



Experimental evaluation of a dual-function solution for reducing flue gas pollutants using low-temperature counter-diffusive combustion of methane with $\text{Au}_{0.06}\text{-Pd}_{0.06}/\text{Al}_2\text{O}_3$ Catalyst

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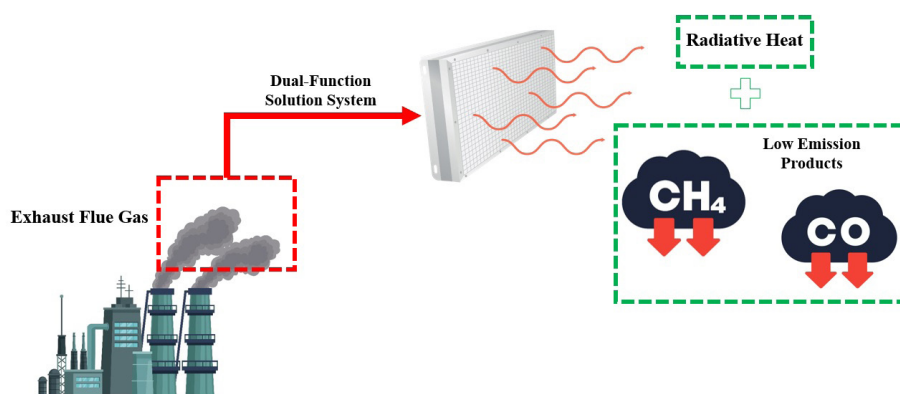
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HIGHLIGHTS

- Catalysts can be employed to reduce CH_4 emission from the exhausts.
- Feasibility of using Catalytic Flameless Combustion with $\text{Au}_{0.06}\text{-Pd}_{0.06}/\text{Al}_2\text{O}_3$ catalyst in a Dual-Function (Heating-Emission Control) application is investigated experimentally.
- Optimum performance along with low production cost are the main criteria of this research.

GRAPHICAL ABSTRACT



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ABSTRACT

The possibility of releasing unburned hydrocarbons and other compounds resulting from incomplete combustion from the exhaust is considered a common industrial issue. Pathing the exhaust gas through catalysts is a common way to lower the emissions. Alternatively, catalytic flameless combustion, used in a wide range of dilute air-fuel mixtures due to its optimal performance, is of interest for local radiative heating applications because of its low emission and high combustion efficiency (over 90%). However, using platinum catalysts in these systems increases production costs, and its surface temperature, ranging from 400-600 °C, faces a temperature limit in some applications. In this study, the feasibility of using a Pd-Au catalyst, with lower temperature and production costs, in a flameless catalytic counter-diffusive system was experimentally evaluated for the first time to reduce pollutants. A methane conversion rate of 98.3% in the 200-250 °C temperature range and a long life without CO and NO_x pollutants were observed. This not only leads to reducing environmental pollutants, but by producing low-temperature radiant heat, it also provides the possibility of producing heat from exhaust gases, making it a dual-function solution in the field.



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1. Introduction

Natural gas (NG), primarily composed of methane, is regarded as a crucial alternative to fossil fuels because it has a higher energy density and produces fewer CO₂ emissions. Nonetheless, residual methane released from NG vehicles, power plants, and various chemical industries poses a significant environmental concern due to methane's substantial global warming potential [1]. The structure of methane is such that its oxidation requires a temperature above 1000 °C in conventional combustion [2]. Hence, minimizing the release of unburned methane is a crucial objective. However, incomplete combustion at high temperatures generates pollutants like CO and NO_x. Catalytic methane combustion (CMC) at low temperatures can effectively decrease emissions of unburned methane, NO_x, and CO pollutants [3-4]. Noble metals and transition metal oxides have been employed as catalysts in methane combustion, with Pt or Pd catalysts demonstrating excellent performance at low temperatures [5].

