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Original Article

Cost-Effective Microwave-Assisted Synthesis of Nanomaterials for Direct Alcohol Fuel Cell Applications

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Abstract *- A low-cost, computer-controlled modification of a domestic microwave oven is developed to achieve precise, programmable control of microwave irradiation during the polyol synthesis of fuel cell catalysts, a level of control previously restricted to costly, laboratory-grade microwave reactors. Platinum-on-Carbon (Pt/C) catalysts are synthesized by the polyol method using two heating sources, a conventional stirring hotplate and the modified microwave oven operated under intermittent heating, and are compared with a commercial Pt/C (20 wt.%) catalyst. The microwave-assisted route reduces the heating stage of the synthesis from 3 h to 15 min and yields a catalyst with crystallite size, electrochemically active surface area, and ethanol oxidation activity comparable to those of the conventionally synthesized and commercial references. These results show that a domestic microwave oven adapted with programmable control is a viable and accessible tool for the laboratory-scale synthesis of fuel cell electrocatalysts.*

Keywords *- Catalyst synthesis, Fuel cells, Microwave-Assisted, Microwave modification, Polyol methods.*

1. Introduction

In recent years, there has been increasing interest in developing efficient and environmentally friendly energy conversion systems. Fuel cells have emerged as a promising solution because of their high-power density and potential for utilizing fuels such as hydrogen (produced from renewable sources) and liquid alcohols. However, one of the main challenges associated with fuel cells is the development of cost-effective, highly active catalysts that can promote efficient electrochemical reactions at low temperatures without getting poisoned by chemical subproducts generated during the electrooxidation of the fuel [1, 2]. Thus, the synthesis of catalysts for fuel cells can be complex, as it requires precise control of the process to achieve the desired physicochemical properties.

Previous studies have reported the physicochemical and electrochemical properties of catalysts synthesized under varying conditions, including different amounts of water, ethylene glycol, and ethanol. These conditions influence key factors such as metal loading, alloying degree, and the formation of unexpected oxide phases in the catalysts. Moreover, the synthesis method itself can significantly impact the structure and composition of the obtained catalysts [3-6]. Also, in the polyol method, the temperature and heating duration are critical parameters; suboptimal control of these variables may result in incomplete precursor salt reduction, changes in the shape of the nanoparticle, or overgrowth of the nanoparticles, thereby affecting the catalytic activity of the nanomaterial [7].

Other authors replaced the traditional heating source (e.g., the stirring heating plate) with microwave irradiation systems. For example, Harish et al. [8] studied Pt-Vulcan-based catalysts synthesized using a commercial microwave oven to assist the polyol method, determining that the method allows the production of active catalysts for the Methanol Oxidation Reaction (MOR) with the expected metal loading and small particle size.

It also helps reduce synthesis time by heating the batch uniformly, with low energy usage. However, they concluded that this method needs to be studied regarding the batch temperature. Sharma et al. [9] also used a microwave heating

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source to synthesize Pt/C catalysts at different reaction times to evaluate the effects of the microwave irradiation over time, concluding that the irradiation time affects the porosity of the carbon support and the physical surface area.

Beyond conventional domestic-oven approaches, other microwave-assisted strategies have been explored for fuel cell electrocatalyst synthesis. Kustov and Vikanova [10] reviewed the application of microwave irradiation to the synthesis of mono-, bi-, and multi-metallic nanoparticles (including Pt, Pd, Cu, Ni, and Co), summarizing its advantages over conventional heating, such as faster and more uniform internal heating.

Karadeniz and Ayas [11] used a sealed, pressurized microwave reactor operating between 140 and 200 °C (1.0-7.5 bar) to synthesize Pt/C catalysts, finding that crystallite size increased nearly linearly with temperature, from 1.95 nm at 140 °C to 13.20 nm at 200 °C, with the highest electrochemical surface area (170.6 m^2 g^{-1} Pt) obtained at 160°C.

Zhang et al. [12] applied a one-pot microwave-assisted process to synthesize Cu/Co-based core–shell nanoheterostructures with tunable composition and size for combined methanol and ethanol oxidation. Feng et al. [13] developed a continuous pipeline microwave reactor for the mass production of Pt/C catalysts, reporting an electrochemical surface area of 103.2 m^2 g^{-1} Pt, a mass activity of 315 mA mg^{-1} at 0.9 V, and 75% retention after 30,000 durability cycles.

