Core-Shell-Like Structured Co$_3$O$_4$@SiO$_2$ Catalyst for Highly Efficient Catalytic Elimination of Ozone

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Co$_3$O$_4$ is an environmental catalyst that can effectively decompose ozone, but is strongly affected by water vapor. In this study, Co$_3$O$_4$@SiO$_2$ catalysts with a core-shell-like structure were synthesized following the hydrothermal method. At 60% relative humidity and a space velocity of 720,000 h$^{-1}$, the prepared Co$_3$O$_4$@SiO$_2$ obtained 95% ozone decomposition for 40 ppm ozone after 6 h, which far outperformed that of the 25wt% Co$_3$O$_4$/SiO$_2$ catalysts. The superiority of Co$_3$O$_4$@SiO$_2$ is ascribed to its core@shell structure, in which Co$_3$O$_4$ is wrapped inside the SiO$_2$ shell structure to avoid air exposure. This research provides important guidance for the high humidity resistance of catalysts for ozone decomposition.

Keywords: ozone decomposition, Co$_3$O$_4$, SiO$_2$, Core@Shell structure, relative humidity

1. introduction

INTRODUCTION

Ozone is widely used in food, medicine, and waste treatment owing to its excellent oxidizing ability (Alameddine et al., 2021; Kim et al., 2022). However, even low concentrations of ozone are harmful to human health, especially to the eyes, nose, and throat (Ferrara et al., 2020; Ferrara et al., 2021). The maximum eight-hour average concentration of ozone allowed by the World Health Organization is 100 μg/m$^3$. Ozone concentrations in the atmosphere near ground level have considerably increased in recent years due to increased levels of volatile organic compounds and nitrogen oxides (Ou et al., 2016). Ozone in the outdoor air can infiltrate into indoor environments. Indoor ozone is considered more harmful than outdoor ozone because modern humans spend most of their time indoors (Abbass et al., 2017; Namdari et al., 2021; Nazaroff and Weschler 2021). The development of environmental technologies to effectively eliminate ozone is therefore necessary.

There are four common treatment methods to eliminate ozone: activated carbon (Yu et al., 2020); absorption (Yang et al., 2017); thermal decomposition and catalytic decomposition (Gopi et al., 2017; Ma et al., 2017; Gong et al., 2018). Catalytic decomposition is considered to be one of the most feasible and effective methods for ozone removal (Li et al., 2020). Noble metals and transitional metal oxides are common catalysts for heterogeneous reactions including decomposition of ozone (Nikolov et al., 2010; Gong et al., 2017; Deng et al., 2019; Tao et al., 2021a; Tao et al., 2021b). Among the transition metal oxides, Co$_3$O$_4$ catalysts with higher oxidation states have exhibited higher ozone decomposition performance than other cobalt oxide catalysts (Tang et al., 2014a). Abdedyam (Abdedyam et al., 2017) demonstrated that the ozone decomposition abilities of Co$_3$O$_4$ support on loaded olivine is proportional to its dispersion degree. However, numerous metal oxide catalysts suffer from interactions with water vapor, and including cobalt oxides (Zhu et al., 2021). It is generally believed that water vapor affects ozone decomposition via competitive adsorption with the transition metal oxides on the active sites (Jia et al., 2016).
In this study, core@shell structure catalysts were synthesized with mesoporous silica as the shell and Co$_3$O$_4$ nanoparticles as the core (Co$_3$O$_4$@SiO$_2$) following the solvothermal route using polyvinylpyrrolidone (PVP) as the capping agent. For comparison, spherical silica supported different additions of Co$_3$O$_4$ and labeled xCo$_3$O$_4$/SiO$_2$, where x = 10, 15, and 20, or 25%. The ozone decomposition performance of xCo$_3$O$_4$/SiO$_2$ increased with increasing Co$_3$O$_4$. The 25Co$_3$O$_4$/SiO$_2$ and Co$_3$O$_4$@SiO$_2$ catalysts yielded high ozone decomposition activity at 20% relative humidity. The ozone elimination activity of 20Co$_3$O$_4$/SiO$_2$ sharply decreased upon increasing the relative humidity to 60%, and the Co$_3$O$_4$@SiO$_2$ catalyst exhibited a better moisture resistance performance for ozone decomposition. This study provides important insights for the further development of coated catalysts for gaseous ozone decomposition.