The oldest catalytic burners are flameless catalytic heaters that were used for ambient heating in open camping spaces. In these types of systems, the fuel passes through a fiber structure whose surface is covered with a catalytic material and reacts with the penetrated ambient oxygen. This design method prevents the formation of flame in the heater's operation, which increases the system's safety. The history of using these systems goes back to 1958 in Germany when the Gogas company produced such burners [6]. Different sizes were produced for various ambient and local heating applications, ranging from about 10 cm × 10 cm for outdoor camp heaters to 200 cm × 60 cm for industrial shed heaters.

Catalytic radiant heaters are a type of heating system in which NG combustion occurs in the presence of a catalyst, even when dilute mixtures of fuel and air are used. Combustion in this type of heater is counter-diffusive, where fuel enters the catalytic layer from one side and the air from the opposite. The oxygen diffusion into the catalyst layer is a critical factor in controlling the heat produced and exhaust gas composition. Fig. 1 shows this process schematically. As can be seen, the direction of oxygen and fuel permeation in this system is opposite; therefore, this type of combustion is also called counter-diffusive. The operating method is fully described in section 2.4.

The most reported noble metals investigated in the literature are as follows:

- **Palladium (Pd):** The low heat resistance and high production cost have limited this catalyst's use in the industry [7]. However, in the absence of sulfur-containing compounds (H₂S, SO₂) in the fuel, the Pd catalyst shows better performance by increasing nitrogen compounds; Water/moisture leads

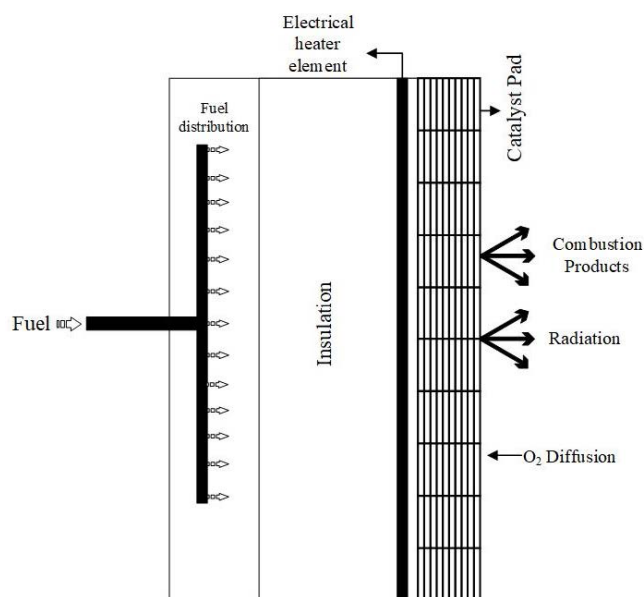


Fig. 1. Combustion Mechanism and Radiant Heat Production in a Counter-diffusive Combustion System.

to reversible inhibition, and carbon monoxide and carbon dioxide do not have a significant effect on its performance [8]. Regarding the catalytic combustion of methane, researchers have paid much attention to noble metals, especially Pd, due to the high activity of this catalyst and its appropriate conversion rate at low temperatures [1, 8-11]. Choudhary *et al.* showed that Pd/Al₂O₃ performs better than Pt/Al₂O₃ in dilute mixtures [13]. Burch *et al.* reported better activity for Pd/Al₂O₃ under lean conditions. They showed that although platinum catalysts are preferred to palladium to oxidize higher alkanes and alkenes, methane combustion is easier on palladium under the same lean fuel conditions [14]. Lyubovsky *et al.* investigated catalytic combustion in rich and lean fuel conditions, reporting that Pd is more active than Pt and Rh under dilute fuel conditions [15].

