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Catalytic oxidation of dichloromethane and toluene over platinum alumite catalyst

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Abstract

Catalytic oxidation technology is one of the most promising technologies for the reduction of volatile organic compound (VOC) emissions. It is very necessary to study the catalytic oxidation of mixture of VOCs and volatile organic compounds (CVOCs), because VOCs are always emitted accompanying with CVOCs. Hence, the catalytic oxidation reaction of toluene and CH_2Cl_2 is explored on a platinum alumite catalyst in this work. The results show that the addition of toluene has no effect on the decomposition of CH_2Cl_2 , although it can suppress CH_3Cl formation because the steam generated from the catalytic combustion of toluene suppresses the formation of CH_3Cl from CH_2Cl_2 . High concentrations of CH_2Cl_2 have a negative effect on the catalytic combustion of toluene.

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1. Introduction

Chlorinated volatile organic compounds (CVOCs) are classified as hazardous to the environment and public health. CVOCs are emitted to the atmosphere from a wide range of industrial processes, including the manufacture of herbicides, plastics and solvents. Furthermore, they are always emitted in mixtures with other VOCs or carbon monoxide. It is known that catalytic oxidation technology is one of the most promising technologies for the abatement of such emissions. Platinum group metals are well-known highly active catalysts for the oxidation of hydrocarbons in general [1,2]. Similarly, we have developed several kinds of platinum catalysts, which are supported on an anodic alumite film for the catalytic combustion of VOCs [3–7].

Recently, metal oxide catalysts, particularly of perovskite structures and zeolites, have been extensively tested in the oxidation of CVOCs. The catalysts for this process should be selective to HCl, carbon dioxide and water, and resistant to chlorine. It is reported that the acidity of the support plays an important role in the catalytic oxidation of chlorinated volatile organic com-

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0304-3894/\$ - see front matter © 2007 Elsevier B.V. All rights reserved. doi:10.1016/j.jhazmat.2007.10.036 pounds. Hence, the commercial catalysts are designed based on certain limitations [8–10]. In the previous work [11], we have moderated the anodic alumite support with the hydration reaction to promote its acidity. Results have shown that the moderated support experiences fine activity and endurance for the catalytic oxidation of the CVOCs. However, it is also known that CVOCs are always emitted accompanied with VOCs. Therefore, to explore the interaction between the catalytic oxidation reaction of CVOCs and the catalytic combustion reaction of VOCs is very important and very necessary. Windawi and Zhang [12] have come to the conclusion that unsaturated and aromatic VOCs strongly enhance the oxidation of unsaturated chlorocarbons, while saturated VOCs only slightly raise the conversion of CVOCs. The enhancement of trichloroethylene (TCE) destruction over alumina-supported Pt or Pd in the presence of hydrogen-rich compounds, i.e. water, hexane and toluene, has been reported by Gonzalez-Velasco et al. [13]. Reports are also available on the enhanced rate of chlorobenzene combustion over Pt-based catalysts in binary mixtures with various hydrocarbons [14,15]. The beneficial effect of acetone and methanol on the conversion of methylene chloride oxidized in two-component mixtures over a Pt-Pd-based catalyst has been detected by Sinquin et al. [16]. The enhanced conversion of CVOCs in the presence of water or hydrocarbons has also been

observed over perovskite-based catalysts [17,18], as well as Cu–Cr and Mn-based catalysts [19].

In this paper, the interaction between the catalytic oxidation of CH₂Cl₂ and the catalytic combustion of toluene is discussed. The study shows that the addition of toluene to the reactant gas of CH₂Cl₂ and air has no effect on the decomposition of CH₂Cl₂, because the catalytic combustion of toluene and the catalytic decomposition of CH₂Cl₂ occur at different activity sites. However, the addition of toluene can suppress the formation of CH₃Cl from CH₂Cl₂. This effect can be attributed to the steam generated from the catalytic combustion of toluene, because steam can suppress the formation of CH₃Cl. The addition of CH₂Cl₂ with low concentration has no effect on the catalytic combustion of toluene. The catalytic combustion of toluene however, can be suppressed when the concentration of CH₂Cl₂ is increased, because the decomposition reaction of CH₃Cl generated from CH_2Cl_2 also occurs on the platinum sites, which impedes the catalytic combustion of toluene on the platinum sites.