**EXPERIMENTAL METHODS**

**Catalyst Preparation**

Co$_3$O$_4$@SiO$_2$ was synthesized in accordance with previously published studies (Khan et al., 2015). First, 0.70 g PVP and
0.35 g Co(NO₃)₂·6H₂O were dissolved in 40 ml ethanol. The solutions were transferred to stainless steel lined with Poly tetra fluoroethylene (PTFE) in an autoclave and heated at 453 K for 4 h. The obtained black powder was dispersed in 103.8 ml ethanol, to which 82.8 ml distilled water, 7.2 ml 25% aqueous ammonia solution, 0.3 g cetyltrimethylammonium bromide, and 1.0 ml tetraethoxysilane were added. The solution was stirred for 48 h at room temperature. The product was collected via filtration, washed three times with distilled water, dried at 333 K, and then calcined at 773 K for 6 h. The finished samples were denoted as Co₃O₄@SiO₂ (wt% = 30%). SiO₂ was impregnated with 10, 15, 20, or 25% cobalt loading in an ethanol solution of cobalt nitrate, and the resulting product was calcined at 773 K for 6 h. The prepared samples were labeled as 10Co₃O₄/SiO₂, 15Co₃O₄/SiO₂, 20Co₃O₄/SiO₂, and 25Co₃O₄/SiO₂, respectively.

**Catalyst Characterization**
The samples were characterized by X-ray diffraction (XRD) using a D/max-RB diffractometer. X-ray photoelectron spectroscopy (XPS) was performed using a Thermo Fisher ESCALAB 250Xi. Morphological and microstructural characterizations were carried out using a Hitachi EM-3010 transmission electron microscope (TEM). The surface areas were calculated by the Brunauer-Emmett-Teller (BET) method. The pore diameters were estimated from the desorption branches of the isotherms based on the Barrett-Joyner-Halenda (BJH) model.

**Catalyst Test**
The ozone decomposition activity of the prepared catalysts was evaluated using a flow-through quartz tube reactor (inner diameter = 10 mm) with 0.10 g of catalyst separated by quartz sand at different temperatures and relative humidity (20, 40, and 60%) under atmospheric pressure conditions. Ozone was generated by flowing 20% O₂/N₂ compressed gas through an ozone generator. The relative humidity of the gas stream was measured using a humidity probe (Benetech, GM1361+). The total gas flow rate passing through the quartz reactor was controlled at 1,500 ml/min and contained 40 ppm O₃. The ozone concentrations at the inlet and outlet were detected using a 106-L ozone online analyzer (2B Technologies, Boulder, Co, United States). The ozone conversion was calculated according to:

\[
\text{Ozone conversion} = \frac{c_i(O_3) - c_o(O_3)}{c_i(O_3)} \times 100\%
\]

where \(c_i(O_3)\) and \(c_o(O_3)\) represent the inlet and outlet ozone concentrations, respectively.

**RESULTS AND DISCUSSION**

**Catalyst Characteristics**
The morphology and nanostructure of the catalysts were observed by TEM. Figure 1A–C show that the Co₃O₄@SiO₂ nanoparticles were relatively dispersible with an average size of 40 nm. This indicates that PVP can prevent Co₃O₄ nanoparticle agglomeration under hydrothermal conditions. Figure 1D–F show that the spherical Co₃O₄/SiO₂ composites prepared via incipient wetness impregnation were highly dispersed with a relatively smooth external surface. This indicates that a
majority of the Co₃O₄ nanoparticles were incorporated into the mesopores (Xie et al., 2011). With regard to the spent Co₃O₄/SiO₂ catalyst, the large aggregates were clearly located on the external surface of the spherical SiO₂ support, and indicating that small Co₃O₄ nanoparticles outside of the mesopores easily agglomerated into large Co₃O₄ aggregates during the reaction.

X-ray photoelectron spectroscopy (XPS) tests were performed to detect the chemical state and composition of the element catalyst surface. According to the previously reported Co₃O₄ spectrum (Gao et al., 2021), the Co 2p spectrum of Co₃O₄ (Figure 2A) consists of two peaks, Co 2p⁰/₂ and Co 2p¹/₂, located at 779.9 and 794.8 eV, respectively. However, the Co 2p⁰/₂ and Co 2p¹/₂ peaks in the Co₃O₄@SiO₂ and Co₃O₄/SiO₂ catalysts shifted to approximately 781.0 and 796.0 eV, respectively, both of which occur at higher energies than those of pure Co₃O₄. This shift is mainly due to the interaction between the silica and Co₃O₄ species, which results in a charge transfer from the Co₃O₄ to the SiO₂ support and has a positive impact on the cobalt catalytic performance.