Yang *et al.* investigated the application of a Pd-Pt catalyst for the catalytic combustion of VOC in storage tanks and observed that under certain conditions, Pd can have more catalytic activity than Pt [16]. Wu *et al.* investigated porous Au-Pd alloy nanoparticles with Co₃O₄ support for methane combustion. In this method, they prepared gold-palladium catalyst samples based on meso-Co₃O₄ and PVA (MW = 1000 g.mol⁻¹), and reported that the best catalyst was 2.94Au0.50Pd/meso-Co₃O₄ with T_{90%} of 324°C [17]. Wang *et al.* investigated porous Au-Pd alloy nanoparticles with CoCr₂O₄ support for methane combustion, with the best catalyst reported as 1.93AuPd1.95/3DOM CoCr₂O₄ with T_{90%} of 394 °C [18]. Goodman *et al.* investigated Pt/Pd bimetallic nanocrystals and the effect of platinum on the activity and stability of methane combustion with palladium catalyst. They observed that in cases of flow without moisture (dry), adding Pt makes Pd work better, but when there is

water vapor, adding Pt reduces the efficiency [19]. Qi *et al.* investigated the reaction of methane combustion during the conversion of metal to metal oxide in the presence of Pd-Pt catalysts. It was observed that in bimetallic catalysts rich in Pd, the amount of active sites increased with increasing chemical potential of oxygen [20]. Chen *et al.* investigated the effects of Pd particle size in methane combustion and the structure-activity relationship of Pd catalyst on γ -alumina base. They reported that when particle size decreased, the activation energy and $T_{50\%}$ decreased [21].

• **Platinum (Pt):** It has a better combustion performance than palladium for combustion in stoichiometric conditions and the rich methane mixtures in feed [14]. Trim and Lam measured the combustion kinetics of platinum catalyst at a temperature lower than 800 K. Agglomeration of platinum and alumina was reported as one of the mechanisms of permanent deactivation of the catalyst [22]. Seo *et al.* studied the application of heating PVC tiles using platinum on alumina catalyst in three working modes: natural diffusion, forced diffusion, and premixed [23]. They found that the thermal load in the premixed state was twice as high as the other states, and the saving in energy consumption in the application of PVC tiles was reported to be 27.7%. Xiong *et al.* investigated the design considerations for hydrocarbon oxidation reactions at low temperatures on Pd-based catalysts. It was observed that the main reason for the improved performance of the Pt-Pd catalyst compared to the Pd-only catalyst is the oxidation of hydrocarbons in excess O_2 [24]. Mussio *et al.* investigated Pt-Pd bimetallic catalysts based on ceria with mechanical-chemical synthesis. They found that adding platinum had a negative effect on the light-off temperature, but for Pd/Pt molar ratios between 1:1 and 8:1, it improves the performance in wet conditions [25]. Saraev *et al.* investigated the atomic structure of catalysts based on Pd, Pt, and Pd-Pt in complete methane oxidation. In their study, 3% by weight Pd/ Al_2O_3 , 3% by weight Pt/ Al_2O_3 , and bimetallic (1% Pd + 2% Pt)/ Al_2O_3 catalysts were investigated in total methane oxidation in the temperature range of 150–400 °C. It was found that platinum and bimetallic palladium-platinum catalysts are more stable than palladium catalysts against sintering. Also, the "core-shell" structure for platinum and bimetallic palladium-platinum catalysts is stable in the temperature range of 150–400 °C [26].

The literature review revealed that Pd and Pt catalysts with a high loading percentage are mainly used in industries' exhaust. This use can dramatically increase production costs, and the resulting high reaction temperature is a limiting factor in some industries. Hence, the present paper aims to investigate the performance of a low-loading Au-Pd catalyst on the alumina support in the application of counter-diffusion combustion. In this regard, the catalyst samples were prepared

and tested. One of the most important goals of this research is to investigate the application of this combustion system for converting unburned hydrocarbons from flue gases and making the reaction temperature as low as possible. In other words, this paper examines the possibility of using a catalyst with the dual function of proper heating and low emission.

