

# Integrated Sensor for Detecting Hydrogen Concentration as Proxy for Ethylene and Propylene Production Using Pt–Fe and Pt–Cu Catalysts

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The increasing demand for ethylene and propylene has stimulated extensive research on propane dehydrogenation (PDH) for short-chain olefin production. However, catalyst deactivation and limited real-time monitoring remain key challenges in microwave-assisted PDH systems. In this study, we investigated waste-derived Pt-based bimetallic catalysts (Pt–Fe and Pt–Cu) and evaluated the feasibility of integrating real-time hydrogen sensing for in situ process monitoring. Waste Pt catalysts were recycled and modified with Fe and Cu, and combined with microwave-absorbing microparticles to enhance energy utilization and catalytic performance. Reactions were conducted at 450 W for 360 min with a propane feed rate of 10 mL/min. Hydrogen concentration was continuously monitored using a Siemens Calomat 62 sensor installed in the tail gas line, enabling the real-time evaluation of catalyst activity and deactivation behavior. This work presents a novel strategy by integrating sustainable Pt catalyst recycling with in situ hydrogen sensing for microwave-assisted PDH monitoring. The Pt–Fe catalyst produced 7.5% ethylene and 3.0% propylene with 69.4% hydrogen, whereas Pt–Cu exhibited superior performance with 9.5% ethylene, 11.9% propylene, and 78.8% hydrogen. SEM analysis revealed distinct coke morphologies associated with catalyst deactivation. The results demonstrate that combining bimetallic catalyst modification with hydrogen sensing significantly improves process monitoring and performance evaluation in microwave-assisted PDH systems. Further studies on long-term stability and scale-up are required for industrial application.

## 1. Introduction

Global economic growth has driven a steady increase in demand for ethylene and propylene, two of the most important building blocks in the chemical industry. Ethane and propane are valuable feedstocks for their production via catalytic dehydrogenation, supplying essential

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precursors for polymers, solvents, and other industrial chemicals.<sup>(1,2)</sup> In 2023, global ethylene demand reached 180 million tons with an annual growth rate of 3.4%, while propylene demand reached 132 million tons in 2025, growing at 4.2%.<sup>(3)</sup> The expansion of shale gas extraction has further increased the availability of low-cost ethane and propane, reinforcing the importance of efficient and selective dehydrogenation technologies.

Conventional methods such as steam cracking and fluidized catalytic cracking are increasingly unable to meet demand, whereas propane dehydrogenation (PDH) enables higher selectivity. However, the reaction is highly endothermic and requires elevated temperatures or reduced partial pressures to achieve efficient conversion.<sup>(4)</sup> PDH is promising in producing low-cost ethane and propane because the process involves converting light alkanes into short-chain olefins, such as ethylene and propane under catalytic conditions.<sup>(5)</sup> These conditions accelerate side reactions, including C–C bond cleavage, thermal cracking, hydrogenolysis, and coke formation, while larger catalyst particles are prone to rapid deactivation. Precision monitoring is therefore essential to suppress side reactions and maintain propylene selectivity, highlighting the role of advanced thermal and gas sensors in real-time process control.<sup>(6)</sup>

Platinum (Pt) catalysts are widely used for PDH owing to their strong reactivity, yet they suffer from poor selectivity and rapid deactivation through coke deposition and sintering.<sup>(7,8)</sup> Regeneration cycles are often required to restore activity, adding complexity to industrial processes. Alloying Pt with a promoter such as copper or iron can mitigate these drawbacks by altering electronic and geometric properties, improving olefin selectivity, and resistance to coking.<sup>(4,9)</sup> Recent studies on Pt<sub>3</sub>In<sup>(10)</sup> and sub-nanometer Pt–Zn<sup>(11)</sup> emphasize the need for advanced analytical tools to balance activity and selectivity. In this context, sensor materials capable of operating under harsh conditions are vital for monitoring reaction progress and catalyst health in real time.<sup>(12)</sup>

