified as type IV with H3-type hysteresis loops, as shown in Figure 2e [21]. Moreover, the surface area, pore volume, pore diameter, and pore width were calculated via the BJH method and are shown in Table 1. The average pore diameter distributions were 13.75, 24.97, 16.00, 18.02, 19.05, and 20.40 nm for C-400, C-600, C-800, AC-400, AC-600, and AC-800, respectively. Moreover, the BET surface areas were 6.49, 2.84, 4.12, 12.19, 4.66, and 14.17 m^2/g , respectively. These results

indicate that all the electric/nanocatalyst structures were mesoporous. It can also be said that a gradual increase in temperature enhances both structure and porosity, but KOH activation is more noticeable when the temperature is constant. However, the lower surface area observed for AC-400 than observed for C-400 suggest that 400 °C was insufficient for effective pore development during KOH activation, whereas higher temperatures (AC-600 and AC-800 °C) promoted better porosity generation and surface area enhancement.

Table 1. The textural properties.

Sample	Surface Area (m ² /g)	Pore Volume (cm ³ /g)	Pore Diameter (nm)	Pore Width (nm)
C-400	6.49	0.02232	13.75	4.30
AC-400	2.84	0.01774	24.97	4.49
C-600	4.12	0.01652	16.00	4.55
AC-600	12.19	0.05496	18.02	4.50
C-800	4.66	0.02225	19.05	4.58
AC-800	14.17	0.07233	20.40	4.52

Scanning electron microscopy (SEM) was used to examine the morphology of C-400, C-600, C-800, AC-400, AC-600, and AC-800, as shown in Figure 3. C-400 showed irregular sizes of tetragonal and pentagonal particles (yellow rectangle) along with particle adhesion and few pores (Figure 3a). As shown in Figure 3b, the C-600 sample consisted of cracks and smaller particle sizes with a relative increase in porosity. Moreover, the C-800 sample consisted of widespread cracks (yellow lines) and a larger spherical particle size (yellow circle) with increased porosity (Figure 3c). Moreover, partially graphitized carbon alongside amorphous carbon could be observed. After the samples were activated, the particles became more spherical (red circle) and porous in samples AC-400, with an increased surface area and a decrease in particle size (Figure 3d–f).

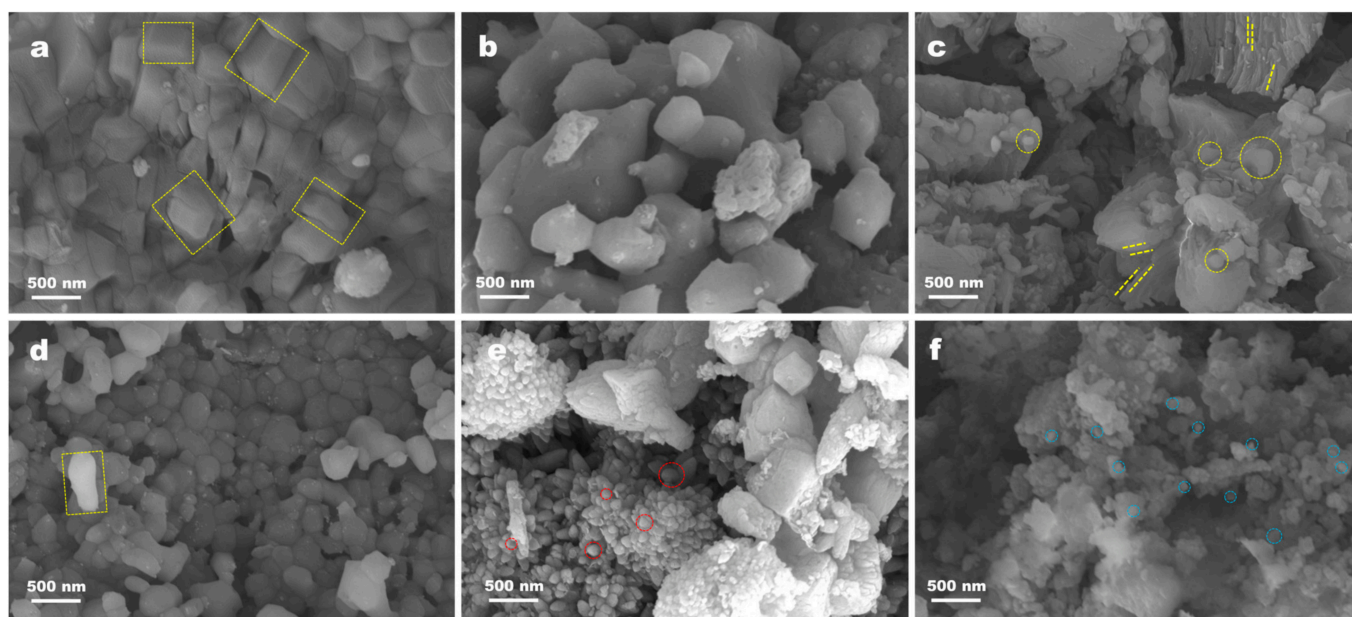


Figure 3. SEM images of (a) C-400, (b) C-600, (c) C-800, (d) AC-400, (e) AC-600, and (f) AC-800 (yellow rectangle: pentagonal particle; yellow line: crack; yellow circle: large spherical particle; red circle: small spherical particle).

3D typography was performed to highlight the areas of difference between elevations and depressions (Figure 4). The 3D surface visualization was reconstructed from SEM micrographs and represents relative contrast variations by grayscale values. White and light blue indicate medium elevations, green and red indicate very high elevations, and dark blue indicates deep porosity. The C-400 sample is white and light blue with a scarcity of dark blue, indicating low porosity and elevated topography, as shown in Figure 4a. In Figure 4b, the C-600 consists of a spread of green plateaus alongside white, light blue, and dark blue, which are relatively higher, which proves that the porosity increased. In C-800, green and red dominate on the surface, and there is a large distinction of dark blue, indicating an increase in surface area with variations in topography and porosity (Figure 4c). After activation, most of the surface area is green and red, with a wider distribution of dark blue than before activation (Figure 4d–f). These findings indicate that activation increases the surface area and porosity, thus improving the performance and efficiency.

