

1 Low-Temperature Oxidative Removal of Benzene from the Air Using Titanium Carbide (MXene)- 2 Supported Platinum Catalysts

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10

11 Abstract

12 MXenes are an emerging class of two-dimensional (2D) inorganic materials with versatile application
13 potentials such as adsorption and catalysis. Here, we describe the synthesis of a platinized titanium carbide
14 MXene (Pt@Ti₃C₂) catalyst with varying amounts of platinum (0.1%–2 wt.%) for the low-temperature
15 oxidation of benzene, an aromatic volatile organic compound often found in industrial effluent. A 1%
16 formulation of Pt@Ti₃C₂-R achieved near-complete (97%) oxidation of benzene at 225 °C with a steady-
17 state reaction rate (r) of 0.119 mol·g⁻¹·h⁻¹. The low-temperature catalytic oxidation of benzene to carbon
18 dioxide was promoted by the lattice oxygen (active sites) of platinized catalysts attained through hydrogen
19 reduction pre-treatment of Pt@Ti₃C₂-R, as revealed by X-ray photoelectron spectroscopy, temperature-
20 programmed reduction, and in situ diffuse reflectance infrared Fourier transform spectroscopy analyses.
21 The oxidation capability of 1% Pt@Ti₃C₂-R against benzene was assessed under the control of the key
22 process variables (e.g., catalyst mass, flow rate, benzene concentration, relative humidity, and time-on-
23 stream) to help optimize the catalytic oxidation process. The results provide new insights into the use of
24 platinum-based 2D MXene catalysts for low-temperature oxidative removal of benzene from the air.

25 **Keywords:** MXenes; Catalytic oxidation; Benzene; VOCs; Air pollution control

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29 **1. Introduction**

30 Industrial emissions of volatile organic compounds (VOCs) in flue gases are a major contributor to
31 air pollution. Among various VOCs, benzene (a typical aromatic hydrocarbon) is of particular concern
32 due to its ubiquity and recalcitrant nature. Industrial sources of benzene include petroleum refining,
33 spraying, organic chemical production, coating, and the manufacturing of textile dyes, pharmaceuticals,
34 leather, pesticides, printing ink, electronic equipment, and adhesives (He et al., 2019). The sources of
35 benzene in indoor air include cosmetics, glass wool insulation, varnishes, paints, curtains, garments,
36 disinfectants, curtains, sterilizers, wall coverings, cigarette smoke, and other consumer goods (Brdarić
37 et al., 2019; Sekar et al., 2019). In light of the adverse health effects of benzene, a time-weighted (10 h)
38 average exposure limit of 0.1 ppm has been fixed by the U.S. National Institute for Occupational Safety
39 and Health (Kim et al., 2022).

40 Multiple treatment technologies have been developed to reduce the negative impact of VOCs in
41 industrial effluents, including adsorption (Li et al., 2020b; Vikrant et al., 2020), absorption (Xu et al.,
42 2021a; Lhuissier et al., 2022), membrane separation (Petrušová et al., 2019; Zhang et al., 2021), and bio-
43 filtration (Vikrant et al., 2017; Lelicińska-Serafin et al., 2019). Among the available options, thermal
44 catalytic oxidation is an effective and durable strategy for treating flue gases contaminated with VOCs
45 without generating hazardous by-products (Zhang et al., 2016; Kim et al., 2022). Supported noble metal
46 (e.g., platinum) catalysts typically display superior activity for the oxidation of most VOCs at relatively
47 low temperatures (< 300 °C) (Zhang and Zhou, 2021). Considerable effort has been devoted to
48 understanding how to fine-tune platinum active sites in support materials, such as metal-organic
49 frameworks (MOFs) (Bavykina et al., 2020), activated carbon (Xu et al., 2021a), metal oxides (Kim et
50 al., 2022), and graphene-based materials (Roy et al., 2018).

51 Two-dimensional (2D) layered transition-metal carbides (MXenes) have attracted considerable
52 research interest in various applications (e.g., energy storage, catalysis, and gas sensing) (Gao et al.,
53 2017; Kim et al., 2018; Nan et al., 2021). The number of research papers returned when searching

54 scientific libraries with the “MXene” keyword has grown dramatically since their first description in
55 2011 (Figure S1). In particular, titanium carbide (Ti_3C_2), a typical 2D MXene, is viewed as a promising
56 active catalyst and support for noble- and transition-metal sites (e.g., platinum, cobalt, and zinc) and
57 metal oxides (e.g., aluminum/copper(II) oxide [CuO] and titanium dioxide [TiO_2]) to accelerate catalytic
58 reactions (e.g., photocatalysis, dehydrogenation, oxygen reduction, thermocatalysis, and electrocatalysis)
59 (Obodo et al., 2020; Parse et al., 2021; Chu et al., 2022; Zhan et al., 2022; Cheng et al., 2023). Ti_3C_2
60 MXene offers abundant surface functional groups (e.g., hydroxyls) and active sites to construct a strong
61 heterojunction with doped metal and metal oxides (Ran et al., 2017; Cai et al., 2018).

62 Due to the high electron conductivity of MXenes, the strong interfacial coupling between Ti_3C_2 and
63 metal/metal oxides was found to improve the electronic structure and catalytic activity of MXene-based
64 catalysts (Chu et al., 2022). The heterojunction formation between MXene and loaded metal could help
65 improve charge separation to enhance the catalytic activity. For example, Ti_3C_2 nanosheets were used as
66 support for platinum- and nitrogen-doped TiO_2 nanoparticles (NPs) to construct a 3% Pt/N- $\text{TiO}_2/\text{Ti}_3\text{C}_2$
67 heterojunction with high photocatalytic activity toward one-pot hydrogenation and amidation of
68 nitroaromatics with carboxylic acid under visible irradiation at room temperature (RT) (Jiang et al.,
69 2021). Ti_3C_2 MXene support for Al/CuO energetic nanocomposites significantly improved microwave
70 combustion at low ignition temperatures in a relatively short time (Cheng et al., 2023). A Co-Zn/N-
71 carbon (NC)- Ti_3C_2 composite catalyst was also synthesized by one-step pyrolysis of zeolitic imidazolate
72 framework (ZIF)-67/ZIF-8 MOFs on a Ti_3C_2 surface to enhance oxygen reduction reactions in anion-
73 exchange membrane fuel cells (Chu et al., 2022). Density functional theory analysis predicted that Ti_3C_2
74 MXene would be an effective catalytic support that would enhance the catalytic activity of the platinum
75 surface toward the dehydrogenation of methylcyclohexane at elevated temperatures, relative to a pristine
76 platinum (1 1 1) surface (Obodo et al., 2020). A platinum-supported Ti_3C_2 catalyst was also utilized for
77 the oxidative removal of formaldehyde (oxygenated VOC) in the air at RT (Vikrant et al., 2023).

78 Pt@Ti₃C₂ composite catalysts can therefore be expected to display high activity for the low-temperature
79 oxidative removal of aromatic VOCs from the air.

80 In the present study, a wetness impregnation method (WIM) was applied to dope Ti₃C₂ MXene with
81 platinum (0.1% to 2 wt.%), followed by thermal reduction pre-treatment using hydrogen (H₂). The
82 developed Pt@Ti₃C₂ catalysts were characterized and tested for their ability to oxidize benzene (a model
83 aromatic VOC) at varying temperatures. The effect of process variables, including platinum loading,
84 catalyst mass (m_{cat}), flow rate (FR), benzene concentration, relative humidity (RH), and time-on-stream
85 (TOS), on the benzene oxidation reaction was also investigated in detail. The extent of benzene
86 mineralization was quantified by measuring the amount of carbon dioxide (CO₂) produced by the
87 catalytic reaction. In situ diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) was
88 utilized to evaluate plausible benzene oxidation pathways. To the best of our knowledge, this study is
89 the first to report on the application of Pt@Ti₃C₂ catalysts for low-temperature oxidative removal of
90 benzene from the air.

91

92 **2. Materials and methods**

93 *2.1. Materials and chemicals*

94 Chloroplatinic acid hexahydrate (H₂PtCl₆·6H₂O; ≥ 37.5% platinum basis), quartz sand (SiO₂), and
95 benzene liquid primary standard (L-PS; ≥ 99.8%) were purchased from Sigma-Aldrich (St. Louis,
96 Missouri, USA). Ti₃C₂ MXene powder (> 98%) was provided by Invisible Co., Ltd. (South Korea).
97 Nitrogen (N₂; 99.999%), H₂ (99.999%), and air (99.999%) cylinders were supplied by Union Gas Co.,
98 Ltd. (South Korea).

99

100 *2.2. Catalyst synthesis*

101 Platinum (0.1 wt%, 1 wt%, and 2 wt.%)@Ti₃C₂ MXene catalysts were synthesized using an IMP
102 protocol described previously (Vikrant et al., 2023). In brief, H₂PtCl₆·6H₂O (3.8, 38, or 76 mg) was
103 dissolved in 20 mL of deionized (DI) water (H₂O) under stirring for 15 min. Subsequently, 1 g of Ti₃C₂
104 was added to the prepared platinum solution and stirred for another 24 h at RT. The obtained suspensions
105 were then transferred to a convection oven (ED-CO150, Han Yang Scientific Equipment Co., Ltd., South
106 Korea) heated to 85 °C (15 h) to yield the 0.1 wt%, 1 wt%, and 2 wt% Pt@Ti₃C₂ catalysts. The
107 instrumental characterization of the synthesized catalysts is detailed in the Supplementary Information
108 (SI).

