

A Study on the Effect of Support on the Catalytic Activity of OMS-2 for Oxidation of Toluene in Gas Phase

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Abstract

In this study, the complete oxidation of an aromatic hydrocarbon compound such as toluene into carbon dioxide and water was carried out in a continuous fixed bed reactor. OMS-2 material synthesized by the refluxing method was used as the catalyst. To reduce the cost of the catalyst, various support materials were employed for supporting the OMS-2 catalyst. The effects of supports (i.e., bentonite, kaolinite, and alumina) and their contents on the catalytic activity of OMS-2 for the oxidation of toluene were investigated. Among the supports, bentonite with Al:Si ratio of 1:2 was the best material with the lowest temperature that reached 100% of toluene conversion at 260 °C. Therefore, 20%OMS-2/bentonite could be a suitable catalyst with high efficiency but low cost for catalytic oxidation of toluene and other organic compounds in the gas phase.

Keywords: OMS-2, kaolinite, bentonite, alumina, toluene, catalytic oxidation.

1. Introduction

Environmental pollution from the exhaust of vehicles, factories, and industrial zones in recent years has become a very serious problem, especially from the vapor of aromatic hydrocarbons, such as benzene, toluene, and xylene. It is worth noting that among these aromatic hydrocarbons, toluene is a volatile organic substance that is often used as a solvent to dissolve a variety of materials such as paints, inks, rubbers, and adhesives. Therefore, it is usually emitted from factories producing these materials. Although toluene is rarely considered as carcinogenic and rarely causes effects in genotoxicity tests, it has a stronger central nervous system inhibitory effect than benzene. If exposed to humans at 200 ppm over 8 h, toluene will often produce symptoms such as fatigue within several hours, frailty, headache, and cutaneous paresthesia. It also causes psychosis at 400 ppm and utmost fatigue, confusion, elation, nausea, and dizziness at 600 ppm for a short time [1].

In terms of solutions for volatile organic compounds (VOCs) control, the catalytic oxidation of hydrocarbons has been studied by scientists over the years [2-4]. A feature that can be noticed is that this method, as Yu *et al.* [5] said, could effectively eliminate VOCs at much lower temperatures than direct combustion. For toluene, most catalytic oxidation methods, to a large extent, use catalysts such

as CeO₂-Fe₂O₃ [6], TiO₂/SBA-16 [7], MnO_x-CeO₂/TiO₂ [5], and a mixture of CuO_x, MnO_x, and CeO_x supported on γ -Al₂O₃ [8]. Basically, these catalysts are transition metal oxides that have high VOCs catalytic oxidation activities, lower costs than precious metal catalysts, high resistance to toxicity, high metal content, and also large surface area of active sites. Therefore, they have been studied in depth in recent years and are considered as effective catalysts and cost savings for the complete oxidation of VOCs [8, 9].

In a study by Sun *et al.* [10], manganese oxide octahedral molecular sieves (OMS-2) were reported to be an oxide of manganese that were widely used in chemical processes due to their microporous structure. To be more precise, result in a study on the complete oxidation of VOCs conducted by Luo *et al.* [11] found that OMS-2 had a high hydrophobic surface, which was capable of exchange of oxygen in the structure with oxygen in the air stream. OMS-2 has a pore size of about 0.46 nm, uniformity in the size of the pores [12], and oxidation state of manganese in the range of 3.68 - 3.92, which is quite high compared to that of OMS-1 (~3.55) and OL-1 (~3.52) [13]. OMS-2 has also a strong affinity for non-polar or weakly polar organic compounds, an advantage over other microporous materials, which can be used as a catalyst for the complete oxidation of VOCs [14-17]. However,

pure OMS-2 material is expensive, difficult to make pellets, and hard to be used in the industry.

There is a need to look for a cheap and available OMS-2/support catalyst that is simple in fabrication, has high activity and durability, and especially can be used in the environment with many impurities, such as compounds of sulfur, halogen, and steam. Therefore, it is reasonable to use supports such as alumina, bentonite, and kaolinite. γ - Al_2O_3 has a large specific surface area [8] and is often used as a desiccant in the treatment of natural gas, adsorbent in the petroleum cracking, and support of catalysts for the oxidation of hydrocarbons. Bentonite and kaolinite are two types of natural clay minerals which are cheap and available in many regions of the world [18]. Although it is worthy of investigation, there has been still no research on adding alumina, bentonite, and kaolinite to OMS-2 to create OMS-2/support materials with a low cost for catalytic oxidation of VOCs, especially toluene.

