Fabrication of CeO_2 based materials by microwave assisted combustion synthesis for treatment of exhaust gas from plastic waste pyrolysis

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Received 5 Aug. 2025; Revised 25 Sep. 2025; Accepted 16 Oct. 2025; Published 18 Nov. 2025.
DOI: https://doi.org/10.54939/1859-1043.j.mst.IMBE.2025.22-28

ABSTRACT

This study presents the synthesis and performance evaluation of CeO_2-based catalysts for the treatment of exhaust gases generated from plastic waste pyrolysis. The catalysts were successfully prepared via a microwave-assisted combustion synthesis method, enabling rapid production of fine powders with high homogeneity. X-ray diffraction (XRD) analysis confirmed the formation of crystalline CeO_2 as the dominant phase, while scanning electron microscopy (SEM) revealed a porous microstructure, favorable for catalytic applications due to its high surface area and accessible active sites. Catalytic activity was assessed by monitoring the composition of pyrolysis exhaust gases before and after treatment, in accordance with the Vietnamese national emission standard QCVN 30:2012/BTNMT. The results demonstrated that CeO_2-based catalysts exhibited excellent removal efficiencies for hazardous gaseous species, including carbon monoxide (CO), nitrogen oxides (NO_x), hydrogen chloride (HCl), sulfur oxides (SO_x), and light hydrocarbons, particularly at elevated operating temperatures. Incorporation of a small proportion of noble metal significantly enhanced low-temperature activity, enabling efficient pollutant removal under milder conditions. Post-treatment measurements indicated that all regulated emissions were reduced to concentrations below the prescribed limits.

Keywords: Plastic waste pyrolysis; Exhaust gas treatment; CeO_2 based catalyst.

1. INTRODUCTION

The rapid accumulation of plastic waste has emerged as a critical environmental challenge due to its non-biodegradable nature and the limitations of conventional disposal methods. Pyrolysis has gained increasing attention as an effective thermochemical process for the conversion of plastic waste into valuable products such as liquid fuels, syngas, and chemical feedstocks [1, 2]. By operating in low-oxygen or oxygen-free environments, pyrolysis avoids complete combustion, thereby reducing direct CO_2 emissions compared with incineration. Furthermore, it enables resource recovery, minimizes landfill use, and offers flexibility in handling mixed or contaminated plastic streams that are unsuitable for mechanical recycling.

Despite these advantages, pyrolysis generates a complex mixture of gaseous by-products, many of which are hazardous to human health and the environment. These emissions may include carbon monoxide (CO), volatile organic compounds (VOCs), nitrogen oxides (NO_x), hydrogen chloride (HCl), sulfur oxides (SO_x), polycyclic aromatic hydrocarbons (PAHs), and various light hydrocarbons [3]. Without effective gas treatment, these emissions contribute to air pollution, toxicity, and potential corrosion of processing equipment. Consequently, the development of efficient exhaust purification technologies is essential to ensure the environmental sustainability of pyrolysis operations.

A variety of catalytic systems have been explored for hazardous gas removal from pyrolysis exhaust streams. Noble metals such as Pt, Pd, and Rh are widely used for oxidation of CO, VOCs, and hydrocarbons due to their high activity and selectivity [4]. Transition metal oxides have been applied for NO_x reduction and VOC oxidation owing to their lower cost and good redox properties [5].

Among oxide-based catalysts, cerium dioxide (CeO_2) has emerged as a particularly promising material for exhaust gas treatment. Its unique oxygen storage capacity (OSC), reversible Ce^{4+}/Ce^{3+} redox cycle, and strong interaction with other active phases enable efficient oxidation of CO, VOCs, and hydrocarbons, as well as decomposition of harmful chlorinated species [6]. CeO_2 also exhibits high thermal stability, making it suitable for the elevated temperatures typical of pyrolysis exhaust streams. The performance of CeO_2-based catalysts can be further enhanced by incorporating small amounts of noble metals (e.g., Pt, Pd, Rh), which improve low-temperature activity and resistance to poisoning [7].

Meanwhile, combustion synthesis (CS) offers an efficient alternative for producing metal oxide-based catalysts and ceramics, as it enables the rapid and straightforward fabrication of nanostructured materials with high specific surface area. Microwave-Assisted Combustion Synthesis, conducted in a microwave oven, further enhances the process by significantly reducing reaction times and promoting uniform heating of the reagent mixture. This homogeneous heating minimizes temperature gradients, thereby reducing the risk of crack formation in the final product. Owing to these advantages, MACS has become a preferred technique for material preparation, particularly for ceramics where structural integrity is critical.