2. Experimental

2.1. Catalyst preparation

A catalyst support was prepared by anodization of a $4 \text{ cm} \times 8 \text{ cm}$ (0.3 mm thick) aluminum plate (JIS A1050). Each plate was first rinsed with acetone and degreased in an aqueous solution of 20 wt.% NaOH for 3 min at room temperature, then successively washed again with deionized water and immersed in a 30 wt.% HNO₃ solution for 1 min at room temperature. The plate was washed again with deionized water and dried. The treated plate was then oxidized as the anode in an oxalic solution against a cathodically polarized graphite plate counter-electrode. The solution was controlled at a constant temperature of 293 K. while the oxalic concentration was set at 4 wt.%. The distance between the two electrodes was set at 8 cm. Under a direct current density of 50 A m⁻², anodic oxidation was carried out for 16 h. After, the plate was dried at an ambient temperature for 12 h and then calcined in air at 623 K for 1 h to decompose the residual oxalic in the film.

Then, in order to moderate the acidity properties of the support, the plates were immersed in deionized water at 353 K for 1.5 h, referred hereafter as hot water treatment (HWT). After HWT, the plates were dried at ambient temperature for 12 h and then calcined in air at 773 K for 3 h. Finally, the platinum was supported on the developed support with the electro-deposition technology [3,4].

2.2. Catalyst characterization

In this study, the anodic alumite film thickness was measured using optical microscopy. The specific surface area and pore distributions could be obtained via a nitrogen adsorption method (SA3100, Beckman Coulter Inc.), where the outgas conditions were set at 573 K for 1 h. An inductively coupled plasma spectrometer (ICPS-7510, Shimadzu Corp.) was used to measure the platinum loading after the catalysts were dissolved in nitrohydrochloric acid. The platinum dispersion and average size of the particles could be measured by CO-pulse adsorption, where pre-treatment conditions were set at 373 K for 30 min in 70 mL min⁻¹ nitrogen gas, and then at 673 K for 30 min in 70 mL min⁻¹ helium gas. Sample reduction was carried out at 673 K in 70 mL min⁻¹ hydrogen gas for 1 h. Finally, the sample was cooled in 70 mL min⁻¹ helium gas. Based on the amount of CO adsorbed and assuming an adsorption stoichiometry of CO:Pt = 1:1, the metal dispersion was obtained.

2.3. Activity experiments

In the activity experiment, air from a cylinder was flowed into a saturator that was filled with liquid mixture of chloromethane and toluene (Kokusan Chemical Co. Ltd.) and then mixed with the volatilized gas. The concentrations were controlled by controlling the temperature of saturator. The catalyst $(1 \text{ cm} \times 1 \text{ cm},$ cut into pieces $2-4 \text{ mm}^2$) diluted with 4 g quartz sand (30-50 mesh, Kishida Chemical Co. Ltd.) was packed into a straighttube Pyrex reactor with an i.d. of 10 mm. The upper and lower parts of the reactor were filled with quartz fibers. The reactor was then placed inside an electric furnace. A K-type thermocouple was placed in the center of the catalytic bed to record the reaction temperature and control the furnace. The reactant flow rate was about 120 mL min⁻¹ under atmospheric pressure. Chlorinated hydrocarbons of the reactants and products were measured using a gas chromatograph with an ECD detector (GC-3900B Hitachi, Hitachi, Ltd., column: CP-Sil 8CB, 0.53 mm × 30 m). Toluene was measured using a gas chromatograph with an FID detector (GC-390B, GL Sciences Inc.).

