

Article

Pt/Al₂O₃ for Efficient CF₄ Hydrolytic Decomposition

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Abstract

Tetrafluoromethane (CF₄) is one of the most stable perfluorinated compounds because of its highly symmetric molecular structure and extremely strong C–F bonds. Catalytic hydrolysis provides a promising route for CF₄ abatement, yet the development of efficient catalysts capable of activating C–F bonds at reduced temperatures remains challenging. Herein, Pt-modified Al₂O₃ catalysts were prepared to promote CF₄ decomposition through the regulation of surface acidity, CF₄ adsorption, and hydroxyl formation. NH₃ temperature-programmed desorption revealed that Pt introduction significantly enhanced the surface acidity of Al₂O₃, while CF₄ temperature-programmed desorption demonstrated strengthened CF₄ adsorption over Pt/Al₂O₃. In situ infrared spectroscopy further showed that Pt incorporation promoted the generation of surface hydroxyl groups under reaction-relevant conditions. These hydroxyl species are proposed to participate in C–F bond activation during CF₄ hydrolysis. As a result, Pt/Al₂O₃ achieved complete CF₄ decomposition at 600 °C and maintained excellent stability during the long-term test, clearly outperforming pristine Al₂O₃. This work demonstrates that Pt/Al₂O₃ is an effective catalyst for CF₄ decomposition by coupling enhanced acidity, improved CF₄ adsorption, and hydroxyl-assisted C–F bond activation.

Keywords: Al₂O₃; C–F bond activation; CF₄ catalytic hydrolysis; hydroxyls

1. Introduction

Perfluorinated compounds have attracted increasing concern because of their chemical inertness, environmental persistence, and strong greenhouse effects. Among them, CF₄ is particularly difficult to decompose due to its tetrahedral structure and highly stable C–F bonds. The carbon–fluorine bond has a bond energy as high as 543 kJ mol^{−1} [1–4]. Conventional thermal decomposition usually requires extremely high temperatures, which leads to high energy consumption and limited practical applicability [5]. Catalytic hydrolysis has therefore been considered a more feasible strategy for CF₄ abatement, as it can convert CF₄ into hydrolysis products at significantly lower temperatures.

Alumina-based catalysts have been widely investigated for CF₄ hydrolysis because of their thermal stability and abundant surface acid sites [6–9]. Previous studies have shown that Lewis acidic Al sites on γ-Al₂O₃ can adsorb CF₄ and initiate C–F bond activation [10–13]. However, pristine Al₂O₃ still suffers from insufficient intrinsic activity and limited long-term stability. Adjusting the surface acidity and introducing additional



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structures are effective ways to improve Al_2O_3 catalysts for catalyzing CF_4 hydrolysis. Previous researchers mostly tried to improve the catalytic hydrolysis ability of aluminum oxide for carbon tetrafluoride by modifying the structure of aluminum oxide itself [14]. However, this usually requires a large amount of doping, which can affect the properties of the aluminum oxide catalyst and is also costly, limiting its large-scale production.

Noble metal species, especially Pt-based sites, can modify the electronic and acidic properties of oxide supports and promote the activation of strongly bonded molecules. In this work, Pt was introduced onto $\gamma\text{-Al}_2\text{O}_3$ to construct $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$ catalysts. Our research shows that introducing Pt not only boosts the adsorption of CF_4 at active Al sites but also significantly increases the surface acidity. In-situ infrared indicates that adding Pt greatly enhances the ability of alumina catalysts to break down water into hydroxyl groups. The structural evolution, acidity, CF_4 adsorption behavior, hydroxyl formation, and catalytic performance were systematically investigated. The results reveal that Pt species, mainly present as platinum oxide-related particles or domains, act as functional active components that enhance the surface acid properties and promote the formation of reactive hydroxyl groups. These effects jointly facilitate CF_4 adsorption and C–F bond cleavage, leading to efficient CF_4 decomposition at 600 °C.