109

110 *2.3. Temperature-programmed reduction*

111 Temperature-programmed reduction (TPR) using hydrogen and carbon monoxide (CO) experiments
112 were performed to characterize the tested materials with an AutoChem II (Micromeritics Instrument
113 Corp., Norcross, Georgia, USA) apparatus. For these experiments, 80.4 ± 0.2 mg of 1%Pt@Ti₃C₂ and
114 1%Pt@Ti₃C₂-R (“R” indicates reduction pre-treatment) catalysts were treated by a flow (FR of 20 mL
115 min⁻¹) of molecular oxygen (5% in argon) at a heating rate of 10 K min⁻¹ up to 383.2 K for activation
116 and removal of weakly adsorbed species (CO₂ and H₂O). The treated samples were cooled to 303.2 K
117 under a 50 mL min⁻¹ argon flow to remove all physically adsorbed oxygen. TPR was carried out under
118 a flow of H₂ or CO in helium (0.10 mol mol⁻¹ and a total FR of 50 mL min⁻¹) at 10 K min⁻¹ up to 1,073
119 K and a constant pressure of 107.1 ± 1.2 kPa. During H₂-based TPR analysis, the reactor outlet was
120 subcooled using isopropyl alcohol at -80 °C to avoid the signal distortion of the thermal conductivity
121 detector (TCD) by the formed H₂O. During the CO-based TPR analysis, the TCD outlet signal was
122 corrected for the formation of CO₂ using the difference in thermal conductivity (SI).

123

124 *2.4. Catalytic activity*

125 For the catalytic experiment, three doses of the prepared catalysts (e.g., 2, 10, and 30 mg) were
126 physically mixed with SiO₂ (at a fixed weight ratio of 1:5) and packed in quartz tubes (90 × 4 mm)
127 between two layers of quartz wool. The fixed-bed catalyst tubes were pre-treated for 180 min at 300 °C
128 in an electric tube heater (model TC200P (K), Misung Scientific Co., Ltd., South Korea) under a flow
129 of 10% H₂/N₂ to reduce the catalyst and remove the pre-adsorbed moisture (Argyle and Bartholomew,
130 2015; Matsumoto et al., 2020). The benzene (547.9 ppm: assuming 100% recovery) gaseous primary
131 standard (G-PS) was prepared by injecting 4 μL of the benzene L-PS into a 2 L polyester aluminum
132 (PEA) bag (Top Trading Co., Ltd., South Korea) filled with N₂. The PEA bag was then left to stand
133 under ambient conditions overnight to ensure complete vaporization and stabilization of the benzene
134 vapor. The obtained G-PS of benzene was then diluted with air in a 20 L PEA bag to prepare a benzene
135 gaseous working standard (G-WS) of varying concentrations (1 to 100 ppm). For the experiments
136 conducted in the presence of moisture, suitable amounts of DI H₂O were injected into the benzene G-
137 WS PEA bags to obtain an RH between 0% and 80% by following the procedure described by works
138 (Liu et al., 2021; Tran et al., 2022)).

139 A schematic for the experimental setup for the catalytic oxidation of benzene is presented in Figure
140 1. The benzene G-WS PEA bag was connected to the reactor inlet while the outlet was linked to a mini
141 pump (MP-Σ300NII, Sibata, Japan) for pulling the G-WS through the catalyst bed at a fixed FR of 300
142 mL min⁻¹. An online gas chromatograph–flame ionization detector equipped with a methanizer system
143 (iGC-7200, DS Science Inc., South Korea) and a large-volume injector (PRG-2010, Shimadzu, Japan)
144 were used to quantify the benzene and CO₂ generated during the catalytic reaction at different time
145 intervals. During the thermocatalytic reaction, the reaction temperature increased at intervals of 25 °C
146 until oxidation of benzene vapor in the gaseous flow was complete. Measurements of benzene
147 conversion (R_{benzene} , %) and the steady-state reaction rate (r , mol g⁻¹·h⁻¹) were employed to evaluate the
148 catalytic performance (Vikrant et al., 2021; Kim et al., 2022; Vikrant et al., 2022) (Equation 1). The

149 value of r was computed using Equation 2 to evaluate the rate of chemical reaction on the catalyst surface.
150 F_{benzene} represents the benzene flowrate (mol s^{-1}).

151 The r calculated using Equation 2 is only valid when the reactor operates under differential conditions,
152 i.e., $R_{\text{benzene}} < 20\%$. Under the differential mode of operation, the reactant partial pressures remain
153 constant along the fixed bed quartz tube reactor. Hence, the benzene disappearance rate ($-r_B$) remains
154 constant under differential conditions inside the fixed bed reactor, irrespective of the position. The r
155 calculated using Equation 2 overestimates the true r under integral conditions, i.e., $R_{\text{benzene}} > 20\%$. As
156 the governing kinetic model and parameters were not derived in the present work, a simple r calculation
157 using Equation 2 was utilized for the performance comparison between similar experiments in relative
158 sense. (It should hence be noted the r obtained under integral conditions can overestimate the actual
159 performance.)

$$160 \quad R_{\text{benzene}}(\%) = \frac{(\text{benzene peak area})_{\text{in}} - (\text{benzene peak area})_{\text{out}}}{(\text{benzene peak area})_{\text{in}}} \times 100 \quad (\text{Equation 1})$$

$$161 \quad r = \frac{3,600 \times 10^3 \times R_{\text{benzene}} \times F_{\text{benzene}}}{m_{\text{cat}}} \quad (\text{Equation 2})$$

162 2.5. Light-off curves

163 The steady-state continuity equation for compound “i” in a packed-bed reactor is given by Equation
164 3, where X_B , $F_{B,0}$, and $r(X_B)$ represent the benzene conversion ($\text{mol}_B \cdot \text{mol}_{B,0}^{-1}$), the inlet molar flow rate
165 of benzene ($\text{mol}_{B,0} \cdot \text{s}^{-1}$), and the reaction rate for benzene total oxidation ($\text{mol}_B \cdot \text{kg}_{\text{cat}}^{-1} \cdot \text{s}^{-1}$), respectively.

$$166 \quad \frac{dX_B}{d m_{\text{cat}} / F_{B,0}} = r(X_B) \quad (\text{Equation 3})$$

167 The “space-time” ratio $m_{\text{cat}} / F_{i,0}$ (Heynderickx et al., 2009, 2010a) is expressed in $\text{kg}_{\text{cat}} \cdot \text{s} \cdot \text{mol}_{i,0}^{-1}$. The
168 apparent first-order reaction rate is calculated using Equation 4. The inlet O_2 concentration was
169 approximately 2,000 times greater than the inlet VOC concentration in the present work. The O_2
170 concentration was therefore assumed to remain constant, and the reaction was assumed to proceed in the

171 first order (concerning the substrate concentration) (Zhang et al., 2020; Zhao et al., 2021). The
 172 compound concentration can be related to the conversion efficiency and the corresponding inlet
 173 concentration, as described in Equation 5. The relationship between the conversion and space-time ratio
 174 is given in Equation 6.

$$175 \quad r = k_{app} C_B \quad (\text{Equation 4})$$

$$176 \quad C_B = C_{B,0} \cdot (1 - X_B) \quad (\text{Equation 5})$$

$$177 \quad X_B = 1 - \exp\left(-k_{app} \cdot C_{B,0} \cdot \frac{m_{cat}}{F_{B,0}}\right) \quad (\text{Equation 6})$$

178 If the concentration for compound “i” is expressed in $\text{mol}_B \text{ m}^{-3}$, the units of k_{app} (and also $k_{app,\infty}$) is
 179 $\text{m}^3 \text{ kg}_{cat}^{-1} \text{ s}^{-1}$. Equation 7 converts ppm_B into $\text{mol}_B \text{ m}^{-3}$ by assuming that the inlet flow conditions are
 180 established at RT. (24.464 is molar volume of gas (L mol^{-1}) at 25 °C)

$$181 \quad (C_B) = (\text{ppm}_B) \cdot \frac{10^{-3}}{24.464} \quad (\text{Equation 7})$$

182 For different isotherm conditions, the so-called “light-off curve” can be constructed based on
 183 Equation 6, where the conversion versus temperature can be considered using the Arrhenius relation
 184 (Equation 8).

$$185 \quad k_{app} = k_{app,\infty} \cdot \exp\left(-\frac{E_{app}}{RT}\right) \quad (\text{Equation 8})$$

186 The pre-exponential factor ($k_{app,\infty}$) and the apparent activation energy (E_{app}) are obtained from non-
 187 linear parameter estimates (Section S4), using the complete conversion versus temperature data set per
 188 catalyst: see Figure 2. The value of $k_{app,\infty}$ in the first-order reaction coefficient ($\text{m}_f^3 \text{ kg}_{cat}^{-1} \text{ s}^{-1}$) can be
 189 converted into s^{-1} through Equation 9, using the catalyst porosity, ε_p ($\text{m}_f^3 \text{ kg}_{pellet}^{-1}$; where “f” denotes free
 190 space and “pellet” refers to the catalyst, including the pores).