Alumina (γ - Al_2O_3), bentonite, and kaolinite are popular supports for catalysts. These substances have the same characteristics as those that are widely used in industry, have a large specific surface area, cheap, and are easily shaped. For γ - Al_2O_3 , results from the BET measurement show that it has a specific surface area of $287 \text{ m}^2/\text{g}$ and has a pore size of approximately 2.5 nm. Bentonite differs from γ - Al_2O_3 in that it is a natural clay mineral, belonging to the montmorillonite group. Its chemical composition is $\text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2 \cdot n\text{H}_2\text{O}$, in which the water content or n value ranges from 4 to 8. Further, in the chemical composition of bentonite, in addition to the two elements of Si and Al, other elements such as Fe, Ca, Mg, Ti, K, and Na are also found [19]. Regarding crystal structure, bentonite is a natural aluminosilicate mineral with a layered structure of 2:1, formed from two tetrahedral networks linked to an octahedral network. Kaolinite is also a natural clay mineral, usually white, but sometimes also has other colors, such as pink, orange, or red, depending on the amount of iron oxide in it. Structurally, kaolinite is also a natural mineral aluminosilicate with a layered structure in the 1:1 form, or in other words, a tetrahedron linked to an octahedron through oxygen atoms. The tetrahedra are formed from $\text{Si}_2\text{O}_5^{2-}$ tetrahedron units and the octahedron are made of octahedral units of $\text{Al}(\text{OH})_6^{-3}$.

OMS-2 is a type of manganese oxide belonging to the Hollandite family and has a porous structure with a pore diameter is lower than 2 nm. While the frame of OMS-2 is made up of octahedral units MnO_6 , the porous structure of the OMS-2 material is formed from the contribution of edges and angles to form double chains of octahedral units $2 \times 2 \text{ MnO}_6$ [20]. These double chains link together vertically, forming a porous structure with a pore size of roughly 0.46 nm. In the structure of OMS-2, manganese has an oxidation state ranging from +3 to +4, in which Mn^{+3} accounts for a very small percentage. Depending on the OMS-2

preparation method, the oxidation state of manganese in OMS-2 is different. According to DeGuzman *et al.* [13], OMS-2 synthesized by the hydrothermal method has the highest oxidation state of manganese, while OMS-2 synthesized by the sol-gel method has the lowest one. The average oxidation state of manganese in OMS-2 is mainly from 3.68 to 3.96. The results in a study of Mai [21] revealed that OMS-2 synthesized by refluxing method had high purity, large specific surface area, and high catalytic activity for oxidation of VOCs as compared to that by sol-gel method.

In this study, the catalyst of OMS-2 on supports was prepared for catalytic oxidation of toluene. The effect of supports such as alumina, bentonite, and kaolinite on the catalytic activity of OMS-2 in the complete oxidation of toluene with the temperature was investigated. The suitable support was selected based on the lowest temperature and cost.

2. Experiment

2.1. Material Synthesis

The refluxing method was used to synthesize the OMS-2 catalyst in this study [22-24]. The solid obtained after refluxing a mixture of KMnO_4 , MnSO_4 , and HNO_3 for 2 h was filtered, washed, then dried, heated, ground, and screened. The final product was OMS-2 material that was black, discrete, and had a uniform distribution of particle size. The morphology of the obtained OMS-2 material is displayed in Fig. 1. The BET surface area of this OMS-2 was determined to be $109.204 \text{ m}^2/\text{g}$ via adsorption-desorption of nitrogen.