3. Results and discussion

The properties of the catalysts used in this work are listed in Table 1. The platinum catalyst prepared with hot water treatment (HWT) is assigned as Cat 1. The support of Cat 1 without Pt is assigned as Cat 3. The platinum catalyst prepared without HWT is assigned as Cat 2. It can be found that a support with a higher BET-area can bring out the higher Pt loading and dispersion, since Cat 1 has a higher Pt loading and dispersion than Cat 2.

3.1. Effect of toluene addition on the CH_2Cl_2 catalytic oxidation

Cat 1 was used in the exploration of the catalytic oxidation reaction of CH_2Cl_2 in the presence of toluene. The concentration of toluene was set at 0 and 1000 ppm. The concentration of CH_2Cl_2 was set at 100 and 310 ppm. The conversions of CH_2Cl_2 are expressed in Fig. 1. From the previous work [11],

Table 1Properties of the catalysts used in the work

No.	HWT	Pt loading $(g m^{-2})$	Dispersion (%)	BET-area $(m^2 g^{-1})^a$
Cat 1	done	0.83	51.3	187.4
Cat 2	none	0.52	21.5	36.1
Cat 3	done	-	-	200.9

^a Calculated based on the mass of Al₂O₃.

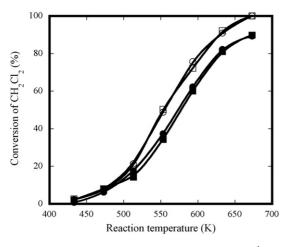


Fig. 1. Conversion of CH_2Cl_2 over Cat 1 (flow rate: 120 ml min⁻¹; concentration of CH_2Cl_2 : 100 ppm; \bigcirc : without toluene; \Box : with toluene of 1000 ppm. concentration of CH_2Cl_2 : 310 ppm; \bullet : without toluene; \blacksquare : with toluene of 1000 ppm).

it is known that CH₃Cl can be generated from CH₂Cl₂, hence the selectivity to CH₃Cl is also explored in this work. The selectivity to CHCl₃ is expressed in Fig. 2. From Fig. 1, it can be found that the conversion of CH₂Cl₂ almost has no change when toluene is added into the reactant gas. Hence, the results show that the addition of toluene has no effect on the decomposition of CH₂Cl₂. Fig. 2 shows that the selectivity to CH₃Cl decreases from 22.5% to 3.4% at 553 K, and from 17.5% to 11.2% at 593 K, when 1000 ppm toluene is added into the 100 ppm CH₂Cl₂ reactant gas. Furthermore, among the whole reaction temperature region, the selectivity to CH₃Cl is decreased by the addition of toluene. When the concentration of CH₂Cl₂ was set at 300 ppm, the similar result can be obtained.

It is known that toluene molecules are oxidized on platinum sites in the catalytic oxidation reaction when a platinum catalyst is used. The addition of toluene has no effect on the catalytic oxidation reaction of CH_2Cl_2 , which maybe explained by the fact that the catalytic oxidation reaction of CH_2Cl_2 does not

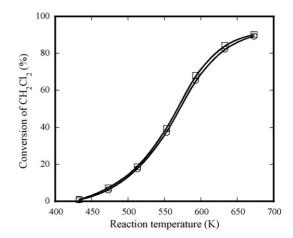


Fig. 3. Conversion of CH₂Cl₂ over Cat 1 and Cat 3 (flow rate: 120 ml min^{-1} ; concentration of CH₂Cl₂: 310 ppm; \bigcirc : on Cat 1; \Box : on Cat 3).

occur on the platinum sites. Brink et al. [20] reported that the rate of CH₂Cl₂ conversion for Pt on γ -Al₂O₃ and plain γ -Al₂O₃ is exactly the same. The only difference is the product distribution. With a platinum-containing catalyst at 723 K, CH₂Cl₂ is converted into CO₂, while on alumina alone, CO is the major carbon-containing product. Pinard et al. [10] also reported that CH₃Cl is formed on Lewis acid sites. Afterwards, CH₃Cl can be oxidized into CO₂ and HCl on platinum sites when the catalytic oxidation reaction of CH₂Cl₂ is carried out on a platinum catalyst. Of course, the products vary with the used catalyst.