191
$$k'_{app,\infty} = k_{app,\infty} \cdot \frac{\rho_{cat} (1 - \varepsilon_p)}{\varepsilon_p} = k_{app,\infty} \cdot \frac{2}{r_{p,av} \cdot S} \quad (\text{Equation 9})$$

192 The units for the conversion are $(\text{kg}_{cat} \text{ m}_{cat}^{-3} \cdot \text{m}_{cat}^3 \text{ m}_{pellet}^{-3}) / (\text{m}_f^3 \text{ m}_{pellet}^{-3}) = \text{kg}_{cat} \text{ m}_f^{-3}$ (Section S5). For
 193 an irreversible reaction, the relationship between the pre-exponential factor ($k'_{app,\infty}$; s^{-1}) and ΔS^\ddagger (entropy
 194 difference between the activated complex and reactants) is given by Equation 10.

195
$$k'_{app,\infty} = \frac{k_B T}{h} \exp\left(\frac{\Delta S^\ddagger}{R}\right) \quad (\text{Equation 10})$$

196 The ΔS^\ddagger value contributes to the shift in equilibrium between the reactants and the activated complex
 197 through the usual thermodynamic relationship $\Delta G^\ddagger = \Delta H^\ddagger - T \cdot \Delta S^\ddagger$ (Vyazovkin, 2021). A positive value
 198 for ΔS^\ddagger suggests that entropy increases upon achieving the transition state, which often indicates a
 199 dissociative mechanism in which the activated complex is loosely bound. Negative values for ΔS^\ddagger
 200 indicate that entropy decreases on forming the transition state, which often indicates an associative
 201 mechanism in which two reaction partners form a single activated complex (Espenson, 1981; Akram et
 202 al., 2016).

203

204 3. Results and discussion

205 Direct thermocatalytic oxidation is an efficient technique for removing aromatic VOCs (e.g., benzene)
 206 from gaseous streams (air and flue gas). In the present section, the effect of high-temperature H_2
 207 reduction is evaluated with respect to the low-temperature catalytic behavior of Pt@Ti₃C₂ against
 208 benzene. In addition, the catalytic performances were studied at various process variables (e.g., platinum
 209 loading, m_{cat} , FR, benzene concentration, RH, and TOS). Figure 2 plots the light-off curves of the

210 benzene oxidation reaction to emphasize the differences in catalytic efficiency between the comparable
211 systems and the effect of platinum loading.

212

213 *3.1. Effect of high-temperature H₂ reduction pre-treatment on the activity of Pt@Ti₃C₂ catalysts*

214 To study the effect of pre-treatment on the catalyst, [Figure 2\(a\)](#) presents the light-off curves for a
215 benzene oxidation reaction over a 1% Pt@Ti₃C₂ catalyst (relative to pristine Ti₃C₂ MXene as a
216 “carrier/support”) before and after H₂ reduction “R” pre-treatment. The 1% formulation of Pt@Ti₃C₂-R
217 achieved higher catalytic activity compared with 1% Pt@Ti₃C₂ and pristine Ti₃C₂-R, as shown in [Figure](#)
218 [2\(a\)](#). From the calculated performance metrics in [Table 1](#), 1% platinum catalysts exhibited the highest
219 catalytic activity against benzene vapor at 200–225 °C. At 200 °C, the r value for benzene over 1%
220 Pt@Ti₃C₂-R was 0.012 mol·g⁻¹·h⁻¹, approximately 1.71 to 12 times higher than that for 1% Pt@Ti₃C₂
221 (0.007 mol·g⁻¹·h⁻¹) and Ti₃C₂-R (0.001 mol·g⁻¹·h⁻¹). The reduction pre-treatment of the platinum-loaded
222 Ti₃C₂ catalyst under high-temperature H₂ flow therefore had a significant promotional effect on the
223 catalytic performance due to support strong metal-support interaction, i.e., facile transfer of electrons at
224 the Pt-MXene interface ([Cui et al., 2020](#); [Li et al., 2022](#)).

225 The reduction pre-treatment was found to increase the amount of mobile lattice oxygen (vacant
226 oxygen centers) while decreasing the chemisorbed oxygen (O_{II}) over the reduced catalysts (as validated
227 later by X-ray photoelectron spectroscopy (XPS)). The platinum metal sites can promote the activation
228 of vacant oxygen sites and O_{II} species on the catalyst surface to synergistically interact with adsorbed
229 benzene molecules and accelerate the diffusion of active oxygen species (O*) from the subsurface or
230 bulk layers ([Yang et al., 2020](#); [Rosli et al., 2022](#)). The catalytic activity of Ti₃C₂-R can be ascribed to the
231 reaction between the benzene molecules and the O* on the surface at elevated temperatures as shown in
232 [Figure S10\(a\)](#). The consumed O* could be replenished by the O₂ in the reaction atmosphere ([Sun et al.,](#)

233 2015). As a result, platinum loading onto Ti_3C_2 and H_2 reduction pre-treatment of $\text{Pt@Ti}_3\text{C}_2$ can
234 synergistically improve the catalytic activity involved in benzene oxidation at low temperatures.

235

236 3.2. Effect of platinum loading and reaction temperature

237 The effect of platinum loading (0.1 wt.%, 1 wt.%, and 2 wt.%) on low-temperature benzene oxidation
238 was assessed using $\text{Pt@Ti}_3\text{C}_2\text{-R}$, as seen in Figure 2(b). The most active catalyst at low temperatures,
239 2% $\text{Pt@Ti}_3\text{C}_2\text{-R}$, completely oxidized the benzene (the temperature at 100% R_{benzene} (T_{100}) = 200 °C).
240 In contrast, the T_{100} increased to 225 °C and 350 °C when platinum loading was decreased to 1 wt.%
241 and 0.1 wt.%, respectively (Table 1). However, both 2% and 1% $\text{Pt@Ti}_3\text{C}_2\text{-R}$ displayed the same r (0.012
242 $\text{mol g}^{-1} \text{h}^{-1}$) at 200 °C, which was 2.4 and 12 times greater than those obtained by 0.1% $\text{Pt@Ti}_3\text{C}_2$ (0.005
243 $\text{mol g}^{-1} \text{h}^{-1}$) and Ti_3C_2 (0.001 $\text{mol g}^{-1} \text{h}^{-1}$), respectively (Table 1). The as-prepared 1% $\text{Pt@Ti}_3\text{C}_2\text{-R}$ had
244 the highest E value (Table S2), although it also had the highest k value. For r per m_{cat} , 1% $\text{Pt@Ti}_3\text{C}_2\text{-R}$
245 (0.012 $\text{mol g}_{\text{cat}}^{-1} \text{h}^{-1}$) and 2% $\text{Pt@Ti}_3\text{C}_2\text{-R}$ (0.012 $\text{mol g}_{\text{cat}}^{-1} \text{h}^{-1}$) performed better than other catalysts
246 (Table 1). For the r per Pt mass, 0.1% $\text{Pt@Ti}_3\text{C}_2\text{-R}$ (3.96 $\text{mol g}_{\text{cat}}^{-1} \text{h}^{-1}$) was the best performer (Table 1).

247 In Table S2, the positive value of ΔS^\ddagger ($16.3 \pm 9.8 \text{ kJ mol}^{-1} \text{ K}^{-1}$) between the activated complex and
248 reactants for the 1% $\text{Pt@Ti}_3\text{C}_2\text{-R}$ catalyst may comply with higher mobility of the surface oxygen
249 species, resulting in a more straightforward oxidation reaction by inserting O^* species. However, the
250 0.1% $\text{Pt@Ti}_3\text{C}_2\text{-R}$ catalyst had a low E and a non-favorable activated complex: see Equation (10). (As
251 the surface reaction intermediate(s) can be considered to be more tightly fixed to the catalyst surface, it
252 is less prone to further oxidation or total oxidation product formation such as H_2O and CO_2 .) Lastly, the
253 2% $\text{Pt@Ti}_3\text{C}_2\text{-R}$ catalyst displayed an active complex with a near-zero ΔS^\ddagger value. In this case, the
254 activated complex for total oxidation (the surface intermediate(s)) may have an entropy value
255 comparable to the reactants. ~~The entropy difference can reflect the adsorption behavior of benzene onto~~

256 ~~the catalyst surface. Strong adsorption is accompanied by a substantial reduction in entropy of the~~
257 ~~benzene surface species, whereas weak adsorption exhibits the opposite behavior. Although 2%~~
258 Pt@Ti₃C₂-R outperformed all the other tested catalysts for low-temperature benzene oxidation, 1%
259 Pt@Ti₃C₂-R was selected for further study due to its lower cost and the similarity of its catalytic activity
260 to that of the 2% catalyst in the 200–225 °C range.

261

262 3.3. TPR results

263 The role of reduction pre-treatment in catalytic activity was evaluated using TPR analysis (Figures 3
264 and 4). Table S3 summarizes the corresponding TPR data of 1% Pt@Ti₃C₂ (relative to Ti₃C₂) as a
265 function of temperature. The peak temperatures can be considered a measure of surface reducibility: the
266 smaller the temperature, the easier it is to consume the corresponding active oxygen surface species. The
267 occurrences of weakly bonded species (O²⁻, O⁻...) were seen with low(er) reduction temperatures (Panov
268 et al., 2006). In contrast, the lattice oxygen consumption for reduction normally corresponded to high(er)
269 reduction temperatures. (In this case, the oxygen is part of a lattice to consume more energy.) As
270 approximately 80 mg of the catalyst is commonly used in TPR experiments, the total available surface
271 oxygen (C_o: mol_O kg_{cat}⁻¹) can be approximated as 10⁻¹ mol_O kg_{cat}⁻¹ during reduction (entries 1 and 4 in
272 Table S3). These values were comparable to those obtained through TPR analysis of other metal-oxide
273 catalysts studied previously (Heynderickx et al., 2010b).

