

A COMPARISON OF THE CATALYTIC OXIDATION OF FORMALDEHYDE OVER THREE TYPES OF CATALYSTS AT AMBIENT TEMPERATURE: THE EFFECT OF SUPERFICIAL VELOCITY

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Abstract. Catalytic oxidation of formaldehyde (HCHO) at ambient temperature is regarded as a promising technology for removal and destruction of HCHO contaminated air. In this study, three types of catalyst which are commercial HCHO catalyst, the catalyst of granular activated carbon supported Pt (Pt/GAC catalyst) and the catalyst of granular activated carbon supported MnO_x (MnO_x/GAC catalyst) were chosen for oxidizing HCHO. The impact of superficial velocity, reaction bed height and the type of catalyst to the removal efficiency of HCHO was investigated. The fact that removal efficiency of HCHO increased firstly and then decreased with the decrease of superficial velocity illustrates its effect on the catalytic oxidation existed at an optimum value, depending on the nature of different catalysts, design of the reaction bed and the operation conditions. The steady state removal efficiencies of HCHO were all above 99% when the superficial velocity was 0.5, 1.0 and 1.5 m/s respectively, yet the needed height shortened with decreasing the superficial velocity. Among the three types of catalysts, Pt/GAC catalyst shows the effective and stable catalytic activity, whose removal efficiencies reached over 90% and the conversion did not change too much with the time increasing.

Keywords: *Pt/GAC catalyst, MnO_x/GAC catalyst, commercial HCHO catalyst, HCHO removal, reaction bed height, room temperature*

Introduction

Formaldehyde (HCHO) is regarded as one of the most common indoor air pollutants, which is emitted from building and furnishing materials (Perry, 1995). Owing to the teratogenicity and carcinogenicity, its pollution problem gets more and more attention (Salthammer et al., 2010; Lu et al., 2017). A series of efforts have been made for abating its emission at room temperature to meet the environmental regulations. Although the physical adsorption or chemical reaction has been proven to remove HCHO, the effectiveness is limited by the material's capacities (Nakayama et al., 2002). On the contrary, due to its low energy consumption and environment-friendly reaction conditions, room-temperature catalytic oxidation, which can completely oxidize HCHO into harmless CO₂ and H₂O, has become a promising technology for removal and destruction of HCHO contaminated air and has attracted extensive attention (Spivey, 1989; Qi et al., 2015; Yan et al., 2016; Ye et al., 2016; Yang et al., 2017).

Recently, dozens of studies have shown that the supported noble metal catalysts (such as Pt, Ru, Pd, Au and so on) exhibit high catalytic activity at room temperature (Yang et al., 2017; Zhang et al., 2006; Huang et al., 2011; An et al., 2013; Quiroz et al., 2013). Among them, the high efficiency of catalytic oxidation by supported Pt catalysts has been proven (Peng and Wang, 2007). It was reported by Zhang et al. (Zhang et al.,

2006) that 100% HCHO decomposition into CO₂ and H₂O was achieved over 1 wt% Pt/TiO₂ catalyst at ambient temperature. However, because of high cost, this catalyst cannot be widely applied in the engineering field (Yusuf et al., 2017).

Another approach for removing HCHO is to combine activated carbon adsorption with catalytic oxidation (Granqvist et al., 2007). Many catalysts have been shown to have high removal efficiency when applied with activated carbon, such as photo-catalysts (Bashkova et al., 2011), silver catalysts (Li et al., 2016), metal oxides catalysts (Fang et al., 2017) and so on. Li et al. (Li et al., 2016) reported that the catalyst of granular activated carbon supported MnO_x had a high activity to remove HCHO at ambient temperature, but its removal efficiency dropped from 75% to 10% after running continuously for 30 h. Fang et al. (Fang et al., 2017) investigated the effectiveness of coconut shell activated carbon supported MnO_x for catalytic oxidation of HCHO at room temperature. The HCHO removal efficiency was kept almost at 100% during a 1000 min period.

