Catalytic oxidation of benzene with manganese oxide supported on Cordierite

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ABSTRACT

Volatile organic compounds (VOCs) are one of the principal causes of air pollution, posing a grave danger to the environment and human health due to their toxicity. In the presence of heat or light, catalytic oxidation has been recognized as a viable and efficient approach for VOCs remediation. Manganese-based oxides are one of the most environmentally benign and cost-effective choices for the catalytic destruction of volatile organic compounds in thermocatalysis. That is the reason why this article focused on catalytic oxidation to control benzene (a VOCs component). The wet impregnation process was used to produce manganese oxide supported on cordierites. Scanning electron microscopy (SEM), energy dispersive spectrometer mapping (EDS mapping), X-ray diffraction (XRD), and Hydrogen Temperature-programmed reduction (TPR-H2) were used to characterize the catalysts. When using the TCD-FID detector, catalytic activity measurements were done on a micro-flow reactor system coupled online to GC. The results showed that MnO_2-Cor potential catalyst for completely oxidizing benzene with a 100% benzene conversion temperature of 350 ^oC to CO_2 and H_2O. This catalyst provides high thermal stability and good reusability due to being carried on cordierite.

Keywords: VOC; Benzene; Oxidation; Manganese oxide; Cordierite.

1. INTRODUCTION

VOCs are a group of carbon-based chemicals with a boiling temperature between 50 and 260 degrees Celsius at standard atmospheric pressure. According to the chemical structure of organic matter, VOCs can be categorized as alkanes, alkenes, alkynes, ketones, halocarbons, aromatics, etc [1-4]. In recent years, particularly in developing nations, the yearly emission of volatile organic compounds has grown considerably due to increasing industrialization and economic expansion. VOCs are a significant class of air pollutants that have severe effects not only on human health but also on the environment. VOCs are generally created by various industrial processes or the combustion of spent organic solvents [1]. In addition to damaging the ozone layer and causing photochemical pollution, aromatic VOCs can also cause physical weakness, nausea, memory loss, vertigo, and even death [6-8].

Consequently, finding efficient and adaptable procedures within the framework of the proper application of pertinent rules is essential for lowering global VOCs emissions. In recent years, several nations have explored and produced VOCs removal methods that are practical, inexpensive, and effective. Several techniques to reduce VOC emissions are categorized as either recovery or destruction [9, 10]. Recovery procedures include absorption, adsorption, membrane separation, and condensation. Thermal incineration, catalytic oxidation, low-temperature plasma, and photocatalytic oxidation are the most prominent means of destruction technologies. Different technologies are appropriate for usage in a variety of operational circumstances. Due to its high removal effectiveness, low energy consumption, and lack of secondary pollutants, catalytic oxidation is often considered an efficient method for removing volatile organic compounds [11-13].

VOCs oxidation catalysts are currently divided into two primary categories: metal-based catalysts and non-noble metal oxide catalysts. The former frequently demonstrate higher catalytic performance but are expensive and easily inactive [14, 15]. Although the activity of non-noble metal oxide catalysts, for example, oxides of Cu, Mn, Co, Fe, etc., may not be as good as that of noble metal catalysts, their low cost and long-term thermal stability have garnered increasing interest, and their future dominance is likely. Manganese-based (Mn-based) oxides are one of the most competitive and promising materials for the catalytic degradation of volatile organic compounds (VOCs) in thermocatalytic systems [2]. In the last decade, a number of Mn-based oxides have been synthesized utilizing various techniques to improve their catalytic performance and examine their catalytic behaviour in the oxidation of volatile organic molecules. The removal of volatile organic compounds by Mn-based oxide catalysts has been studied. However, the work's scope is limited to either the thermocatalytic method or a specific class of volatile organic compounds (VOCs).