274

275 3.3.1. CO-based TPR and H₂-based TPR

276 In the present study, 1% Pt@Ti₃C₂ and 1% Pt@Ti₃C₂-R exhibited significantly higher oxygen storage
277 capacities during CO-based TPR and H₂-based TPR analyses than did Ti₃C₂. In the case of the CO-based
278 TPR, reduction pre-treatment of 1% Pt@Ti₃C₂ achieved a slight improvement, increasing the C_o value

279 from 4.13 ± 0.04 to 4.84 ± 0.03 mol_O kg_{cat}⁻¹ for 1% Pt@Ti₃C₂ and 1% Pt@Ti₃C₂-R, respectively (Table
280 S3). Comparatively, the C_o value of the 1% Pt@Ti₃C₂-R catalyst ($4.44 \cdot 10^{-1}$ mol_O kg_{cat}⁻¹) was 1.5 times
281 higher than that of the 1% Pt@Ti₃C₂ catalyst ($2.97 \cdot 10^{-1}$ mol_O kg_{cat}⁻¹) under H₂-based TPR analysis (Table
282 S3). The possible reason for the higher oxygen uptake can be attributed to the prior reduction of
283 platinized catalysts by H₂, followed by reoxidation during the synthesis of 1% Pt@Ti₃C₂-R. As oxygen
284 is removed during reduction pre-treatment at high temperatures, it can be removed rapidly in the
285 subsequent reduction experiment. In contrast, the 1% Pt@Ti₃C₂ catalyst removed considerably fewer
286 oxygen atoms, as it did not go through a prior reduction treatment.

287 The occurrences of multiple reduction peaks in the low- and high-temperature ranges in the H₂-based
288 TPR and CO-based TPR profiles (Figure 3) indicate the presence of different oxygen-containing groups
289 (e.g., OH) on the as-prepared platinized MXene catalysts. In the case of the CO-based TPR analysis,
290 reduction peaks for the Ti₃C₂ carrier were observed between 170 °C and 350 °C. In contrast, the
291 reduction peaks for the 1% Pt@Ti₃C₂ and 1% Pt@Ti₃C₂-R catalysts appeared between 120 °C and
292 560 °C. During the H₂-based TPR analysis, the temperature for H₂ reduction was much higher than that
293 for CO for the Ti₃C₂ MXene carrier. However, lower reduction temperatures were observed for the first
294 reduction peak in the case of the Pt@Ti₃C₂ catalysts. In particular, reduction peaks in the H₂-based TPR
295 profiles (Figure 3) of the Ti₃C₂ carrier and Pt@Ti₃C₂ catalysts were observed in temperature ranges of
296 170 °C to 600 °C and 40 °C to 550 °C, respectively. These observations indicate the formation of
297 additional reducible surface oxygen species due to doped platinum atoms on the MXene surface.

298 As can be seen in the CO-based TPR profiles (Figure 3), the low-temperature peaks (below 350 °C)
299 contributed to a significant percentage (e.g., 62% and 91%) of the total signal for the 1% Pt@Ti₃C₂ and
300 1% Pt@Ti₃C₂-R catalysts, respectively. The additional peaks at higher temperatures (> 350 °C:
301 compared to the carrier material) contributed approximately 38% and 9% of the total signal for 1%
302 Pt@Ti₃C₂ and 1% Pt@Ti₃C₂-R, respectively. However, the first reduction peak for the Ti₃C₂ carrier was

303 observed at a higher temperature (191.6 °C) compared with 1% Pt@Ti₃C₂ (158.4 °C). The peak
304 reduction temperature for 1% Pt@Ti₃C₂-R was relatively high (222.1 °C). The enhanced contribution of
305 the first peak for 1% Pt@Ti₃C₂ (20% relative to 7% recorded by 1% Pt@Ti₃C₂-R) in the total signal of
306 reducible oxygen atoms could explain the superior activity of the latter. The greater oxygen
307 concentration available for reduction could also explain the synergistic role of reduction pre-treatment
308 in the enhanced performance of Pt@Ti₃C₂-R (relative to the Ti₃C₂ MXene carrier and Pt@Ti₃C₂).
309 Because a similar amount of oxygen was used for the reduction with CO (33.1 ± 3.3 and 39.1 ± 2.5
310 μmol_o), reduction treatment at the catalyst preparation stage may lead to a significant shift of O* species
311 toward lower reduction temperatures.

312 According to the H₂-based TPR analysis, the first peak reduction temperature for 1% Pt@Ti₃C₂-R
313 (106.9°C) was significantly higher than that for 1% Pt@Ti₃C₂ (67.7°C), while it was 2.5-fold lower than
314 that for the Ti₃C₂ carrier (269.5 °C). The reducible oxygen atom for 1% Pt@Ti₃C₂-R was approximately
315 17%, which was 3.4 times higher than that for 1% Pt@Ti₃C₂. This observation may explain the superior
316 activity of 1% Pt@Ti₃C₂-R compared with 1% Pt@Ti₃C₂, and the MXene support at low temperatures.
317 Besides, the high-temperature peaks (> 350 °C) decreased after platinum deposition (from
318 approximately 70% to 6% and 11%, as shown in [Table S3](#)). Three peaks were observed for Ti₃C₂ in the
319 high-reduction-temperature region, whereas only two were observed for the platinum catalysts ([Figure](#)
320 [3](#)). An extra peak was observed for the platinum catalysts in the lower-temperature region. Interaction
321 with the deposited platinum may have caused a shift in reduction temperature, lowering the
322 corresponding surface oxygen concentration.

323

324 *3.3.2. Effect of reduction pre-treatment on the catalytic activity*

325 A linear relationship was found in the peak reduction temperatures of both CO-based TPR and H₂-
326 based TPR data between the 1% Pt@Ti₃C₂-R and the 1% Pt@Ti₃C₂ as can be seen in Figure 4 (a). The
327 reduction temperatures appeared to be higher for the former until approximately 400 °C, while such a
328 pattern was reversed at higher temperatures. As a benzene aromatic ring contains more electrons
329 compared with CO or H₂, the overall reaction may be subject to the complicated effects of adsorption
330 (benzene + O₂) and surface reactions. For example, if the peak temperatures for both catalysts are
331 compared relative to the Ti₃C₂ carrier (ranked in increasing order), CO should require equal or higher
332 reduction temperatures. In contrast, the opposite result should be obtained for H₂-based TPR, i.e., lower
333 reduction temperatures for the platinum catalysts, as shown in Figure 4 (b) and (c). For the results of
334 CO-based TPR, the Ti₃C₂ carrier appears to achieve superior reducibility at lower temperatures
335 compared with platinum catalysts. Instead, the opposite H₂-based TPR results suggest that, due to the
336 complexity of surface catalysis, the performance of catalysts cannot be analyzed by a single
337 characterization. It is better to characterize the catalysts by combining other methods (e.g., XPS and in
338 situ DRIFTS) with TPR. Because the best-performing catalyst achieved a higher reduction temperature
339 for the first peak (H₂ and CO reduction), benzene adsorption and activation, rather than oxygen
340 consumption in VOC oxidation, is likely the rate-determining step of the overall reaction.

341

342 3.4. Effect of process variables on catalytic activity

343 3.4.1. Effect of m_{cat} and flow rate

344 The effect of 1% Pt@Ti₃C₂-R m_{cat} (2, 10, and 30 mg) on the oxidation of benzene vapor at 225 °C
345 can be assessed as a function of reaction time. As seen in Figure 5(a), R_{benzene} increased from 75% to
346 100% at 225 °C upon increasing the m_{cat} from 2 to 30 mg. The value of r increased from 0.004 to 0.046
347 mol g⁻¹ h⁻¹ with a decrease in m_{cat} from 30 to 2 mg (Table S4). The decreased activity with increased m_{cat}
348 was likely due to a lowering of the utilization efficiency of the active sites of the catalysts. If the m_{cat}

349 was 10 mg, R_{benzene} decreased gradually from 99% to 96% with an increasing FR (50 to 200 mL min⁻¹),
350 as shown in [Figure 5\(b\)](#) (Wang et al., 2019). However, r increased from 0.012 to 0.047 mol g⁻¹ h⁻¹ with
351 an increase in the FR ([Table S4](#)). The increased catalytic activity at a higher FR suggests that more
352 benzene molecules reacted with the active sites on the Pt@Ti₃C₂-R surface.

353

354 3.4.2. Effect of benzene concentration and RH

355 The catalytic activity of 1% Pt@Ti₃C₂-R was further studied as a function of varying benzene
356 concentrations (2 to 100 ppm) and RH levels (0 to 80%). [Figure 5\(c\)](#) shows that R_{benzene} increased from
357 90.8% to 99% upon increasing the inlet benzene concentration from 2 to 10 ppm. Increasing the inlet
358 benzene concentration another 10-fold showed no significant effect on R_{benzene} , which was 99.4% and
359 97.3% at 50 and 100 ppm benzene, respectively, even after 210 min TOS. The increase in r from 0.002
360 to 0.119 mol g⁻¹ h⁻¹ with the increase in benzene concentration from 2 to 100 ppm can be attributed to
361 an increase in the utilization efficiency of the active sites on the catalyst surface ([Table S4](#)).