2. Results and Discussion

2.1. Structural Characterization of $\text{Pt}/\text{Al}_2\text{O}_3$ Catalysts

The $\text{Pt}/\text{Al}_2\text{O}_3$ catalyst was made using the classic equal-volume impregnation method [15]. The actual Pt loadings determined by inductively coupled plasma optical emission spectroscopy (ICP-OES) are summarized in Table 1. And through BET analysis of specific surface area and pore size, it was found that adding Pt reduced the catalyst's surface area, ruling out the effect of increased surface area on the catalyst's performance (Table 2). The crystal structures of Al_2O_3 and $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$ catalysts with different Pt contents were first examined by X-ray diffraction (XRD). The pristine Al_2O_3 sample displayed characteristic diffraction peaks of $\gamma\text{-Al}_2\text{O}_3$ [Figure 1], indicating that the support maintained its typical alumina phase. After Pt introduction, the main diffraction features of $\gamma\text{-Al}_2\text{O}_3$ were still preserved, suggesting that Pt loading did not destroy the crystalline framework of the support [16,17].

Table 1. Elemental composition of Al_2O_3 and $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$ catalysts.

Catalysts	Al (wt.%)	O (wt.%)	Pt (wt.%)
Al_2O_3	27.1	72.9	/
$\text{Pt}_{0.3}/\text{Al}_2\text{O}_3$	26.9	71.9	1.2
$\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$	26.8	71.4	1.8
$\text{Pt}_1/\text{Al}_2\text{O}_3$	26.6	69.7	3.7

Table 2. BET analysis of Al_2O_3 and $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$ catalysts.

Catalysts	BET Surface Area (m^2/g)
Al_2O_3	134.6
$\text{Pt}_{0.3}/\text{Al}_2\text{O}_3$	131.9
$\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$	127.3
$\text{Pt}_1/\text{Al}_2\text{O}_3$	122.4

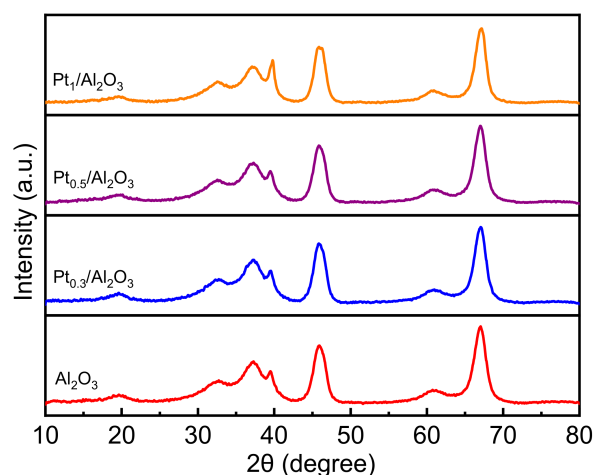


Figure 1. XRD pattern of catalysts as prepared (Al_2O_3 , $\text{Pt}_{0.3}/\text{Al}_2\text{O}_3$, $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$, $\text{Pt}_1/\text{Al}_2\text{O}_3$).

Transmission electron microscopy (TEM) was further used to examine the morphology and microstructure of the $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$ catalyst. The high-resolution TEM image showed clear lattice fringes with interplanar spacings of approximately 2.40 and 1.97 Å, which can be attributed to the $\gamma\text{-Al}_2\text{O}_3$ lattice planes (Figure 2). This result confirms that the support retained the $\gamma\text{-Al}_2\text{O}_3$ phase after Pt modification. In addition, no severe structural collapse or sintering was observed, indicating that the catalyst possessed good structural integrity. Elemental mapping demonstrated that Pt, Al, and O were uniformly distributed throughout the observed region (Figure 3). The homogeneous Pt distribution implies strong dispersion of Pt species over the Al_2O_3 surface, which is favorable for exposing accessible Pt-containing active sites during CF_4 hydrolysis. The electronic structure of Pt was analyzed using XPS (Figure 4). The results show that Pt is mainly loaded on the Al_2O_3 surface in the +2 oxidation state, with Pt 4f characteristic peaks at around 74.2 eV, indicating that the Pt species exist in this form rather than as metallic Pt^0 particles. The formation of Pt^{2+} species likely comes from coordination between Pt and surface hydroxyl or oxygen species on Al_2O_3 . In this process, electron transfer between Pt and the support leads to an electron-deficient state at the Pt center, enhancing its ability as a Lewis acid site. Additionally, introducing Pt species may change the electronic environment of nearby Al sites, increasing the polarization of Al–O bonds and promoting the formation of surface acid sites [18].