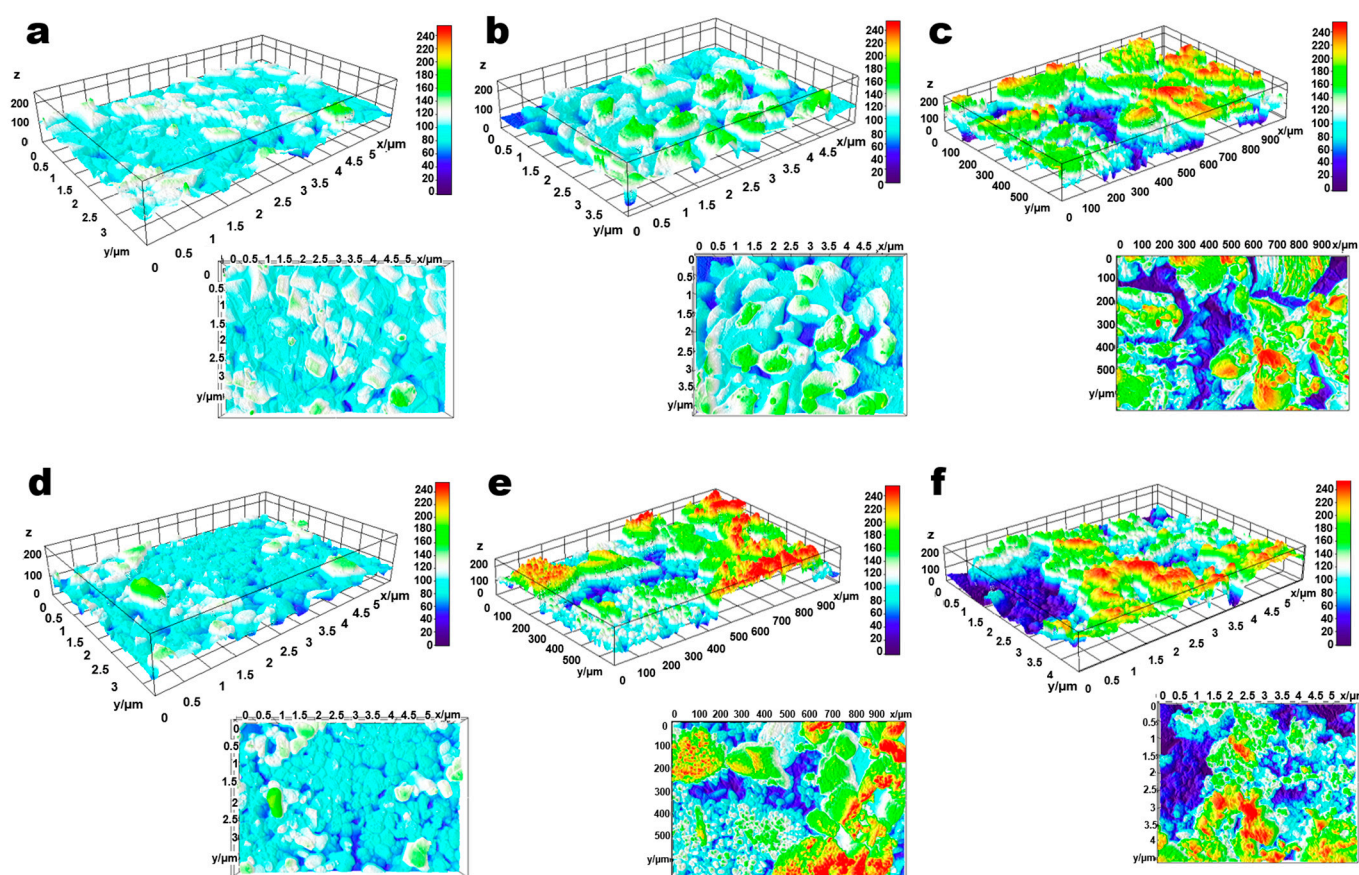


Figure 4. 3D typography images of (a) C-400, (b) C-600, (c) C-800, (d) AC-400, (e) AC-600, and (f) AC-800.

Energy-dispersive X-ray spectroscopy (EDX) analysis of the snail shell powder revealed a high-purity biomineral composition, as shown in Figure 5. There were peaks for calcium, oxygen, and carbon, along with small amounts of other substances. Calcium had the highest elemental concentration, with a weight percentage of 52%, followed by oxygen at 29%. This strong elemental distribution confirmed the presence of calcium carbonate CaCO_3 in the raw material form. The remaining fraction, with a weight percentage of 19%, was attributed to carbon, originating from both the organic protein matrix of the shell (conchiolin) and the conductive carbon tape used for sample fixation.

The elemental composition of the synthesized materials was investigated using EDX, as shown in Figure 6. The analysis confirmed the presence of the major constituent elements

of C, Ca, and O, indicating the successful formation of the prepared nanocomposites. These elements were consistently detected in all analyzed samples, demonstrating the effectiveness of the synthesis process. In addition, trace amounts of Al, Si, and K were also identified. The presence of these minor elements was attributed to the natural composition of the precursor material and residual mineral constituents rather than to the formation of undesirable secondary phases. Therefore, the EDX results provide strong evidence of the expected elemental composition and further support the successful preparation of the synthesized materials.

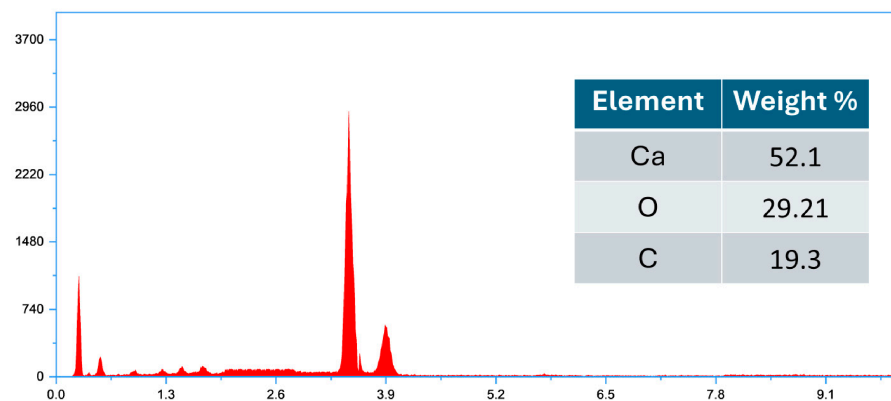


Figure 5. EDX analysis of snail shell powder.