362 [Figure 5\(d\)](#) shows the CO₂ detected upon catalytic oxidation of benzene at the highest (100 ppm) and
363 lowest (2 ppm) tested concentrations. At 225 °C, the thermocatalytic oxidation of 100 and 2 ppm benzene
364 over 1% Pt@Ti₃C₂-R yielded 648 and 29 ppm of CO₂ production after 210 min TOS, with corresponding
365 Y_{CO_2} values of 108 and 242%, respectively, as shown in [Figure 5\(d\)](#). These measured CO₂ values were
366 higher than the expected CO₂ yield (600 and 12 ppm, respectively). If the inlet benzene vapor has been
367 entirely mineralized for CO₂, the balanced equation should be $2\text{C}_6\text{H}_6 + 15\text{O}_2 \rightarrow 12\text{CO}_2 + 6\text{H}_2\text{O}$. These
368 findings suggest that the mineralization of surface-accumulated benzene molecules with reaction time
369 should yield excess CO₂ in the effluent (Xu et al., 2021b; Vikrant et al., 2022).

370 It is critical to evaluate the effect of RH (0%, 50%, and 80%) on the benzene oxidation performance
371 of 1% Pt@Ti₃C₂-R at 225 °C due to the ubiquity of moisture in real-world conditions, as depicted in
372 [Figure 5\(e\)](#). Compared with a dry gas stream (0% RH), the presence of water vapor (50% and 80% RH)

373 significantly suppressed the catalytic activity of 1% Pt@Ti₃C₂-R toward benzene oxidation (R_{benzene}
374 value of 5.1%–8.9%) at the beginning of the thermocatalytic reaction process (TOS = 0–50 min). After
375 50 min TOS, the catalytic activity of 1% Pt@Ti₃C₂-R rapidly recovered at all tested RH levels. R_{benzene}
376 values were 93.4% and 91.6% at 50% and 80% RH, respectively. A steady state was maintained even
377 after 210 min TOS at 225 °C. In addition, the values of r displayed a slight decline of 0.08% from dry
378 (0% RH) to humid (50% and 80% RH) conditions (Table S4), indicating that moisture had a slight
379 inhibitory effect on the catalytic activity of Pt@Ti₃C₂-R. The catalyst also exhibited acceptable
380 reusability without alteration in the catalytic activity even after seven reuse cycles under dry conditions
381 (0% RH), as shown in Figure 5(f).

382 The initial drop in the catalytic activity of 1% Pt@Ti₃C₂-R under humid conditions is likely explained
383 by competition among H₂O, benzene, and O₂ molecules for active surface sites (Liu et al., 2019; Yang
384 et al., 2019; Kong et al., 2020; Saqlain et al., 2021). With an increase in the TOS at 225 °C, the oxygen
385 vacancies (lattice oxygen) on the catalyst surface may be activated, initiating the desorption and
386 activation of H₂O and O₂ reactants and generating reactive oxidative species for benzene oxidation. The
387 thermocatalytic oxidation over Pt@Ti₃C₂-R may therefore eliminate aromatic VOCs under real-world
388 conditions in dry and moist environments at low temperatures.

389

390 3.4.3. Pt@Ti₃C₂-R catalytic stability

391 The catalytic stability of 2 mg of 1% Pt@Ti₃C₂-R was explored under dry conditions at 200 °C against
392 10 ppm benzene and an FR of 50 mL min⁻¹. As shown in Figure 6, the catalyst maintained high stability
393 and durability, with less than a 6% reduction in catalytic activity over 5 h TOS. The low-temperature
394 catalytic process may be the primary reason for the prolonged survival of the catalyst under oxidizing
395 conditions. The tested catalyst therefore appears to be sufficiently robust for the oxidative removal of
396 benzene under real-world conditions.

397 Transmission electron microscope (TEM) images and selected area electron diffraction (SAED)
398 analyses indicated no significant changes in morphology and an average particle-size distribution of 1%
399 Pt@Ti₃C₂-R when a comparison was made between before (9 ± 0.32 nm) and after 5 h (7.85 ± 0.17 nm)
400 of TOS oxidation reactions (Figure S13). However, powder X-ray diffraction (PXRD) patterns of the
401 spent 1% Pt@Ti₃C₂ catalyst, when compared with the fresh catalyst, exhibited significant changes after
402 benzene oxidation, as depicted in Figure S4 and Figure S13(d). Specifically, after oxidation, the intensity
403 of the 2θ diffraction peaks for MXene at 18.62° and 60.93° increased, whereas the intensity of the 2θ
404 diffraction peaks for Pt₃Ti at 25.26° , 48.14° , 54.69° , 68.59° , and 76.45° decreased. Such observations
405 may be attributable to poor crystallinity caused by the accumulation of strongly bounded by-products
406 on the catalyst surface (Kumar et al., 2018).

407 According to the XPS data for 1% Pt@Ti₃C₂-R after 5 h TOS shown in Figure S14(a), the Ti 2p
408 spectrum can be deconvoluted into four peaks at 459.5 (Ti-C), 461.22 (Ti³⁺), 465.19 (Ti⁴⁺), and 472.4
409 eV (satellite peak) (Konstantinova et al., 2018). The peaks for Ti-C, Ti³⁺, and Ti⁴⁺ showed slight shifts
410 toward lower binding energy (-0.09 to -0.61 eV) compared with those observed for fresh 1% Pt@Ti₃C₂-
411 R, possibly due to the reduction of titanium species under continuous heating at 200 °C during the
412 reaction (Jiang et al., 2007). In the case of the C 1s spectrum in Figure S14(b), the C 1s core level can
413 be deconvoluted into four peaks at 284.64 (C-Ti), 285.7 (C-C), 287.13 (C-O), and 289.65 eV (O-C=O),
414 with slight blue shifts compared to fresh 1% Pt@Ti₃C₂-R. The blue shifts in the binding energies may
415 reflect the effects of chemical interactions between the catalyst and the benzene molecules (Li et al.,
416 2020a).

417 In Figure S14(c), deconvolution of the O 1s spectrum yields two peaks at 531.09 and 532.78 eV,
418 corresponding with Ti-O (O*) and C-Ti-(OH)_x/H₂O_{ads}/O_{ads} (adsorbed oxygen), respectively. The amount
419 of O* in 1% Pt@Ti₃C₂-R increased from 45.3% to 71% after reduction pre-treatment. However, the
420 amount of O* in the catalyst decreased to 63% after benzene oxidation (Table S5). These findings are

421 consistent with the experimental results shown in [Figure 2\(a\)](#) and [Figure 6](#). For the Pt 4f spectrum in
422 [Figure S14\(d\)](#), four peaks were observed at 72.27 (4f_{7/2} Pt⁰), 73.37 (4f_{7/2} Pt²⁺), 75.82 (4f_{5/2} Pt⁰), and 78.59
423 eV (4f_{5/2} Pt²⁺). The XPS analysis of the catalyst after benzene oxidation also produced a new peak (for
424 the Pt²⁺ species), with a blue shift for other peaks due to the intrinsic charge transfer after 5 h TOS.
425 Similarly, the amount of Pt²⁺ in the catalyst increased from 34.6% to 61.7% after reduction pre-treatment.
426 However, the amount of Pt²⁺ in the catalyst decreased to 40.5% after benzene oxidation ([Table S5](#)),
427 which is consistent with the experimental results shown in [Figure 2\(a\)](#) and [Figure 6](#). The platinum
428 clusters and NPs dispersed on the Ti₃C₂ surface during thermal reduction pre-treatment at 300 °C to form
429 more Pt²⁺ species ([Figure S2](#)) ([Poerwoprajitno et al., 2022](#)). Combined with the experimental results
430 depicted in [Figure 2\(a\)](#), the Pt²⁺ species may have catalyzed benzene oxidation more favorably than the
431 Pt⁰ ([Guo et al., 2020](#)).

432

433 *3.5. Reaction mechanism and pathway*

434 In situ DRIFTS was conducted to evaluate the benzene oxidation pathway and mechanism for 1%
435 Pt@Ti₃C₂-R at varying temperatures from RT to 300 °C. As seen in [Figure 7](#), similar intermediates were
436 observed in the DRIFTS spectra for benzene (250 ppm) oxidation over 1% Pt@Ti₃C₂-R across varying
437 temperatures (RT–300 °C). The reaction temperature therefore did not alter the benzene oxidation
438 pathway. The DRIFTS spectra produced prominent bands for (i) formate (1528 cm⁻¹), (ii) phenolate
439 (1595 cm⁻¹), (iii) C=O vibration of benzoquinone (1703 cm⁻¹), and (iv) adsorbed H₂O and surface OH
440 (3647-3880 cm⁻¹) ([Qin et al., 2020](#); [Kim et al., 2022](#)). The benzene ring first adsorbs on the surface of
441 the catalyst. The O* (active sites) on the catalyst surface induce oxidation to generate oxygen vacancies,
442 which can be replenished by O₂ molecules adsorbed dissociatively on the platinum active sites ([Sun et
443 al., 2015](#)). Benzene oxidation by O* forms phenolate species, followed by a ring-opening reaction to

444 produce smaller aliphatic intermediates (e.g., formate). Eventually, the synergistic interaction between
445 O* and platinum on the MXene surface promotes the conversion of formate to CO₂ and H₂O.