2. EXPRIMENTAL

2.1. Materials

2.1.1. Catalyst synthesis

- Cerium nitrate $Ce(NO_3)_3.6H_2O$, glycine (NH_2-CH_2-COOH), Chloroplatinic acid (H_2PtCl_6) and Palladium(II) chloride ($PdCl_2$)

2.1.2. Pyrolysis process

- Plastic waste collected from Nam Son landfill (Soc Son, Hanoi) and Nghi Yen (Nghi Loc, Nghe An) and then classified, cleaned, cut into 1-3 mm pieces. The obtained plastic waste are the raw materials for pyrolysis process.

- Carbon black (Macklin) is used as a microwave absorber.

- HZSM-5 is used as catalyst.

- N_2 gas (99,99%).

2.2. Experiment preparation

2.2.1. Catalyst synthesis

Synthesis of CeO_2: The material was prepared using the microwave assisted combustion synthesis method, employing nitrate salts and a reducing agent (glycine). The nitrate salt was mixed with the reducing agent in the stoichiometric ratio, dissolved in a sufficient amount of water, and then placed in a microwave oven. The device was operated until the combustion reaction occurred, at which point it was stopped. The synthesis of CeO_2 employed glycine as reducing agents according to the following equations:

$$9Ce(NO_3)_3 + 14C_2H_5NO_2 = 9CeO_2 + 41/2N_2 + 28CO_2 + 35H_2O$$

Synthesis of Pt/CeO_2 and Pd/CeO_2: The Pt- and Pd-containing precursors (H_2PtCl_6 and $PdCl_2$, respectively) were dissolved in solution of cerium nitrate salt and glycine, with the calculated content of 10 wt%. The experimental procedure was carried out in the same manner as for the synthesis of CeO_2.

2.2.2. Pyrolysis process and exhaust gas treatment

In the reaction chamber, place cut-up plastic waste together with a carbon-based microwave absorber, ensuring the mixture is thoroughly homogenized. In the catalyst container, add HZSM-5 pyrolysis catalyst. To discharge the oxygen in the system, N_2 gas was blown into the reaction

chamber at a flow rate of 0.5 L/min. Turn on the microwave to react for 90 minutes; the power consumption is 500 W. Under the influence of temperature, plastics pyrolyzed into lighter products and also gases like CO, NO_x, HCl were formed. The exhaust gas from the equipment will be analyzed according to the parameters specified in QCVN 30:2012/BTNMT, including: total dust, hydrogen chloride (HCl), carbon monoxide (CO), sulfur dioxide (SO_2), nitrogen oxides (NO_x), mercury, cadmium, lead, as well as the total content of other heavy metals (As, Sb, Ni, Co, Cu, Cr, Sn, Mn, Tl, Zn), total hydrocarbons, and total dioxins/furans, in order to determine any parameters exceeding the permissible limits.

The gas generated by the equipment is subsequently directed through a treatment unit containing the synthesized CeO_2 catalyst. The unit is equipped with a heating system, with temperature precisely monitored by a thermocouple, in range of 100 - 400 °C. A small pump supplies air to the unit at a constant flow rate of 1 L/min, thereby promoting the catalytic oxidation of toxic gases. The efficiency of the exhaust gas treatment is determined by comparing the gas quality before and after treatment. The emission gas concentrations were determined using a Testo 350 XL analyzer (Germany).

3. RESULTS AND DISCUSSION

3.1. CeO_2 based catalyst synthesis

3.1.1. Catalyst composition

Photographs of the synthesized catalyst powders are shown in figure 1. The CeO_2 sample appears white, whereas the noble-metal-loaded samples exhibit a black or dark brown coloration.

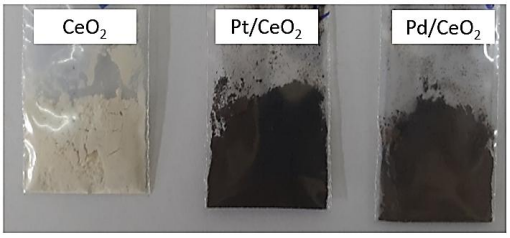


Figure 1. *Images of synthesized catalysts.*

The crystal structures of CeO_2, Pt/CeO_2, and Pd/CeO_2 were characterized using X-ray diffraction (XRD). The results are presented in figure 2.