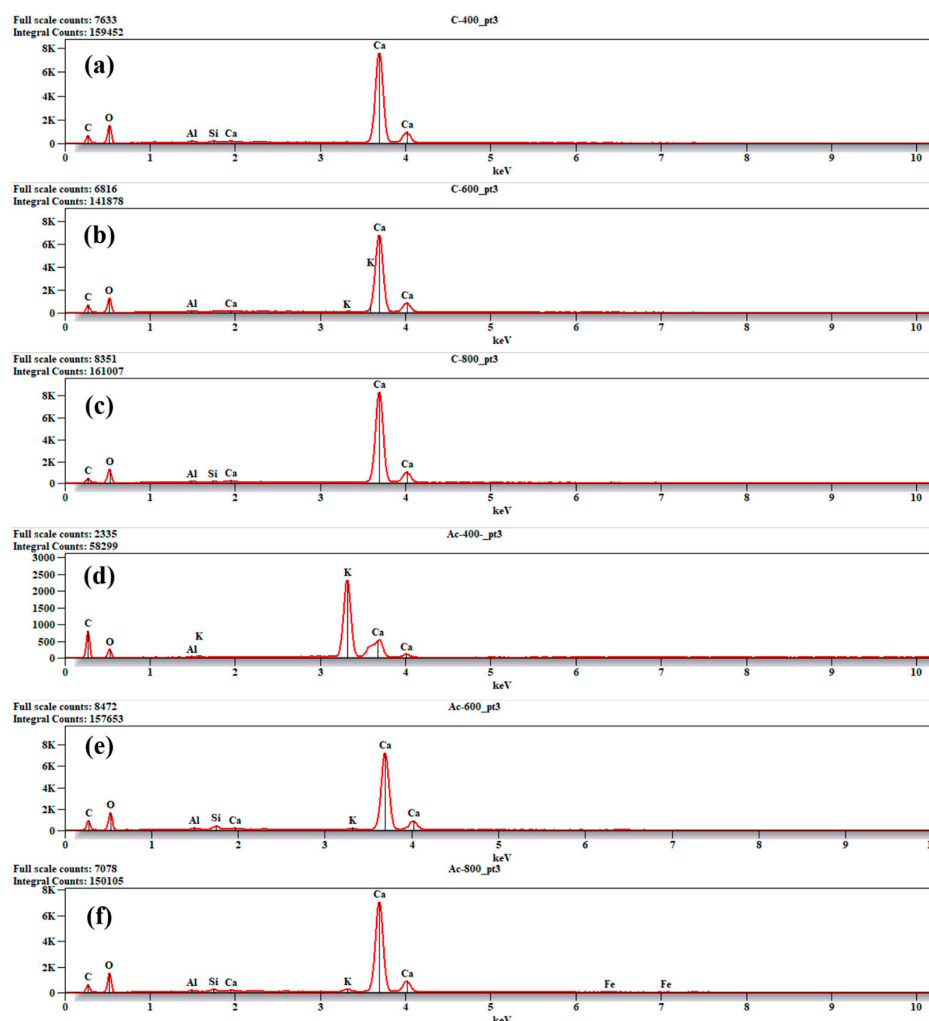


Figure 6. EDX analysis of (a) C-400, (b) C-600, (c) C-800, (d) AC-400, (e) AC-600, and (f) AC-800.

Furthermore, quantitative elemental mapping was conducted to examine the distribution of elements within the six nanocomposites, as illustrated in Figure 7. The results demonstrate a uniform distribution of the principal elements of C K α , Ca K α , and O K α across the nanocomposite surfaces. This homogeneous elemental dispersion of C, Ca, and O promoted efficient diffusion, enhanced milling, and improved heat transfer throughout the composites. The consistent distribution of C among all six samples confirms that the organic constituents were successfully converted into C across the entire nanocomposite rather than being localized in specific regions, providing further evidence of carbon formation. Similarly, the uniform Ca distribution, supported by the results of the elemental analysis, indicates that Ca was present in all the samples in the form of either CaCO₃ or CaO. The absence of Ca-rich domains further suggests that no distinct impurity phases were present within the nanocomposites.

