

LOW-TEMPERATURE CATALYTIC OXIDATION OF WHITE SPIRIT VAPOR IN AIR BY BINARY OXIDES OF CuO-MnO_x SUPPORTED ON OMS-2 MATERIAL

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Abstract

White spirit is mainly used as a solvent which contains volatile organic compounds (VOCs) that contribute to air pollution and pose health risks. In this study, the binary oxides of manganese and copper were prepared through a facile impregnation of the corresponding salts on a octahedral molecular sieve (OMS-2) and $\gamma\text{-Al}_2\text{O}_3$ was prepared via hydrothermal and calcination methods. The characteristics of materials were determined by BET, EDX, FTIR, XRD, and SEM analyses. Among the catalysts, $\text{CuO-MnO}_x/\text{OMS-2}$ containing Cu/Mn mole ratio at 6:4, and the weight ratio of CuO and MnO with OMS-2 of 15 wt% provided the best performance for VOC removal, with an efficiency of 95%. Additionally, a durability test of the $\text{CuO-MnO}_x/\text{OMS-2}$ catalyst showed that it can be a potential catalyst for low-temperature oxidation of VOCs in air.

Keywords: VOCs; white spirit; catalytic oxidation; OMS-2; binary oxides

Introduction

Volatile organic compounds (VOCs) are mixtures of carbon-based volatile gases that exist in the air. VOCs can be found in products used in homebuilding and industries. Most of them are toxic or have an unpleasant odour and can cause photochemical reactions to pollutants already in the air (e.g., O_3 , NO_x , and SO_x), forming unwanted compounds (Zhang *et al.*, 2016). Since VOCs can diffuse rapidly in the air, the occurrence of air

pollution in a small area will quickly cause the remaining air to become polluted (Zhang *et al.*, 2016). Moreover, depending on the amount inhaled, VOCs can cause eye, nose, and throat irritation, breathing difficulties and nausea, and even damage to the central nervous system and other body parts in humans (Zhang *et al.*, 2016). In this context, white spirit evaporates into VOCs and has the same adverse effects as VOCs in general. White spirit is

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a colourless solvent commonly used in paint, is insoluble in water, and has a distinctive odour. It is a mixture of hydrocarbons from C7 to C12 and is highly flammable (Anonymous, 1996). There are six main methods of treating VOCs in the industrial exhaust stream, including biofiltration, adsorption, adsorption, condensation, thermal incineration or catalytic oxidation, and flare. The biofiltration method can also eliminate VOC but is usually only effective for low concentrations and large installation areas. Meanwhile, absorption and adsorption are methods of mass transfer application to collect pollutants from the air stream. The pollutants are transferred from the gas phase to the liquid or solid phase depending on their characteristics and their intended use after collection. If the gas contains acidic or water-soluble components, an absorption method should be used to treat those pollutants. Condensation is the process of changing a contaminated molecule from a gas phase to a liquid phase. Here, the phase change can be achieved by increasing the pressure when the temperature is fixed or changing the temperature while the pressure of the gas stream is fixed. However, this method requires a suitable coolant, and the pollutant concentration must be high enough for the collection of the contaminated molecule to reach its highest value. For pollutant gases that are low to moderate in concentration and do not need to be collected, thermal incineration, catalytic oxidation and flares are suitable. The basic principle of these methods is to convert the pollutants in the gas stream into CO₂ and water. Thermal incineration and flares require a large amount of heat for treatment. The latter can be used to control a wide variety of flammable VOCs streams and often to treat pollutants in petroleum refineries. Similarly, catalytic oxidation also uses temperature to treat emissions, but at a lower temperature than the others due to the catalyst-induced oxidation. Therefore, a low level of fuel is required for the heating and system operation. In addition, the treatment equipment require low temperatures, so the raw materials for producing the equipment do not need to be subjected to high temperatures and the production cost is accordingly cheaper than that of conventional heat treatment equipment (Schnelle and Brown, 2002).

A catalyst is a material that speeds up a reaction process by reducing the activation energy of the involved components. In the formation of an intermediate compound with reactants, the catalysts will reactivate when the reaction is complete. A catalytic reaction is a chemical reaction where the reaction speed depends on the catalytic mass and activity (Hagen, 2006). Based on the characteristics and combination of metals, heterogeneous catalysts

can be classified into two basic types: noble metal and transition metal catalysts (Zhang *et al.*, 2016). Although the transition metal oxides require higher temperatures for removing VOCs compared to those of the noble metal catalysts, their ability to treat VOCs containing S or Cl is better than those of the latter (Zhang *et al.*, 2016). In fact, gases containing Cl⁻ and S²⁻ often coexist with VOCs in polluted air (Zhang *et al.*, 2016). Therefore, transition metal oxide catalysts have a great potential to treat VOC contaminated gasses with high concentrations and low-cost requirements, for example, in industrial areas.