More recently, Li et al. [14] applied microwave-assisted polyol synthesis to Pd–Ni/C bimetallic catalysts for anion exchange membrane fuel cells, showing that polyol type and irradiation time govern nanoparticle dispersion and hydrogen oxidation activity.

The application of microwave-assisted heating in the polyol synthesis process reduces the synthesis time. This approach not only represents an improvement in efficiency and productivity but also advances toward more sustainable and environmentally friendly practices, as it reduces energy consumption and resources used in production [10-12].

However, there are several limitations regarding microwave-assisted synthesis, e.g., the limited penetration depth of microwaves may restrict their application to small-scale or low-volume synthesis, some materials may not be suitable for microwave irradiation due to their transparency or poor coupling with the electromagnetic field, which can result in uneven heating and potential degradation of the catalyst precursors [13].

In addition, the limited understanding of the fundamental mechanisms underlying microwave-assisted reactions can make it difficult to accurately predict and optimize reaction conditions. Furthermore, under certain conditions, the high temperatures and rapid heating rates involved in microwave-assisted processes may lead to the formation of hazardous byproducts or waste materials, potentially offsetting some of the environmental benefits associated with this method [15].

Independent of the specific catalyst system, the degree of control over the microwave irradiation has been addressed through different strategies. At the high end, fully automated platforms integrate professional-grade, software-controlled microwave modules for programmable synthesis under standardized protocols, but their cost and complexity restrict their use to well-resourced laboratories.

At the opposite end, domestic microwave ovens such as those used by Harish et al. [8] and Sharma et al. [9] rely on native, non-programmable timer and power controls. Low-cost, microcontroller-based automation of commercial microwave ovens has been reported outside the catalysis field, for example, an Arduino-controlled system for tissue decalcification [16], demonstrating that programmable, safety-controlled operation of a domestic oven is feasible at low cost. To the best of the authors' knowledge, this strategy has not been used for the polyol synthesis of fuel cell catalysts under microwave heating.

In this work, a low-cost approach involving the modification of a domestic microwave oven is presented for easy implementation in catalyst synthesis, aiming to reduce reaction times compared to the traditional polyol method. Three Pt/C catalysts are compared: a commercial one (D0), one prepared by the polyol method on a hotplate (D1), and one prepared by the polyol method under microwave irradiation (D2).

The resulting Pt/C catalysts were characterized using X-ray Diffraction (XRD), Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES), and electrochemical characterization for the Ethanol Oxidation Reaction (EOR) in acid media.

2. Materials and Methods

A 600 W domestic microwave oven was modified to provide heating during catalyst synthesis, and an electronic system was developed to precisely control the duration of microwave radiation via a Personal Computer (PC). This system included a custom-designed control circuit board and dedicated software. Figure 1 shows, on the left side, the traditional heating setup, and on the right, the modified microwave oven for the microwave-assisted method.

The detailed descriptions of the physical modifications, the PC microwave oven electronic interface, the developed control software, and the procedure for synthesizing Pt/C catalysts are outlined as follows.

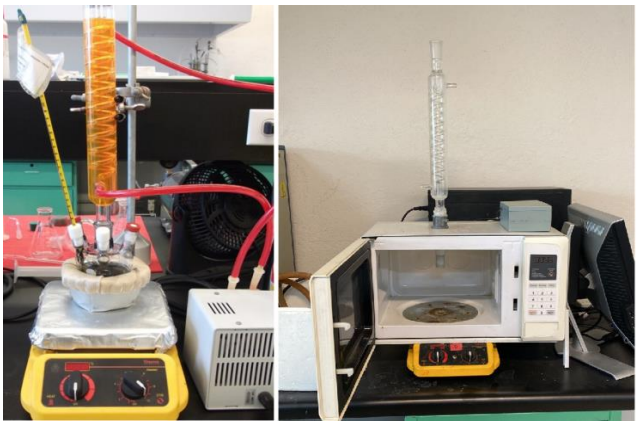


Fig. 1 Traditional heating vs. microwave irradiation setup

2.1. Physical Modification of the Microwave Oven

As Figure 2 shows, it was necessary to make a hole at the bottom of a 600 W microwave oven to match the diameter of a stirring hotplate. A nonmagnetic metallic patch was placed over this hole to enable the use of the hotplate's magnetic stirrer. Another hole was created at the top for inserting a Teflon bushing, which supports the reaction flask with a reflux condenser. The Teflon bushing was lathe-machined to allow attachment of the reflux condenser to the microwave oven and to provide a tight seal around the round flask to prevent any leakage of the synthesis solution.