446

447 3.6. Catalytic performance comparison

448 The results obtained in the present work were compared with those reported previously (including
449 the supported platinum and other catalysts) to assess catalytic performance differences (Table S6). The
450 value of r at T_{100} was used as the critical metric to compare catalytic performance based on an earlier
451 recommendation (Vikrant et al., 2022). We observed that the 1% Pt@Ti₃C₂-R used in the present study
452 could attain 100% R_{benzene} at a relatively lower temperature (225 °C) compared with some of the
453 previously reported catalysts (Deng et al., 2018; Guo et al., 2019; Sophiana et al., 2020; Liu et al., 2021).
454 Similarly, the r -value of 0.119 mol g⁻¹ h⁻¹ for 1% Pt@Ti₃C₂-R was higher than that of most previously
455 reported catalysts for low-temperature benzene oxidation (Shim and Kim, 2010; Li et al., 2016; Zhu et
456 al., 2017; Zhang et al., 2018). In general, the supported platinum catalysts outperformed other catalysts
457 (e.g., manganese-based catalysts) for the oxidation of benzene (Huang et al., 2015).

458

459 4. Conclusions

460 In the present work, platinum-loaded 2D Ti₃C₂ MXene catalysts were synthesized by a one-pot IMP
461 for benzene oxidation at varying operating conditions (e.g., reaction temperature, platinum loading, m_{cat} ,
462 benzene concentration, FR, and RH). The catalytic oxidation activity of the platinized catalyst
463 (Pt@Ti₃C₂) was improved through reduction pre-treatment using H₂. The catalytic activity was further
464 enhanced by increasing the platinum loading from 0.1 wt.% to 1 wt.%. Specifically, the 1% Pt@Ti₃C₂-
465 R was the best candidate for completely oxidizing benzene (10 ppm) to CO₂ at 225 °C under dry
466 conditions. At 225 °C, the value of r decreased in the order of 1% Pt@Ti₃C₂-R (0.012 mol g⁻¹ h⁻¹) > 1%
467 Pt@Ti₃C₂ (0.007 mol g⁻¹ h⁻¹) > Ti₃C₂-R (0.001 mol g⁻¹ h⁻¹). Moisture was found to exert inhibitory effects

468 on the catalytic oxidation of benzene, especially at the initial reaction stage. In contrast, catalytic activity
469 recovered for an extended reaction time at 225 °C to produce a $R_{\text{benzene}} > 90\%$ at 50%–80% RH after
470 210 min TOS. The reusability study also proved the high catalytic oxidation stability of 1% Pt@Ti₃C₂-
471 R over seven cycles when tested for 300 min TOS. Based on the results of XPS, TPR, and in situ DRIFTS
472 analyses, we postulated that the surface O* active sites attacked the adsorbed benzene ring on the 1%
473 Pt@Ti₃C₂-R surface, inducing a series of thermal oxidation reactions and generating various
474 intermediates (phenolates and formate) that subsequently oxidized to CO₂ and H₂O as end products. The
475 present work demonstrates the significant potential of platinized MXene for the effective oxidation of
476 gaseous benzene at lower temperatures. The results of this study are expected to open a path for a better
477 understanding of the role of the platinum-loaded 2D Ti₃C₂ MXene catalysts during benzene oxidation.

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481

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Table 1. The benzene removal performance of the tested catalysts.^a

Order	Catalyst	T ₅₀ (°C) ^b	T ₉₀ (°C) ^c	T ₁₀₀ (°C) ^d	r (mol g _{cat} ⁻¹ h ⁻¹) ^e	r (mol g _{Pt} ⁻¹ h ⁻¹) ^f
1	Ti ₃ C ₂ -R	–	–	–	0.001	-
2	1% Pt@Ti ₃ C ₂	195	230	325	0.007	0.82
3	1% Pt@Ti ₃ C ₂ -R	167	191	225	0.012	1.27
4	0.1 %Pt@Ti ₃ C ₂ -R	207	255	350	0.005	3.96
5	2% Pt@Ti ₃ C ₂ -R	139	162	200	0.012	0.58

^a Reactant composition: benzene (10 ppm) + air (balance), FR: 50 mL·min⁻¹, mass: 10 mg MXene + 50 mg sands, and reduction pre-treatment (under the flow (50 mL·min⁻¹) of 10% H₂/N₂ at 300 °C for 3 h).

^b Reaction temperature at 50% R_{benzene}.

^c Reaction temperature at 90% R_{benzene}.

^d Reaction temperature at 100% R_{benzene}.

^e Reaction rate per catalyst mass obtained at 200 °C.

^f Reaction rate per Pt mass obtained at 200 °C.

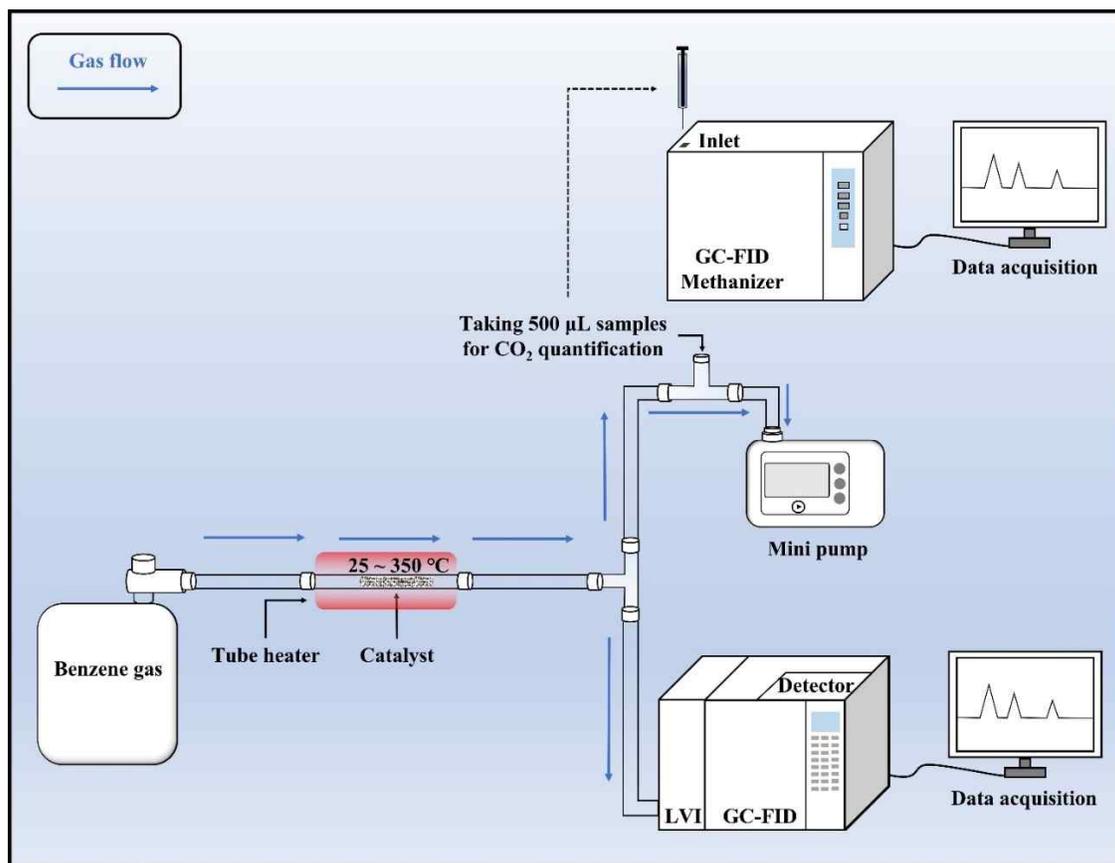
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Table 2. The ratio of the lattice oxygen, adsorbed oxygen, Pt⁰, and Pt²⁺ of tested materials (analysis by XPS).

Order	Catalyst	Lattice oxygen (O [*])	Adsorbed oxygen (O _{ads})	Pt ⁰	Pt ²⁺
1	1% Pt@Ti ₃ C ₂	45.3%	54.7%	65.4%	34.6%
2	1% Pt@Ti ₃ C ₂ -R	71.0%	29.0%	38.9%	61.1%
3	1% Pt@Ti ₃ C ₂ -R (after reaction)	63.0%	37.0%	59.5%	40.5%

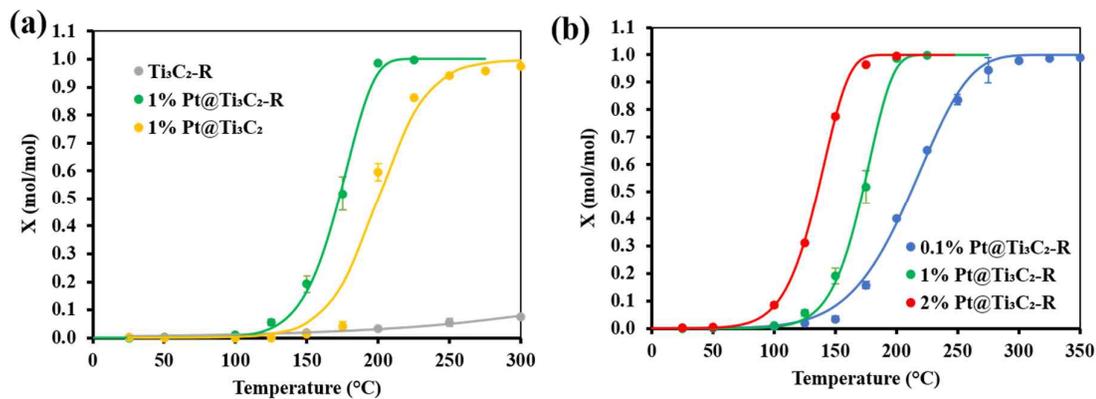
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666 **Figure 1.** Schematic of the benzene catalytic oxidation experimental setup.