Cordierite honeycomb ceramic is widely used as a monolithic supporter or carrier in environmental protection due to its numerous benefits, including excellent thermal shock resistance, low air resistance, high transformation efficiency, and low cost [17, 18]. In addition, Cordierite has been utilized as the substrate to load the active components, such as noble metals or transition metal oxides, in order to prevent their loss during the removal process of volatile organic molecules. In JiananZhu et al.’s research, studies of space velocity and catalytic stability over nanorod Ag/MnO_2-cordierite molded catalyst confirmed the good catalytic performance, excellent mechanical strength, and satisfied anti-toxicity to Cl at higher space velocity, indicating that this molded catalyst has the potential for industrial application [19]. Meanwhile, Phuong Thi Mai Pham et al.’s study suggested that This approach produces a catalyst with a layer of MnO_2–NiO–Co_3O_4 mixed oxides on top of a cordierite substrate that has an excellent activity for the treatment of CO, NO, and C_3H_6 in exhaust gases (CO conversion of 100% at 250 °C, C_3H_6 conversion of 100% at 400 °C, and NO conversion of 40% at 400 °C) [20]. Therefore, in this paper, we synthesize MnO_2 and MnO_2 catalysts on cordierite to improve the catalyst's stability for the complete oxidation of benzene.

2. EXPERIMENT

2.1. Material

Manganese nitrate solution of 50% and Standard benzene solution were purchased from Xylong Ltd. And the Cordierite from kaolin was purchased from China. Meanwhile, Standard gas cylinders H_2, Ar, He, N_2, and O_2 are also used of domestic origin.

2.2. Preparation of catalysts

A manganese oxide catalyst based on cordierite was synthesized via impregnation. 10 ml of distilled water was added to 10 ml of a 50% $Mn(NO_3)_2$ solution, and the mixture was agitated and heated at 50 °C for two hours. In addition, five grams of cordierite were added to the solution to absorb for one hour. The impregnated cordierite was dried for one hour at 110 °C (in 161l UN160mplus Memmert oven) before being calcinated for three hours at 300 °C at a rate of 3 °C/min (using 100 liters 1100 °C LMF-B70 LABTRON furnace).

The impregnation process was repeated three times to improve the concentration and dispersibility of the MnO_2 catalyst when coated on cordierite. The solid was finally calcined at a rate of 2 ^oC/min to 500 ^oC for three hours. The sample MnO_2 supported on cordierite was marked as MnO_2-Cor. The obtained catalyst consisted of 17.5 percent of the activated phase.

After the impregnation process, the remaining solution was dried at 120 ^oC for 1 h and calcined at 500 ^oC for 3 hours at a heating rate of 2 min/°C. MnO_2 powder was designated as the powder catalyst.

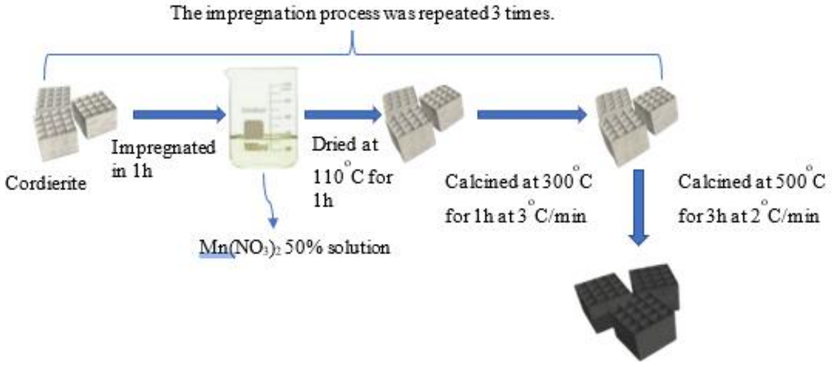

Figure 1. *Flow chart of catalyst preparation using cordierite by impregnation method.*

2.3. Characterization of catalyst

In this study, the morphology and microstructure of catalysts were examined using a scanning electron microscope (SEM) Hitachi S-4800 (Japan). Using a JEOL JCM-7000 (Japan) scanning electron microscope equipped with an X-ray microprobe analyzer (EDS) spectrometer, the chemical compositions and metal concentrations of catalysts were analyzed. The cordierite-supported catalyst was also characterized by X-ray diffraction (XRD) using a D8 Advance Bruker instrument. On a chemisorption analyzer, H2-TPR readings were taken (Micromeritics, Autochem II 2920). Prior to the TPR test, 50 mg of catalyst was treated with He gas for one hour at 300 °C and then cooled to 50 ^oC. Heating the sample from 50 °C to 500 °C at a rate of 10 °C/min in H_2/Ar (5% H_2, 50 mL/min) with cold trap propanol in N2 liquid generated the TPR profiles.