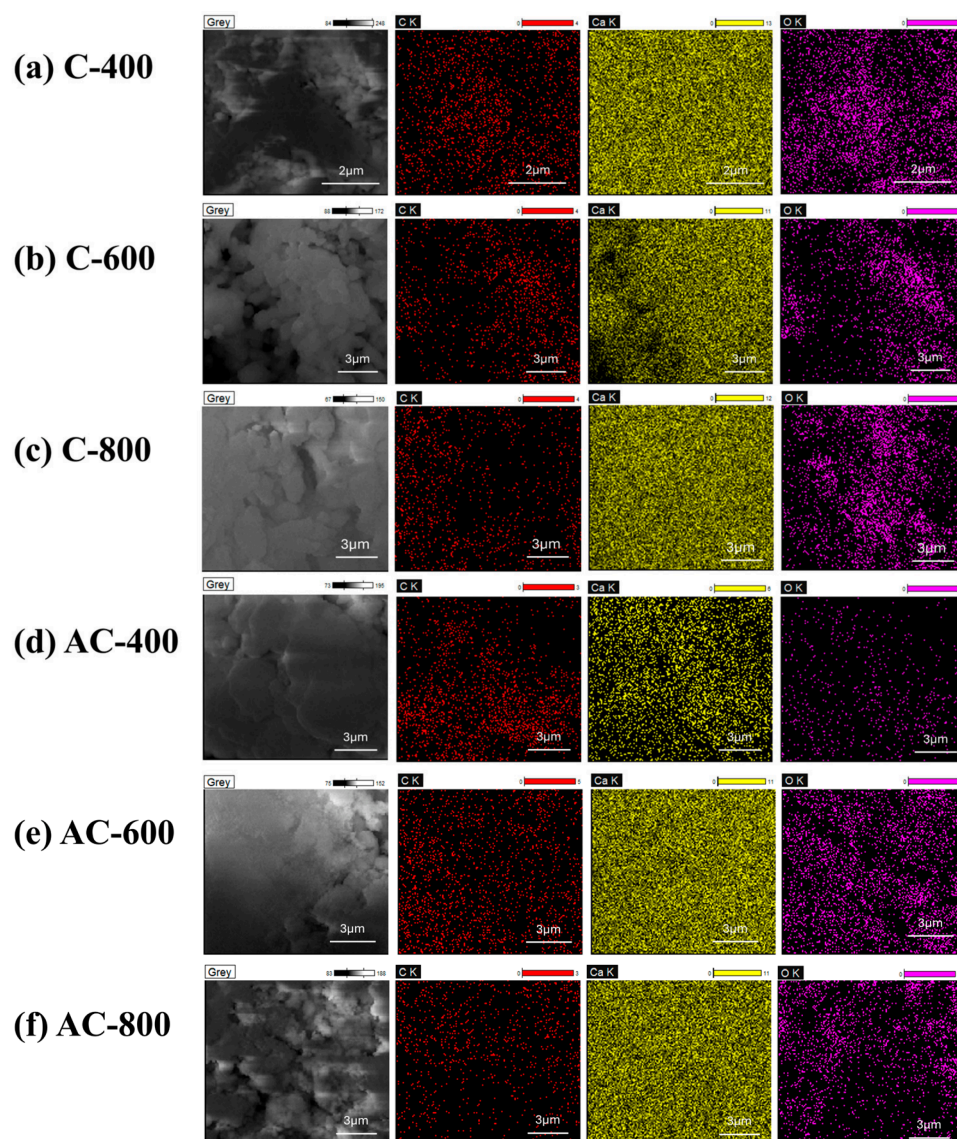


Figure 7. Mapping images of (a) C-400, (b) C-600, (c) C-800, (d) AC-400, (e) AC-600, and (f) AC-800.

An electrochemical cell was used to analyze the electrochemical properties of the electro/nanocatalyst in 1 M KOH at room temperature. Cyclic voltammetry (CV) profiles alter the potential to varying current by changing identical scan rates to determine electron transfer rates and redox mechanisms [22]. Different concentrations of methanol were added to the electrolyte solution to establish the optimum methanol concentration (0–3 M) for the

oxidation process using the prepared catalysts. CV measurements were then performed for each catalyst, as shown in Figures 8 and 9. Moreover, the variation in the samples was tracked during the analysis before and after the addition of methanol, as shown in Figure 8. The current density of the sample increased with increasing efficiency in the absence of methanol up to 3 M, with the values increasing for C-400 (6.46 to 21.12 mA/cm²), C-600 (12.5 to 30.43 mA/cm²), and C-800 (22.76 to 49.89 mA/cm²). Thus, the results indicate that C-400, C-600, and C-800 had good electrochemical properties. Moreover, doubling the temperature more than doubled the electrochemical properties. CV analysis revealed a leaf-shaped expansion of the properties of the electro/nanocatalyst where the behavior was influenced by a combination of the double-layer capacitance current and Faraday processes associated with the methanol oxidation reaction.

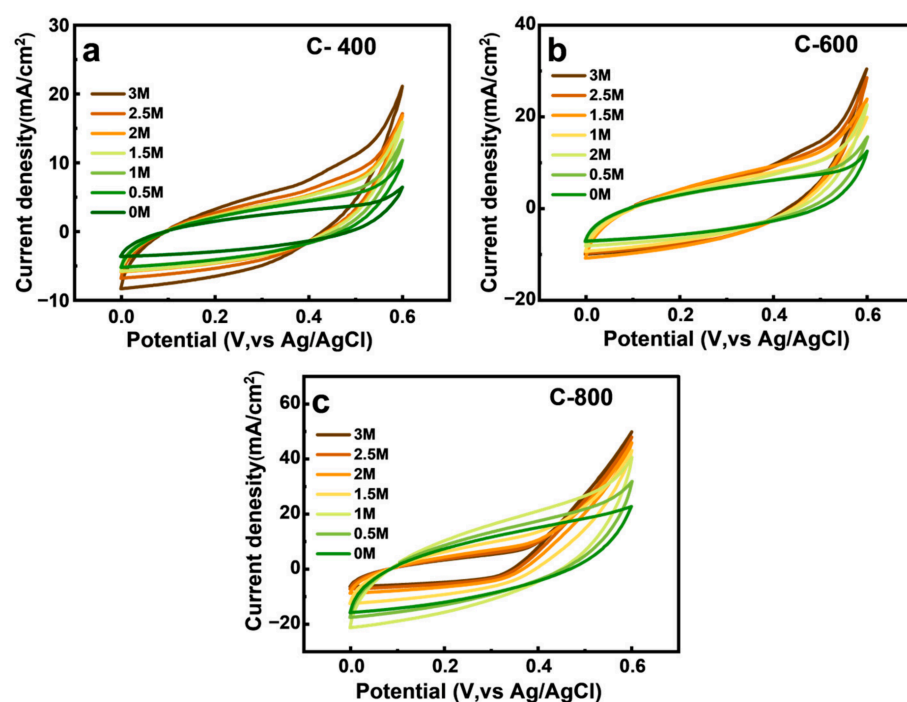


Figure 8. CV curves of different catalysts at a scan rate of 20 mV/s in solutions of (a) C-400, (b) C-600, and (c) C-800 at various methanol concentrations (voltages of 0–0.6 V, concentrations of 0–3 M, and surface areas of 1 cm²).

The catalytic activity of AC-400, AC-600, and AC-800 compared to that of C-400, C-600, and C-800 is shown in Figure 9. Before the methanol concentration increased, the maximum current densities of AC-400, AC-600, and AC-800 were 9.7, 13.6, and 27.05 mA/cm², respectively, as shown in Figure 9a–c. After the methanol concentration increased, the maximum current densities of AC-400, AC-600, and AC-800 were 26.19, 38.1, and 65.24 mA/cm², respectively. The oxidation peaks associated with the scan rate were consistent with methanol oxidation, confirming that the electrochemical performance was controlled by both charge storage and catalytic activity. Furthermore, the disappearance of the ideal rectangular shape resulted in internal resistance and kinetic limitations in the electrochemical response. As shown in Figure 6d, compared with those of C-400 (21.12 mA/cm²), C-600 (30.43 mA/cm²), and C-800 (49.89 mA/cm²), the current densities of AC-400, AC-600, and AC-800 increased with increasing methanol concentration. Therefore, all electro/nanocatalysts are suitable for direct methanol oxidation applications. Moreover, the enhanced electrochemical activity can be attributed to the porous carbon framework generated through CaCO₃ decomposition and KOH activation.