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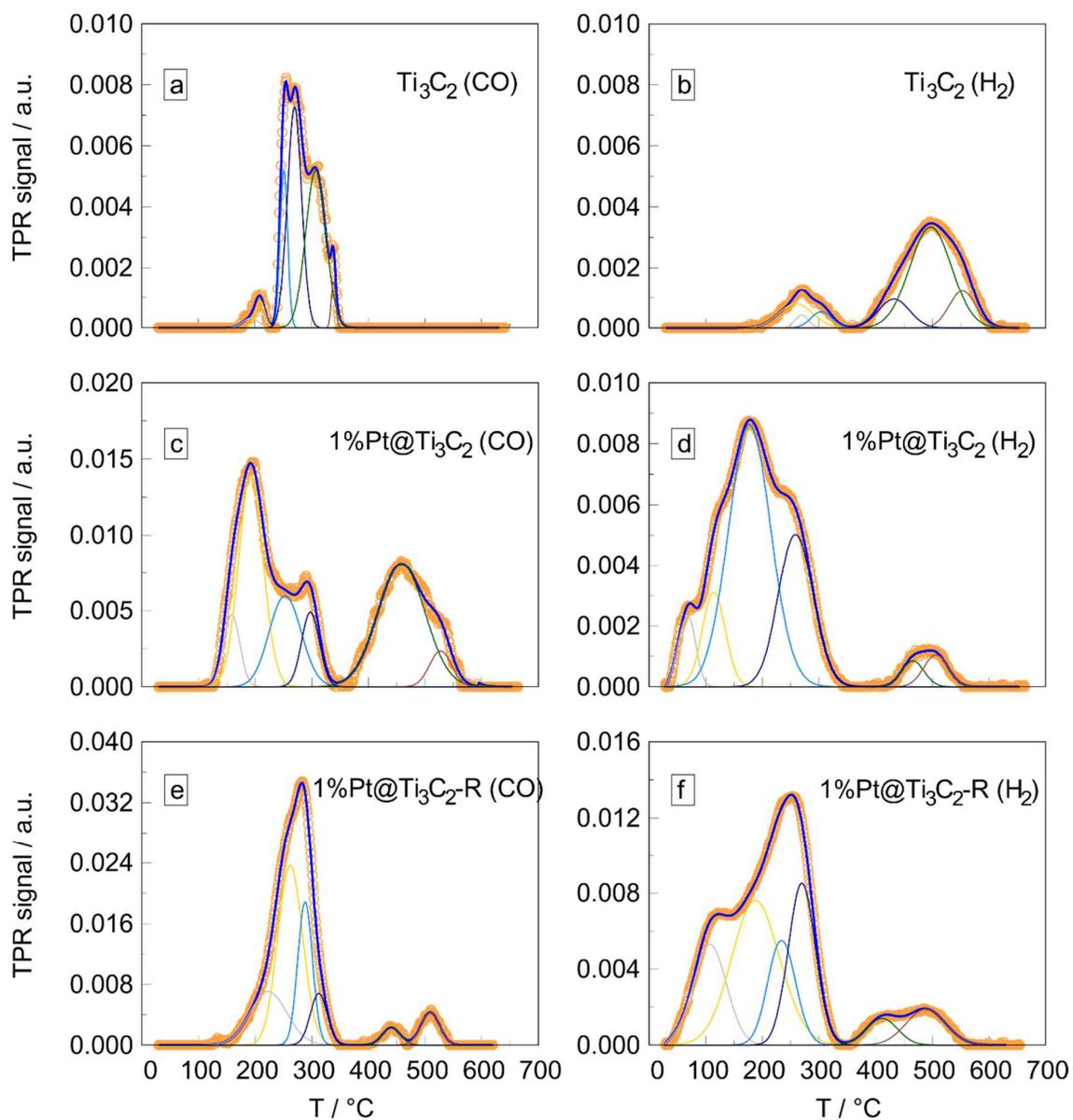
668 **Figure 2.** Light-off curves for the conversion performance of benzene (10 ppm in dry air and RH = 0%):

669 (a) comparison between the catalysts of the control group (bed mass: 10 mg catalyst + 50 mg sand and

670 FR: 50 mL·min⁻¹) and (b) effect of different platinum loading (bed mass: 10 mg catalyst + 50 mg sand

671 and FR: 50 mL·min⁻¹) on Pt@Ti₃C₂-R.

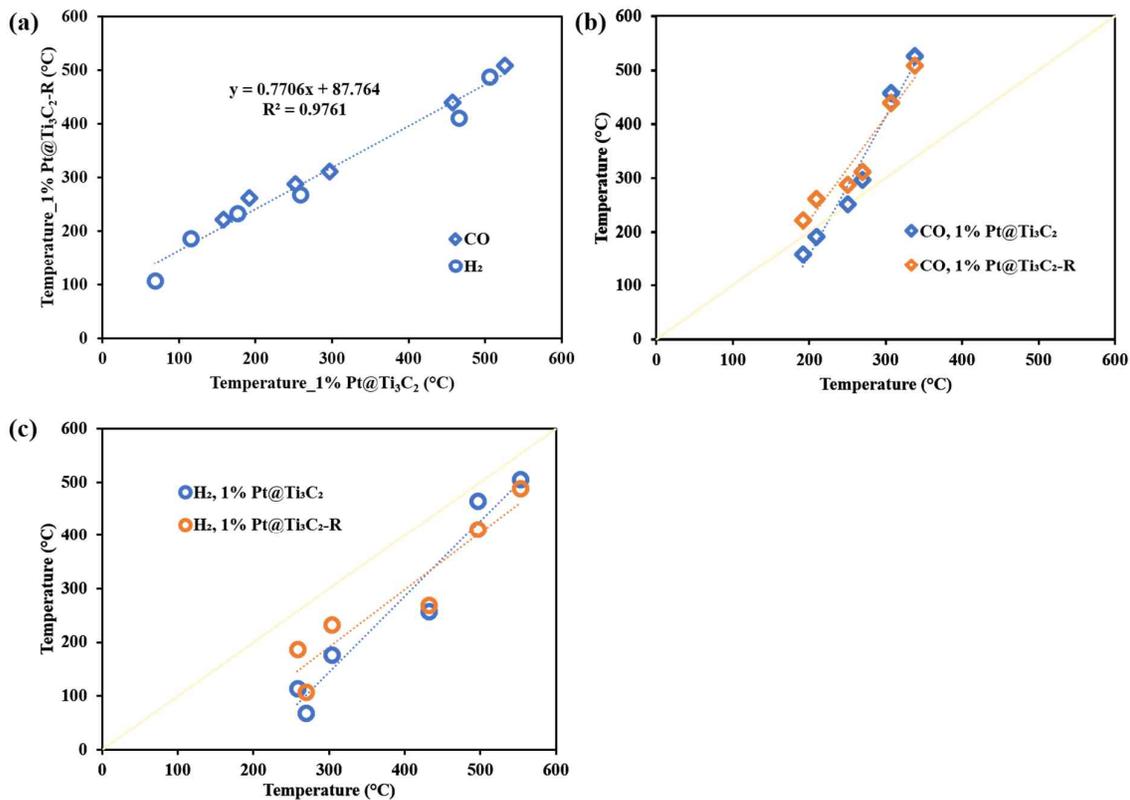
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674 **Figure 3.** TPR analysis (profiles =○) of the tested materials: (a) Ti_3C_2 (under CO), (b) Ti_3C_2 (under H_2),
 675 (c) $1\%\text{Pt}@Ti_3C_2$ (under CO), (d) $1\%\text{Pt}@Ti_3C_2$ (under H_2), (e) $1\%\text{Pt}@Ti_3C_2\text{-R}$ (under CO), and (f)
 676 $1\%\text{Pt}@Ti_3C_2\text{-R}$ (under H_2). Gaussian model, according to Equation S2, for peak 1 (—), peak 2 (—), peak
 677 3 (—), peak 4 (—), peak 5 (—), peak 6 (—), and total calculated signal (—). The ramp rate was 10 K/min.