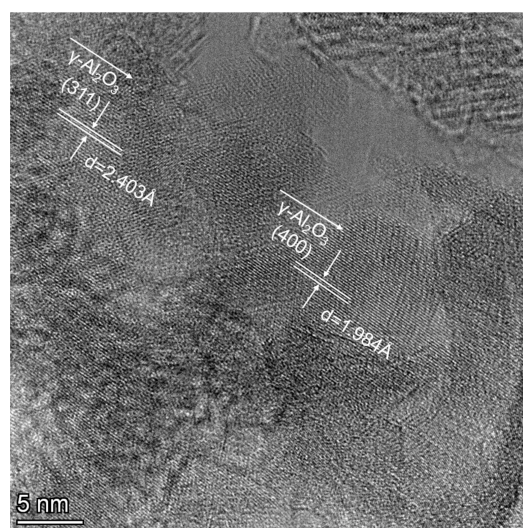


Figure 2. TEM pattern of $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$ as prepared.

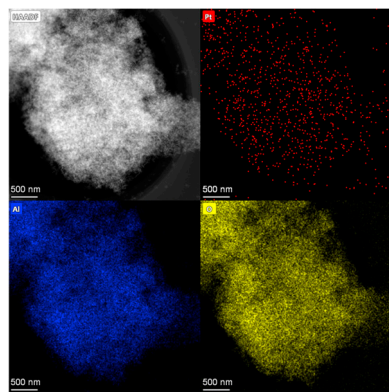


Figure 3. EDS-mapping pattern of $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$ as prepared.

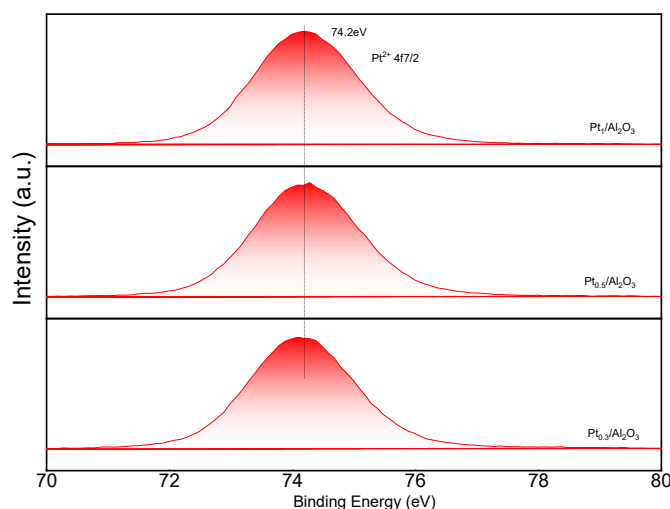


Figure 4. X-ray photo electron spectroscopy (XPS) spectra of catalyst (Pt 4f).

2.2. Effect of Pt Introduction on Surface Acidity and CF_4 Adsorption

Surface acidity plays a critical role in CF_4 hydrolysis because acid sites can provide adsorption and activation centers for CF_4 molecules. NH_3 -TPD (Temperature Programmed Desorption) was therefore conducted to evaluate the acidity of pristine Al_2O_3 and $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$ [17–19]. As shown in Figure 5, pristine Al_2O_3 exhibited NH_3 desorption signals mainly in the low- and medium-temperature regions, corresponding to weak and moderate acid sites. After Pt incorporation, the total NH_3 desorption intensity increased significantly. In particular, the $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$ catalyst showed enhanced desorption signals over a broader temperature range, indicating an increase in both the amount and strength of acid sites [20,21].

The enhanced acidity can be attributed to the interaction between Pt species and the Al_2O_3 support. The introduction of Pt species may modify the local electronic environment of surface Al–O sites and generate additional acidic centers. These newly formed or strengthened acid sites can improve the interaction between the catalyst surface and CF_4 molecules, thereby facilitating the initial adsorption step during hydrolysis.

To verify this assumption, CF_4 -TPD measurements were performed [22,23]. The CF_4 desorption signal of $\text{Pt}/\text{Al}_2\text{O}_3$ was much stronger than that of pristine Al_2O_3 [Figure 6]. Moreover, the main CF_4 desorption peak shifted to a higher temperature after Pt introduction, indicating stronger CF_4 adsorption on $\text{Pt}/\text{Al}_2\text{O}_3$. This result demonstrates that Pt modification not only increases the number of adsorption sites but also strengthens the interaction between CF_4 and the catalyst surface [24]. The improved CF_4 adsorption is

beneficial for subsequent C–F bond activation, which is generally considered the key step in CF_4 decomposition [25,26].