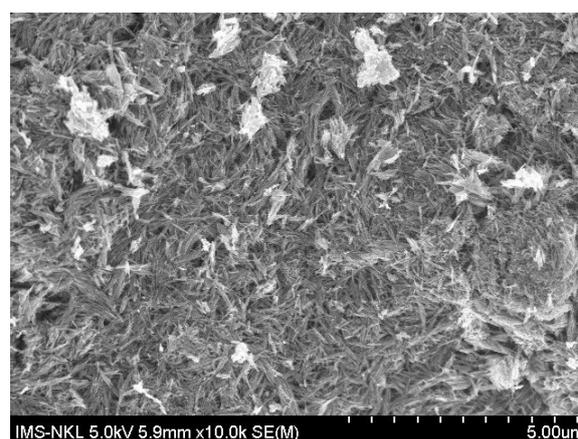


Fig. 1. SEM image of OMS-2 catalyst

The supports (alumina, bentonite, and kaolinite) are industrial grade. Alumina was supplied by the Ho Chi Minh City Institute of Applied Materials Science and directly applied without any pretreatment. Bentonite and kaolinite were pretreated by soaking in 20% sulfuric acid solution for 3 days, then rinsed many times with distilled water until reaching neutral pH,

dried, and ground. The OMS-2/supports were prepared according to the following steps. At first, an OMS-2/support mixture with a percentage by mass of supports (i.e., 5, 10, 15, or 20%) was put into a small beaker containing distilled water at the rate of 15 mL distilled water/2 g of the mixture and stirred by a magnetic stirrer for 8 h. The obtained black slurry was then dried at 120 °C for 8 h. Next, this mixture was heated at 450 °C with a gradient temperature of 1 °C/min for 3 h. Finally, the resulting mixture was OMS-2 catalyst/support whose color varied from black to brown depending on the mass ratio of OMS-2 in the mixture and was a very fine powder after being ground.

2.2. Catalytic Oxidation of Toluene

Air-containing toluene was prepared by bubbling pure nitrogen (>99%) in toluene liquid to generate a toluene vapor flow. This flow was then mixed with pure oxygen (>99%) with an N₂/O₂ ratio of 4:1 to form a synthetic air containing toluene. The physicochemical parameters of toluene catalytic oxidation used in the reactor are listed as follows: the lowest temperature in the investigated temperature range (180 °C), catalyst amount (200 mg), catalytic activation in N₂ stream with a flow rate of 8 L/h at 400 °C for 3 h, the flowrate of reactant (4 L/h of synthetic air containing toluene), and time for collecting samples (after 30 min).

In order to calculate the toluene conversion, follow the following steps: Collect raw material and product samples at the appropriate point in the reactor; analyze the samples using gas chromatography with FID detector; calculate the toluene conversion according to the formula below:

$$C = \frac{S_r - S_p}{S_r} \times 100 (\%)$$

where C is the conversion (%), S_r is the peak area of toluene in the inlet gas, and S_p is the peak area of toluene in the outlet gas.

3. Results and Discussion

Fig. 2 compares toluene conversion between different OMS-2/ γ -Al₂O₃ catalysts. It can be seen that in the high-temperature zone at around 400 °C, the Al₂O₃ support can only convert 72% of the toluene presenting in the gas stream, whereas all the OMS-2/ γ -Al₂O₃ catalysts had toluene conversion of 100% at this temperature. By comparison, the Al₂O₃ support was almost inert with the oxidation reaction. In contrast, the OMS-2/ γ -Al₂O₃ catalysts showed its catalytic activity in different degrees depending on their OMS-2 contents with a steadier leap in toluene conversion against temperature. This indicated that pure OMS-2 catalyst had a much higher toluene catalytic activity than OMS-2/ γ -Al₂O₃. More specifically, in the low-temperature range, such as 200 °C, toluene conversion

of below 60% was found with OMS-2/ γ -Al₂O₃ catalysts having different amounts of OMS-2, while 98% toluene was successfully converted by OMS-2. In short, toluene conversion increased with the increase in OMS-2 content in the OMS-2/ γ -Al₂O₃ catalyst, from 5% to 20%, and reached a peak of 100% at 300 °C.