2. Experimental

2.1. Materials

The $H AuCl_4$ (tetrachloroauric(III) acid trihydrate, 99%) and $PdCl_2$ (palladium(II) chloride, ReagentPlus, 99%) were prepared by Merck and Sigma-Aldrich, respectively. Alumina fiber insulation (BET surface area $> 100 \text{ m}^2 \cdot \text{g}^{-1}$) as the catalyst support was employed for catalyst preparation.

2.2. Instruments

The elemental analysis was performed using ICP-OES with a VISTA-PRO model device to determine the catalyst percentage on the support. Moreover, FE-SEM (TESCAN MIRA\LMU) was employed to evaluate the catalyst particles' size and distribution. The collected combustion product samples were kept in gas sampling bags and analyzed using a gas chromatograph Younglin, YL-6100 GC. It should be mentioned that CO pollutants were measured by a TESTO 350 portable gas emission analyzer.

2.3. Catalyst preparation

First, 0.12 weight percentage of $H AuCl_4$ and 0.10 weight percentage of $PdCl_2$ were dissolved in 100 ml of distilled water for a 10 g support. Then, the immersion method was applied to layer the support. Lastly, the catalytic samples were dried for 12 h at 120 °C and calcined at 600 °C for 3 h.

2.4. Combustion setup

The setup shown schematically in Fig. 2 was used to investigate the performance of these catalysts. In this test, the gas and electric element for each panel was turned on, the panel temperature was allowed to reach about 100 °C, and then the element was turned off. By initiating catalytic combustion in the catalytic samples (temperature increment with a steep slope even with the element off), the optimal state for each catalyst was determined by adjusting the input flow. After the temperature was stable, the data were recorded for 30 min. Type K thermocouples, with an accuracy of 0.1 °C, were used to measure the temperature of the depth of the catalytic layer. These thermocouples were calibrated at five points: 100, 200,

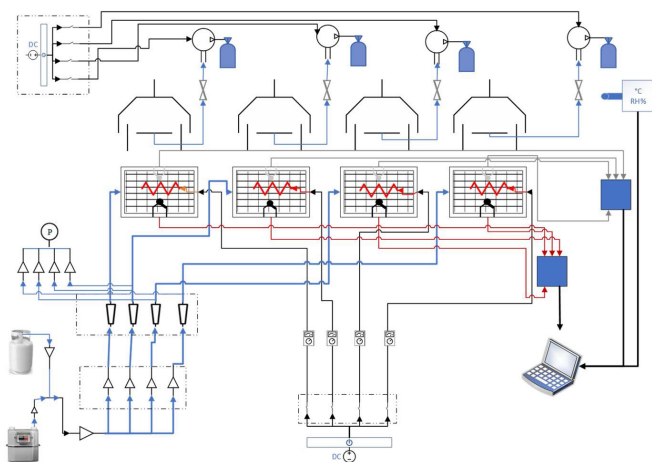


Fig. 2. Schematic of the setup for testing the performance of catalytic samples.

300, 400, and 500 °C. The surface temperature was measured at 5 points using a TES 1326S thermometer from Taiwan with an accuracy of 0.1°C. The combustion product samples were collected with the aid of a pump and from the hood section. The main test setup is shown in Fig. 3. As can be seen, all four catalytic samples could be run simultaneously. Hence, catalyst preparation was repeated four times to prepare four catalysts using the same procedure described above. It should be mentioned that each test was repeated three times to check the repeatability.

3. Results and discussion

3.1. Catalyst synthesis and characterization

To better spray the solution containing catalytic particles on the support, the solution was stirred using a FUNDAMIX® Vibromixer mechanical vibrator while spraying (Fig. 4).