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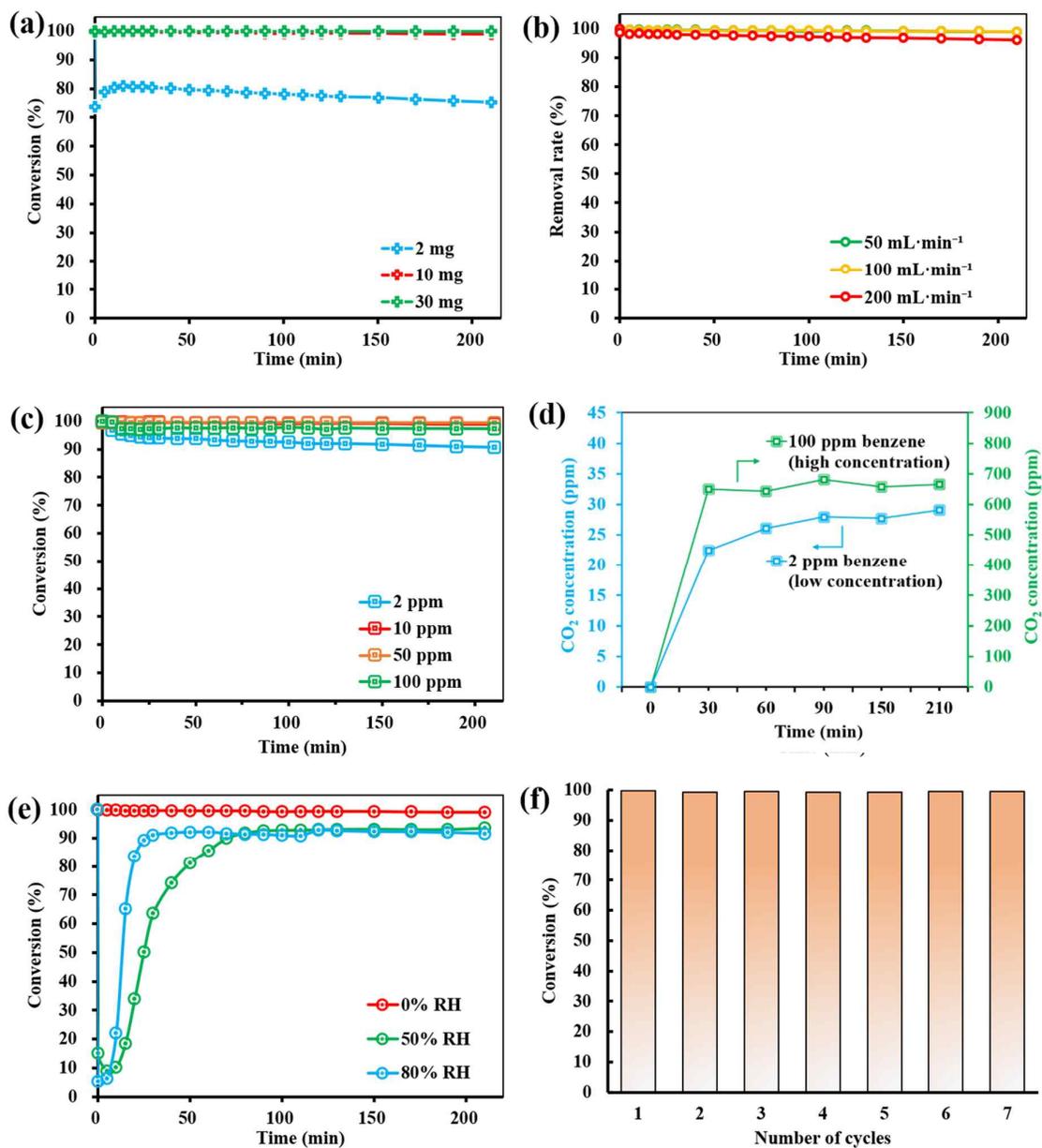


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680 **Figure 4.** Comparisons of peak temperatures of 1% Pt@Ti₃C₂ and 1% Pt@Ti₃C₂-R based on TPR

681 analysis: (a) CO (□) and H₂ (○). (b) CO results between 1% Pt@Ti₃C₂ (□) versus 1% Pt@Ti₃C₂-R (◇).

682 (c) H₂ results between 1% Pt@Ti₃C₂ (○) versus 1% Pt@Ti₃C₂-R (◊).



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Figure 5. The effects of process variables on the catalytic activity of 1% Pt@Ti₃C₂-R (bed mass: 10 mg

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catalyst + 50 mg sand) against benzene at a gaseous FR of 50 mL·min⁻¹: (a) effect of bed mass (bed

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mass: 2–30 mg catalyst mixed with sands (at a fixed weight ratio of 1:5), benzene: 10 ppm, and FR: 50

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mL·min⁻¹), (b) effect of FR (bed mass: 10 mg catalyst + 50 mg sand and 10 ppm benzene), (c) effect of

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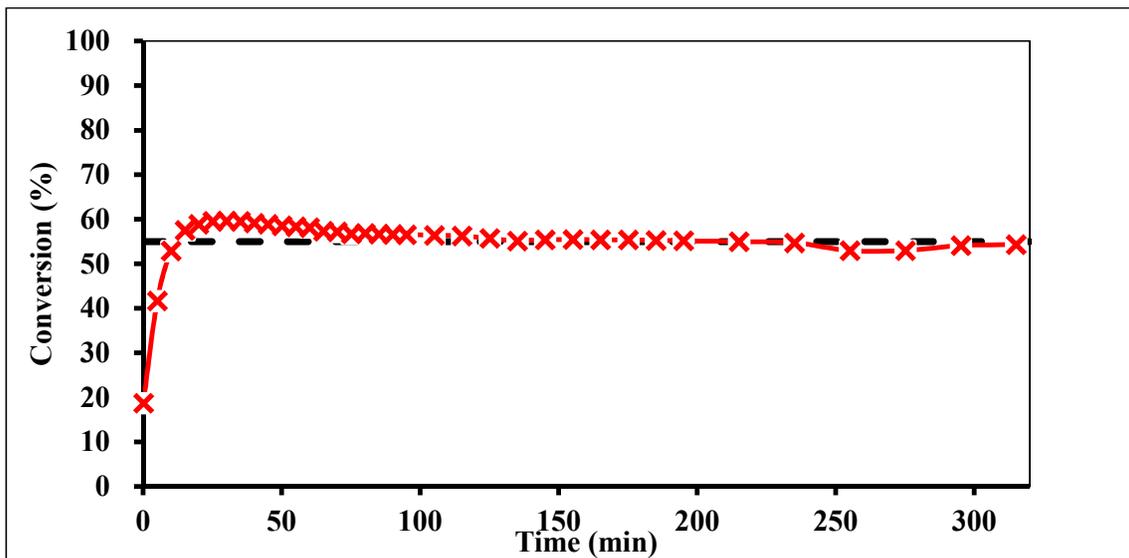
benzene concentrations (2–100 ppm) at RH= 0%, (d) CO₂ yield at benzene concentration of 2 and 100

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ppm (in air) and RH: 0%, (e) effect of RH (0%, 50%, and 80%) against 10 ppm benzene oxidation (in

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air), and (f) recycle test against 10 ppm benzene oxidation (in dry air; RH: 0%).



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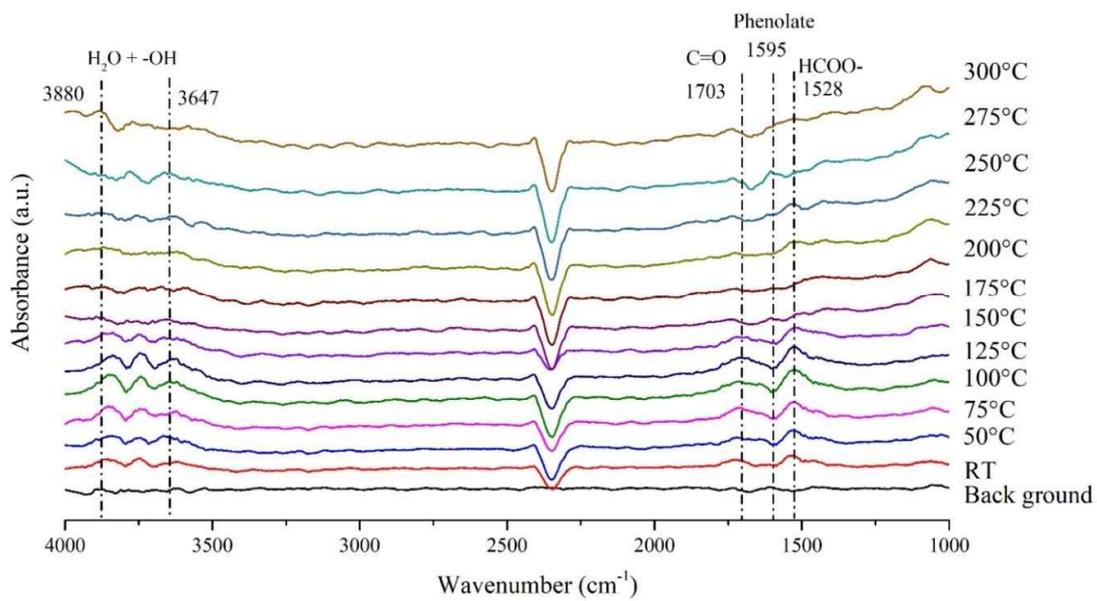
692 **Figure 6.** Benzene oxidation stability test of 1% Pt@Ti₃C₂-R (mass: 2 mg catalyst + 10 mg sand,
693 benzene concentration: 10 ppm (in dry air, RH: 0%), FR: 50 mL·min⁻¹, and reaction temperature: 200 °C).

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699 **Figure 7.** In situ DRIFTS spectra over 1% Pt@Ti₃C₂-R at RT–300 °C (benzene + air, 1:1 mixture).

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