The atomic surface contents of cobalt were 0.8 and 7.6% for the Co₃O₄@SiO₂ and Co₃O₄/SiO₂ catalysts, respectively. This significant difference further confirms that the preparation of Co₃O₄@SiO₂ successfully encapsulated Co₃O₄ into the SiO₂ matrix. The O 1s spectra of the catalysts are shown in Figure 2B. The main O 1s peak centered at 533.0 eV represents the lattice oxygen of Co₃O₄ and SiO₂, but is difficult to be accurately distinguished. The oxygen in the unreducible silica has no notable effect on the catalysis of ozone.

Figure 3 shows the nitrogen isothermal adsorption-desorption curves and pore size distributions of the Co₃O₄@SiO₂ and 25Co₃O₄/SiO₂ catalysts. The nitrogen adsorption-desorption isotherms clearly show that both samples have typical hysteresis loops and are classified as type-IV isotherms, thus indicating that the samples have a mesoporous structure. The average pore diameter of the two samples ranges between 6 and 9 nm. The pore volume of Co₃O₄@SiO₂ (0.15 cm³/g) is larger than that of Co₃O₄/SiO₂ (0.11 cm³/g). The specific surface area of Co₃O₄@SiO₂ is 94.8 m²/g, which is 1.5 times greater than that of Co₃O₄/SiO₂ (68.8 m²/g). The specific surface area of a catalyst is

FIGURE 4 | (A) Ozone removal rates and (B) XRD patterns of Co₃O₄@SiO₂ and xCo₃O₄ (x = 10, 15, 20, and 25). Temperature = 25°C; relative humidity = 20%.

FIGURE 5 | Ozone removal rate of 25Co₃O₄@SiO₂ (A) and Co₃O₄/SiO₂ (B). Temperature = 25°C; relative humidity (RH) = 20, 40, and 60%.
generally believed to have a substantial impact on the catalytic activity, in which catalysts with larger specific surface areas usually have higher catalytic activities. Effect of Co$_3$O$_4@$SiO$_2$ and Co$_3$O$_4$/SiO$_2$ on ozone decomposition.

The ozone decomposition rates of Co$_3$O$_4@$SiO$_2$ and Co$_3$O$_4$/SiO$_2$ with different Co$_3$O$_4$ loadings were evaluated in a gas flow with 40 ppm ozone at 20% relative humidity. The activity of the 10Co$_3$O$_4$/SiO$_2$ catalyst dropped sharply within 1 h, and the 15Co$_3$O$_4$/SiO$_2$ and 20Co$_3$O$_4$/SiO$_2$ catalysts dropped to 94% ozone conversion after 4 h. The time to achieve 100% ozone removal rate increased to 9 h for a Co$_3$O$_4$ load of 25%. However, the Co$_3$O$_4@$SiO$_2$ catalyst with 30 wt% loading achieved the same ozone removal rate as that of 25Co$_3$O$_4$/SiO$_2$. This indicates that the ozone elimination rate is proportional to the Co$_3$O$_4$ catalyst load. The XRD patterns of the as-prepared catalysts are shown in Figure 4B, in which all of the obtained samples exhibit the same peaks, corresponding to pure Co$_3$O$_4$ (JCPDS No. 42-1,467) (Agilanadeswari and Rubankumar 2016). This indicates that the crystalline phase is well maintained during the treatment. The diffraction peaks of both Co$_3$O$_4@$SiO$_2$ and xCo$_3$O$_4$/SiO$_2$ are sharp and intense, and the peak intensities gradually increase with increasing Co$_3$O$_4$ catalyst load. The 25Co$_3$O$_4$/SiO$_2$ catalyst exhibits more intense peaks at 36.5 than Co$_3$O$_4@$SiO$_2$ for a similar Co$_3$O$_4$ content. This indicates that the Co$_3$O$_4@$SiO$_2$ core-shell structure weakens the intensity of the characteristic peaks, and that the Co$_3$O$_4$ crystalline material is well inside the mesoporous silica particles. Effect of 25Co$_3$O$_4@$SiO$_2$ and Co$_3$O$_4$/SiO$_2$ on ozone decomposition under different relative humidity conditions.