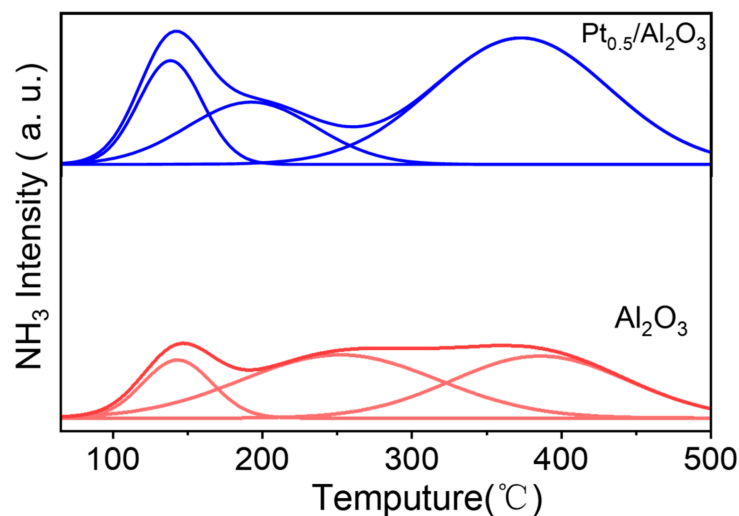


Figure 5. NH_3 -TPD pattern of Al_2O_3 and $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$.

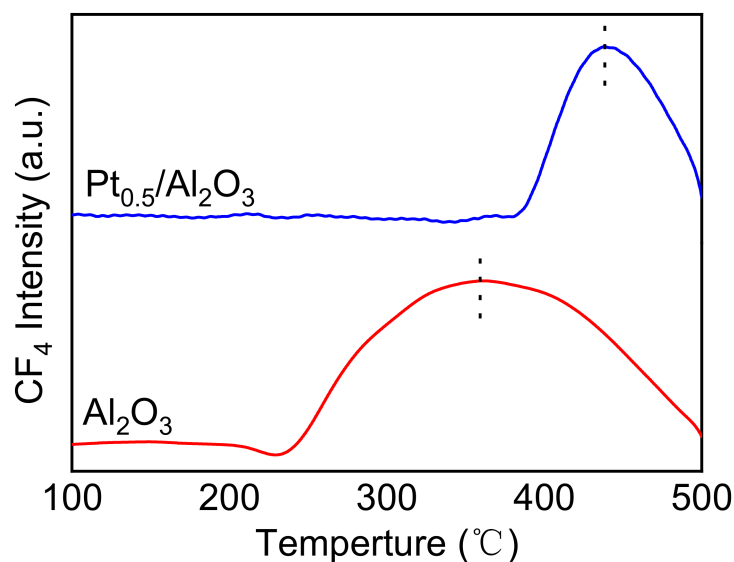


Figure 6. CF_4 -TPD pattern of Al_2O_3 and $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$.

2.3. Identification of Active Pt-Containing Particles and Hydroxyl Formation

The role of Pt species was further evaluated by combining microscopic characterization and in situ infrared spectroscopy. The STEM image of $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$ showed a highly dispersed Pt-containing structure on the alumina support (Figure 7). These Pt-containing particles or domains are proposed to act as the primary functional components responsible for the improved catalytic behavior. Rather than serving only as passive additives, Pt species alter the surface chemical environment of Al_2O_3 and participate in the activation process through their interaction with adsorbed reactants and surface hydroxyl groups [27,28].

In situ infrared spectroscopy was then used to monitor the evolution of hydroxyl species over $\text{Pt}/\text{Al}_2\text{O}_3$ and pristine Al_2O_3 at 600 °C. For both catalysts, hydroxyl-related bands appeared in the range of 3600–3800 cm^{-1} and gradually intensified with time, indicating the formation of surface hydroxyl groups during water activation. However, the

OH signal over $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$ was more pronounced than that over pristine Al_2O_3 , suggesting that Pt introduction promoted the generation of surface hydroxyl species (Figure 8a,b).