In order to explore the effect of platinum in the catalytic oxidation reaction of CH_2Cl_2 , the reactions were carried out on Cat 1 and Cat 3 (which is the support of Cat 1 without Pt), respectively. The conversion of CH_2Cl_2 is expressed in Fig. 3, and the selectivity to CH_3Cl is expressed in Fig. 4. Fig. 3 shows that there is no obvious change in the conversion of CH_2Cl_2 on the platinum catalyst and the support. Fig. 4 shows that the selectivity to CH_3Cl decreases greatly when the platinum catalyst is used. The selectivity to CH_3Cl decreases from 32.1% to 22.8% at 593 K and decreases from 33.2% to 18.1% at 633 K when the platinum catalyst is used instead of the support in the reaction. Hence, it can be induced that the catalytic oxidation reaction of

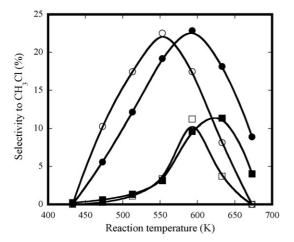


Fig. 2. Selectivity to CH₃Cl over Cat 1 (flow rate: 120 ml min^{-1} ; concentration of CH₂Cl₂: 100 ppm; \bigcirc : without toluene; \Box : with toluene of 1000 ppm concentration of CH₂Cl₂: 310 ppm; \bullet : without toluene; \blacksquare : with toluene of 1000 ppm).

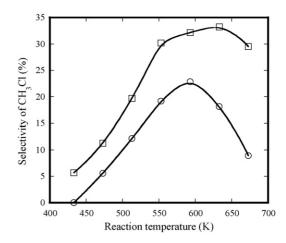


Fig. 4. Selectivity of CH₃Cl over Cat 1 and Cat 3 (flow rate: 120 ml min^{-1} ; concentration of CH₂Cl₂: 310 ppm; \bigcirc : on Cat 1; \Box : on Cat 3).

 CH_2Cl_2 occurs on the acid sites only, and platinum sites almost have no effect on the decomposition of CH_2Cl_2 . The similar result also has been reported by Brink et al. [20]. Fortunately, the catalytic combustion of toluene occurs on the platinum sites. Hence, it can be explained that the addition of toluene has no effect on the catalytic decomposition of CH_2Cl_2 with it.

From Fig. 2, it can be concluded that the addition of toluene cannot enhance the decomposition of CH₂Cl₂; however it can suppress the formation of CH₃Cl. In our previous research [11], the effect of water steam on the catalytic oxidation of CH₂Cl₂ has been studied. It shows that steam cannot enhance the decomposition of CH₂Cl₂; however it can change the production distribution of the reaction, especially, it can suppress the formation of CH₃Cl. It is thought that the steam can combine with the intermediate formed during the decomposition of CH₂Cl₂ to form HCl rather than CH₃Cl. Lopez-Fonseca et al. [21] also reported that the activities of the catalysts were promoted slightly with the addition of water. However, the presence of steam has a remarkable effect on the reaction product distributions. It is known that the catalytic combustion reaction of toluene can generate steam. One thousand parts per million toluene can generate about 3500 ppm of steam when toluene is combusted completely and also can generate several hundred ppm of steam at the low temperature. The generated steam may suppress the formation of CH₃Cl, thus it is observed that the selectivity to CH₃Cl decreases when toluene is added.