Figure 5A,B show the ozone removal rates of 25Co$_3$O$_4@$SiO$_2$ and Co$_3$O$_4$/SiO$_2$, respectively, at relative humidity conditions of 20, 40, and 60%. The 25Co$_3$O$_4@$SiO$_2$ and Co$_3$O$_4$/SiO$_2$ catalysts exhibit similar ozone removal rates at 20% relative humidity. The 25Co$_3$O$_4$/SiO$_2$ catalyst shows 99% ozone conversion for 11 h at 20% relative humidity. The removal rate then sharply drops and ultimately stabilizes at 60%. It is noted that the ozone removal rate sharply decreases with increasing relative humidity, especially when the relative humidity is increased from 40 to 60%. For Co$_3$O$_4@$SiO$_2$, the ozone removal rate begins to decrease during the first 12 h of the reaction runs with a gas flow of 40 ppm ozone, and then decreases to 60% when the reaction has been maintained for 24 h. The ozone removal rate of Co$_3$O$_4@$SiO$_2$ shows a different trend from that of 25Co$_3$O$_4$/SiO$_2$ at 40% relative humidity. When the relative humidity is increased to 60%, the ozone removal rate sharply decreases and remains at 30%, which is approximately 10% higher than that of 25Co$_3$O$_4$/SiO$_2$. This indicates that the main reason for the different performance of the two catalysts is their differing structures. The Co$_3$O$_4$ loaded on the surface of SiO$_2$ is directly exposed to the reaction environment. The accumulation of oxygen atoms and adsorption of water vapor thus lead to catalyst deactivation. In contrast, in the Co$_3$O$_4@$SiO$_2$ catalyst, the Co$_3$O$_4$ is wrapped by SiO$_2$, and which isolates water vapor and prevents it from directly contacting with the Co$_3$O$_4$. The deactivation can thus be attributed to the accumulation of oxygen atoms.

**Proposed Mechanism**

According to the experimental results, we proposed a possible mechanism involving oxygen vacancies (O$_{\text{V}}$) as depicted below. Initially, the ozone molecule is adsorbed on the oxygen vacancy of the surface of Co$_3$O$_4$ and the ozone decompose into oxygen, while another oxygen atom is left on the surface of Co$_3$O$_4$ and form lattice oxygen (O$^{2-}$). Subsequently, the ozone molecule reacts with lattice oxygen and form oxygen and O$_2^{2-}$. Finally, the O$_2^{2-}$ breaks off the Co$_3$O$_4$ surface in form of oxygen.

\[
\begin{align*}
O_3 + O_{\text{V}} & \rightarrow O_2 + O^{2-} \quad (R1) \\
O_3 + O^{2-} & \rightarrow O_2 + O_2^{2-} \quad (R2) \\
O_2^{2-} & \rightarrow O_2 + O_{\text{v}} \quad (R3)
\end{align*}
\]

**CONCLUSION**

In this work, Co$_3$O$_4@$SiO$_2$ and xCo$_3$O$_4$/SiO$_2$ (x = 10, 15, 20, and 25) catalysts were successfully synthesized using the hydrothermal method. Under similar loading conditions, the ozone removal rates of Co$_3$O$_4@$SiO$_2$ and 25Co$_3$O$_4$/SiO$_2$ were nearly the same under flow conditions of 40 ppm ozone and 20% relative humidity. When the relative humidity increased to 60%, the ozone removal rate of Co$_3$O$_4@$SiO$_2$ was higher than that of 25Co$_3$O$_4@$SiO$_2$. XRD, XPS, and BET characterizations indicate that the high Co$_3$O$_4@$SiO$_2$ performance is related to the core@shell structure. This study thus provides insight for developing catalysts to effectively remove gaseous ozone.

**DATA AVAILABILITY STATEMENT**

The original contributions presented in the study are included in the article/Supplementary Material, further inquiries can be directed to the corresponding authors.

**AUTHOR CONTRIBUTIONS**

FC and QX conceived the idea. JD, YC, and FH designed and fabricated the sample, ZM, DC, MC, GZ, JK, and SX conducted the experiment. All the authors contributed to analysis of the data and draft of the manuscript.

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**SUPPLEMENTARY MATERIAL**

The Supplementary Material for this article can be found online at: https://www.frontiersin.org/articles/10.3389/fchem.2021.803464/full#supplementary-material
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