In this study, microwave-induced PDH was investigated using waste-derived Pt catalysts recycled from petrochemical sources and modified with Fe and Cu to form Pt–Fe and Pt–Cu bimetallic catalysts. Unlike most previous studies relying on newly synthesized Pt-based catalysts, in this work, we demonstrated the reutilization of industrial waste Pt as sustainable precursors for bimetallic catalyst design, providing a cost-effective and environmentally friendly strategy. In addition, unlike conventional studies based on *ex situ* or off-line analysis, we integrated a real-time hydrogen sensor as an *in situ* diagnostic tool for the continuous monitoring of reaction dynamics and catalyst deactivation behavior.

Microparticles with high dielectric properties were introduced to enhance microwave absorption and localized heating, thereby promoting C–H bond activation. Since hydrogen formation is stoichiometrically correlated with olefin production, real-time hydrogen monitoring provides an effective indicator of reaction progress and catalyst stability. Compared with conventional off-line gas analysis, the proposed sensor-assisted approach enables a faster detection of catalyst deactivation and dynamic changes during operation. Overall, by combining waste Pt catalyst recycling, bimetallic modification, and *in situ* hydrogen sensing, we established a novel and practical framework for microwave-assisted PDH systems, extending beyond conventional catalytic and diagnostic approaches.

## 2. Methods

### 2.1 Equipment and sensor integration

Microwave irradiation creates localized hot spots on the catalyst surface, supplying the energy required for C–H bond cleavage in propane. This rapid, volumetric heating was continuously monitored by an integrated hydrogen sensor (Siemens Calomat 62) (Fig. 1). Because microwave power was tightly controlled by the proportional-integral-derivative (PID) system, changes in hydrogen yield detected by the sensor directly reflected the efficiency of microwave-to-thermal energy conversion and the stability of the bimetallic catalyst. While gas chromatography (GC) provided detailed compositional analysis, its 60 min sampling interval was insufficient to capture transient changes in catalyst behavior. In contrast, the Calomat 62 sensor, operating on thermal conductivity detection (TCD), enabled the instantaneous quantification of hydrogen evolution.

In the experiment, the MOB-P23 microwave oven (SAMPO, Taiwan) was used as the energy delivery system for the bimetallic catalytic process. Operating at 2.45 GHz, the magnetron generates electromagnetic waves that penetrate the quartz reactor, enabling the selective dielectric heating of microparticles mixed with Pt–Fe or Pt–Cu catalysts. The oven was modified with a PID controller to ensure the precise regulation of microwave power.

The dehydrogenation of propane ( $\text{C}_3\text{H}_8 \rightarrow \text{C}_3\text{H}_6 + \text{H}_2$ ) proceeds with a 1:1 stoichiometry, allowing hydrogen concentration to serve as a real-time proxy for propylene production. This capability provided the immediate detection of catalyst coking or deactivation events that would otherwise be missed between GC sampling points.<sup>(13)</sup> The catalytic reactions were carried out in