For HCHO, the efficiency of the catalytic reaction is strongly affected by factors, such as temperature, humidity, gas hourly space velocity (GHSV), inlet HCHO concentration and the properties of the catalyst (preparation method, morphology and structure, specific surface active area, active sites, low-temperature reducibility and surface active oxygen species) and so on. It was reported that the removal efficiency of HCHO decreased with the increase of the inlet HCHO concentration and GHSV (Xia et al., 2010; Han et al., 2016). Chen et al. (Chen et al., 2013) examined the effect of GHSV on HCHO conversion over 1 wt% Au/CeO₂ (DPU) at room temperature in the range of 34000-143000 h⁻¹. The steady conversion of HCHO reached 100% at all GHSV (34000, 95500, 143000 h⁻¹), but the reaching time was reduced with the increase of GHSV. Besides, Han et al. (Han et al., 2016) investigated that HCHO catalytic efficiencies for all the prepared catalysts increased with the rising reaction temperature. Though various factors influencing the HCHO decomposition have been reported in many kinds of literature, there are few studies related to the effect of superficial velocity. Besides, finding high activity of catalyst to oxidize low-concentration HCHO at room temperature also needs to be investigated further (Zhang et al., 2005; Tang et al., 2008).

On the bases of the reports above, studies on the catalytic oxidation of low-concentration HCHO at room temperature still need to be further developed and completed. Therefore, in this study, we investigated the properties and efficiencies of two different typical catalysts (Pt/GAC catalyst and MnO_x/GAC catalyst) for removing HCHO, compared with the commercial HCHO catalyst. Moreover, the effects on catalytic oxidation were studied concerning the factors of superficial velocity, reaction bed height and the types of catalysts.

Materials and methods

Catalyst preparation

The catalysts used in this study were commercial HCHO catalyst (10 × 10 × 10 cm³, five blocks), Pt/GAC catalyst (10 × 10 × 10 cm³, one block), and the MnO_x/GAC catalyst. The activated carbon used in this study was coal-based columnar carbon with an averaged diameter of 4 mm. The carbon tetrachloride adsorption activity of GAC was 82.95%. The details of Pt/GAC catalyst preparation were as follows: Chloroplatinic acid solution with Pt was prepared and used as the impregnation liquid, then GAC (10 × 10 × 10 cm³ per block) was immersed in the solution at room temperature till they

are mixed completely. A series of steps including dehumidification, purification and reduction reaction were completed in the preparation (Zhang et al., 2015; Zhu et al., 2016). The MnO_x/GAC catalyst was obtained via in-situ reduction of potassium permanganate with GAC. The MnO_x/GAC catalyst was prepared by the reaction of in-situ synthesis. The details of MnO_x/GAC catalyst preparation were as follows: the GAC was immersed in the KMnO_4 solution (concentration of 1%) for 1 h. The obtained MnO_x/GAC catalyst was then filtered and dried at $120\text{ }^\circ\text{C}$ for 1 h (Fang et al., 2017).

Experimental setup

The general configuration of the apparatus used to test the catalytic performance with the commercial HCHO catalyst or the Pt/GAC catalyst is depicted in *Figure 1*. The apparatus consisted of three parts: gas distribution system, catalytic system and detection system. Liquid HCHO (ACS reagent grade) was delivered by a syringe pump (LongerPump, LSP01-2A, United Kingdom), evaporated and mixed with contaminant-free compressed air. The air mixture flowing through a rotameter and a buffer to measure and regulate the gas flow contained 9 ppm HCHO and clean air, which was introduced as the reactants. The catalytic system included a wind turbine and catalytic reactor. Owing to the large catalyst resistance, the catalytic system had a wind turbine. The catalytic oxidation of HCHO was performed in a quartz tubular fixed-bed reactor (five levels) under atmospheric pressure at room temperature ($25 \pm 1\text{ }^\circ\text{C}$). The catalyst was respectively loaded in the five-story reactor, installing a perforated stainless-steel plate at the bottom of each section. Valves were installed in the front of and behind every catalytic unit to measure contaminant concentrations.