As a result, it can be informed that the catalytic combustion reaction of toluene occurs on the platinum sites when a platinum catalyst is used and the catalytic decomposition reaction of CH_2Cl_2 occurs on the acid sites of support. Two reactions occur at different activity sites; hence the addition of toluene has no effect on the conversion of CH_2Cl_2 . Of course, the interaction between the CH_2Cl_2 molecules and toluene molecules also has an effect on these reactions, but this interaction can be neglected based on the research results. However, the changes in the selectivity to CH_3Cl are very obvious, although the addition of toluene has no effect on the catalytic oxidation of CH_2Cl_2 . It can be explained that the water steam generated from the catalytic combustion of toluene suppresses the formation of CH_3Cl from CH_2Cl_2 .

3.2. Effect of CH_2Cl_2 addition on the toluene catalytic oxidation

The conversion of toluene in a CH_2Cl_2 atmosphere is expressed in Fig. 5, which shows that the conversion of toluene in CH_2Cl_2 of 100 ppm is almost the same as that without CH_2Cl_2 . However, when the concentration of CH_2Cl_2 was increased to 310 ppm, the conversion was found to decrease. The conversion at 513 K is about 88.7% when CH_2Cl_2 is absent, but this conversion decreased to 57.9% when 310 ppm CH_2Cl_2 was added into the reactant gas. The conversion also decreases from 96.0% to 88.5% at 533 K. The obtained results indicate that the addition of CH_2Cl_2 with low concentration has no effect on the catalytic combustion reaction of toluene. When the concentration of CH_2Cl_2 is increased, the CH_2Cl_2 can suppress the reaction of toluene.

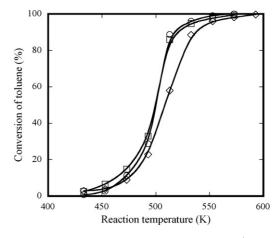


Fig. 5. Conversion of toluene over Cat 1 (flow rate: 120 ml min⁻¹; concentration of toluene: 1000 ppm; \bigcirc : without CH₂Cl; \Box : with CH₂Cl₂ of 100 ppm; \Diamond : with CH₂Cl₂ of 310 ppm).

It is known that steam will be generated from the catalytic combustion reaction of VOCs and that the steam can impede the adsorption of VOC molecules on platinum sites. Hence, hydrophobic support materials are always used in these reactions [22]. The previous work proves that the alumite support has strong hydrophilic property after the HWT treatment [11]. Hence, the TG analysis of Cat 1 and Cat 2 were carried out, respectively, and the result is expressed in Fig. 6. The results show that the mass of Cat 1 decreased with increasing temperature, while the mass of Cat 2 showed almost no change. Therefore, it can be induced that Cat 1 has stronger hydrophilic properties than Cat 2.

The catalytic combustion reactions of toluene were carried out on Cat 1 and Cat 2, respectively, the results of which are expressed in Fig. 7. The results show that Cat 2 almost has the same activity as Cat 1 at low reaction temperature, although the platinum loading and dispersion of Cat 2 are lower that those of Cat 1. Hence, it can be found that a hydrophobic support is desirable for the catalytic combustion of VOCs. It can be induced that the steam generated from the catalytic combustion reaction of toluene can also suppress the reaction at the same time when the anodic alumite catalyst is applied.

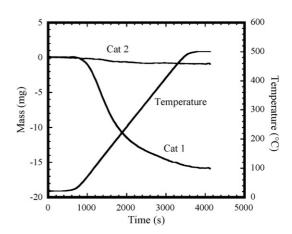


Fig. 6. TG analysis result (carrier gas: N_2 ; catalyst: 500 mg; heating rate: 10 K min⁻¹).

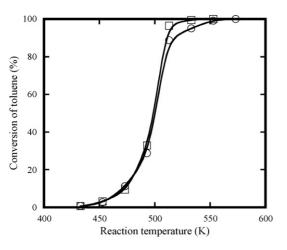


Fig. 7. Conversion of toluene over Cat 1 and Cat 2 (flow rate: 120 ml min^{-1} ; concentration of toluene: 1000 ppm; \bigcirc : on Cat 1, \Box : Cat 2).