To examine the repeatability of the fabrication process, four samples were prepared, and elemental analysis was conducted on all four samples. Table 1 represents the test

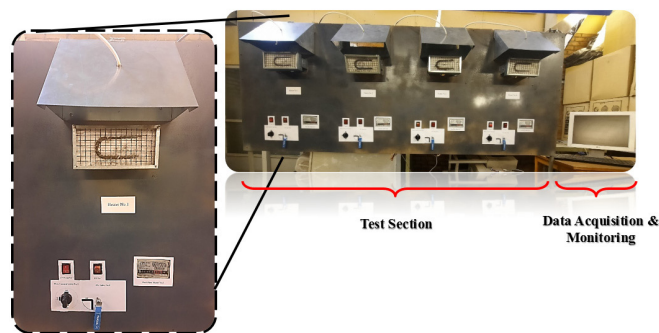


Fig. 3. Main setup for testing the performance of four catalytic samples simultaneously.

Table 1. ICP-OES results for four samples prepared with the same procedure.

Sample No.	Au wt%	Pd Wt%
1	0.052	0.055
2	0.049	0.062
3	0.054	0.059
4	0.064	0.053
Ave.	0.055	0.057

results for the prepared samples. The average weight percent of Au and Pd from the four prepared samples are 0.055% and 0.057%, respectively. Additionally, to examine the coating method and analyze the catalytic particles on the alumina fiber support, FE-SEM was employed before and after the performance test and after the aging test and the results will be discussed in sections 3.3 and 3.4.

3.2. Catalyst performance

After ensuring the proper distribution of the catalyst metals on Al_2O_3 support, the combustion performance of the prepared catalytic samples was investigated. The

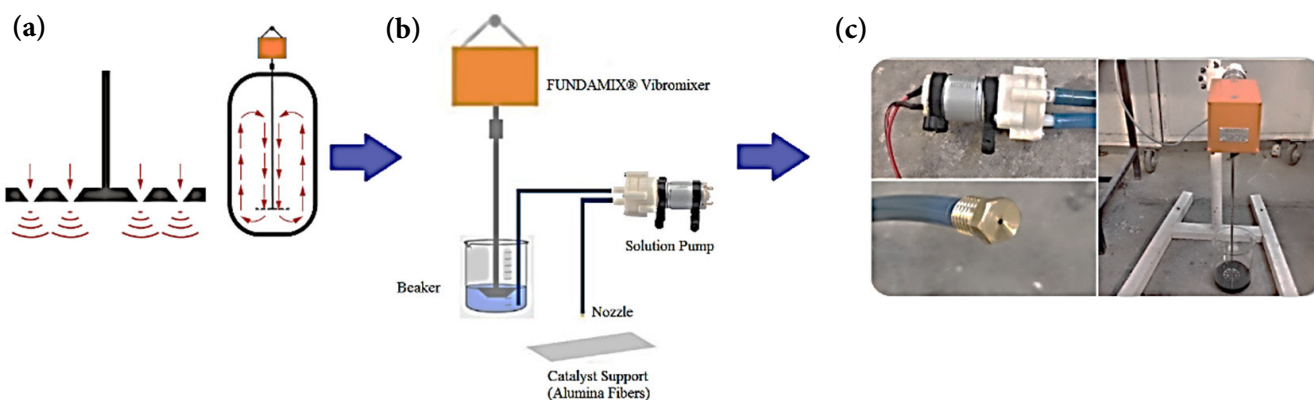


Fig. 4. The method of spraying catalytic solution on the support.

Note. (a) Circulation inside the beaker. (b) Schematic of the spraying setup. (c) The spraying setup's main parts.

comparison criteria in the combustion performance test include the internal temperature of the catalytic layer, the surface temperature of the panel, analysis of combustion products, and fuel consumption at steady state conditions. All measurements were repeated three times, and the average results for each sample are presented in this section. Fig. 5 shows the temperature inside the catalytic layer at 3 mm from the front surface for all four samples. It can be seen that the range of stable temperature in the depth of the catalytic layer is 230 to 265 °C, and this is true for all four samples.

Fig. 6 represents the average surface temperature for the four samples. As can be seen, the minimum temperature of 209 °C and the maximum temperature of 246 °C were recorded. In other words, a reliable surface temperature range of 200 to 250 °C can be considered. Due to the heat transfer with the surrounding environment, the surface temperature is about 20 °C lower than the depth of the samples.