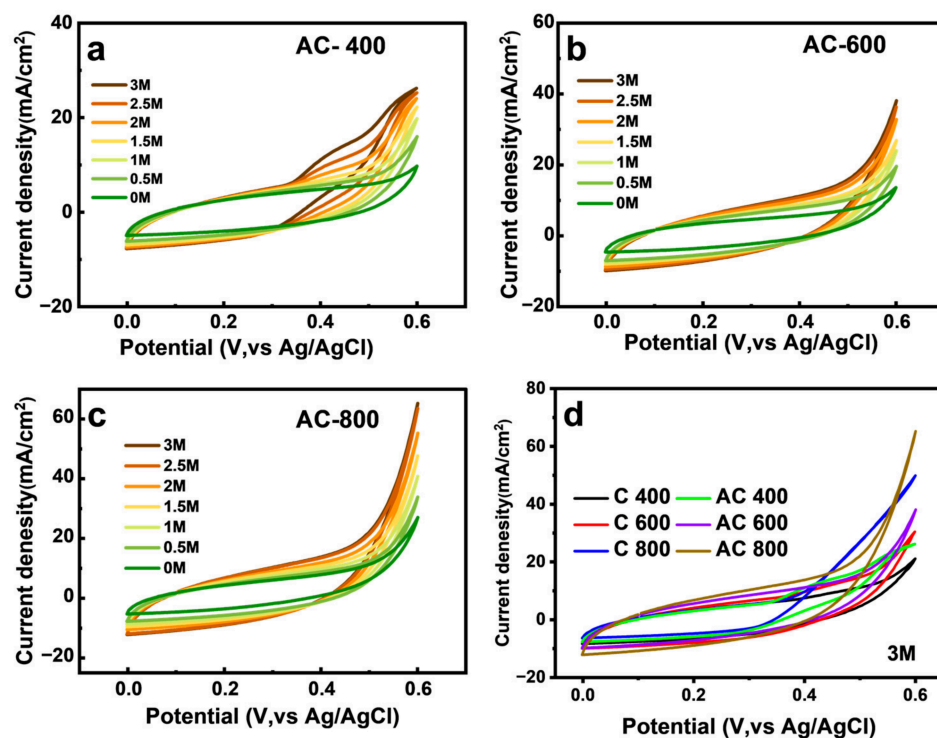


Figure 9. CV curves of different catalysts at a scan rate of 20 mV/s in solutions of (a) AC-400, (b) AC-600, and (c) AC-800 at different methanol concentrations of 0–3 M. (d) CV curves of different nanoelectric catalysts at 5 mV/s (voltage of 0–0.6 V and surface area of 1 cm²).

CV analysis of C-400, C-600, and C-800 was performed at 5–50 mV/s, as shown in Figure 10. The CV profiles of AC-400, AC-600, and AC-800 increased from 6.84 to 21.12 mA/cm² (Figure 10a), from 17.9 to 30.43 mA/cm² (Figure 10b), and from 32.55 to 49.89 mA/cm² (Figure 10c), respectively, with increasing scan rate, indicating strong electrochemical properties and conductivity.

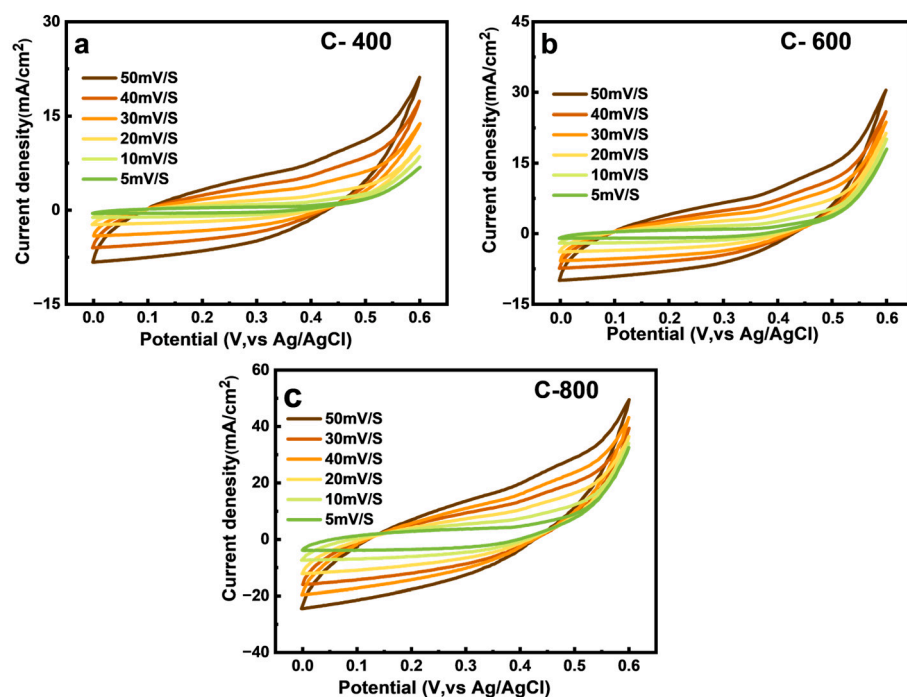


Figure 10. CV analysis of (a) C-400, (b) C-600, and (c) C-800 at various scan rates of 5–50 mV/s (voltages of 0–0.6 V and a surface area of 1 cm²).

The results of the CV analysis of AC-400, AC-600, and AC-800 increase from 16.79 to 27.76 mA/cm² (C-400) in Figure 11a, from 26.99 to 38.1 mA/cm² (C-600) in Figure 11b, and from 37.01 to 64.2 mA/cm² (C-800) in Figure 11c, respectively, with increasing scan rate. A comparison of the behavior of the materials at different temperatures and activation levels in Figure 11d reveals that activation acts as a prerequisite after the appropriate temperature factor is applied. The results of the CV analysis revealed a rounded-angled square shape with all the electrocatalytic characteristics where the behavior was a combination of double-layer capacitive current and Faradic processes. Furthermore, AC-800 had the best electrochemical properties, a large surface area, high performance, extra capacitance, and excellent conductivity.