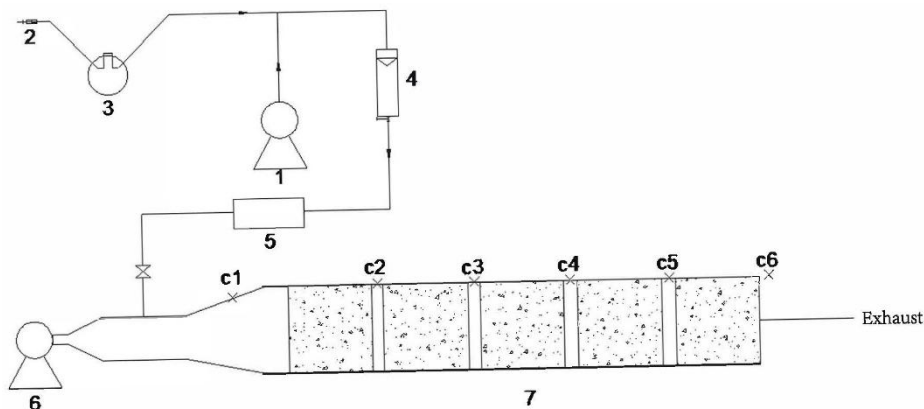


Figure 1. Schematic diagram of apparatus used for catalytic performance with the commercial HCHO catalyst or the Pt/GAC catalyst. (1, 6: blower; 2: syringe pump; 3: temperature-controlled magnetic stirrer; 4: rotameter; 5: surge flask; 7: catalytic unit; c1-c6: sampling port)

The Catalytic performance with the MnO_x/GAC catalyst in the experiment was set up as shown in *Figure 2*. As in the above process, the air mixed with HCHO, then adjusted to the desired concentration. The catalyst (the depth of 10 cm) was installed in the packed-columns which were made of glass (i.d. 40 mm) and operated in the downflow mode. Valves were installed before the catalytic unit inlet and after the catalytic column to measure contaminant concentrations.

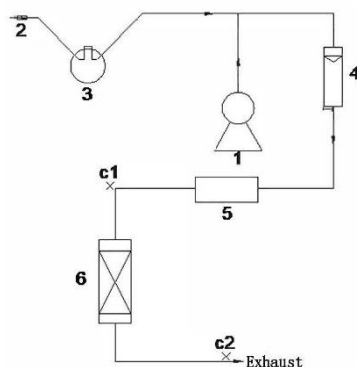


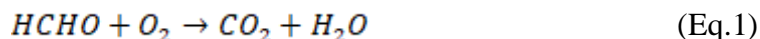
Figure 2. Schematic diagram of apparatus used for catalytic performance with the MnO_x/GAC catalyst. (1: blower; 2: syringe pump; 3: temperature-controlled magnetic stirrer; 4: rotameter; 5: surge flask; 7: catalytic unit; c1-c2: sampling port)

Experimental testing and analytical techniques

During the catalytic oxidation of HCHO, ~9 ppm HCHO was injected mixed with clear air as a contaminated gas. The superficial velocity was set by 0.5, 1.0, 1.5 m/s for the commercial HCHO catalyst and Pt/GAC catalyst and 0.2, 0.5, 1.0 m/s for the MnO_x/GAC catalyst. The HCHO in the airstream was analyzed by an HCHO monitor (Formaldemeter 400, PPM Technology, United Kingdom).

The results of removal efficiency of HCHO under different parameters were compared and analyzed statistically. The multivariate analysis was applied to identify the significance of selected differencing factors.

Equation 1 below shows the complete reaction process of HCHO decomposition in the catalytic oxidation:



The results of each selected influencing factors of removal efficiency were analyzed statistically. The HCHO removal efficiency was calculated by Equation 2 as follows:

$$\text{The Removal efficiency of HCHO} = \frac{[\text{HCHO}]_{\text{in}} - [\text{HCHO}]_{\text{out}}}{[\text{HCHO}]_{\text{in}}} \times 100\% \quad (\text{Eq.2})$$

where [HCHO]_{in} and [HCHO]_{out} are the inlet and outlet HCHO concentration respectively (both expressed in ppm).

Results and discussion

The influence factors of the catalytic reaction, including superficial velocity, reaction bed height, the types of catalysts, were studied to evaluate the catalytic activity and stability of the catalysts.