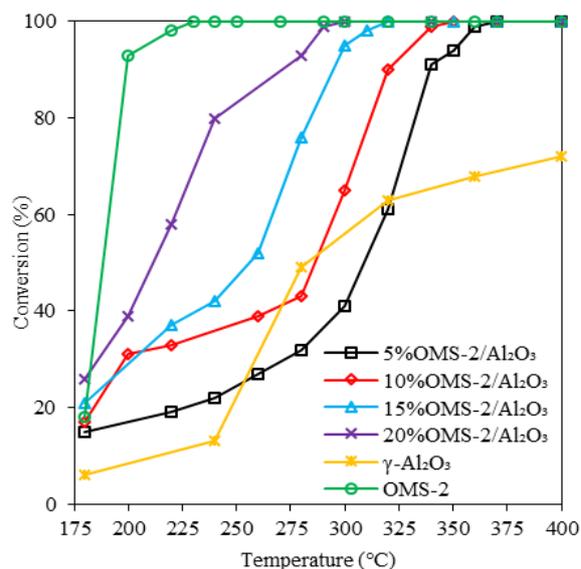


Fig. 2. Conversion of toluene with different OMS-2/ γ -Al₂O₃ catalysts

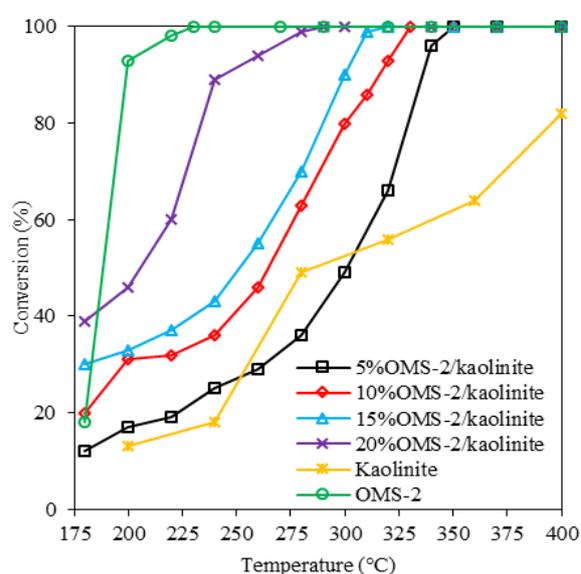


Fig. 3. Toluene conversion with different OMS-2/kaolinite catalysts

Fig. 3 illustrates the toluene conversion of OMS-2/kaolinite catalyst with different amounts of OMS-2 in the temperature range from 180 to 400 °C. Similar to Al₂O₃ support, the OMS-2 activity showed its main catalytic role in toluene oxidation, which experienced an increase in catalytic activity when the amount of

OMS-2 increased. In other words, as observed in Fig. 3, it was apparent that with the highest OMS-2 percentage of 20%, the OMS-2/kaolinite catalyst gave the greatest leap in toluene conversion of 29% in the temperature range of 220 to 240 °C, much lower than 75% which was the highest leap in toluene that OMS-2 achieved in the lower temperature range, 180 to 200 °C, to be precise. A toluene conversion of 100% was achieved at 290 °C with 20% OMS-2/kaolinite, while OMS-2 gave a total conversion of toluene at only 230 °C, 1.26 times lower than 290 °C. Thus, the catalytic activity difference between OMS-2 and OMS-2/kaolinite was shorter than that between OMS-2/Al₂O₃ and OMS-2.

Fig. 4 provides a comparison of the toluene conversion using OMS-2/bentonite catalysts having different amounts of OMS-2. In general, the toluene conversion with OMS-2/bentonite catalyst increased with the OMS-2 amount, virtually identical to OMS-2/ γ -Al₂O₃ and OMS-2/kaolinite. However, there was a similarity in the catalytic activity between the OMS-2/bentonite catalysts with different OMS-2 amounts of 5, 10, and 15%. OMS-2/bentonite with 20% differed from the remaining OMS-2/bentonite in that it gave a toluene conversion of 100% at 260 °C, which was lower than that of 290, 300, and 310 °C for OMS-2/bentonite catalysts with 15, 20, and 5% of OMS-2, respectively.

Based on the results above, it could be concluded that changes in toluene conversion with OMS-2/supports (i.e., supports of γ -Al₂O₃, kaolinite, and bentonite) catalysts were a function of temperature. The curves that represent toluene conversion against temperature were all S-shaped, which was quite similar to many complete oxidation reactions (e.g. combustion reactions) of other organic compounds in the presence of oxidation catalysts in previous reports [21, 25, 11]. In addition, catalytic activity was directly

proportional to the amounts of OMS-2 in OMS-2 supported γ -Al₂O₃, kaolinite, and bentonite. The changes in toluene conversion with 5% of OMS-2 catalyst in OMS-2/bentonite, OMS-2/kaolinite, and OMS-2/ γ -Al₂O₃ are illustrated in Fig. 5. As seen in this figure, with an amount of 5% of OMS-2, the OMS-2/bentonite yielded the highest catalytic activity and a large difference in catalytic activity between it and the other two catalysts. 5% OMS-2/bentonite reached the highest point of toluene conversion (i.e 100%) at 310 °C. Both 5% OMS-2/kaolinite and 5% OMS-2/ γ -Al₂O₃, on the other hand, yielded a toluene conversion of less than 60% at the same temperature. Therefore, the catalytic activity of the catalysts could be arranged in descending order of 5% OMS-2/bentonite, 5% OMS-2/kaolinite, and 5% OMS-2/ γ -Al₂O₃.