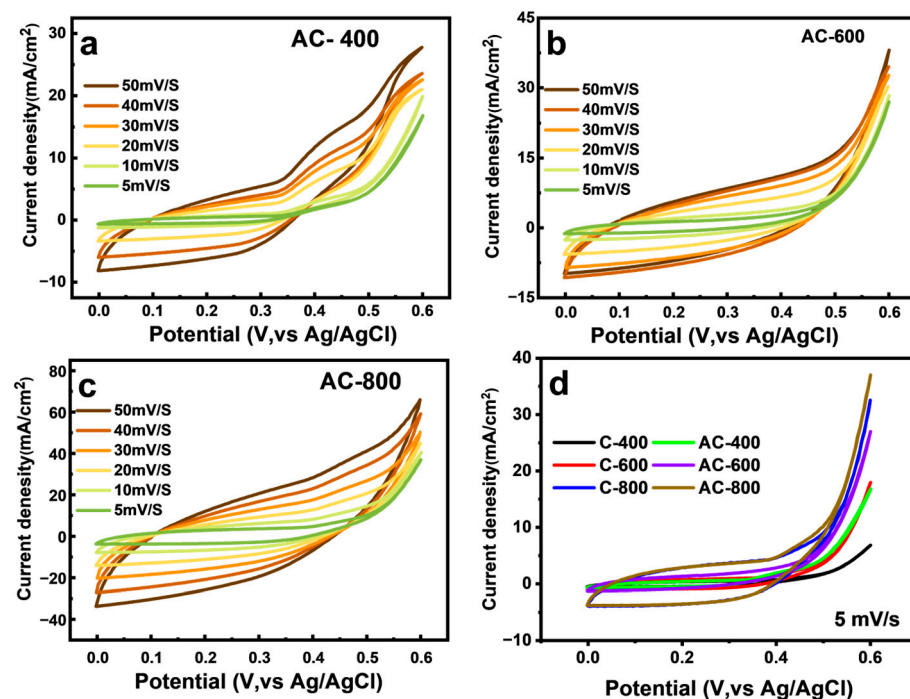


Figure 11. CV analysis of (a) AC-400, (b) AC-600, and (c) AC-800 at various scan rates of 5–50 mV/s. (d) CVs of different nanoelectrics at 5 mV/s (voltage of 0–0.6 V and surface area of 1 cm²).

In Figure 12a, the current density (*j*) is plotted versus the scan rate, resulting in a linear relationship whose slope signifies the electrical double-layer capacitance (*C_{dl}*) [23,24]. The *C_{dl}* values for the C-400, C-600, C-800, AC-400, AC-600, and AC-800 samples are 310, 250, 360, 200, 230, and 630 µF/cm², respectively (Figure 12a). The *C_{dl}* values markedly improved with activation and temperature, increasing from 310 µF/cm² to 630 µF/cm² for C-400 to AC-800. The results show that it was characterized by a high electrochemically active surface area (ECSA) with high capacitive behavior [25]. In Figure 12b, the diffusion-controlled plot illustrates the linear relationship between the peak current and the square root of the scanning rate with respect to the degree of diffusion in the samples [26]. The values are greater for the AC-600 and AC-800 samples than for the C-600 and C-800 samples. The linear relationship indicates that a difference between the electrochemical processes existed, which presented diffusion control and increased with activation. As shown in Figure 9c, the current increased gradually with increasing methanol concentration, with the current values for C-400, C-600, C-800, AC-400, AC-600, and AC-800 increasing from 6.46, 12.54, 22.76, 9.74, 13.61, and 27.05 to 10.33, 15.64, 31.87, 15.97, 19.59, and 33.87 mA/cm² (from 0 to 0.5 M methanol, respectively). As shown in Figure 12c, when the maximum methanol concentration was compared among the reconstituted C-400 (21.12 mA/cm²), C-600 (30.43 mA/cm²), C-800 (49.89 mA/cm²), AC-400 (26.19 mA/cm²),

AC-600 (38.1 mA/cm^2), and AC-800 (65.24 mA/cm^2) samples, the addition of methanol resulted in an increase in the maximum current density. In addition, the R^2 values were high, confirming that the oxidation current was improved by the electrocatalytic kinetics and the high efficiency of the electro/nanocatalyst materials in oxidizing methanol, which was highly dependent on the methanol concentration [27]. The polarization curve shows the variation in the power density with the scan rate in Figure 12d. The minimum power densities of the C-400, C-600, C-800, AC-400, AC-600, and AC-800 electro/nanocatalyst were 4.1, 10.7, 19.5, 10.7, 16.19, and 22.2 mW/cm^2 , respectively, at 5 mV/s . On the other hand, the maximum power densities of the C-400, C-600, C-800, AC-400, AC-600, and AC-800 electron nanocatalysts were 12.6, 18.2, 29.7, 16.6, 22.86, and 39.56 mW/cm^2 , respectively, at 50 mV/s . Consequently, high power densities for the materials were obtained, whereas in the AC-800 grade, the power density was excellent for providing activated porous carbon, a more porous structure, a larger pore size, and an increased surface area.

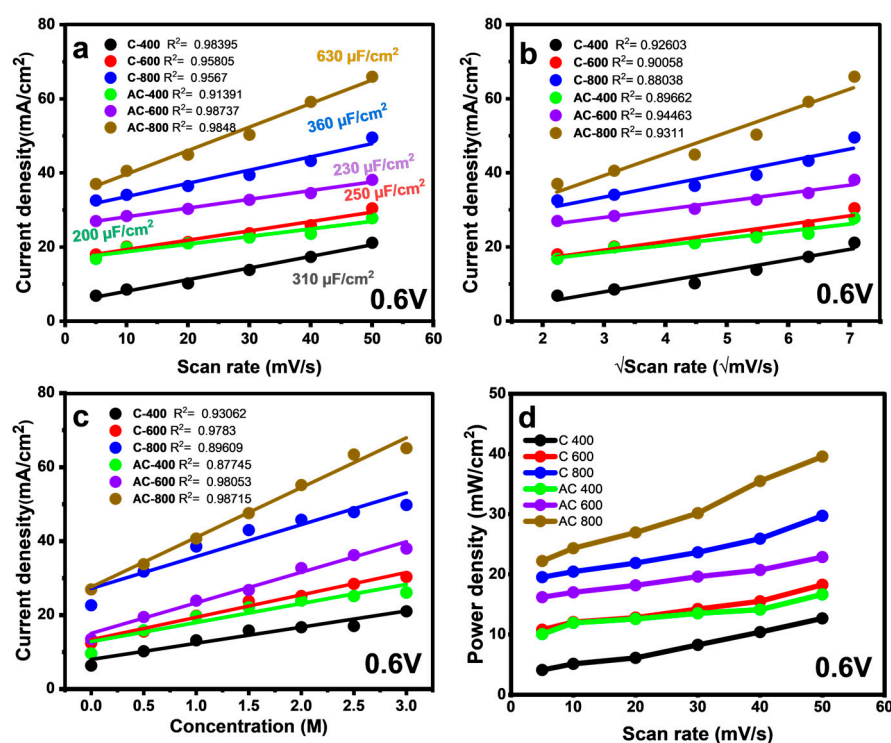


Figure 12. (a) Current density vs. scan rate of 5–50 mV/s, (b) diffusion-controlled plot, (c) concentration dependence plot at 0.5–3 M methanol, and (d) polarization curve at 5–50 mV/s (voltage of 0.6 V and surface area of 1 cm²).