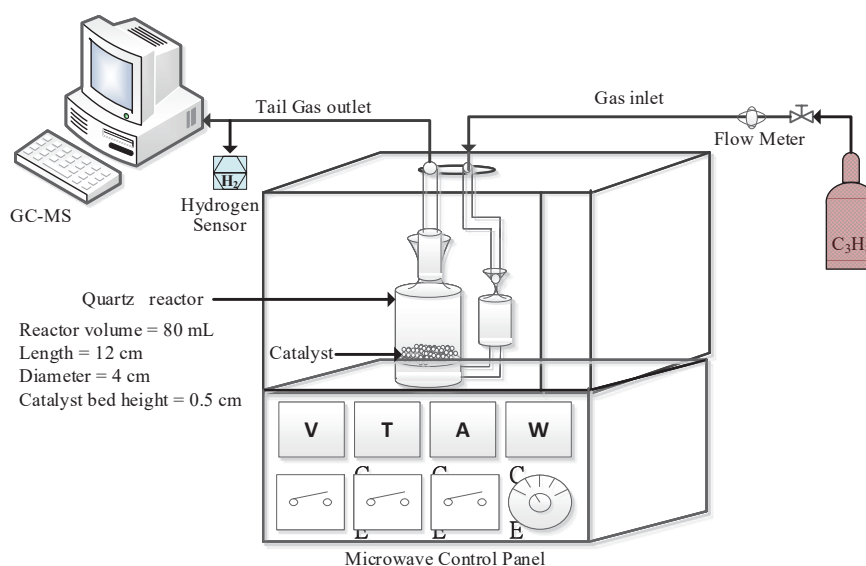


Fig. 1. (Color online) Equipment used in the experiment in this study [GC-TCD: gas chromatography-thermal conductivity detector, V: volt (V), T: temperature ( $^{\circ}\text{C}$ ), A: current (A), and W: power (W)].

a cylindrical quartz glass reactor (80 mL) placed inside the microwave-assisted reaction system shown in Fig. 1. The quartz reactor had a length of 9 cm and a diameter of 4.5 cm, whereas the catalyst bed height was fixed at 0.6 cm. To achieve uniform gas flow distribution, 40 perforations were constructed at the bottom section of the reactor. Platinum catalysts (0.3 wt% Pt) with initial coke contents of 1.4–1.5 wt% were reclaimed from oil-refining waste and modified for reuse, with particle sizes ranging from 1.5 to 1.6 mm.

The Calomat 62 sensor was used to measure heat loss from a heated element to the surrounding gas stream, translating conductivity changes into a continuous electrical signal. Owing to the large disparity in thermal conductivity between hydrogen (0.18 W/m·K), propane (0.018 W/m·K), and propylene (0.019 W/m·K), the sensor provides the accurate quantification of hydrogen concentration up to 100% without the delays inherent to GC analysis. In the microwave-induced catalytic process, real-time hydrogen monitoring ensured kinetic tracking and stability assessment. In addition, rapid declines in hydrogen evolution signaled catalyst deactivation or coke formation, offering critical insight into bimetallic catalyst performance under microwave irradiation.

The integration of the hydrogen sensor enables a closed-loop monitoring system. In simultaneously quantifying hydrogen and indirectly tracking propylene yield, the sensor's role is vital for catalyst evaluation and process optimization.

## 2.2 Pt catalyst modification to bimetallic catalysts

### 2.2.1 Pt–Fe catalyst

A 0.135 M ferric chloride ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ) solution was prepared, into which the waste Pt catalyst was immersed using the impregnation method. The mixture was agitated in a water bath at 150 rpm for 8 h at 60 °C. Metal ions were subsequently reduced using a 0.61 M sodium borohydride solution under ultrasonic agitation for 30 min. The resulting material was washed with ultrapure water, dried at 120 °C, and calcined at 500 °C for 3 h.

### 2.2.2 Pt–Cu catalyst

Pt catalyst modification with Cu was achieved by dissolving 0.23 g of copper chloride ( $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ ) in 100 mL of water (1000 ppm Cu solution). The waste Pt catalyst underwent an identical impregnation and reduction process as described in Sect. 2.2.1. Final thermal treatment included drying at 120 °C and calcination at 500 °C for 3 h.

## 2.3. Experiment and analysis

6g of the Pt–Fe or Pt–Cu was combined with 0.5 g of microparticles, which acted as microwave absorbers owing to their high dielectric constant (11.7 F/m) and dielectric loss (13.7 F/m). The reactor was insulated with cotton, with the iron particles placed beneath the catalyst layer to enhance microwave coupling. Propane ( $\text{C}_3\text{H}_8$ ) was introduced at a constant feed

rate of 10 mL/min, and the system was irradiated for 360 min under a fixed microwave power of 450 W. Gas samples were collected every 60 min using a 1 mL gas-tight syringe for gas composition analysis. Product distributions and individual component yields were determined on the basis of carbon mass balance calculations using Eq. (1), while product yields were quantified according to Eq. (2).