Effect of superficial velocity on removal efficiency of HCHO

Superficial velocity has a vital effect on the adsorption time and catalytic oxidation time, but only limited studies focused on it. Keeping the inlet HCHO concentration of

9 ppm, the impact of superficial velocity on the catalytic performance was investigated by setting the velocity from 0.5 to 1.5 m/s for the commercial HCHO catalyst and Pt/GAC catalyst and from 0.2 to 1.0 m/s for the MnO_x/GAC catalyst. The results are shown in *Figure 3*. For the Pt/GAC catalyst (*Fig. 3b*) and the MnO_x/GAC catalyst (*Fig. 3c*), when the superficial velocity was increased, the removal efficiency for HCHO would be decreased. However, for the commercial HCHO catalyst (*Fig. 3a*), the removal efficiencies from the highest to the lowest were SV = 1.0 m/s > SV = 1.5 m/s > SV=0.5 m/s.

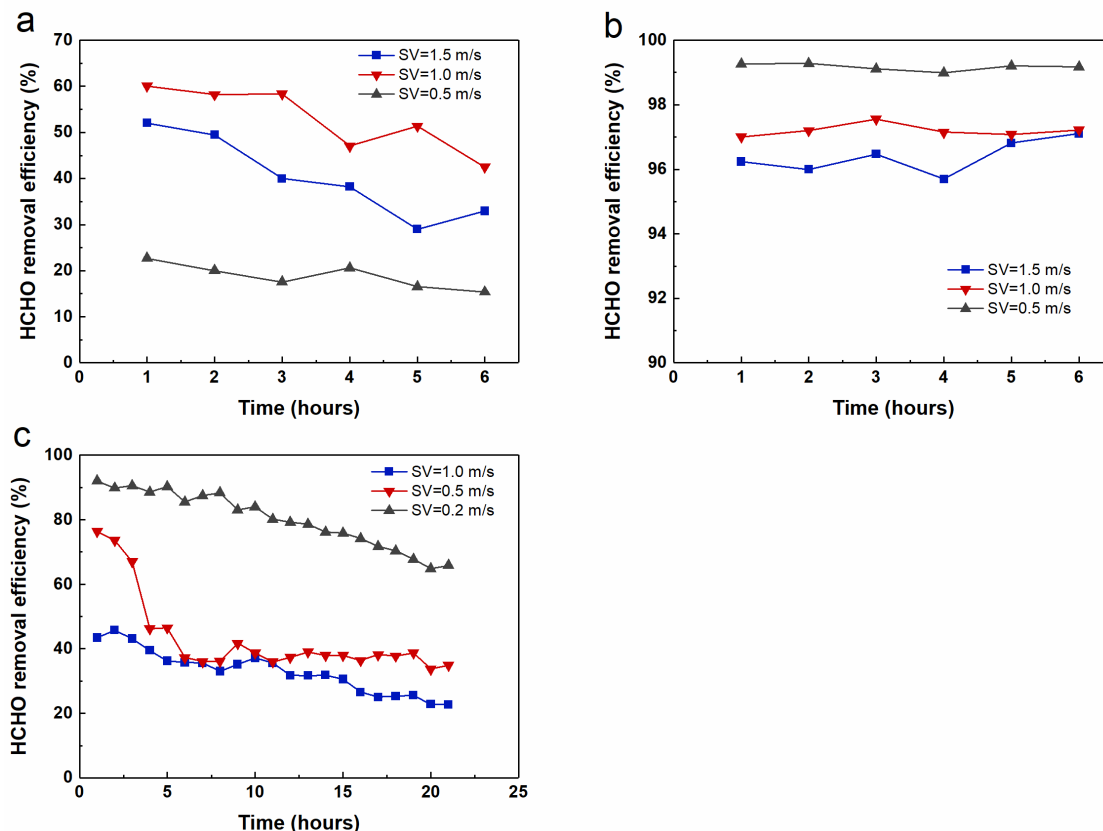


Figure 3. Removal efficiencies of HCHO over three types of catalysts under different superficial velocities. (a: the commercial HCHO catalyst; b: the Pt/GAC catalyst; c: the MnO_x/GAC catalyst; SV refers to superficial velocity)