Consequently, compared with the other prepared samples, the sample prepared at 800 °C (AC-800) exhibited the highest electrochemical performance. This improved performance was attributed to the optimized carbonization and activation processes at higher temperatures, which promoted the development of a well-connected porous structure, a greater specific surface area, and enhanced electrical conductivity. As a result, AC-800 provided more accessible active sites and improved charge transfer kinetics, which can lead to superior catalytic activity in DMFC applications.

Compared with the catalysts from the literature shown in Table 2, the present electro/nanocatalyst materials offer significant advantages, including lower cost, environmental sustainability, and improved resistance to poisoning, although noble metals still provide higher intrinsic catalytic activity. Thus, this study utilizes snail shell waste as a dual-source raw material containing biocalcious compounds and carbonaceous components, enabling the formation of a unique porous carbon structure after heat treatment. Furthermore, the

developed material could exhibit enhanced electrochemical activity and power density against methanol oxidation in DMFC applications.

Table 2. Previous literature review of electrocatalysts for DMFCs.

Sample	Power Density mW/cm ²	Reference
5C-NP-Fe	10.8	[28]
rN/LS-HAP	22.45	[29]
N117	9.66	[29]
rN117	8.31	[29]
Co@NG	34.7	[30]
NiCo/N-CNFs@800	29.15	[31]
15 wt% Fe N/C (CS) at 30 °C	7.21	[32]
15 wt% Fe N/C (CS) at 60 °C	15.94	[32]
15 wt% Fe Pt/C at 30 °C	5.718	[32]
15 wt% Pt/C at 60 °C	7.56	[32]
C-400	12.6	This work
C-600	18.2	This work
C-800	29.7	This work
AC-400	16.6	This work
AC-600	22.86	This work
AC-800	39.56	This work

Tafel analysis reveals physical properties such as the Tafel slope and exchange current density, as shown in Figure 13 [33,34]. At 5 mV/s, the Tafel slopes of the C-400, C-600, C-800, AC-400, AC-600, and AC-800 catalysts are 14, 11, 10, 8, 54, and 19 mV/dec, respectively, at low potential (Figure 13a). In addition, the Tafel slopes of the C-400, C-600, C-800, AC-400, AC-600, and AC-800 catalysts are 166, 147, 156, 175, 154, and 155 mV/dec, respectively, at high potential. With 3 M methanol, the Tafel slopes of the C-400, C-600, C-800, AC-400, AC-600, and AC-800 catalysts are 19.4, 19.5, 18.2, 14.6, 15.6, and 19.4 mV/dec, respectively, at low potential (Figure 13b). In addition, the Tafel slopes of the C-400, C-600, C-800, AC-400, AC-600, and AC-800 catalysts are 152, 137, 382, 186, 154, and 156 mV/dec, respectively, at high potential. Consequently, the Tafel slope indicates that the reaction is faster, involves the transfer of several electrons, requires less effort, and affects the surface coverage of the material.

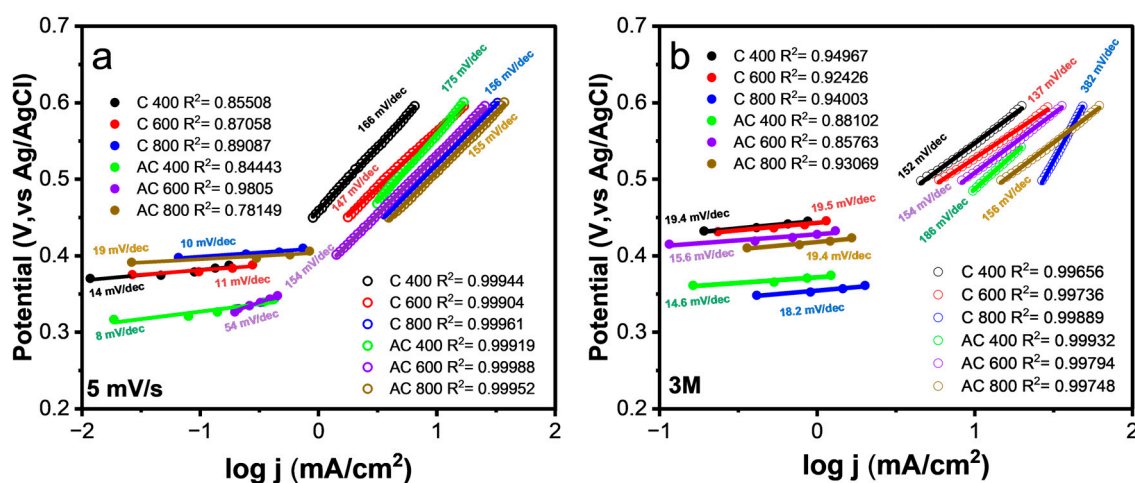


Figure 13. Tafel slopes (a) at 5 mV/s and (b) in 3 M methanol (surface area of 1 cm²).

Nyquist plots include the charge-transfer resistance (R_{ct}) and uncompensated resistance (R_u), which represent the electrolyte resistance, and the kinetic and mechanistic data

for various electrochemical systems (Figure 14) [35]. The circuit comprises an uncompensated resistor (R_u), a charge-transfer rate resistor (R_{CT}), a double-layer capacitance (C_{dl}), intermediate adsorption (R_{ads}), and an adsorption capacitance (C_{ads}). In this case, the total current through the uncompensated resistor (R_u) was divided into the current associated with the charging/discharging of the bilayer. The R_u values were 11.59 Ω (C-400), 6.83 Ω (C-600), 6.16 Ω (C-800), 5.17 Ω (AC-400), 4.52 Ω (AC-600), and 3.55 Ω (AC-800). Accordingly, AC-800 had a lower solution resistance gradient, higher conductivity, more ion aggregation, and shorter internal pathways. Moreover, the $R_u + R_{ct}$ values were 78.96 Ω (C-400), 77.47 Ω (C-600), 54.64 Ω (C-800), 28.12 Ω (AC-400), 20.83 Ω (AC-600), and 19.43 Ω (AC-800). Therefore, the electrochemical reaction of AC-800 was faster, and charge transfer was easier, indicating excellent performance. The results indicate that the reduction in R_{ct} was a consequence of the improved porous structure and surface functionalities of the activated carbon, which resulted in an increased contact area between the electrolyte and electrons. This finding supports the availability of the electrolyte, the active site, and the electron transport kinetics.