2.4. Measurement of catalytic activity

A micro-reactor reactor system was utilized for the treatment of benzene. Figure 1 depicts the Schematic diagram. The formation of gaseous benzene was accomplished by bubbling N_2 into liquid benzene. It was then combined with N_2 and O_2. The total reactant mixture flow rate was set at 50 mL/min. When the benzene concentration at the intake reached a constant state of 6,000 ppm and the GHSV (Gas hourly space velocity) reached 11,000 h^{-1}. The sample (0.1 grams) of each catalyst was put into the center of the 60-centimeter-long, ¼-inch reactor. CO_2 and benzene were analyzed for their concentrations using an online Focus-Thermo Scientific gas chromatograph equipped with a thermal conductivity detector (TCD) and a flame ionization detector (FID).

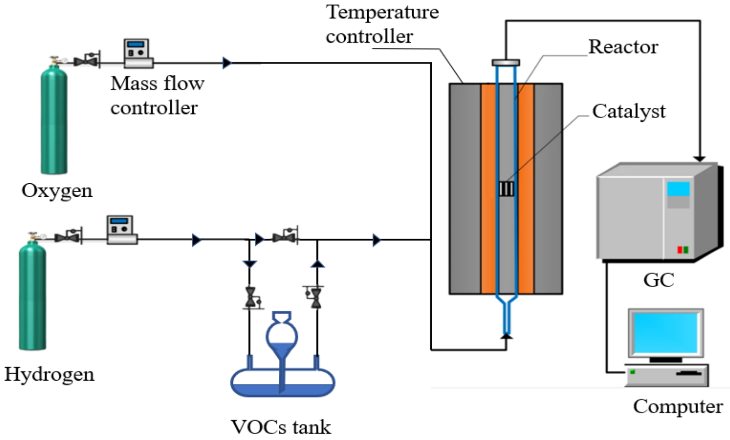

Figure 2. *The schematic diagram of the benzene oxidation experiment.*

Analysis and calculation of the result

- Benzene conversion was evaluated as equation 1

$$\eta_B = \frac{C_T^1 - C_T^2}{C_T^1} \text{x } 100 \qquad (1)$$

Whereas:

η_B: Benzene conversion (%);

C_T^1: Benzene concentration of inlet flow at a temperature T (ppm);

C_T^2: Benzene concentration of inlet flow at a temperature T (ppm).

- The conversion of Benzene into CO_2 was determined as equation 2

$$\gamma_{CO2} = \frac{C^2_{CO2.T}}{6(C_T^1 - C_T^2)} \text{x } 100 \qquad (2)$$

Whereas:

$C^2_{CO2.T}$: CO_2 concentration of outlet flow at a temperature.

3. RESULTS AND DISCUSSION

3.1. Catalyst characterization

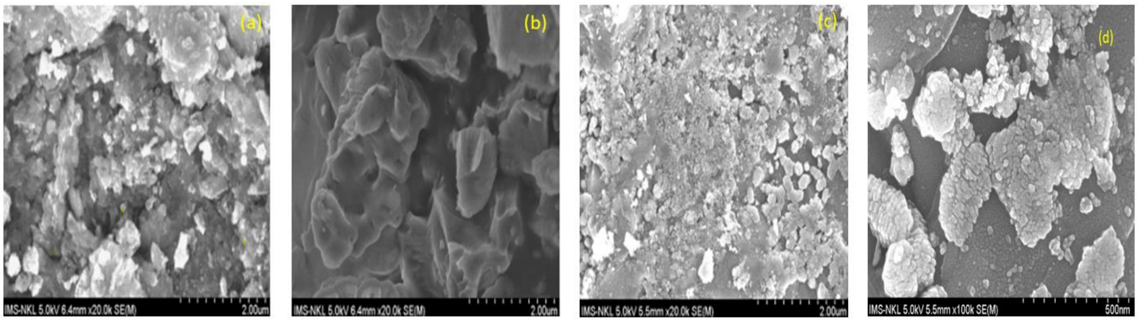

Figure 3. *SEM images of (a) MnO_2 power; (b) Cordierite; and (c,d)MnO_2-Cor.*

As illustrated by Figure 3b, the surface of the pure Cordierite substrate was smooth.