Many types of transition metal oxides have been studied for VOCs oxidation such as CuO_x, MnO_x, FeO_x, and NiO_x (Larsson *et al.*, 1996; Larsson and Andersson, 2000; Hettige *et al.*, 2001; Kim, 2002; Wang *et al.*, 2006). These studies have found that in addition to the catalyst characteristics, the structure and surface area of the support also affect the oxidation capacity of the catalyst. Among the supports, γ -Al₂O₃ with a nanostructure that can be controlled through preparation and has a high surface area is a good support material for impregnating with other metal oxides. Wang (2004) showed that when a CuO/ γ -Al₂O₃ catalyst was impregnated with 5 wt% (weight percentage) CuO to remove toluene and n-hexane separately, the removal efficiency using the CuO/ γ -Al₂O₃ catalyst reached nearly 100% at 360°C. In another study, Sager *et al.* (2009) used CuO on γ -Al₂O₃ and impregnated with MnO and CeO₂, then applied this material for toluene removal. The results showed that the catalysts containing γ -Al₂O₃ impregnated with MnO and CuO provided a treatment efficiency of nearly 100% at temperatures below 360°C. In addition, the study found that the more wt% of MnO impregnated, the higher the treatment efficiency. Further research by Sager *et al.* (2011) showed that when a mixture of MnO and CuO was impregnated into γ -Al₂O₃, with a 10 wt% of CuO and 60 wt% of MnO, the treatment efficiency was nearly 100% for the toluene-propane mixture at 300°C. Salari *et al.* (2010) also showed that when impregnating MnO into γ -Al₂O₃, the treatment efficiency for 2-propanol was over 90%. In 2012, Binh *et al.* (2012) reported that a mixture of Ni, Zn, and Al impregnated into γ -Al₂O₃ resulted in a xylene treatment efficiency of 88% at 400°C. Similarly to Sager *et al.* (2011), Loc *et al.* (2014) noted that impregnating 10 wt% CuO into γ -Al₂O₃ led to a removal efficiency of p-xylene of nearly 60% and about 73% with further addition of 20% CeO₂. Among transition metal oxides, manganese oxide has been found to have the highest catalytic activity (Huang *et al.*, 2015). OMS-2, with a structure comprising a peculiar sharing of 2 x 2 (MnO₆)

octahedral chains that form one-dimensional tunnel structures and has a relatively large surface area (Hou *et al.*, 2013a), has been found to be a promising manganese oxide catalyst in recent years. Wang and Li (2010) further reported the high effectiveness of OMS-2 in treating some VOCs such as ethanol, benzyl alcohol, ethyl acetate, benzene, 2-thiophene methanol, and formaldehyde. Chen *et al.* (2001) used OMS-2 catalysts impregnated with a series of metal ions (Cu^{2+} , Zn^{2+} , Ni^{2+} , Co^{2+} , Al^{3+} , and Mg^{2+}) to remove 2-propanol, and achieved the highest removal efficiency by OMS-2 catalyst impregnated with Cu^{2+} . Other studies have shown good removal ability of OMS-2 catalyst when prepared by hydrothermal method for benzene at reaction temperature below 240°C (Hou *et al.*, 2013a; Hou *et al.*, 2013b). Moreover, OMS-2 prepared via the hydrothermal method was found to have a nanorod structure and a relatively large surface area as a carrier. Similarly, Wu *et al.* (2017) also found that the o-xylene treatment efficiency of the Cu^{2+} -impregnated OMS-2 catalyst was higher than that of the Ag^{+} -impregnated one.

Overall, $\gamma\text{-Al}_2\text{O}_3$ with a large specific surface area can be a good support. Similarly, in addition to being a catalyst, OMS-2 can also be a good support with a rather large surface and porous structure. With the raw material to prepare $\gamma\text{-Al}_2\text{O}_3$ and OMS-2 being easily available in Vietnam, the two have the potential to expand the research on catalytic oxidation methods at low temperatures, as studies on the treatment of VOCs by transitioning metal oxide catalysts at low temperatures are limited. Therefore, this study used the $\gamma\text{-Al}_2\text{O}_3$ and OMS-2 preparation methods to determine suitable, low-cost catalysts for VOC treatment efficiency that can be used in developing countries. It also investigated the effects on the removal efficiency of the catalyst.