2.2. Electronic Materials for Control Interface Board

The PC-Microwave interface board was designed to send control signals through the computer output interface. The software developed is responsible for sending these signals. The controlling circuit implemented, consisting of an optocoupler and a digital relay, was evaluated. The optocoupler fulfills two roles: first, it receives the on/off signal from the software, and second, it redirects this signal to the digital relay, which activates the internal energy control circuit of the microwave oven's magnetron.

The use of an optocoupler to interface low-power and high-power systems ensures safe and effective communication between them. Optocouplers function by using light to transfer signals while electrically isolating the input from the output, which protects the sensitive low-power components of the computer from the potentially damaging voltages and currents present in the high-power systems of the microwave oven.

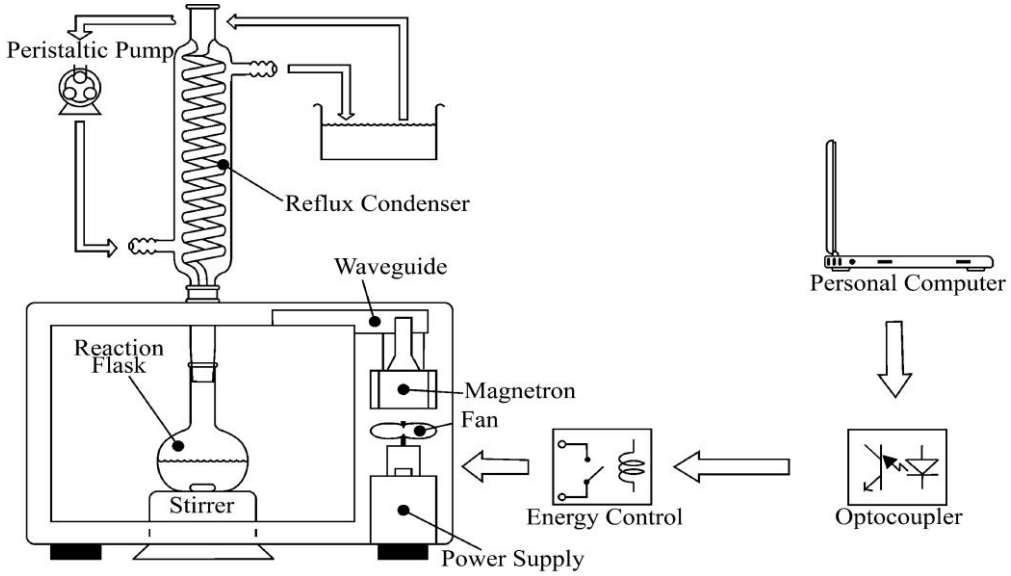


Fig. 2 Overall design of the modified microwave-assisted synthesis system

2.3. Control Software

Figure 3 shows the LabVIEW™ user interface developed to control the modified microwave oven during the catalyst synthesis. The system was designed to regulate the duration of microwave radiation according to user-defined parameters. The time-control algorithm implemented in the software is illustrated as a simplified flowchart in Figure 4.

At startup, the software begins by setting the output control signal to zero to prevent the activation of the microwave magnetron while resetting the circuit. It then enters a standby mode, awaiting instructions or the selection of the control method (Figure 4).

Once the Time option is selected on the main screen, all experimental parameters must be configured: the overall heating time, the duration of the initial heating pulse, the delay time, and the duration of the heating pulse. Pressing the Start/Stop button starts the heating cycle. The magnetron is activated during the initial heating pulse and then deactivated for the delay time; afterwards, heating pulses and delay times alternate until the overall heating time is completed (Figure 4).