As can be seen from Fig. 6, the similarity in the catalytic activity between the three catalysts (i.e., OMS-2/bentonite, OMS-2/kaolinite, and OMS-2/ γ -Al₂O₃) with 10% of OMS-2 and these three catalysts with 5% of OMS-2 were experienced at the temperature ranging from 180 to 350 °C.

When the OMS-2 content was 10%, however, the toluene conversions with these three catalysts were almost equal to each other at a temperature of 200 °C. When the reaction was performed in a high-temperature range, the catalytic activity was most clearly separated from the OMS-2/bentonite. The difference in the catalytic activity of 10% OMS-2/supports was shortened as compared to 5% OMS-2/supports. For example, 10% OMS-2/bentonite catalyst produced a toluene conversion of 100% at 300 °C, while 10% OMS-2/kaolinite catalyst and 10% OMS-2/ γ -Al₂O₃ catalyst only achieved 80 and 65% of toluene conversion at the same temperature, respectively.

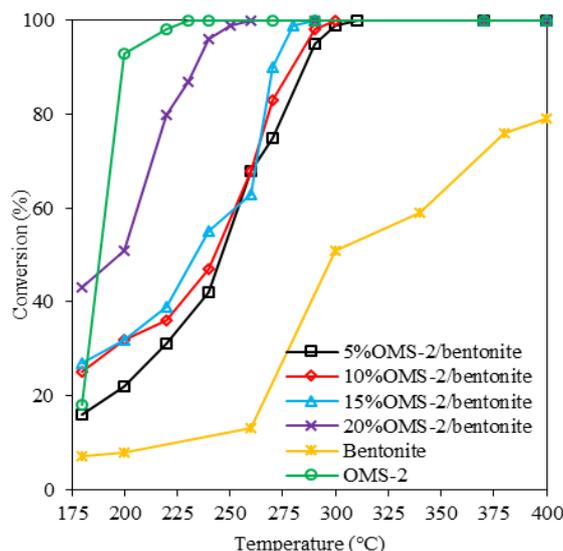


Fig. 4. Toluene conversion with different OMS-2/bentonite catalyst

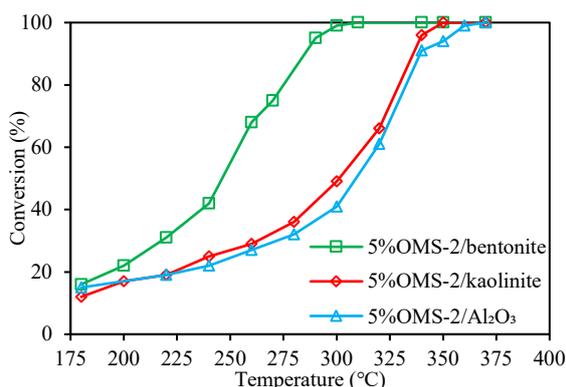


Fig. 5. Effect of support with 5% of OMS-2

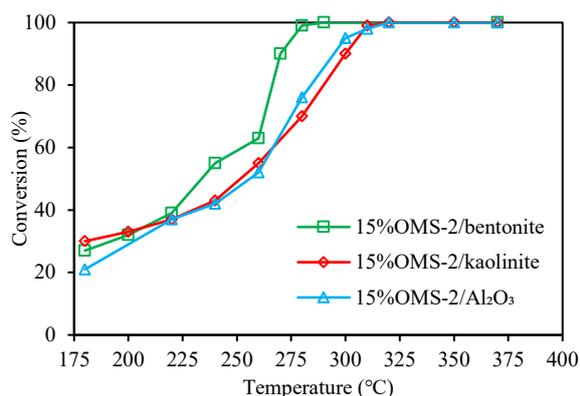


Fig. 7. Effect of support with 15% of OMS-2

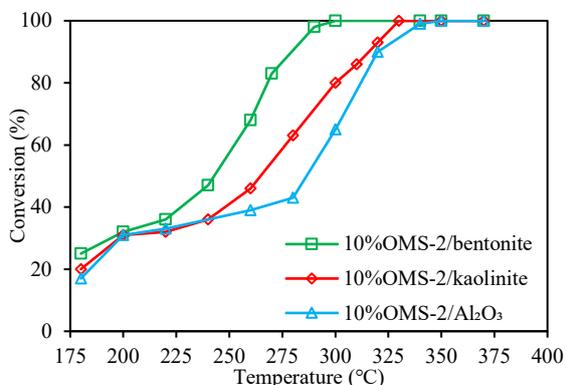


Fig. 6. Effect of support with 10% of OMS-2

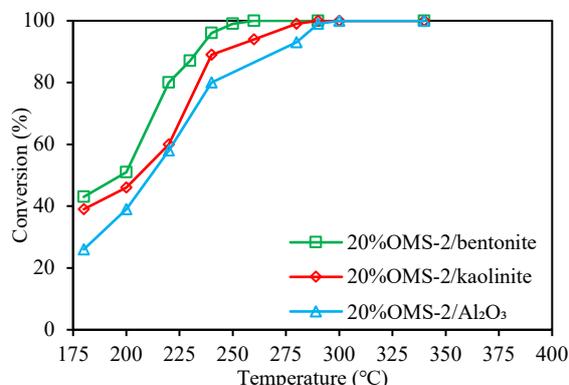


Fig. 8. Effect of support with 20% of OMS-2

When 15% (Fig. 7) and 20% (Fig. 8) of OMS-2 were investigated, the curves representing the toluene conversion against temperature were closer together with all three kinds of catalysts, but the total toluene conversion with OMS-2/bentonite peaked at a temperature lower than that at which the two other catalysts (e.g., 15%OMS-2/bentonite at 290 °C and 20%OMS-2/bentonite at 260 °C). Therefore, it was proved that the important role in the toluene catalytic oxidation of OMS-2 was indisputable.