Materials and Methods

Experimental Setup and Methods

Figure 1 provides the schematic diagram of the experimental model, which consists of three parts: (1) gas supply, (2) oxidation reaction, and (3) sampling and analysis systems. The air pump in the gas supply system pulled fresh air from the surrounding environment. This air was then regulated by the mass flow controllers (MFC), which controlled both the total air flow rate and the air pressure within the system. Next, the airflow was divided into two distinct streams: one passed through the system's flowmeter and the other through the gas bubbler. The flowmeter regulated the clean air flow rate to ensure it was sufficient to blend with the concentrated VOC air stream, which

was derived from the stream passing through the bubbler containing the solvent (i.e., liquid white spirit). Here, the objective was to generate a synthetic exhaust air stream with a predetermined VOC concentration for the experiment. Next, the air stream generated by the gas supply system was passed through the reactor. The reactor contained a U-shaped tube that housed both a catalyst and a supporting material. The former, weighing 1g, had a particle size ranging from 20 to 40 mesh. The supporting material was a layer of glass wool that was inert and could withstand high temperatures. Additionally, this reactor was thermally regulated and maintained by a temperature control apparatus, capable of adjusting the temperature within a range of 0°C to 250°C , as required. Method of sampling and determination of concentration according to Olkhovskaya (1971). The absorbance was analysed by a spectrophotometer (Hach DR 5000). All the experiments were repeated in triplicate, and the results were derived from the average values.

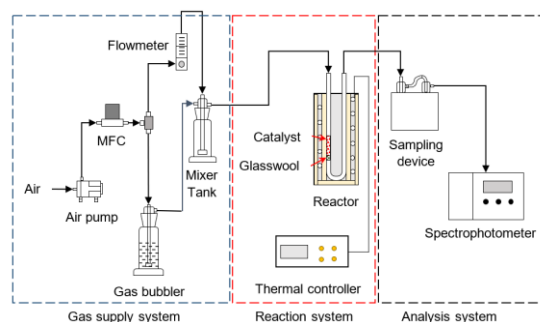


Figure 1. Schematic Diagram of the Experimental Setup

Catalyst Preparation and Characterization

In this study, the two types of catalyst support material, OMS-2 and $\gamma\text{-Al}_2\text{O}_3$, were prepared. $\gamma\text{-Al}_2\text{O}_3$ was prepared by calcining $\text{Al}(\text{OH})_3$ at 500°C for 6 h, and OMS-2 was prepared via the hydrothermal method. First, 11.33 g of $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ and 7.57 g of KMnO_4 were dissolved in 158 mL of distilled water. Next, 4 mL of HNO_3 was added to the mixture, which was stirred for 30 min and then sonicated for the next 30 min. The mixture was then hydrothermally treated at 160°C for 24 h, filtered, and washed with distilled water until the pH in the mixture was equal to that of distilled water. The resulting mixture was dried at 120°C for 12 h and finally calcined at 400°C for 6 h in air. Two metal oxides (CuO and MnO) were then impregnated on the support materials to form the main catalysts. Total mass of the impregnated Cu and Mn was

equal to 15 wt% of the catalyst support mass, and the Cu:Mn molar ratio was 6:4. After this step, there were six materials created (γ -Al₂O₃, CuO/ γ -Al₂O₃, CuO-MnO_x/ γ -Al₂O₃, OMS-2, CuO/OMS-2, and CuO-MnO_x/OMS-2).

The Brunauer-Emmett-Teller (BET) specific surface of material was measured by CBET-210A using N₂ adsorption. The crystalline nature of the material was analysed using an X-ray diffractometer (XRD, D2 Phaser, Bruker) equipped with CuK α , and the 2 θ was from 5 to 80°. The morphology of the catalyst was obtained from a high-resolution scanning electron microscope (SEM, JSM-IT200, JEOL). The species on the catalyst surface after the adsorption experiment were analysed using an FTIR spectrometer (Alpha, Bruker).