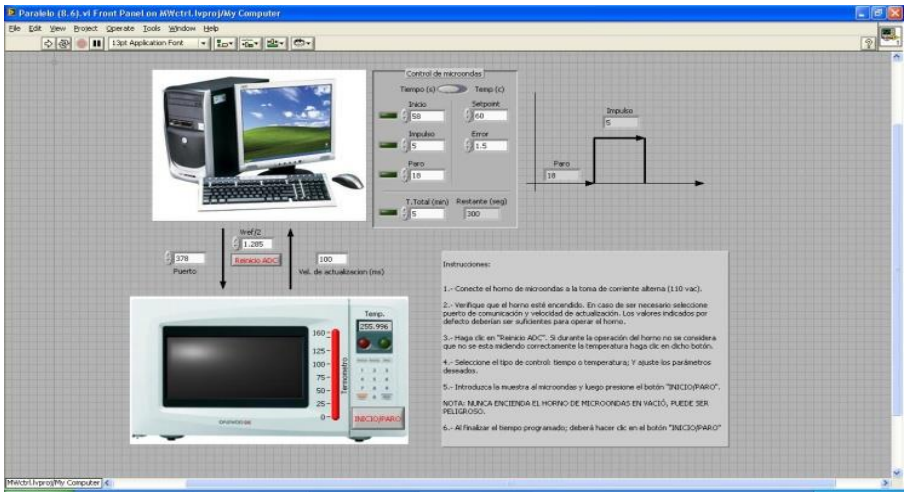


Fig. 3 Graphical user interface of the developed LabVIEW™ software

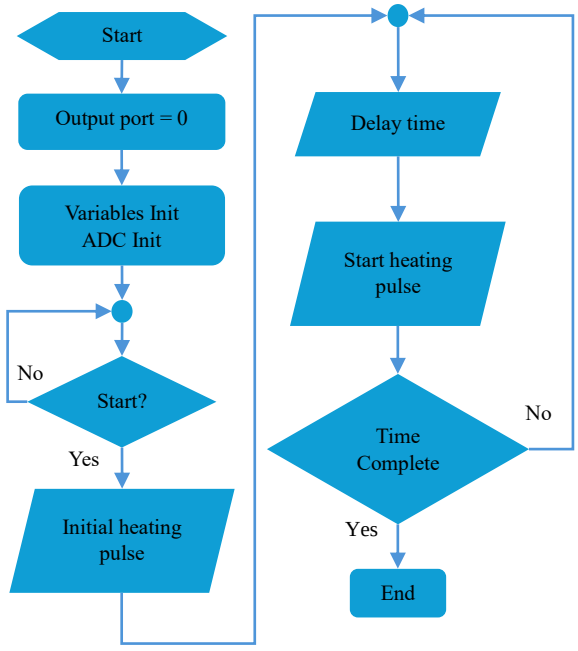


Fig. 4 Simplified flowchart of microwave time control system

2.4. Reactants and Gases

Chloroplatinic acid hexahydrate $H_2PtCl_6 \cdot 6H_2O$ (Sigma-Aldrich) was used as the Pt precursor and Vulcan XC-72® (Cabot Corp) as the carbon support. Their amounts were set to target a 20:80 wt-% Pt/C composition. Ethylene glycol (EG, $C_2H_6O_2$) served as a solvent and reducing agent.

NaOH and H_2SO_4 (Sigma-Aldrich) were used to raise and lower the pH, respectively, during the reduction, and C_2H_5OH and 2-propanol (Sigma-Aldrich) completed the reagents. All reagents were of analytical grade and were used as received. During the electrochemical tests, an inert atmosphere was maintained by bubbling N_2 (Infra Gas Company, >99.9% purity)-a commercial 20 wt.% Pt/C catalyst (E-TEK) served as the reference (D0).

2.5. Conventional Polyol Synthesis of Pt/C (D1)

The D1 catalyst was prepared following the traditional polyol synthesis method illustrated in Figure 5. The amounts of salts and reagents were used to achieve a Pt/C catalyst composition of 20:80 wt.%. Briefly, 63 mg of $H_2PtCl_6 \cdot 6H_2O$ was dissolved in 2 mL of water, and, separately, 96 mg of Vulcan XC-72® was dispersed in 48 mL of EG and sonicated for 30 min.

The platinum solution was then added dropwise to the carbon dispersion under constant stirring, which was kept until the end of the synthesis. After 1 h, the pH was raised to 12 with 1 M NaOH (dropwise), and the mixture was immediately heated to 130 °C and held for 3 h. It was then cooled to room temperature under stirring. After 3 h of cooling, the pH was lowered to 2 with 1 M H_2SO_4 (dropwise), and stirring continued for another 3 h. The solid was collected by vacuum filtration, washed extensively, and dried overnight in a desiccator before characterization.