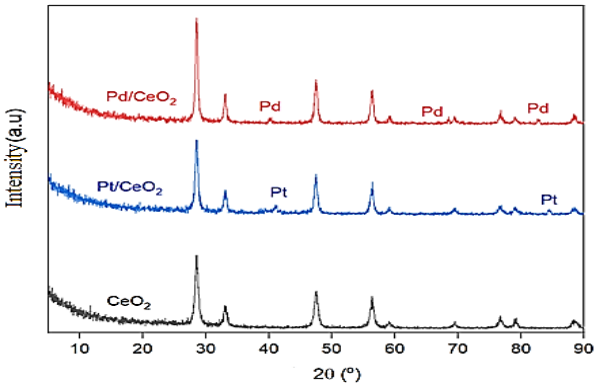


Figure 2. *X-ray diffraction pattern of synthesized catalysts.*

The XRD pattern of CeO_2 indicates a high degree of crystallinity, as evidenced by the sharp and intense characteristic peaks with narrow bases. The characteristic peaks at 2θ = 28.5°, 33°, 47.5°, and 56° are indexed to the (111), (200), (220), and (311) planes, respectively, of cubic CeO_2 crystal structure. The absence of peaks from other compounds confirms the successful synthesis

of pure CeO_2. For the Pd/CeO_2 sample, in addition to the characteristic peaks of CeO_2, additional peaks appear at $2\theta = 40°$, 68.5°, and 82.7°, corresponding to the (111), (200), and (311) planes of the Pd crystal lattice. Similarly, the Pt/CeO_2 sample exhibits distinct peaks at $2\theta = 41°$, 48°, and 84.5°, which are attributed to the (111), (200), and (311) planes of the Pt crystal lattice.

The incorporation of noble metals into CeO_2 was further confirmed by EDX analysis (table 1). The measured Pt content closely matched the nominal loading value of 10% determined from the experimental design. In contrast, the Pd-loaded sample exhibited a lower noble metal content than the theoretical value. This discrepancy is attributed to the relatively low solubility of $PdCl_2$, which partially crystallized during the water evaporation stage, thereby reducing the final Pd content in the product.

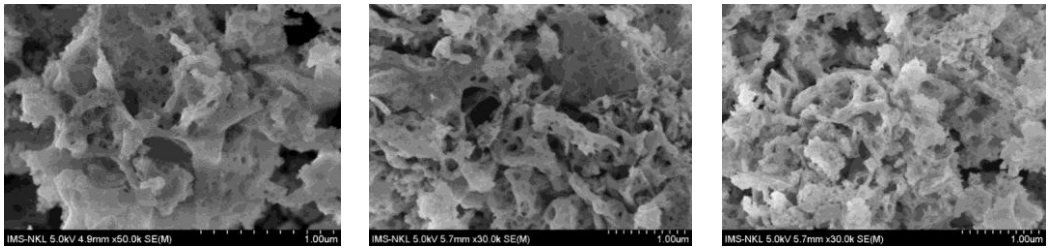
Table 1. *Element composition of synthesized catalysts.*

Sample	Element	Weight %	Atom %
CeO_2	O	22,63	71,92
	Ce	77,37	28,08
Pd/CeO_2	O	26,09	75,23
	Ce	69,82	22,99
	Pd	4,10	1,78
Pt/CeO_2	O	21,16	70,91
	Ce	68,75	26,31
	Pt	10,09	2,77

3.1.2. Catalyst morphology

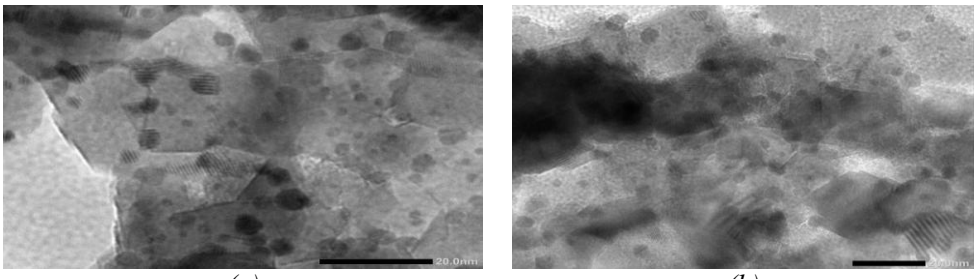
The morphological structures of CeO_2, Pt/CeO_2, and Pd/CeO_2 catalysts were examined using field-emission scanning electron microscopy (FE-SEM) (figure 3). The fabricated materials exhibit a thin, plate-like morphology with numerous surface pores, a characteristic feature of products synthesized via combustion methods, resulting from the release of a large volume of gases during the reaction.