Figure 3c is a SEM image of the MnO_2-Cor catalyst, which reveals that other components wholly covered the Cordierite substrate. In addition, as shown in the high-magnification SEM picture of the catalyst in Figure 3d, the MnO_2 particle on the cordierite surface exhibited an uneven shape. It comprised a significant number of nanoparticles ranging in size from 0.5 µm to 5 µm.

In addition to the Si, Al, and Mg ascribed to the cordierite substrate, the elemental analysis of the MnO_2-Cor catalyst using EDS revealed the presence of Mn and O, as shown in Figures 4a and 4b. The elemental analysis revealed that Mn, Mg, Al, Si, and O are the most abundant components on the surface of the MnO_2-Cor catalyst. It also announced that the MnO_2 component of the MnO_2-Cor catalyst had efficiently developed on the surface of the cordierite. Due to the fact that cordierite is synthesized from Ca-containing dolomite precursors, traces of Ca were detected. Nonetheless, during MnO_2-Cor production, this Ca component was dissolved and removed.

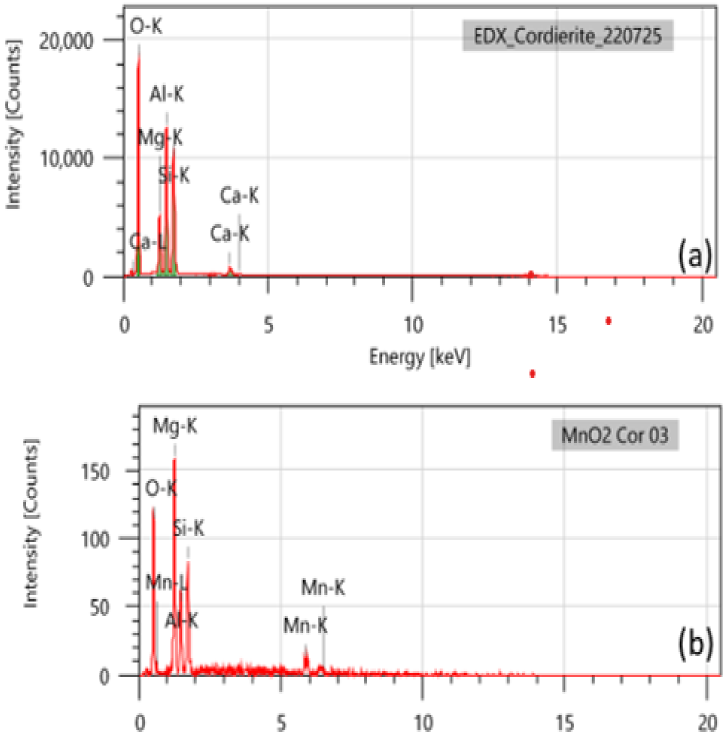

Figure 4. *EDS spectrum of a surface area of*
(a) the cordierite substrate
(b) MnO_2-Cor catalyst.

The catalysts' EDS results are given in figure 4. In the EDS results, the peaks of catalytically active components were observed. The strength of these peaks was found to vary amongst catalysts. EDS has a known scan depth of hundreds of nanometers. A catalytic region is chosen as the scanning area, and the proportion of atoms in this region is calculated. Therefore, EDS scans do not precisely depict the surface. However, the presence of all covered parts inside the picture might provide information about the entire surface. According to the EDS study, catalytically active components are distributed throughout the catalyst surface.