According to the *Figure 3*, the nature of different catalysts showing different catalytic activities was related to the influence of superficial velocity on removal efficiency of HCHO. For the commercial HCHO catalyst, in a certain scale, the removal efficiency increased with the reduction of superficial velocity. Further decrease of the superficial velocity to 0.5 m/s appeared no positive effect on HCHO conversion, even a sharp decrease. These results can be understood by considering the fact that HCHO molecules fully exposed to the catalyst and participated in the removal reaction per unit would increase with the decrease of superficial velocity, which resulted in improving the removal efficiency correspondingly. Zhang et al. (Zhang et al., 2005) investigated the impact of air-flow rate on catalytic performance, which illustrates that the removal efficiency of HCHO in the catalytic oxidation was inversely proportional to the air-flow

rate and the correlation coefficient (R^2) reached 0.99. However, below a certain range of superficial velocity, because of the increase of residence time and HCHO molecules, the competition for the active catalytic sites on the catalyst surface would strengthen, leading to a decline of the removal efficiency. The Pt/hydrophobic catalyst was tested under different gas flow rates by Chuang et al. (1994), where HCHO conversion percentage showed a small increase when the gas flow rate increased from 6 to 18 L/h. The fact shows that the catalytic oxidation was dominated by the diffusion rate of HCHO molecules onto the surface of catalyst. For the Pt/GAC catalyst and the MnO_x /GAC catalyst, the fact that removal efficiency of HCHO rose with a decrease in the superficial velocity indicated that the catalytic oxidation was dominated by HCHO molecules onto the catalyst surface, and the competitive reactions between molecules were not obvious. Therefore, the optimal superficial velocity of the Pt/GAC catalyst and the MnO_x /GAC catalyst needed to be investigated further. Huang et al. (Huang et al., 2011) confirmed the conversion of HCHO decreased with the increase of GHSV through examining the effect of GHSV on the catalytic oxidation of HCHO in the range of 40000-240000 h^{-1} . The steady conversion of HCHO was 100% at 40000 h^{-1} which dropped to 90.5% at 240000 h^{-1} .

Overall, these results show that superficial velocity is an important factor which influences the removal efficiency of HCHO, which has the two-side influence on the catalytic oxidation. For one thing, due to the external mass transfer, higher superficial velocity can increase the removal efficiency of HCHO. For another thing, the residence time reduces with the increase of the velocity, resulting in the decrease of removal efficiency. Therefore, the effect of the superficial velocity on the catalytic oxidation existed an optimum value, depending on the nature of different catalysts, design of the reaction bed and the operation conditions (Pei and Zhang, 2011).

Effect of reaction bed height on removal efficiency of HCHO

The effect of reaction bed height on the removal efficiency over the commercial HCHO catalyst at ambient temperature was further investigated by keeping the inlet HCHO concentration at 9 ppm. A plot of removal efficiencies versus reaction bed height determined in the present study operated under different operation times and superficial velocities were conducted as shown in *Figure 4*. The reaction bed heights of 0-50 cm correspond to the height of measured sampling point 1-6, respectively.

For each superficial velocity, the similar trend of removal efficiency appears at different operation time – that the removal efficiency of HCHO at different operation time increased with the rise of reaction bed heights and kept almost unchanged over a certain height. This result suggests that higher reaction bed height provides more reaction time as a result of the fact that the removal efficiency of HCHO increases more.

From *Figure 4a*, keeping the superficial velocity of 1.5 m/s, removal efficiency of HCHO in the height of 10 cm follows the order: $T = 1\text{ h} > 2\text{ h} > 3\text{ h} > 4\text{ h} > 6\text{ h} > 5\text{ h}$ at different operation time, and the efficiency was 52.06% at 1h, dropping to 29.01% at 5 h. With the increase of reaction bed height, the difference of removal efficiency at different operation time decreased gradually, and the efficiencies all reached to above 98% in the height of 40 cm.