Fig. 9 shows the dependence of the temperature that is necessary for a toluene conversion of 100% to be achieved on OMS-2 amount and type of support (i.e., kaolinite, bentonite, and γ -Al₂O₃). According to this figure, 260 °C was the lowest temperature and 370 °C was the highest temperature required for the 100% toluene conversion in the gas stream with the catalysts of 20% OMS-2/bentonite and 5% OMS-2/ γ -Al₂O₃, respectively. Thus, among the supports used, bentonite could be considered as the most suitable material for OMS-2 because of the lowest decline in the catalytic activity of OMS-2 in OMS-2/bentonite

The toluene conversion using catalysts with a high amount of OMS-2 is illustrated in Fig. 10. Accordingly, the toluene conversion with the OMS-2 amount of 50% was greater than that of 20%. With 50% of OMS-2 in OMS-2/bentonite, the toluene conversion of 100% was obtained at 240 °C, only 10 °C difference from 230 °C at which the pure OMS-2 catalyst yielded the total toluene conversion. The use of up to 50% of bentonite in the catalyst forming stage, cylindrical granulation, or pelletizing could be done without significantly changing the temperature of the oxidation process.

The study was also conducted to reveal the effect of the Al/Si ratio in the support on its effectiveness as a catalyst for toluene oxidation. As mentioned above, the decline in the catalytic activity of OMS-2/supports could be arranged according to the presence of the supports in the following order: γ -Al₂O₃ > kaolinite > bentonite corresponding to Al/Si ratio of 1/0, 1/1, and 1/2 (Table 1). Therefore, it could be seen that the catalytic activity of OMS-2/supports decreased with the increase in the Al content of the supports. The main conclusion to be drawn from this finding was that

a decrease in the Al/Si ratio of the support directly contributed to a rise in the catalytic activity of OMS-2.