Table 1. *EDS analysis of Cordierite and MnO$_2$-Cor.*

Cordierite		
Element	**Mass %**	**Atom %**
O	53.92±0.43	66.68±0.53
Mg	6.78±0.12	5.52±0.10
Al	18.77±0.20	13.76±0.15
Si	18.58±0.21	13.09±0.15
Ca	1.95±0.08	0.96±0.04
Total	100.00	100.00
MnO$_2$-Cor		
O	35.73±3.63	49.80±5.05
Mg	24.92±2.45	22.86±2.24
Al	12.48±1.95	10.31±1.61
Si	15.81±2.18	12.55±1.73
Mn	11.06±3.25	4.49±1.32
Total	100.00	100.00

To determine the distributions of the active catalytic elements, EDS mapping was performed. EDS mapping revealed that both catalysts had the components O, Mg, Al, and Si in their Cordierite material structure. Similarly, Mn was detected solely in the MnO$_2$-Cor catalyst. This study indicated that cordierite components were covered with chemicals with catalytic properties. On the surface of every catalyst, the carrier substance Cordierite was equally dispersed. A surface with an even distribution of Cordierite provides additional binding sites for catalytically active components. Despite the low catalyst concentration on cordierite ceramics (about 17.5 wt%), the dispersion of Mn on the surface of the catalyst was observed to be almost equal to that of the other characteristics.

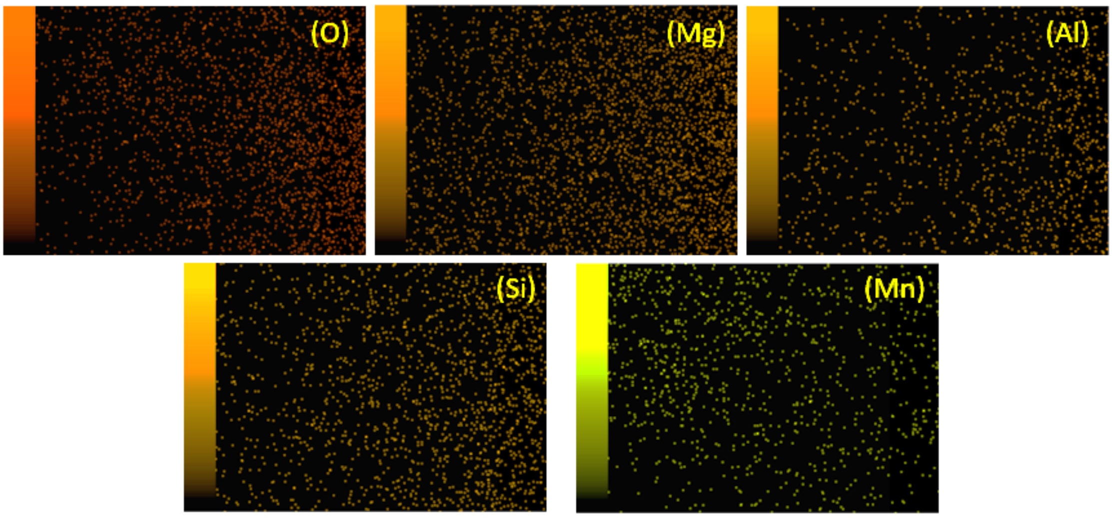

Figure 5. *Elemental mapping results of MnO$_2$-Cor catalyst.*

The XRD characteristic measuring method was utilized to assess the phase composition of MnO_2 when carried on Cordierite. The results are shown in figure 6. The prominent peaks of the XRD pattern (Fig.6) are assigned cordierite. And at peak $2\theta = 37.34^o$ and 55.56^o, MnO_2 could also be found. The existence of MnO_2 did not change the framework of cordierite. In addition, at $2\theta = 10.43^o$ and 30.09^o.