The presence of noble metals was confirmed by transmission electron microscopy (TEM) (figure 4). The results showed that Pt and Pd particles are relatively uniformly dispersed on the CeO_2 surface, with sizes ranging from 2 to 5 nm.



(a) *(b)* *(c)*

Figure 3. *SEM images at 30.000 magnification of (a) CeO_2, (b) Pt/CeO_2, (c) Pd/CeO_2.*



(a) *(b)*

Figure 4. *TEM images at magnification of 600.000 (a) Pt/CeO_2, (b) Pd/CeO_2.*

3.2. Exhaust gas treatment

The pyrolysis gas was analyzed for key parameters in accordance with QCVN 30:2012/BTNMT, including: total dust, hydrogen chloride (HCl), carbon monoxide (CO), sulfur dioxide (SO_2), nitrogen oxides (NO_x), mercury, cadmium, lead, as well as the total concentration of other heavy metals (As, Sb, Ni, Co, Cu, Cr, Sn, Mn, Tl, Zn), total hydrocarbons, and total dioxins/furans. The analysis results of pyrolysis gas derived from collected plastic waste are as follows:

Table 2. *Exhaust gas compostion.*

Gas composition (mg/Nm^2)	Total dust	HCl	CO	SO_2	NOx	Hg	Cd	Pb	Total heavy metals	Total hydrocacbons	Total dioxin/furan (ngTEQ/Nm^2)
Measured	BDL	0	190	205	14,3	BDL	BDL	BDL	< 0,06	**302**	0,127
Permissable limit	100	50	250	250	500	0,2	0,16	1,2	1,2	50	1,2

**Note: BDL stands for below detection limit.*

As shown in table 2, the pyrolysis gases generally meet most of the requirements set by QCVN 30:2012/BTNMT. Notably, the concentrations of total dioxins/furans and NO_x are significantly lower than the permissible limits. However, the hydrocarbon (CxHy) concentration remains high. The presence of CxHy is attributed to the thermal cracking of polymers during pyrolysis, resulting in the formation of low-molecular-weight hydrocarbons. While CO and SO_2 levels comply with the standard limits, their concentrations are still relatively high. Specifically, the CxHy concentration reached 302 mg/Nm³ (six times the permissible limit), CO reached 190 mg/Nm³, and SO_2 reached 205 mg/Nm³.

The results presented in table 3 indicate a significant reduction in CxHy gas concentrations following catalytic treatment. For the CeO_2 catalyst, a temperature of 400 °C was required to achieve effective oxidation of CxHy, resulting in an outlet concentration of 48 mg/Nm², which complies with regulatory standards (< 50 mg/Nm²). In contrast, for the Pt/CeO_2 and Pd/CeO_2 catalysts, a lower temperature of 350 °C was sufficient to meet the required emission limits.

At the same operating temperature, the gas treatment efficiency of the catalysts follows the order: Pt/CeO_2 > Pd/CeO_2 > CeO_2. At 350 °C, the CxHy concentration decreased from 302 mg/Nm² to 60 mg/Nm² with CeO_2, 40 mg/Nm² with Pd/CeO_2, and 33 mg/Nm² with Pt/CeO_2. The enhanced performance of Pd- and Pt-containing catalysts can be attributed to their superior ability to interact with CxHy compounds, facilitating their adsorption onto the catalyst surface. This interaction prolongs the residence time of the reactants, thereby improving the overall treatment efficiency. The Pt/CeO_2 catalyst exhibited the highest efficiency, which is likely due to its higher platinum loading (10%) compared to the palladium content in Pd/CeO_2 (4%).

At lower temperatures (e.g., 100 °C), the catalytic systems still demonstrated a measurable degree of activity, with reductions in CxHy concentrations of 5% for CeO_2 and 18% for Pt/CeO_2. As the temperature increased, gas treatment efficiency improved markedly. At 350 °C, over 70% of the CxHy emissions were treated using CeO_2, and nearly 90% with Pt/CeO_2. Further temperature increases to 400 °C continued to reduce CxHy concentrations, but the improvements were marginal beyond 350 °C.