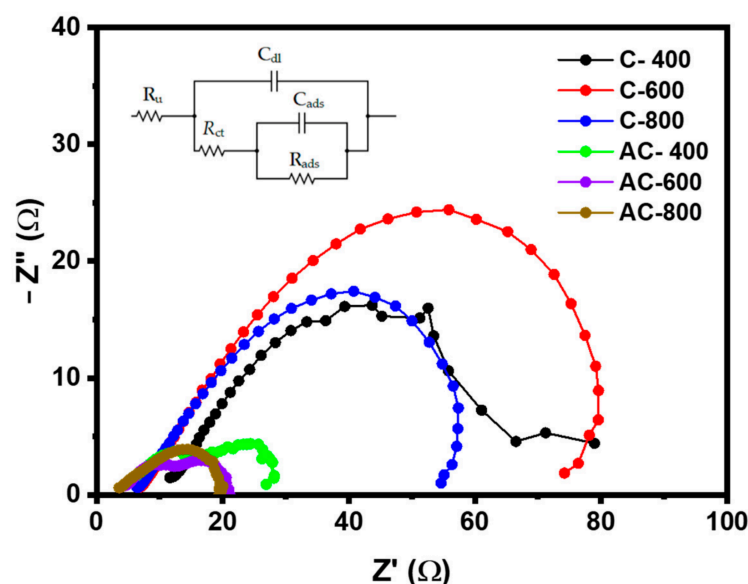


Figure 14. Nyquist plots (voltage of 0.0 V).

Here, the enhanced nanoelectrocatalytic performance of the prepared carbon electrodes is attributed to the combined effect of the carbonization temperature and activation with potassium hydroxide. During carbonization, the initial shell compound transforms into a conductive carbon structure. Increasing the temperature (400–800 $^{\circ}\text{C}$) increases the degree of carbonization and graphitization, thus increasing the electrical conductivity. Subsequent activation with potassium hydroxide then results in the formation of a highly diffuse porous structure with a large surface area, facilitating electrolyte penetration and ion diffusion. The presence of numerous pores and defects provides more active sites for electrochemical reactions. Thus, the synergy between improved conductivity and increased porosity leads to faster charge transfer and enhanced electrocatalytic activity.

3. Experimental

3.1. Synthesis of Nanoelectrocatalysts

The snails were collected and then ground in a ball mill to obtain powder at 300 $^{\circ}\text{C}$ for 2 h. Moreover, it was confirmed that the samples were thoroughly ground to a fine texture. The next step involved placing the powder in the furnace in N_2 at different temperatures,

such as 400°, 600°, and 800°, to obtain the C-400, C-600, and C-800 samples. Second, the C-400, C-600, and C-800 samples were activated using KOH at a 1:3 ratio (sample:KOH). It was thoroughly washed several times to remove excess hydroxyl groups and to reach a pH of 7. After that, it was heated in a tube furnace at 800 °C for 2 h in N₂ to obtain the AC-400, AC-600, and AC-800 samples.

3.2. Characterization

X-ray diffraction (XRD, PANalytical Empyrean, 202964, Sweden) analysis was performed using Cu K α radiation ($\lambda = 0.15405$ nm), 40 kV, and 30 mA. Scanning electron microscopy (SEM, JEOL JSM-5900, Tokyo, Japan) and the Brunauer–Emmett–Teller (BET) technique (N₂ adsorption–desorption at 273 K) were used to characterize the textural properties and calculate the surface area. N₂ adsorption–desorption isotherms were established by TriStar II 3020 (Micrometrics, Norcross, GA, USA). The pore size distribution was evaluated using the Barrett–Joyner–Halenda (BJH) method. The volume of the mesopores was calculated from the N₂ adsorption branch using the t-plot method.

3.3. Electrochemical Measurements

The graphite sheet electrode (GSE) was prepared in the required dimensions and cleaned by ultrasonication in acetone, ethanol, and deionized water for a few minutes. The cleaned electrode was then dried under N₂. To increase the wettability and improve catalyst attachment, the GSE surface was polished and rinsed with deionized water before catalyst deposition.

The catalyst ink was prepared by dispersing 5 mg of the active material in a mixture of 200 μ L of ethanol and 50 μ L of Nafion[®] solution (5 wt%, Sigma–Aldrich). The mixture was ultrasonicated for half an hour until a uniform suspension was obtained. Afterward, 10 μ L of the ink was deposited onto the cleaned GSE surface by drop-casting and dried under an infrared lamp, resulting in a catalyst loading of 0.1 mg/cm². The electrodes used for the reference, counter, working electrodes, and fuel were saturated Ag/AgCl, a Pt sheet, GSE, and methanol, respectively. The working electrode had an active surface area of 1 cm² with dimensions of 1 cm $\times$ 1 cm.

4. Conclusions

Eco-friendly carbon materials were successfully prepared from snail shell waste through controlled carbonization and chemical activation processes for direct methanol fuel cell (DMFC) applications. Snail shells were collected, cleaned, dried, and ground into a fine powder. They were then calcined at different temperatures (400, 600, and 800 °C) to produce the C-400, C-600, and C-800 samples. These samples were then activated (KOH) to obtain the AC-400, AC-600, and AC-800 activated carbon samples. The results show that increasing the calcination temperature enhances the carbonization process and pore formation, resulting in highly porous activated carbon structures with improved surface properties. The activation process significantly increased the surface area and produced porous structures suitable for electrochemical applications. The maximum current densities of C-400, C-600, C-800, AC-400, AC-600, and AC-800 were 21.12, 30.43, 49.89, 26.19, 38.1, and 65.24 mA/cm², respectively, in the presence of methanol. In addition, the power densities of the C-400, C-600, C-800, AC-400, AC-600, and AC-800 electron nanocatalysts were 12.6, 18.2, 29.7, 16.6, 22.86, and 39.56 mW/cm², respectively. Manufactured materials have also become effective sustainable carbon materials for waste-to-energy conversion by transforming snail shell biomass into valuable functional carbon materials for renewable energy applications. Future work will focus on full DMFC testing to evaluate practical fuel cell performance.

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