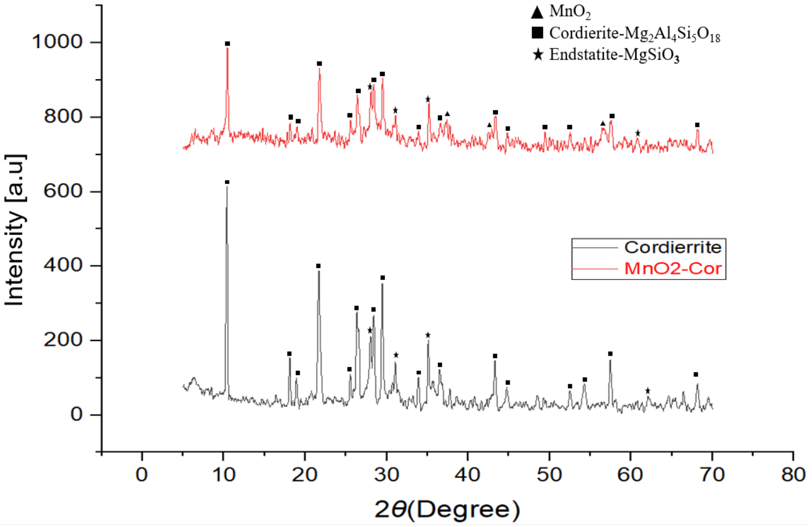

Figure 6. *XRD pattern of Cordierite and MnO_2-Cor.*

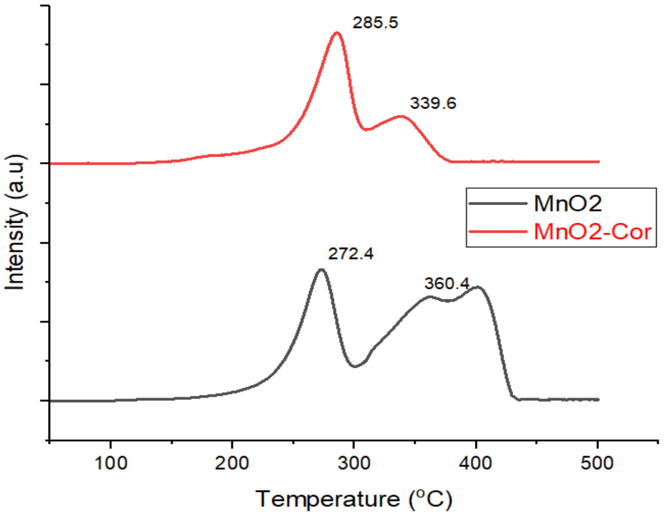

Figure 7. *H_2-TPR profile of different morphology MnO_2 catalysts.*

H_2-TPR is an excellent method to evaluate the redox properties of solid materials, which can be correlated with their catalytic performances for oxidation reactions. The H_2-TPR results of the power MnO_2 and MnO_2-Cor are shown in figure 7. H_2-TPR techniques were used to study the redox properties of catalysts further. In both samples (MnO_2 and MnO_2-Cor), two reductive board peaks are observed, which are located at around 280 and 350 °C, respectively. It has been reported that the two board peaks located between 100 to 400 °C should be attributed to the stepwise reduction of Mn^{4+} to Mn^{3+} and Mn^{3+} to Mn^{2+}, respectively [20, 21]. Apparently, the MnO_2 has a better redox ability. The MnO_2 emerged with the lowest reduction temperature at 272.4 °C. The lowest reduction temperature meant the most amounts of surface oxygen and active sites, which contributed to the excellent reduction ability of the catalyst. These observations implied that higher redox property was conducive to higher catalytic activity. The higher reducibility of MnO_2 indicated its higher mobility ability of oxygen species and then enhanced the catalytic efficiency for the catalytic combustion of benzene [3].

3.2. Catalytic activity

To examine the catalytic activity of the as-prepared catalysts, the toxic gas benzene was used as a model for catalytic oxidation. The experiments involved the complete oxidation of 6000 ppm of benzene vapor at a GHSV of 10000 h^{-1}. The results are illustrated in figure 8. As we can see in figure 8, the benzene conversion increased with the temperature increased. Evidently, the conversion increased slowly under lower temperatures while increasing sharply under higher temperatures; the intrinsic reaction of toluene and the transformation of the intermediates might influence this. From Fig. 8a, it is apparent that MnO_2 shows higher activity than that of MnO_2-Cor at low temperatures (below 350 °C). It is fully compatible with the data obtained from the TPR-H_2 measurement, which indicates that MnO_2 has a better redox capacity than MnO_2-Cor. Both of the samples, which contain Manganese oxide, achieved a toluene conversion of 100% at a temperature higher than 350 ℃. Meanwhile, Cordierite has a virtually negligible activity for benzene oxidation at low temperatures and only converts 8.97% of benzene at 400 °C.