$$\text{Carbon balance: } [3 \times (F_{C_3H_8, outlet}) + \sum(n_i \times F_{i, outlet})] / (3 \times F_{C_3H_8, inlet}) \quad (1)$$

$$\text{Yield: } X_i / F_{C_3H_8} \quad (2)$$

Here,  $F_{in}$  and  $F_{out}$  are the volumetric flow rates of the feed and product gases, respectively,  $X$  is the amount of the outlet gas component,  $i$  represents the products  $C_3H_6$ ,  $C_2H_4$ ,  $CH_4$ ,  $CO$ , and  $CO_2$  in the outflow gas,  $n_i$  is the number of carbon atoms in component  $i$ , and  $F_i$  is the volumetric flow rate under standard temperature and pressure (STP).

The chemical compositions of organic intermediates and final products were analyzed using a GC-TCD system (Shimadzu GC-2014). Gas samples were extracted every 60 min in triplicate using a 1 mL gas-tight syringe. In parallel, hydrogen concentration was continuously monitored by using the Calomat 62 sensor that was integrated into the tail gas line for real-time sensor-based measurement. The equipment provided the minute-by-minute tracking of hydrogen evolution, serving as a dynamic proxy for reaction kinetics and catalyst stability. Coke accumulation on spent catalysts was determined using Eltra CS 800, an elemental analyzer that quantifies the coke (carbon) content deposited on the catalyst surfaces before and after the microwave-induced reaction, while surface morphology and structural changes were characterized by SEM (Hitachi SU8000). Using these analytical techniques, we precisely measured product distribution, hydrogen generation, and catalyst performance under microwave irradiation.

### 3. Results and Discussion

#### 3.1 Effect of Pt–Fe catalyst

In this study, 6 g of a Pt–Fe catalyst was employed under microwave irradiation at a fixed power of 450 W. Ethylene ( $C_2H_4$ ) yield decreased over time, while propylene yield remained low and stable, indicating limited selectivity toward propylene and progressive catalyst deactivation (Fig. 2). Pure propane was introduced at a feed rate of 10 mL/min, and the reaction was sustained for 360 min. The catalyst exhibited moderate activity, with an average ethylene yield of up to 7.5% and a relatively low propylene yield of up to 3.0%. The ethylene yield reached 10% but gradually declined over time, reflecting catalyst deactivation and coke accumulation. In contrast, propylene yield remained consistently low (3–4%), underscoring the challenge of achieving high selectivity toward propylene in Pt-based systems. These results highlight the need for catalyst optimization to balance activity and selectivity while mitigating coke formation.

Sensor data revealed a gradual decline in hydrogen concentration, consistent with the observed decrease in ethylene yield. This correlation validates the sensor's role as a kinetic