As shown in *Figure 4b*, keeping the superficial velocity of 1.0 m/s and removal efficiency of HCHO in the height of 10 cm follows the order: $T = 1\text{ h} \approx 2\text{ h} \approx 3\text{ h} > 5\text{ h} > 4\text{ h} > 6\text{ h}$ at different operation time, and the efficiency was 60.06% at 1 h, dropping to

42.52 % at 6 h. Compared with the efficiency under the superficial velocity of 1.5 m/s, the difference among different operation time narrowed, and the efficiency had improved some. When the height was above 20 cm, the efficiency has little difference at different operation time, reaching to about 95%.

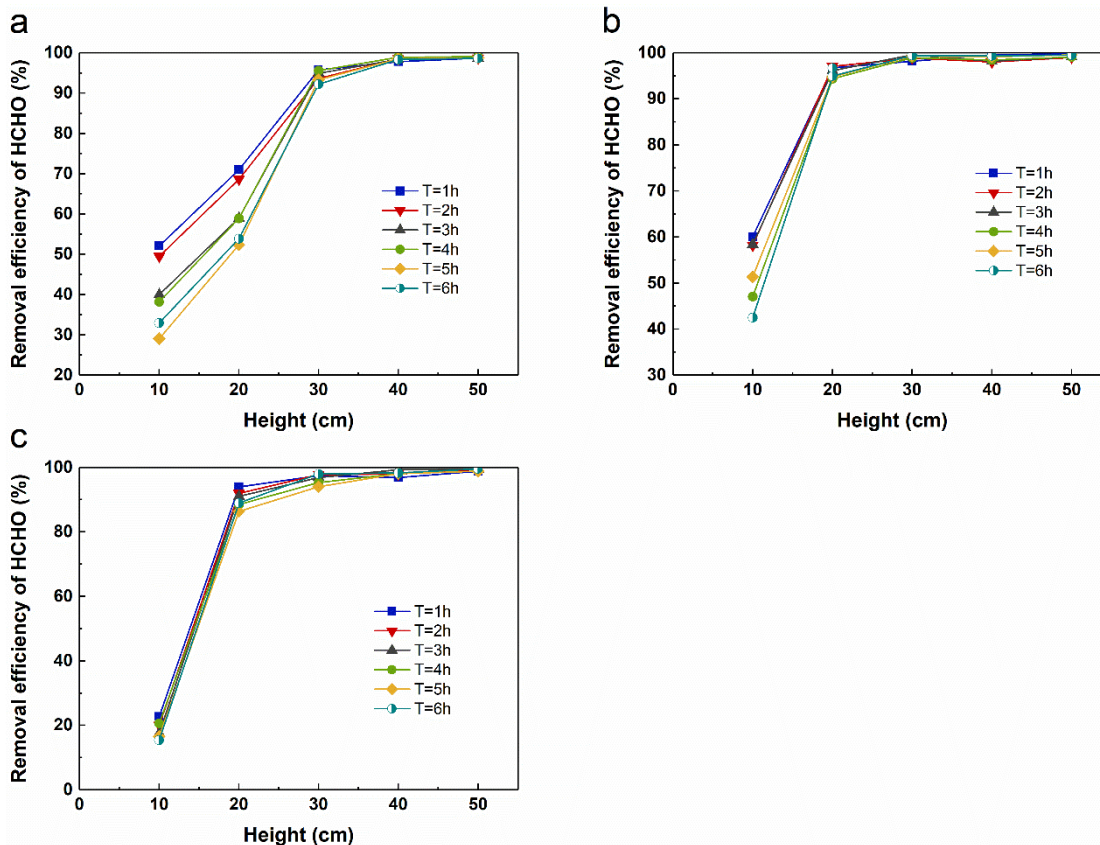


Figure 4. Removal efficiencies of HCHO over the commercial HCHO catalysts under different reaction bed heights. (a: superficial velocity = 1.5 m/s; b: superficial velocity = 1.0 m/s; c: superficial velocity = 0.5 m/s)

The change of removal efficiency with reaction bed height under the superficial velocity of 0.5 m/s was seen in *Figure 4c*. There was little difference at the different operation time in the same height. However, the removal efficiency of HCHO decreased when superficial velocity dropped to 0.5 m/s, only 20% or so at the height of 10 cm.