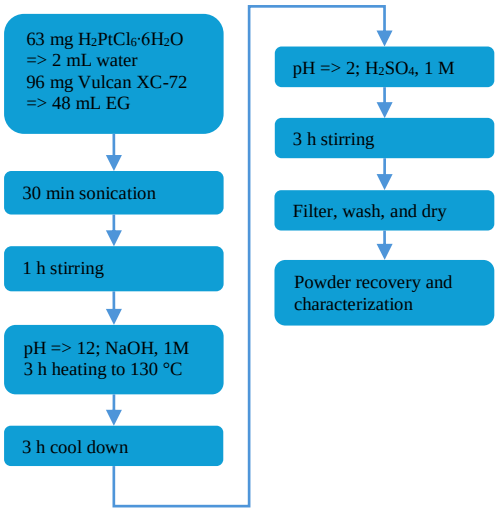


Fig. 5 Flowchart and setup of the synthesis of Pt/C with the traditional method

2.6. Microwave-Assisted Synthesis of Pt/C (D2)

The D2 catalyst synthesized by an Intermittent Microwave Heating (IMH) polyol process was obtained similarly to the procedure described above, using the same amount of reagents and EG: Water ratio (96:4 v/v). Figure 6 shows the flowchart of the procedure, where the heating hotplate was replaced with a microwave oven. The IMH procedure was conducted with an initial heating time of 58 seconds to reach ca. 130 °C, then the IMH was stopped for 18 seconds to avoid overheating. After this, the microwave irradiation was pulsed for 5 seconds to maintain a stable temperature of ca. 130 °C, and the cycle was repeated for 15 minutes. The time intervals used were determined experimentally.

2.7. Physicochemical Characterization

Structural characterization was carried out by X-ray Diffraction (XRD) on a Philips X'Pert diffractometer (Cu Kα radiation, λ = 1.5406 Å). Crystallite size was calculated from the diffraction patterns following the Scherrer and Bragg equations [17, 18]. ICP-AES determined the catalyst chemical composition independently.

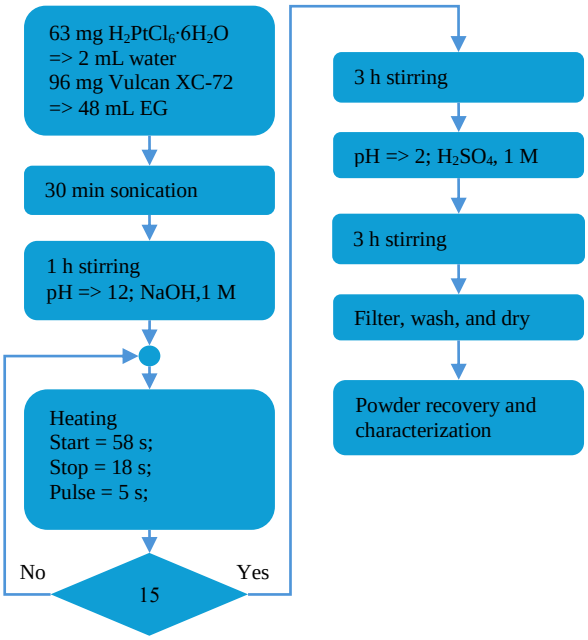


Fig. 6 Flowchart of the microwave-assisted synthesis of Pt/C

2.8. Electrochemical Set-Up and Characterization

Electrochemical measurements were carried out in a three-electrode cell connected to a VoltaLab PGZ 301 potentiostat/galvanostat. The working electrode was a glassy carbon disk onto which 10 µL of catalytic ink was deposited. The ink was prepared by sonicating 10 mg of Pt/C and 5 µL of Nafion in 1 mL of 2-propanol. A Pt foil and an Ag/AgCl electrode were used as counter and reference electrodes, respectively, and all potentials are given versus the Standard Hydrogen Electrode (SHE). The bare catalyst surface was first characterized by cyclic voltammetry in N_2-saturated 0.5 M H_2SO_4 (20 mV s^{-1}, 50–1200 mV vs. SHE). The Ethanol Oxidation Reaction (EOR) response was then recorded with the same scan rate and potential window after adding 0.5 M C_2H_5OH.

3. Results and Discussion

The 600 W domestic microwave oven was modified and tested with a solution containing ethylene glycol and water with a 96:4 v/v ratio in order to determine the starting and on/off steps of the irradiation time to reach and maintain the desired temperature (130 °C), resulting in an initial heating time of 58 seconds at starting and on/off steps of 5 and 18 seconds, respectively. The tests resulted in precise time control, leading to a uniformly heated solution without leakage or loss.