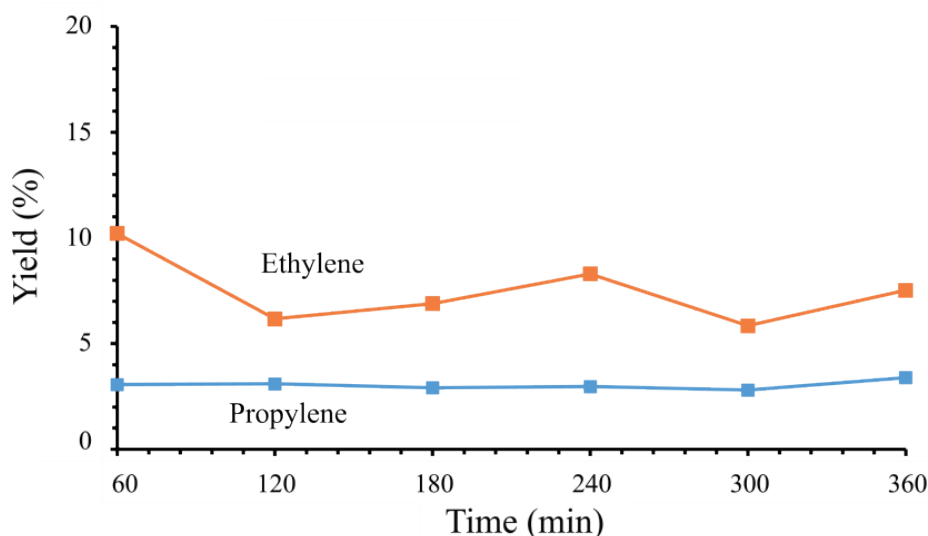


Fig. 2. (Color online) Ethylene and propylene yields during propane dehydrogenation over a Pt–Fe catalyst under microwave irradiation (450 W, 360 min).

proxy, confirming that real-time hydrogen monitoring can capture catalyst deactivation trends earlier than periodic GC sampling. The PDH reaction requires a highly active catalyst to promote the cleavage of the strong  $sp^3$  C–H bonds in propane (bond dissociation energy: 415.7 kJ/mol). The  $sp^3$  C–H bonds have four equivalent sigma bonds (the strongest type of covalent chemical bond) in a tetrahedral structure. These bonds are extremely stable and saturated, making their activation the challenge in producing ethylene and propylene. In contrast, the weaker C–H bonds in propylene (322.1 kJ/mol) make it susceptible to over-dehydrogenation, accelerating coke formation. Although platinum catalysts are active for C–H bond activation, they suffer from poor selectivity toward propylene and rapid deactivation due to coke deposition and sintering at elevated temperatures. To overcome these limitations, catalysts such as Fe are used to modify the electronic environment of Pt sites. This promotes the dispersion of Pt atoms, enhances C–H bond activation, and suppresses the formation of coke precursors.<sup>(14–16)</sup>

### 3.2 Effect of Pt–Cu catalyst

The modification of Pt catalysts with Cu significantly improved olefin selectivity and catalyst stability. This enhancement is attributed to the electronic effects of the Pt–Cu alloy, which weaken propylene adsorption and suppress over-dehydrogenation. In single-atom alloy configurations (Pt–Cu/ $Al_2O_3$ ), isolated Pt atoms surrounded by Cu stabilize surface-bound propylene, preventing its conversion to coke precursors and enhancing olefin selectivity while moderating overall dehydrogenation activity.

In this study, 6 g of the Pt–Cu catalyst was employed under microwave irradiation at 450 W for 360 min, with pure propane introduced at a feed rate of 10 mL/min. As shown in Fig. 3, the catalyst demonstrated superior performance compared with Pt–Fe, achieving average yields of

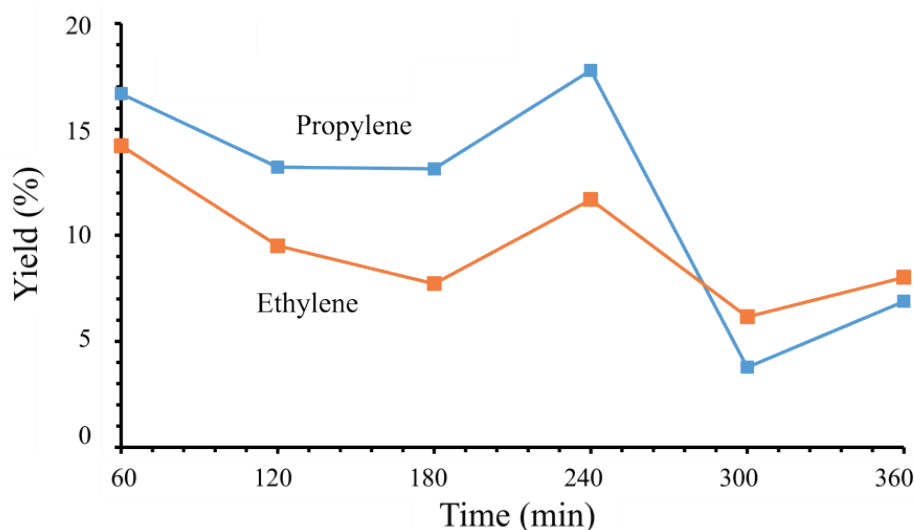


Fig. 3. (Color online) Ethylene and propylene yields during propane dehydrogenation over a Pt–Cu catalyst under microwave irradiation (450 W, 360 min).