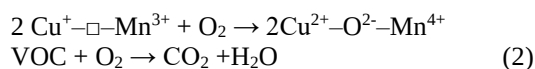
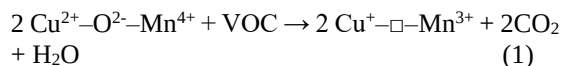
Results and Discussion

Catalytic activity and characterization

The prepared catalysts, γ -Al₂O₃, CuO/ γ -Al₂O₃, CuO-MnO_x/ γ -Al₂O₃, OMS-2, CuO/OMS-2, and CuO-MnO_x/OMS-2, were applied to the thermal catalytic oxidation of white spirit. In this experiment, the air flow rate was set to 1 L/min, and the white spirit concentration was set to 2000 ppm. Figure 2 shows the effectiveness of the six materials in removing the white spirit. From 50°C to 150°C, the white spirit removal efficiency of CuO-MnO_x/OMS-2 catalysts had the highest value. Their removal efficiency reached a steady state at a reaction temperature of 150°C or higher, and their highest efficiency was 95.29% at 250°C. In general, the catalysts based on OMS-2 had higher removal efficiency than those based on γ -Al₂O₃, due to the Mn⁴⁺ and Mn³⁺ atoms in OMS-2. Moreover, CuO-impregnated catalysts had a higher removal efficiency than catalyst supports (e.g., OMS-2 and γ -Al₂O₃). The catalysts impregnated with both MnO and CuO (with lower content of CuO) such as CuO-MnO_x/ γ -Al₂O₃ and CuO-MnO_x/OMS-2 had even higher removal efficiency than those with only CuO (with higher content of CuO). This is because CuO/Cu²⁺ with a sufficient amount ensures effective VOC removal. Conversely, too much CuO can inhibit the ability of catalytic VOCs removal (Wu *et al.*, 2017).

Meanwhile, the reduced oxide on the surface may be reoxidized by free oxygen in the feed gas in the reaction pathway, which incorporates lattice oxygen in the support oxide with VOCs. Therefore, the reaction greatly depends on the mobility and reducibility of the active lattice oxygen. Cu species provide chemisorption sites for the oxidation of VOCs while oxygen vacancies serve as activation

centres for oxygen. A reaction pathway of VOC oxidation on CuO/OMS-2 can combine the reducible lattice oxygen in the Cu²⁺–□–Mn⁴⁺ sites with the activities of CuO/OMS-2 catalysts for VOC oxidation at low temperatures, as follows:



where □ represents an oxygen vacancy.

The highly reducible bridge oxygen present in the Cu²⁺–O²⁻–Mn⁴⁺ sites may combine with the VOC to form CO₂ and partially reduced Cu⁺–□–Mn³⁺ sites. Subsequently, the Cu⁺–□–Mn³⁺ sites may undergo rapid reoxidation by the O₂ present in the feed gas.

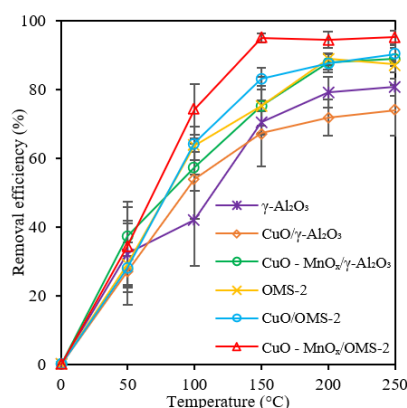


Figure 2. Catalytic activities of various catalysts for white spirit oxidation in air

Figure 3(a) contains the SEM image of the CuO-MnO_x/OMS-2 catalyst. The material has a rod-like form, measuring approximately 12nm in diameter and 300 nm to 500 nm in length. Typically, this form exhibits a significantly elevated level of catalytic activity in the material. Meanwhile, the EDS pattern in Figure 3(b) shows that the three main elements present in CuO-MnO_x/OMS-2 are O, Mn, and Cu.

Figure 3(c) provides the XRD pattern of the CuO-MnO_x/OMS-2 material and its characteristic peaks. These peaks at 2 θ of 12.6°, 17.9°, 28.8°, 37.5°, 41.9°, 49.9°, and 60.1°, corresponding to the crystal planes of (269), (327), (384), (432), (258), (215), and (207), respectively, show that CuO-MnO_x/OMS-2 had a cryptomylane structure and crystal phase characteristics of KMn₈O₁₆ (Liu *et al.*,