3.1. Physicochemical Characterization of the Catalysts

Figure 7 presents the XRD patterns of the catalysts. In all of them, the reflection at 26° (2θ) corresponds to the (002) plane of the Vulcan carbon support, and the reflections at ca. 40°, 46°, 67°, and 81° (2θ) are assigned to the (111), (200), (220), and (311) planes of fcc Pt (JCPDS #04-0802), respectively. The crystallite size estimated from the XRD patterns was 1.9 nm for all catalysts (Table 1).

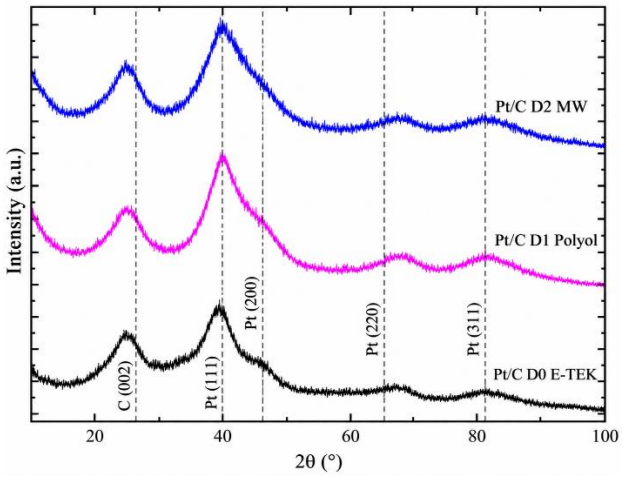


Fig. 7 X-Ray Diffraction patterns of Pt/C catalysts

The chemical composition of the catalysts obtained varied among the catalysts. The D0 and D1 catalysts were closest to the expected value, whereas the D2 catalyst exhibited a metallic loading of 16.67 wt.%. The crystallite size and chemical composition of the reported catalysts are summarized in Table 1.

3.2. Ethanol Oxidation Activity of the Catalysts

Figure 8 shows the CVs of the Pt/C catalysts in 0.5 M H_2SO_4. The Pt/C catalysts showed the typical Pt electrochemical profile [19]. The Electrochemically Active Surface Area (ECSA) was obtained using the charge of the

hydrogen desorption region (Q_{Hdes}), the Pt loading on the electrode (L_{Pt}), and the geometrical area of the working electrode [20].

Table 1. Physicochemical characteristics of the catalysts

Sample	Crystallite size (nm)	Chemical composition ICP-AES(Pt wt.%)
D0 (E-TEK)	1.90	19.12
D1 (Polyol)	1.90	18.41
D2 (MW)	1.90	16.67

The catalysts showed similar current densities associated with the H_2 adsorption/desorption region. The estimated ECSA values for D0, D1 and D2 catalysts were 55.42, 52.46, and 58.58 $m^2 g^{-1}$ Pt, respectively, indicating similar catalytic activity, but enhanced for D2. Table 2 summarizes the catalysts' ECSA characteristics.

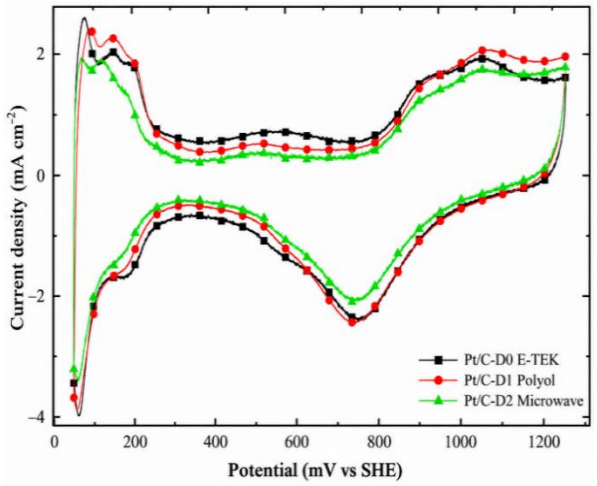


Fig. 8 CVs of Pt/C catalysts at 20 mV s^{-1} in 0.5 M H_2SO_4 under N_2

Figure 9 shows the catalytic activity for the EOR of the catalysts. The polarization curves of the EOR for the Pt/C synthesized samples showed a similar catalytic activity compared to the commercial Pt/C electrocatalyst.