Overall, the steady state removal efficiencies of HCHO were all above 99% when the superficial velocity was 0.5, 1.0 and 1.5 m/s respectively, yet the needed height shortened with decreasing the superficial velocity, which means that superficial velocity and reaction bed height both play important roles in the catalytic oxidation for HCHO.

To further discuss the effect of reaction bed height to the HCHO destruction, experimentally measured HCHO concentration changing curves, conducted under different superficial velocities and reaction bed heights are shown in *Figure 5*. Each data point in the graph illustrates the average of outlet concentration measured when it ran for one hour.

From *Figure 5*, it is evident that HCHO concentration collected when superficial velocity was 0.5 and 1.0 m/s reduced to 0.2 ppm under the height of 20 cm. However,

HCHO concentration measured when the superficial velocity was 1.5 m/s dropped to 0.2 ppm under the height of 30 cm. With the increase of superficial velocity, the residence time and contact time reduced, which led to the fact that it decreased the removal efficiency of HCHO and needed higher reaction bed to meet the emission standard. After the contaminated air was treated under a certain value, no influence of reaction height was observed on the HCHO concentration.

From the micro-perspective, the slope of that curve represents removal rate of HCHO. According to the results, in the certain height range, the removal rate of HCHO increased with the rise of reaction bed height. When the concentration dropped to certain levels, the removal rate began to decrease rapidly and down to zero finally. This result was consistent with the variation of removal efficiency with reaction bed height.

Multivariate analysis was applied to determine the significance of height and superficial velocity which indicates that reaction bed height has important influence in the removal efficiency of HCHO ($F = 46.362 > F_{crit} = 3.326$) and superficial velocity has no reliable effect ($F = 0.969 < F_{crit} = 4.103$).

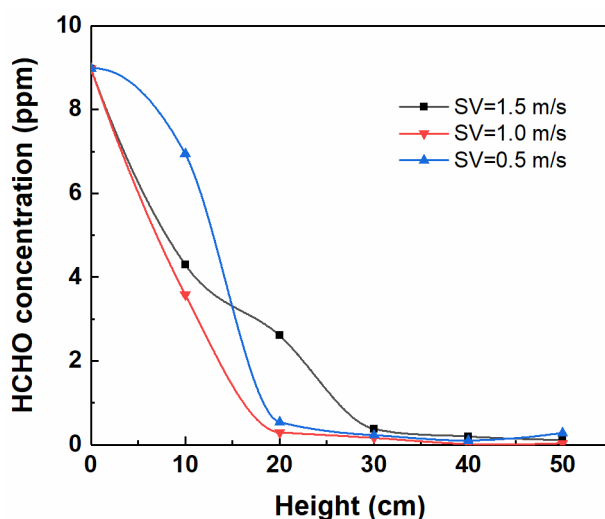


Figure 5. HCHO concentration over the commercial HCHO catalysts under different reaction bed heights. (SV refers to superficial velocity)

Effect of catalyst types on removal efficiency of HCHO

The effect of catalyst types on the removal of HCHO was studied to enhance the removal efficiency and improve the catalytic oxidation method. The catalytic activities of three types of catalysts were evaluated for HCHO conversion under the same superficial velocity (0.5 and 1.0 m/s respectively) as shown in *Figure 6*.

In the case of Pt/GAC catalyst, HCHO could be almost degraded into CO₂ and H₂O under both superficial velocities, whose removal efficiencies reached over 90% and the conversion did not change too much with the time increasing. However, the other catalysts showed lower effects in the same conditions. While the superficial velocity was 1.0 m/s, the commercial HCHO catalyst had a better removal effect than the MnO_x/GAC catalyst, but the conclusion was opposite when superficial velocity dropped to 0.5 m/s. As shown in *Figure 6a*, the removal efficiency of commercial HCHO catalyst dropped slowly from 60.06% to 42.52% after continuously running 6 h, while the efficiency over MnO_x/GAC catalyst fluctuated around 40%. From the *Figure 6b*, the

removal efficiency of HCHO over MnO_x/GAC catalyst at the superficial velocity of 0.5 m/s dropped dramatically from 76.42 to 37.27% within 6 h, while the efficiency over commercial HCHO catalyst changed little, always taking up 20% or so. Therefore, it can be concluded that the influence of catalyst on HCHO removal rate was related to superficial velocity. Besides, these findings illustrated that different activities of different types of catalysts depend on the different capacities for oxidizing HCHO (Zhang and He, 2007). In addition, the support makes a difference in the catalytic oxidation of HCHO at ambient temperature (Colussi et al., 2015).