Table 1. Chemical composition and Al/Si ratio of γ -Al₂O₃, kaolinite, and bentonite

Support	Chemical composition	Al/Si
γ -Al ₂ O ₃	Al ₂ O ₃	1/0
Kaolinite	Al ₄ (Si ₄ O ₁₀)(OH) ₈	1/1
Bentonite	Al ₂ O ₃ .4SiO ₂ .nH ₂ O	1/2

In brief, in the presence of bentonite support, the OMS-2 catalyst gives the most stable toluene oxidation efficiency compared with other studied supports. To validate the complete oxidation, the product gas was analyzed to determine its composition using a GC-MS system for 20%OMS-2/bentonite catalyst. As presented in Fig. 11, the oxidation at temperatures below 220 °C produced some products such as benzoic acid, benzaldehyde, CO₂, and a small amount of other organics, in which, CO₂ always accounts for the highest percentage. However, with the reaction temperature of 230 °C and above, the gaseous

product contains almost only CO₂. Thus, it can be said that the OMS-2 catalyst can completely oxidize toluene at a temperature of 230 °C and above.

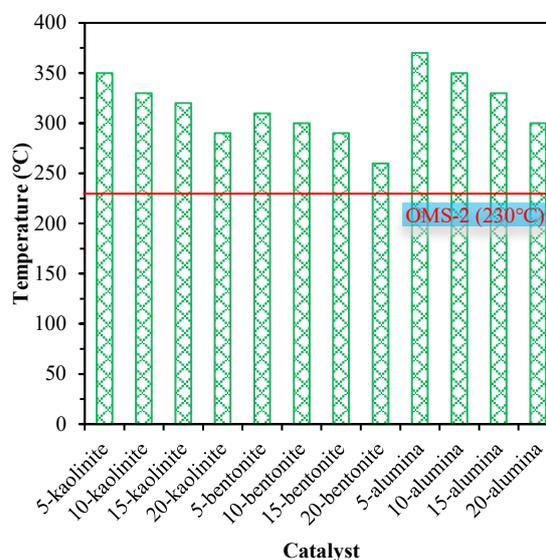


Fig. 9. The lowest temperature that reaches 100% toluene conversion (T_{100}) of different catalysts

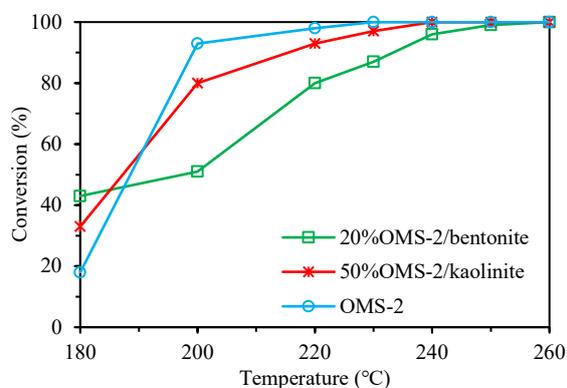


Fig. 10. Conversion of toluene with large amounts of OMS-2 in OMS-2/bentonite catalyst

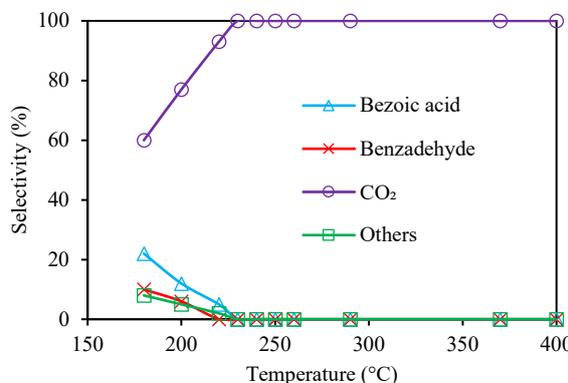


Fig. 11. Major components of the gas products from the oxidation of toluene using 20%OMS-2/bentonite at different reaction temperatures

4. Conclusion

An investigation on the catalytic activity of the OMS-2/support was initially conducted to elucidate the influence of supports such as γ -Al₂O₃, bentonite, and kaolinite on the catalytic activity for toluene oxidation. Results showed that bentonite was the most appropriate support for OMS-2, in which the greater the amount of OMS-2, the higher the catalytic activity the system had. It was possible to use up to 95% of bentonite, while the minimum temperature required to reach the total toluene conversion was not too

high at 310 °C. In terms of technical and economical perspectives, the use of 20% OMS-2 with support of bentonite gave 100% toluene conversion at 260 °C, which could be a suitable catalyst for practical applications.

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