However, the catalysts obtained with the microwave-assisted method (D2) showed a lower onset potential of the EOR (448 mV), and a higher current density in the forward scan (459.11 mA mg^{-1} Pt at 902 mV, see Table 3).

For the EOR, the catalyst D2 showed a slightly higher catalytic activity in comparison with the commercial reference (D0) and polyol procedure (D1). Beyond the electrocatalytic performance, the relevance of the IMH method lies in the accessibility of the synthesis platform itself.

Table 2. ECSA characteristics of the catalysts

Sample	Q_{Hdes} (µC)	L_{Pt} (mg Pt)	ECSA ($m^2 g^{-1}$ Pt)
D0 (E-TEK)	12.15	0.0204	55.42
D1 (Polyol)	10.86	0.0193	52.46
D2 (MW)	10.46	0.0166	58.58

Among the microwave-assisted routes to Pt/C catalysts discussed above, several rely on dedicated microwave synthesizers [9], sealed pressurized reactors [11], or continuous industrial-scale pipeline systems [13], all of which require specialized equipment beyond the reach of many research groups. In contrast, the PC-controlled modification presented here is built on a low-cost domestic microwave oven, yet still produces a Pt/C catalyst (D2) that matches the physicochemical properties with a slightly superior EOR activity, both the conventionally synthesized (D1) and commercial (D0) references. This indicates that precise, low-cost control of the microwave irradiation, rather than access to specialized high-pressure or industrial-scale equipment, is sufficient to obtain catalysts of comparable or superior quality for this application.

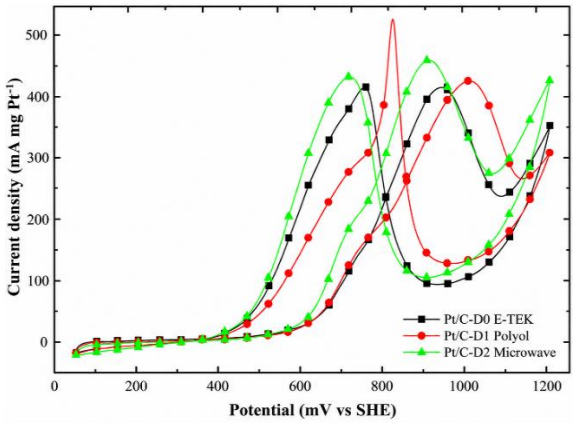


Fig. 9 EOR at Pt/C catalysts at 20 mV s^{-1} in 0.5 M H_2SO_4 + 0.5 M C_2H_5OH under N_2

Table 3. Electrocatalytic parameters of the EOR for each Pt/C catalyst

Sample	Onset potential (mV)	j_f (mA mg^{-1} Pt)
D0 (E-TEK)	449	416.80 at 930 mV
D1 (Polyol)	450	427.57 at 1002 mV
D2 (MW)	448	459.11 at 902 mV

4. Conclusion

A 600 W domestic microwave oven was successfully modified and interfaced with a personal computer. This modification allowed programmable control of the microwave irradiation time through an optocoupler and relay circuit and a LabVIEW™ interface. With Intermittent Microwave Heating (IMH), the heating step of the polyol synthesis was shortened from 3 h to 15 min, while the other steps were unchanged. The

IMH catalyst (D2) showed the same crystallite size (1.9 nm) as the conventionally synthesized (D1) and commercial (D0) catalysts.

Its Pt loading determined by ICP-AES was lower (16.67 wt.% vs. 18.41 and 19.12 wt.%), and its ECSA was 58.58 m^2 g^{-1} Pt, compared with 52.46 and 55.42 m^2 g^{-1} Pt for D1 and D0. For the EOR in acid media, D2 reached the mass activity of 459.11 mA mg^{-1} Pt, slightly above D1 (427.57) and D0 (416.80), with onset potentials within 2 mV of each other. The less defined Pt(200) reflection of D2 was not interpreted in terms of surface facet, since XRD alone does not establish the surface facet distribution.

The optimal heating-time intervals depend on factors such as volume, catalyst precursors, microwave oven power, and mixture ratio of the reduction solution, and must be determined experimentally for each system. These results indicate that a domestic microwave oven with programmable control is a viable platform for the laboratory-scale synthesis of Pt/C electrocatalysts.

Conflicts of Interest

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

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