Table 2 presents the analysis of combustion products during steady-state conditions. Sampling at the beginning, middle, and end of stable performance was collected and analyzed. The results show zero NO_x, ~1.5% CH₄, and <3 ppm CO.

Five natural gas analyses were used to calculate conversion efficiency, as reported in Table 3. The average methane in the samples is equal to 91.32%. The percentage of methane

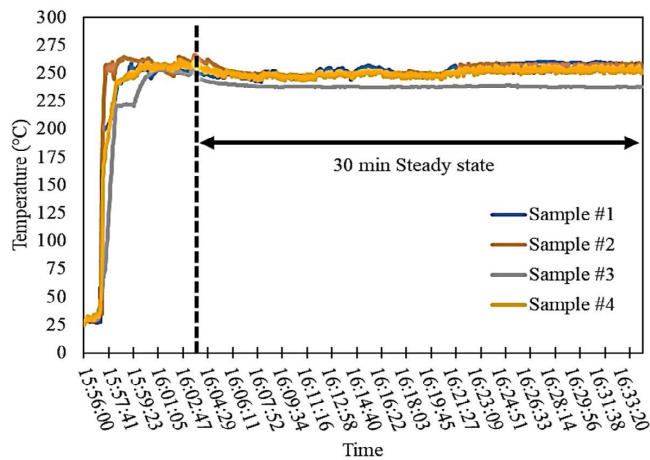


Fig. 5. The depth temperature of the catalytic samples at steady-state conditions.

Table 2. Analysis of combustion products during stable operations.

Sample	Start			Middle			End			Average		
	CH ₄ (%)	CO (ppm)	NO _x (%)	CH ₄ (%)	CO (ppm)	NO _x (%)	CH ₄ (%)	CO (ppm)	NO _x (%)	CH ₄ (%)	CO (ppm)	NO _x (%)
#1	1.50	1	0	1.57	1	0	1.55	2	0	1.54	1	0
#2	1.48	3	0	1.51	1	0	1.57	2	0	1.52	2	0
#3	1.42	2	0	1.44	0	0	1.49	0	0	1.45	1	0
#4	1.40	1	0	1.44	1	0	1.57	1	0	1.47	1	0

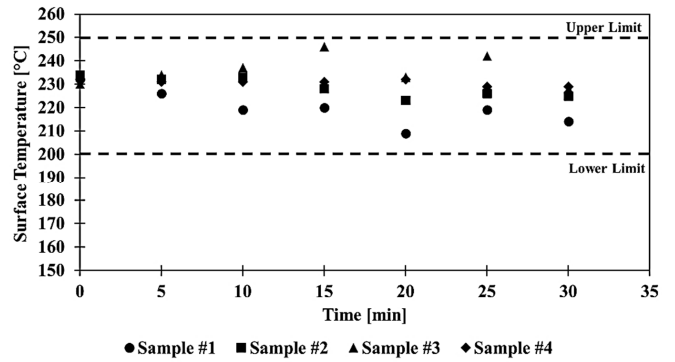


Fig. 6. Comparison of the average surface temperature for samples 1-4 in steady state conditions.

conversion rate is calculated according to Eq. (1) [27]. The results show a minimum conversion rate of 98.31%.

$$Conversion\ Rate = \frac{Inlet\ \%CH_4 - Outlet\ \%CH_4}{Inlet\ \%CH_4} \quad (1)$$

Table 3. NG characteristics from five different stations in tehran.