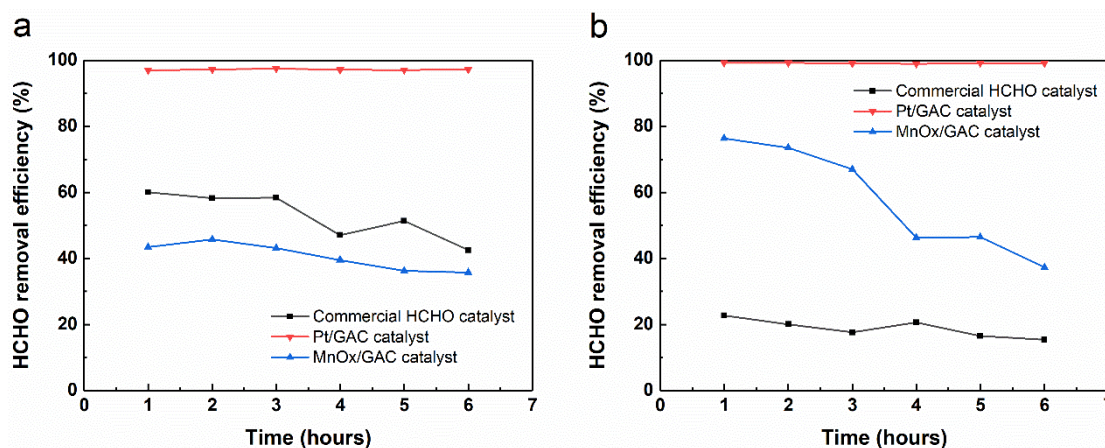


Figure 6. Removal efficiencies of HCHO over three types of catalysts under different superficial velocities. (a: superficial velocity = 1.0 m/s; b: superficial velocity = 0.5 m/s)

Among the three catalysts, the fact that Pt/GAC catalyst shows the effective and stable catalytic property suggests that it is a promising catalyst for HCHO oxidation, but the expensive cost blocked its application from commercialization. The commercial HCHO catalyst possessed the best economical interest and certain catalytic ability but had poor stability. There was no advantage in both the price and catalytic performance of commercial HCHO catalyst. Hence, the selection of catalyst needs to consider the effects of multiple factors such as removal performance, residence time and economy comprehensively.

Conclusions

In this study, compared with the commercial HCHO catalyst, two typical catalysts (Pt/GAC catalyst and MnO_x/GAC catalyst) were studied for catalytic oxidation of HCHO at ambient temperature. Different influencing factors of catalytic oxidation on the removal efficiency such as superficial velocity, reaction bed height and the type of catalyst were investigated.

According to the results, the superficial velocity influenced the removal efficiency of HCHO, and there was an optimum value for catalytic oxidation which depends on the type of catalyst. The removal efficiency of HCHO increased firstly and then decreased with the decrease of superficial velocity. For the commercial HCHO catalyst, the removal efficiencies of HCHO from the highest to the lowest were SV = 1.0 m/s > SV = 1.5 m/s > SV = 0.5 m/s. The removal efficiency over the commercial HCHO catalyst could be enhanced by increasing the reaction bed heights.

HCHO concentration measured when superficial velocity was 0.5 and 1.0 m/s reduced to 0.2 ppm under the height of 20 cm, but when the superficial velocity was 1.5 m/s, the height has to be 30 cm to meet the emission standard. Among the three types of catalysts, Pt/GAC catalyst shows the effective and stable catalytic property, whose removal efficiencies reached over 90% and the conversion did not change too much with the time increasing.

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