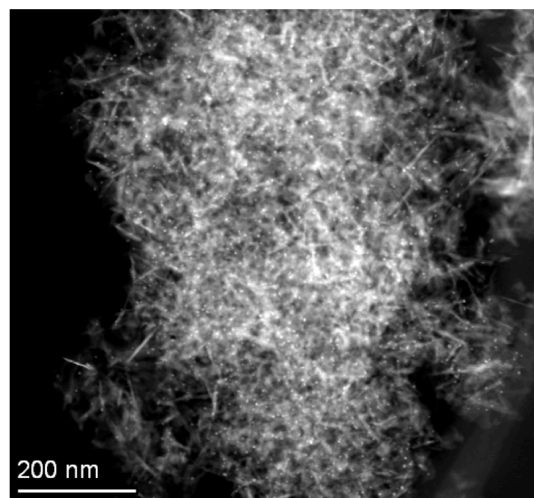


Figure 7. TEM pattern of $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$ as prepared (200 nm).

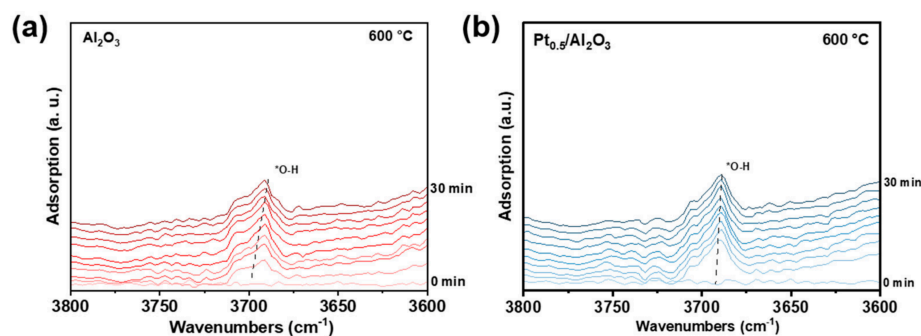


Figure 8. In situ H_2O -DRIFTS (Diffuse Reflectance Infrared Fourier Transform Spectroscopy) spectra on (a) Al_2O_3 (b) $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$. Experimental process: Prior to experimentation, the samples underwent a pretreatment process involving heating at $600\text{ }^\circ\text{C}$ for 2 h under an Ar flow. At this temperature, background spectra were recorded. Following this, the samples were exposed to a controlled atmosphere of $\text{H}_2\text{O}/\text{Ar}$ at $600\text{ }^\circ\text{C}$ for 0.5 h. The recording interval is once every 3 min and 20 s.

The enhanced hydroxyl formation is highly relevant to CF_4 hydrolysis. Surface hydroxyl groups can act as reactive proton-donating or nucleophilic species that interact with adsorbed CF_4 -derived intermediates, thereby weakening and cleaving C–F bonds. Therefore, the superior activity of $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$ can be rationalized by the synergistic effect between enhanced acid sites, stronger CF_4 adsorption, and Pt-promoted hydroxyl formation. In this process, acid sites are responsible for anchoring and polarizing CF_4 , while surface hydroxyl groups participate in the cleavage of C–F bonds. Pt-containing particles further promote this process by increasing the availability and reactivity of these surface species [29].

2.4. Catalytic Performance for CF_4 Decomposition

To prove that introducing Pt can promote CF_4 decomposition, CF_4 decomposition performance tests were carried out with different Pt loadings [Figure 9a]. It was found that CF_4 decomposition shows a clear volcano-shaped trend, with the optimal loading being a 0.5% molar ratio. Lower loadings might not provide enough hydroxyl groups due to insufficient Pt water dissociation active sites, while higher loadings could cause Pt aggregation that covers the active Al sites. The catalytic performance of $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$ and pristine

Al_2O_3 was evaluated for CF_4 hydrolytic decomposition. The CF_4 decomposition efficiency increased with reaction temperature for both catalysts [Figure 9b]. However, $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$ consistently showed higher activity than pristine Al_2O_3 over the entire temperature range. At relatively low temperatures, $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$ already exhibited measurable CF_4 conversion, whereas pristine Al_2O_3 showed much lower activity. With increasing temperature, the activity gap between the two catalysts remained evident.