Additionally, table 3shows a considerable decline in CO and SO_2 concentrations at 350 °C. With the CeO_2 catalyst, CO concentration dropped from 190 mg/Nm² to 52 mg/Nm², corresponding to a 70% reduction. For the Pd/CeO_2 and Pt/CeO_2 catalysts, the CO concentrations decreased to 48 mg/Nm² and 40 mg/Nm², respectively—equivalent to 75% and 79% removal efficiencies.

Similarly, SO_2 removal efficiencies were 73% (CeO_2), 75% (Pd/CeO_2), and 80% (Pt/CeO_2). Based on these findings, the study concludes that Pt/CeO_2 and Pd/CeO_2 catalysts, when operated at 350 °C, are the most suitable for the treatment of pyrolysis gas emissions from plastic waste.

Table 3. *Exhaust gas composition after treatment.*

Catalyst	Temperature (°C)	CO (mg/Nm^2)	SO_2 (mg/Nm^2)	CxHy (mg/Nm^2)
CeO_2	100	167	180	288
	150	144	155	228
	200	129	139	168
	250	89	96	92
	300	55	59	74
	350	52	56	60
	400	50	53	48
Pt/CeO_2	100	143	154	248
	150	119	128	199
	200	87	93	145
	250	65	70	80
	300	41	44	59
	350	40	43	33
	400	39	42	28
Pd/CeO_2	100	160	172	275
	150	130	140	220
	200	121	130	160
	250	82	88	90
	300	50	53	65
	350	48	51	40
	400	47	50	39

4. CONCLUSIONS

Catalytic materials based on CeO_2 were successfully synthesized using a microwave-assisted combustion method**.** The resulting materials exhibited high purity and a porous structure, which is well-suited for catalytic applications. This synthesis method also proved effective in uniformly dispersing noble metals (Pt and Pd) onto the CeO_2 support. Furthermore, the combustion synthesis technique demonstrated high efficiency, as the actual loading of Pt closely matched the theoretical value.

Catalytic performance tests indicated that the synthesized materials possess good oxidation capability for the treatment of toxic gases generated during the pyrolysis of plastic waste. After treatment, pollutant gas concentrations were significantly reduced, with all emission indicators falling within the permissible limits set by QCVN 30:2012/BTNMT. Notably, the incorporation of noble metals enhanced the catalytic efficiency, enabling effective treatment at lower operating temperatures.

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TÓM TẮT

Chế tạo vật liệu nền CeO_2 bằng tổng hợp đốt cháy có hỗ trợ vi sóng để xử lý khí thải từ quá trình nhiệt phân chất thải nhựa

Nghiên cứu này trình bày quá trình tổng hợp và đánh giá hiệu suất của các xúc tác trên cơ sở CeO_2 nhằm xử lý khí thải sinh ra từ quá trình nhiệt phân rác thải nhựa. Các vật liệu xúc tác đã được chế tạo thành công bằng phương pháp tổng hợp đốt cháy hỗ trợ vi sóng, cho phép tạo ra bột mịn trong thời gian ngắn với độ đồng đều cao. Phân tích nhiễu xạ tia X (XRD) xác nhận sự hình thành của pha tinh thể CeO_2, trong khi ảnh hiển vi điện tử quét (SEM) cho thấy vật liệu có cấu trúc xốp, phù hợp cho ứng dụng xúc tác nhờ diện tích bề mặt lớn và nhiều vị trí hoạt hóa dễ tiếp cận. Hoạt tính xúc tác được đánh giá thông qua việc theo dõi thành phần khí thải nhiệt phân trước và sau xử lý, tuân theo quy chuẩn kỹ thuật quốc gia về khí thải QCVN 30:2012/BTNMT. Kết quả cho thấy các xúc tác trên cơ sở CeO_2 có hiệu quả xử lý cao đối với các khí độc hại như carbon monoxide (CO), các oxit nitơ (NO_x), hydro clorua (HCl), các oxit lưu huỳnh (SO_x) và các hydrocacbon nhẹ, đặc biệt khi vận hành ở nhiệt độ cao. Việc bổ sung một lượng nhỏ kim loại quý đã cải thiện đáng kể hoạt tính ở nhiệt độ thấp, giúp xử lý hiệu quả các chất ô nhiễm hiệu quả hơn. Các phép đo sau xử lý cho thấy nồng độ tất cả các chất khí nằm trong phạm vi cho phép theo quy định.

Từ khoá: Nhiệt phân nhựa; Xử lý khí thải; Xúc tác trên cơ sở CeO_2.