Component	Babayi	Rey	Shahriar	Takht Rostam	Vardavard
Methane	91.2	91.4	91.4	91.2	91.4
Nitrogen	3.4	3.6	3.5	3.5	3.5
CO ₂	0.9	0.9	0.9	0.9	0.9
Ethane	3.6	3.5	3.5	3.6	3.5
Propane	0.6	0.42	0.49	0.51	0.49
Water	0	0	0	0	0
H ₂ S	0	0	0	0	0
Hydrogen	0	0	0	0	0
CO	0	0	0	0	0
Oxygen	0	0	0	0	0
i-Butane	0.1	0.06	0.07	0.1	0.07
n-Butane	0.14	0.08	0.1	0.13	0.1
i-Pentane	0.03	0.02	0.02	0.03	0.02

The feed gas flow rate for the samples (10 cm × 20 cm) at steady state is described in Table 4. The feed gas consumption of these systems is presented per unit of panel surface area. Hence, the range of 1.90 to 2.05 standard liters per hour (at 1 bar pressure and 15.56 °C temperature) per square meter of catalytic panel can be considered.

Table 4. Feed gas flow rates for samples 1-4 in steady state conditions.

Sample	#1	#2	#3	#4
Flow rate (St. L.h ⁻¹)	0.040	0.038	0.039	0.041

3.3. Catalyst properties

The FE-SEM images of the prepared samples after the combustion test are shown in Fig. 7. The size of the particles was reported to be less than 100 nm, and there was no sign of sintering or significant accumulation of particles.

3.4. Catalyst ageing

Another critical parameter in evaluating the presented catalyst's performance is the catalyst's lifetime and the change of properties during operation. All four prepared catalysts were operated for six weeks (1000 h), and flow rates

and surface temperatures are presented in Figs. 8(a) and (b). Then, FE-SEM was used to observe possible changes in the structure. The results are shown in Fig. 9. As can be seen, the temperature range of 200 to 250 °C was maintained during the aging test, and no effect of particle sintering was reported during this period.

The analysis of combustion products at the end of the aging test is also presented in Table 5. The amount of NO_x is still zero, and the maximum amount of CO is reported to be 4 ppm. It should be noted that according to Eq. (1), the rate of methane conversion is still more than 98%.

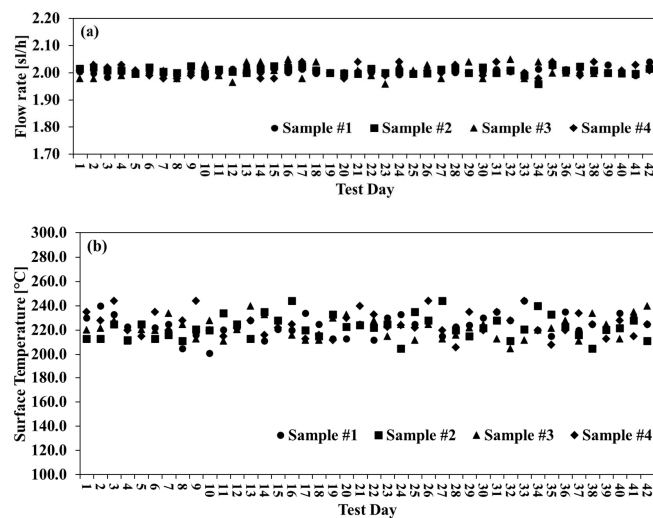


Fig. 8. Data for four samples during ageing test. Note. (a) Flow rate [St. L.h⁻¹], (b) Surface Temperature [°C].