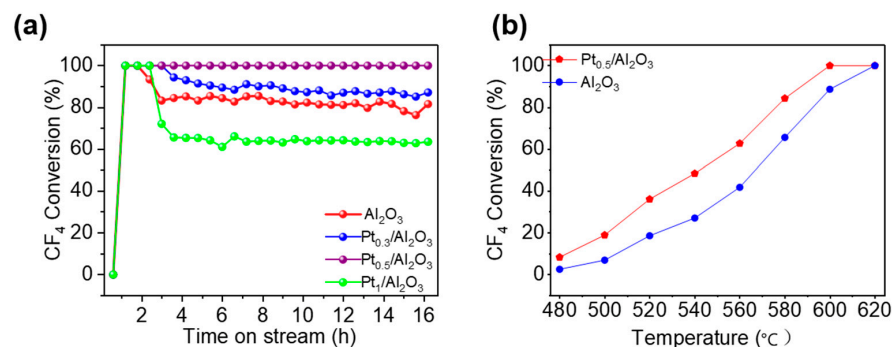


Figure 9. (a) Catalytic decomposition rate of CF_4 on Al_2O_3 with different Pt loadings. (b) CF_4 conversion reaction over Al_2O_3 and $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$.

Notably, $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$ achieved complete CF_4 decomposition at $600\text{ }^{\circ}\text{C}$. In contrast, pristine Al_2O_3 required a higher temperature to reach full conversion ($620\text{ }^{\circ}\text{C}$), this may be due to F poisoning caused by insufficient supply of surface-active species. This activity enhancement confirms the positive role of Pt modification in promoting CF_4 hydrolysis. The superior performance of $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$ can be attributed to the combined effects demonstrated above: Pt introduction increases surface acidity, strengthens CF_4 adsorption, and promotes hydroxyl generation. These properties jointly facilitate C–F bond activation and accelerate CF_4 decomposition.

Long-term stability is another critical criterion for practical CF_4 abatement catalysts [30]. $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$ maintained nearly 100% CF_4 decomposition during the stability test, indicating excellent durability under high-temperature reaction conditions [Figure 10]. In comparison, pristine Al_2O_3 gradually lost activity with time on stream. The stable performance of $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$ suggests that Pt-containing active sites and the $\gamma\text{-Al}_2\text{O}_3$ framework remain effective during prolonged operation. The improved stability may also be related to the stronger activation of water and continuous generation of hydroxyl species, which can help maintain surface reactivity during CF_4 hydrolysis.

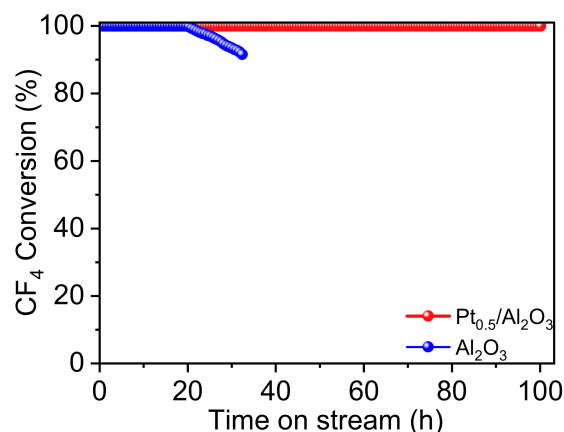


Figure 10. Stability test of $\text{Pt}_{0.5}/\text{Al}_2\text{O}_3$ and Al_2O_3 catalysts at T_{100} (decomposition reaction conditions: 2500 ppm CF_4 , Ar as balance gas and 8.16 mL min^{-1} of water vapor under atmospheric pressure, gas hourly space velocity (GHSV) = 1000 h^{-1}).

2.5. Proposed Mechanism

Based on the above characterization and catalytic results, a plausible mechanism for CF₄ decomposition over Pt_{0.5}/Al₂O₃ is proposed. First, CF₄ molecules are adsorbed and polarized on the enhanced acid sites of Pt_{0.5}/Al₂O₃. The stronger CF₄ adsorption observed in CF₄-TPD indicates that Pt modification improves the ability of the catalyst to capture CF₄. Second, water molecules are activated on the catalyst surface to generate hydroxyl species. In situ infrared spectroscopy confirms that Pt_{0.5}/Al₂O₃ produces more surface OH groups than pristine Al₂O₃ under reaction-relevant conditions. Third, the adsorbed CF₄ species interact with adjacent hydroxyl groups, leading to C–F bond weakening and cleavage. Pt-containing particles serve as active components that promote both surface acidity and hydroxyl formation, thereby accelerating the overall hydrolysis process. This synergistic pathway enables Pt_{0.5}/Al₂O₃ to achieve complete CF₄ decomposition at 600 °C with high stability.