9.5% for ethylene and 11.9% for propylene. The propylene yield was higher, peaking near 240 min before declining, while ethylene yield decreased from 15 to 8% and then partially recovered, reflecting dynamic changes in catalyst activity and stability.

The continuous monitoring of hydrogen concentration using the integrated sensor-based gas analysis system revealed that the Pt–Cu catalyst achieved a hydrogen concentration of 78.8%, compared with 69.4% for Pt–Fe. This improvement indicates that Cu incorporation promotes dehydrogenation pathways while suppressing undesired side reactions, enhancing olefin and hydrogen production. A summary of ethylene and propylene yields, along with hydrogen concentrations for Pt–Fe and Pt–Cu systems, is provided in Table 1.

The sensor-based monitoring revealed a significant disparity in steady-state hydrogen concentration between the two bimetallic systems. The sustained 78.8% hydrogen concentration observed for the Pt–Cu catalyst led to a layered, uniform carbon distribution, suggesting a more controlled cracking mechanism. In contrast, the lower and more erratic hydrogen levels recorded for the Pt–Fe system (69.4%) provided the early sensor-based evidence of the spherical carbon nucleation causing rapid active-site obstruction. This demonstrates that sensor technology is an indispensable tool for characterizing the performance and longevity of waste-derived catalysts.

### 3.3 Surface structural analysis of modified waste catalyst after microwave treatment

The surface morphology of the modified Pt–Fe catalyst after microwave treatment is shown in Fig. 4. The SEM image reveals spherical carbon deposits protruding from the catalyst surface, with gaps between particles. The average diameter was 30 nm, with the largest deposits reaching 38 nm. These features suggest localized carbon nucleation and growth, which obstruct active sites and contribute to catalyst deactivation. The SEM observations of coke deposition align with

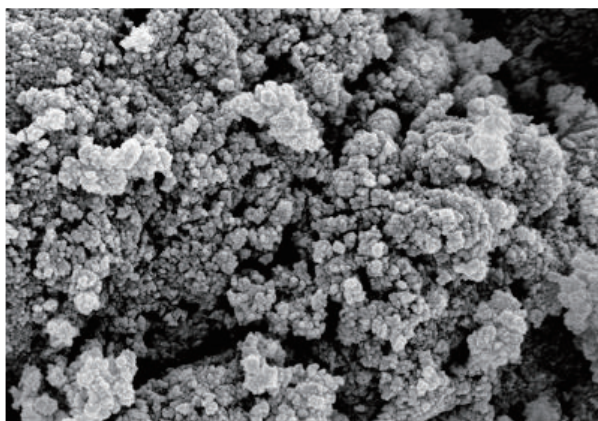
sensor-based hydrogen monitoring: spherical deposits in Pt–Fe corresponded to declining hydrogen signals, while layered deposits in Pt–Cu coincided with sustained hydrogen evolution. This synergy between sensor data and structural analysis highlights the importance of integrating sensor technology into catalyst characterization workflows.