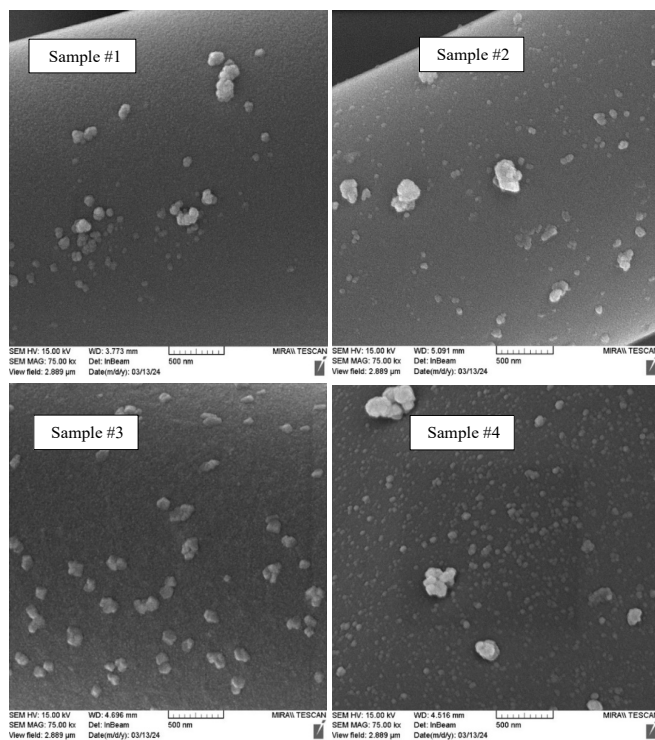


Fig. 7. FE-SEM images representing the distribution and size of the catalytic particles on the support.

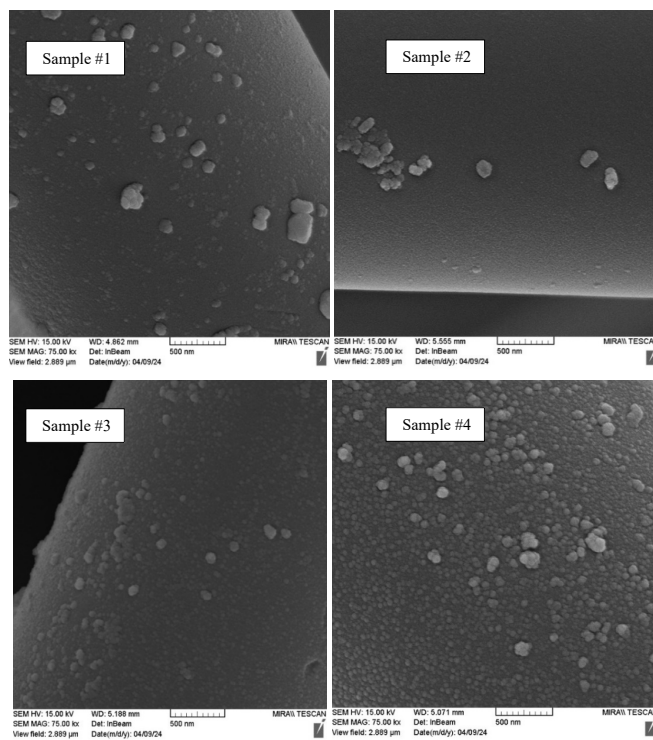


Fig. 9. FE-SEM images representing the distribution and size of the catalytic particles on the support after 1000 operational hours.

Table 5. Analysis of combustion products at the end of 1000 operational hours.

Sample	CH ₄ (%)	CO (ppm)	NO _x (%)
#1	1.51	2	0
#2	1.54	4	0
#3	1.49	2	0
#4	1.50	3	0

4. Conclusion

Purifying exhaust gases and preventing the release of unburned hydrocarbons into the atmosphere is one of the challenges of combustion industries. This research introduced a Pt replacement catalyst combining Pd and Au with low loading. Four samples of the catalyst were prepared, and their operation was evaluated. With a surface temperature distribution between 200 to 250 °C (a low and safe temperature source), the proposed catalyst showed potential for exploitation in industrial sites. The conversion rate of methane as the primary hydrocarbon was recorded as high as 98.31%. System performance was re-evaluated after 1000 h of continuous operation. The methane conversion rate and CO in the reaction products were 98.34% and less than four ppm, respectively. The FE-SEM images did not show a significant change in the structure even after continuous operation for 1000 h. The proposed catalyst can be used as a suitable and less expensive substitute for the platinum catalyst as an outlet filter for combustion industries. Hence, the proposed catalyst can be considered a dual-function solution for low-temperature heating and lowering emissions for industries with unburned hydrocarbons in combustion products.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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Additional information

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