In the above discussion, it has been proved that the steam generated from the catalytic combustion reaction of toluene can be consumed in the catalytic oxidation reaction of CH_2Cl_2 . Moreover, it has also been proved that the oxidation reaction of CH_3Cl occurs on the platinum sites. Hence, it can be induced that the consumption of steam generated from toluene will promote the catalytic combustion reaction of toluene and the oxidation reaction of CH_3Cl will suppress the catalytic combustion reaction of toluene.

In the oxidation reaction of CH_2Cl_2 , the concentration of CH_3Cl generated from CH_2Cl_2 increases with increasing CH_2Cl_2 concentration. When low concentration CH_2Cl_2 is added into the catalytic combustion reaction of toluene, the generated CH_3Cl is also very low and the suppression effect of CH_3Cl on the catalytic oxidation reaction of toluene is weaker than the promotion effect of the steam consumption on the catalytic oxidation reaction of toluene. Hence, there is almost no change in the conversion of toluene. However, when the concentration of CH_2Cl_2 is increased, the suppression effect of CH_3Cl on the catalytic oxidation reaction of toluene becomes dominant; hence the conversion of toluene decreases in the reaction.

3.3. Endurance test

It is known that organic compounds with Cl or Si always have a detrimental effect on the catalyst in the catalytic oxidation reaction of VOCs, especially when Pt or Pd catalysts are used. Hence, the endurance test was also carried out in this work to test the endurance of the developed catalyst. The concentration of toluene was set at 1000 ppm and the concentration of CH_2Cl_2 was set at 310 ppm. The reaction temperature was set at 573 K. The experimental results are expressed in Fig. 8.

In the initial reaction time, the conversion of toluene increases from 60.2% to 93.4% and keeps constant, as has been discussed in previous work [5,6]. The conversion of CH₂Cl₂ is steady and is about 45.7%. The research result indicates that the catalyst has fine endurance in the catalytic oxidation reaction of the mixture gas.

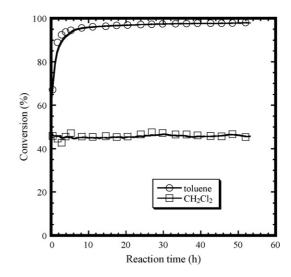


Fig. 8. Result of the endurance experiment (flow rate: 120 ml min⁻¹; toluene: 1000 ppm; CH₂Cl₂: 310 ppm; temperature: 573 K).

4. Conclusions

The catalytic oxidation reaction of CH_2Cl_2 on the platinum alumite catalyst can be expressed as

$$CH_2Cl_2 \underset{acid \ sites}{\longrightarrow} CH_3Cl \underset{Pt}{\longrightarrow} CO_2 + H_2O + HCl$$

Firstly, CH_2Cl_2 is adsorbed on the acid sites of the catalyst and then decomposed to CH_3Cl . The generated CH_3Cl can be oxidized into CO_2 on the platinum sites. Hence, the addition of toluene almost has no effect on the decomposition of CH_2Cl_2 . However, it is known that steam can suppress the formation of CH_3Cl . It is also found that the selectivity to CH_3Cl is decreased by the steam generated from the added toluene. Therefore, the addition of toluene can benefit the transformation of CH_2Cl_2 to HCl rather than CH_3Cl .

Since CH₃Cl is oxidized into CO₂ on platinum sites, the oxidation reaction will suppress the catalytic combustion reaction of toluene. However, the steam generated from the catalytic combustion reaction of toluene is consumed in the catalytic oxidation reaction of CH₂Cl₂, which can promote the catalytic combustion reaction of toluene. When the concentration of CH₂Cl₂ is low, the change in conversion of toluene cannot be observed. However, when the concentration of CH₂Cl₂ increased, the suppression effect of CH₃Cl on the catalytic oxidation reaction of toluene becomes dominant and the conversion of toluene is observed to decrease.

The endurance experiment shows that the developed platinum alumite catalyst experiences excellent endurance for the catalytic combustion of VOCs and CVOCs.

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