In contrast, Fig. 5 illustrates the Pt–Cu catalyst after microwave treatment. Layered carbon deposits were observed, densely filling the interparticle gaps and forming a compact distribution

Table 1

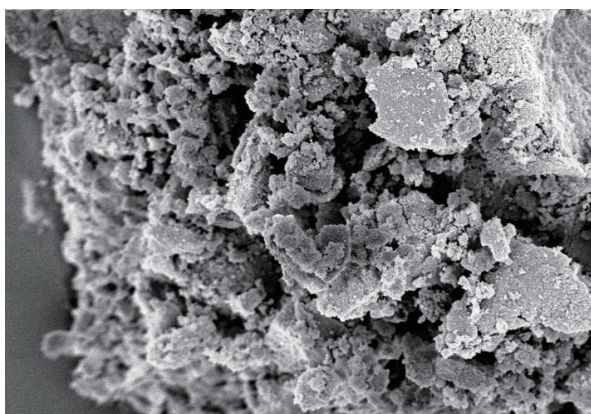
Catalytic performance of Pt–Fe and Pt–Cu with real-time sensor-based hydrogen monitoring.

Bimetallic catalyst	Ethylene yield (%)	Propylene yield (%)	Hydrogen concentration (%)	Sensor application
Pt–Fe	7.5	3.0	69.4	Integrated sensor-based gas analysis system
Pt–Cu	9.5	11.9	78.8	Integrated sensor-based gas analysis system



200 nm

Fig. 4. (Color online) SEM image of the Pt–Fe catalyst after microwave treatment, showing spherical carbon deposits protruding from the surface, with average particle sizes of approximately 30 nm. The scale bar represents 200 nm, and analysis was performed at 30000× magnification.



200 nm

Fig. 5. (Color online) SEM image of Pt–Cu catalyst after microwave treatment, showing layered carbon deposits densely filling interparticle gaps and forming a compact distribution. The scale bar represents 200 nm, and analysis was performed at 30000× magnification.

across the surface. This morphology reflects a different coke formation pathway, likely affected by the electronic properties of the Pt–Cu alloy, which alters hydrocarbon adsorption and cracking behavior. These carbon deposits originate from hydrocarbon cracking reactions occurring on the catalyst surface during propane dehydrogenation. While elevated temperatures increase reaction rates, they also accelerate carbon generation and accumulation. The distinct morphologies observed for Pt–Fe and Pt–Cu highlight that catalysts considerably affect coke formation mechanisms, catalyst stability, and long-term performance.

#### 4. Conclusion

We investigated microwave-induced PDH using waste-derived Pt–Fe and Pt–Cu catalysts enhanced with microparticles to improve microwave absorption and heat generation. While Pt catalysts are intrinsically active for C–H bond cleavage, they suffer from poor propylene selectivity and rapid deactivation due to coke formation and sintering. By alloying Pt with Fe or Cu, the electronic environment of Pt sites was modified, dispersing active atoms and suppressing coke precursor formation. The results demonstrated that Pt–Cu outperformed Pt–Fe in activity and selectivity. The Pt–Cu catalyst achieved average yields of 9.5% ethylene and 11.9% propylene, with hydrogen concentrations reaching 78.8%. In contrast, Pt–Fe produced 7.5% ethylene and only 3.0% propylene, accompanied by a lower hydrogen concentration of 69.4%. SEM analysis revealed distinct coke morphologies: spherical deposits on Pt–Fe correlated with rapid deactivation, while layered deposits on Pt–Cu were associated with sustained hydrogen evolution and improved stability.

The integration of a hydrogen sensor (Siemens Calomat 62) enables continuous, high-precision measurements of hydrogen concentration and the real-time monitoring of reaction kinetics, catalyst stability, and coke formation that conventional GC could not capture. The sensor integration also ensured the direct hydrogen quantification and indirect monitoring of propylene yield, transforming the experimental setup into a closed-loop monitoring system.

The catalytic potential of waste-derived Pt–Fe and Pt–Cu catalysts systems was validated in this study, and the sensor's critical role in advancing PDH technology was also confirmed. To introduce sensor technology into catalyst science, high-fidelity sensors need to be developed to enable efficient, selective, and sustainable olefin production under microwave irradiation. Future work will focus on long-term catalyst stability and large-scale reactor applications.

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