3. Materials and Methods

3.1. Chemicals

γ-Al₂O₃ and chloroplatinic acid were purchased from Shanghai Aladdin Reagent Co., Ltd. (Shanghai, China). All chemicals were used as received, without any further purification.

3.2. Synthesis of Pt/Al₂O₃

A Pt/Al₂O₃ catalyst was prepared using the wet impregnation method. Typically, 5 g of Al₂O₃ are dispersed in 300 mL of deionized water and sonicated for 60 min. Then, aqueous solutions of chloroplatinic acid at 1 mmol/mL are added dropwise 222 μL, 333 μL, 666 μL for preparing Pt_{0.3}/Al₂O₃, Pt_{0.5}/Al₂O₃, Pt₁/Al₂O₃ catalysts. After sonication for another 30 min, the suspension is evaporated at 85 °C for 2 h using a rotary evaporator. Finally, the sample is calcined in a tube furnace at 600 °C for 5 h.

3.3. Catalytic Reaction

The CF₄ hydrolysis reaction was conducted in a continuous flow reaction system with a quartz fixed-bed reactor (20 mm i.d.) under atmospheric pressure. A 33.3 mL min^{−1} of gas flow (0.25% CF₄ in Ar) controlled by a mass flow controller, while a micro feeder delivered 0.006 (liquid) or 8.16 (vapor) mL min^{−1} of water over 2.0 g of catalyst.

According to the following equations, the CF₄ decomposition is calculated:

$$\text{CF}_4 \text{ Decomposition \%} = \frac{[\text{CF}_4]_{\text{in}} - [\text{CF}_4]_{\text{out}}}{[\text{CF}_4]_{\text{in}}} \times 100\%$$

where [CF₄]_{in} and [CF₄]_{out} indicate the input and output relative gas concentrations, respectively.

3.4. Characterization of Catalysts

The X-ray diffraction (XRD) patterns were obtained with Ni-filtered Cu-Kα (λ = 1.540598 Å) (40 kV, 40 mA) radiation in the 2θ range of 10° to 90° with a scan rate of 1 °/min. The XRD is made by Japan's Rigaku Smart Lab SE (Rigaku Corporation, Tokyo, Japan). The morphologies of the samples were determined by TESCAN MIRA3 field emission scanning electron microscope (LMH, Brno, Czech Republic) and high resolution transmission electron microscopy with a spherical aberration corrector (HRTEM, Titan G2 60-300) equipped with energy dispersive X ray spectroscopy (EDS) mapping. The HRTEM is made by Japan's JEOL, model JEM-F200 (Tokyo, Japan). NH₃-TPD and CF₄-TPD were performed by using a PCA-1200 on a chemisorption analyzer equipped with a thermal

conductivity detector (TCD). The chemisorption analyzer was carried out on the PCA-1200 from Beijing Builder electronic technology Co., Ltd. (Beijing, China).

4. Conclusions

In summary, Pt-modified γ - Al_2O_3 catalysts were developed for efficient CF_4 hydrolytic decomposition. XRD and TEM results confirmed that the γ - Al_2O_3 structure was maintained after Pt introduction, while platinum oxide-related species gradually formed with increasing Pt loading. Elemental mapping demonstrated the homogeneous distribution of Pt on the Al_2O_3 support. NH_3 -TPD and CF_4 -TPD results revealed that Pt incorporation enhanced the surface acidity and strengthened CF_4 adsorption. In situ infrared spectroscopy further showed that Pt/ Al_2O_3 generated more surface hydroxyl groups than pristine Al_2O_3 , indicating improved water activation capability. These hydroxyl species, together with enhanced acid sites and Pt-containing active particles, promoted C–F bond activation during CF_4 hydrolysis. Consequently, Pt/ Al_2O_3 achieved complete CF_4 decomposition at 600 °C and exhibited excellent long-term stability. This work provides an effective strategy for designing alumina-based catalysts for perfluorinated compound decomposition through Pt-induced regulation of acidity, adsorption, and hydroxyl-mediated activation.

Author Contributions: All authors contributed to the study conception and design. Z.W., X.H. developed and designed the methodology of this experiment. M.X., R.C. and T.Y. prepared the original draft. J.L. and W.L. supervised the project and had leadership responsibility for the research. R.C. is in charge of various tests as well as the related data processing and analysis. W.L. is in charge of coming up with new ideas, organizing the thinking, and editing the paper. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no competing interests.

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