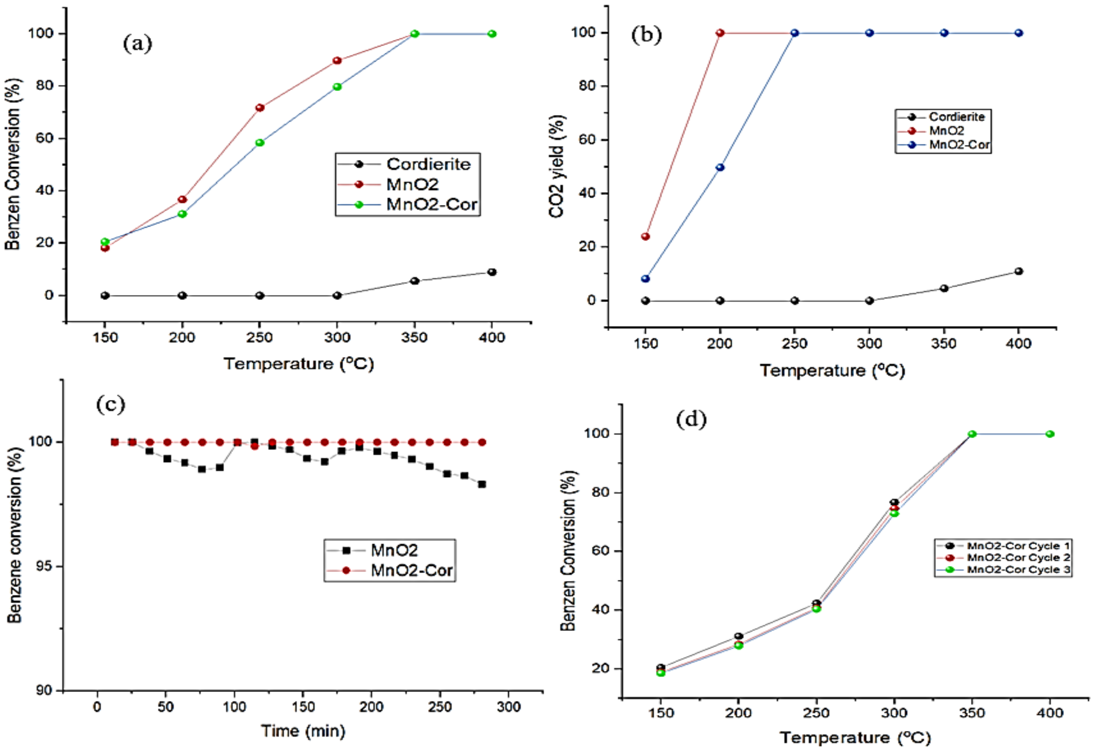

Figure 8. *Profile of (a) Benzene conversion over different samples;*
(b) CO_2 yield over all samples;
(c) Catalysts stability during 300 minutes continuous test over MnO_2 and MnO_2-Cor;
(d) Cycling performance of MnO_2-Cor.

Figure 8b indicates whether oxidized benzene is totally transformed to CO_2 and H_2O or just converted to intermediate chemicals. At a low temperature of less than 250 °C, both catalysts have not yet fully oxidized benzene but have transformed it into intermediate molecules. In this CO_2 yield graph, the MnO_2 sample still has a better ability to convert benzene ultimately.

The two samples, MnO_2 and MnO_2-Cor, both had higher benzene conversion rates as the temperature rose. As this is not the ideal temperature range for the complete oxidation of benzene, the conversion efficiency progressively rises at temperatures between 150 and 200 ^oC. Since benzene was oxidized and transformed into intermediate products in this situation, the percentage of benzene converted to CO_2 steadily rose but remained low. Mainly when the temperature was 200 ^oC, the conversion of both samples was only around 30-35%, but when the temperature went up to 350 ^oC, the conversion rate of both samples was only about 30-35%. At these temperatures, the conversion of benzene grew more rapidly. The benzene gas used as an input has undergone complete conversion. By using the manganese oxide catalyst system and the manganese oxide carried on cordierite, it can be found that 350 ^oC is the ideal temperature for the complete oxidation of benzene.

A number of studies were conducted to examine the catalyst's stability and capacity to be reused following reaction cycles. The experimental procedures used to assess the catalyst's stability are identical to those used to examine its catalytic activity, with the exception that the temperature under investigation is the chosen optimum temperature at 350 ^oC. 300 minutes were spent doing the experiment, and the stability test results are exhibited in figure 8c. The benzene conversion of MnO_2-Cor was almost 100% after 300 minutes and did not show a significant decline, while the MnO_2 catalyst gives the ability to convert Benzene less stable. Therefore, the materials delivered via Cordierite will be more heat-resistant and durable.

Meanwhile, after each cycle of the catalytic reaction was desorbed to reconstitute with a stream of N_2 at 10 ml/min for three h and then continued with the following reaction cycles, the experiments to test the reusability of the catalyst was carried out in the same manner as the test of catalytic activity. After three cycles of the benzene oxidation process catalyzed by MnO_2 carried on cordierite, there appears to be no trace of decreased activity, as seen in figure 8d. Hence, it is clear that the catalysts were able to boost their mechanical and thermal stability compared to their granular form by using cordierite ceramic materials. Since the catalyst is still in the form of granules, continuing with ceramic also makes the process more practical for large-scale manufacturing because it prevents pipeline obstructions and avoids blowing out while handling the gas stream with high velocity.

4. CONCLUSIONS

The impregnation technique efficiently produced the MnO_2 catalyst system comprised of Cordierite and MnO_2. TPR-H_2 measurements revealed that the powdered MnO_2 catalyst has a greater redox capacity than the MnO_2-Cor catalyst. This has been shown in practice through measurements of catalytic activity. Although the MnO_2-Cor catalyst has the power to totally oxidize benzene at lower temperatures, within a wide temperature range of 350 ^oC, powdered oxide has the same capacity to convert benzene entirely. In addition, the MnO_2-Cor catalyst has improved thermal stability over time and may be reused after three cycles without exhibiting any evidence of activity degradation. Thus, it can be seen that the Manganese oxide catalyst supported on a cordierite ceramic carrier has many characteristics that make it suitable for use as a catalyst to treat VOCs in industry, such as high-temperature resistance, high strength and the ability to oxidise benzene at temperatures above 350 °C completely.

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TÓM TẮT

Xúc tác oxi hóa benzen bằng mangan oxit được hỗ trợ trên Cordierite

Quá trình oxy hóa xúc tác được biết đến như một công nghệ hiệu quả và điển hình với nhiều ưu điểm để xử lý các hợp chất hữu cơ dễ bay hơi (VOCs). Các oxit mangan là một trong những vật liệu tiềm năng và phù hợp nhất cho quá trình oxy hóa xử lý VOC. Do đó, nghiên cứu này tập trung vào quá trình oxy hóa xúc tác để kiểm soát benzen (một chất VOC tiêu biểu có trong không khí). Hệ xúc tác Mangan oxit hỗ trợ trên cordierit được điều chế bằng phương pháp ngâm tẩm đã được tổng hợp. Chất xúc tác được kiểm tra đặc trưng bởi kính hiển vi điện tử quét (SEM), phương pháp tán sắc năng lượng tia X (ánh xạ EDS), nhiễu xạ tia X (XRD) và khử theo chương trình nhiệt độ hydro (TPR-H_2*). Các phép thử hoạt tính xúc tác được tiến hành trên hệ thống vi dòng được kết nối với GC trực tuyến với đầu dò TCD-FID. Kết quả cho thấy xúc tác MnO2-Cor là một hệ xúc tác đầy tiềm năng cho quá trình oxi hóa hoàn toàn VOC (benzen) với nhiệt độ chuyển hóa 100% benzen ở 350* ^oC*. Chất xúc tác này mang lại độ ổn định nhiệt cao sau thời gian dài và khả năng tái sử dụng tốt sau nhiều lần sử dụng.*

Từ khoá: VOC; Benzen; Oxi hóa; Mangan oxit; Cordierite.