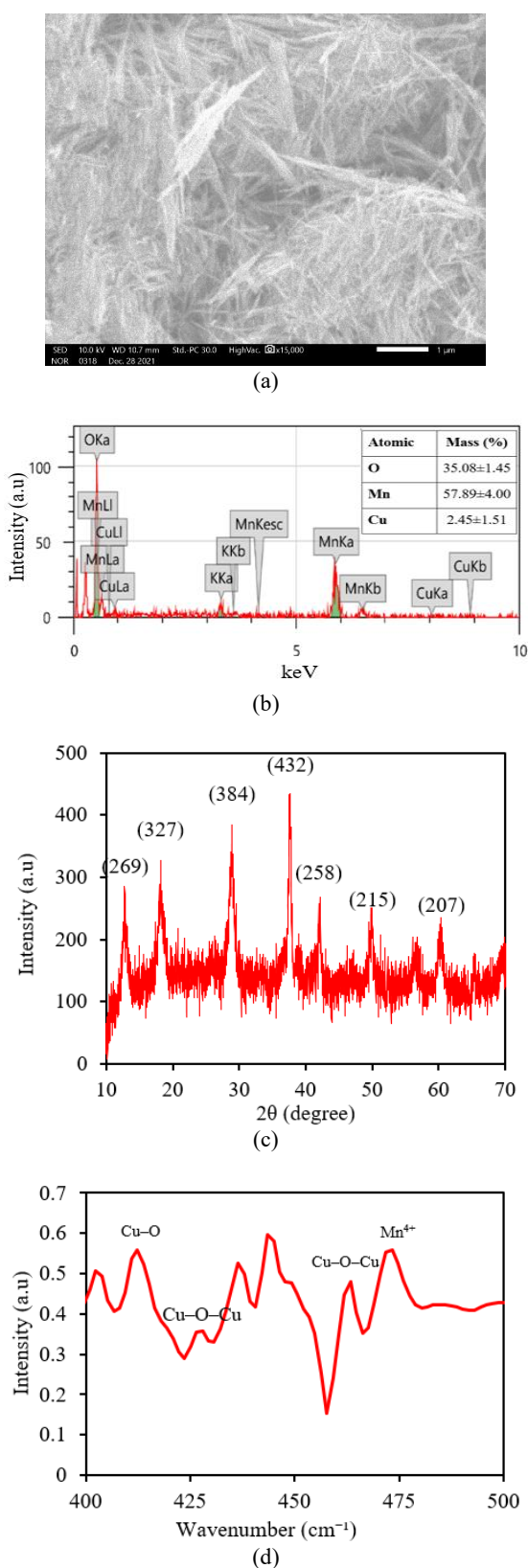


Figure 3. (a) SEM image, (b) EDX result, (c) XRD pattern, and (d) FTIR spectrum of CuO-MnO_x/OMS-2

2010). Figure 3(d) shows the FTIR spectrum of the CuO-MnO_x/OMS-2 material. The spectrum shows strong vibrations in the range of 500 to 400 cm⁻¹, corresponding to the cryptomylane structure of CuO-MnO_x/OMS-2 as mentioned in Garces *et al.* (2003). The wavenumbers in the range 470 to 475 cm⁻¹ suggest that Mn⁴⁺ appeared in the catalyst (Abulizi *et al.*, 2014). The vibration at 415 cm⁻¹ indicates the Cu-O bond while the vibrations at 432 and 461 cm⁻¹ are assigned to the Cu-O-Cu bonds (Chang, 1992; Rahman *et al.*, 2009). Additionally, the specific surface area of the CuO-MnO_x/OMS-2 catalyst, measured by BET, was 52.76 m²/g.

Effects of the operating condition

This experiment was conducted by increasing the gas flow rate from 0.5 to 2 L/min. Figure 4(a) shows that when the flow rate increased from 0.5 to 1 L/min, the removal efficiency increased slightly from 94.21% to 95.22%. When the flow rate was further increased from 1 to 2 L/min, the removal efficiency of the catalyst reduced from 95.22% to 59.9%. The retention time decreased as the air flow rate increased, with the maximum retention time being 0.36 s and the minimum time being 0.09 s. In contrast, the space velocity (GHSV) increased as the flow increased from 0.5 to 2 L/min. This is because when the air flow rate increased, the time may be not sufficient for the catalyst to adsorb and oxidize all the white spirit molecules in the air stream. In particular, the removal efficiency reached the highest value of 95.22% with the air flow rate was 1 L/min (GHSV of around 20,000 h⁻¹, retention time of 0.18 s). At a GHSV of 19894 h⁻¹, the result was similar to those of Gandhe *et al.* (2007) who investigated the ability of OMS-2 to remove ethyl acetate at a concentration of 1410 ppm.

Figure 4(b) illustrates the effects of the input concentration on the removal efficiency with 1 L/min air flow rate. When the concentration increased from 500 ppm to 2500 ppm, the removal efficiency also increased. However, with a further increase from 2500 ppm to 3000 ppm, the removal efficiency reduced. At low concentrations, in the beginning of the reaction process, the white spirit molecular density was not enough to trigger the continuous reaction process, thus making the removal efficiency low. At 2500 ppm, the removal efficiency reached the highest value because the white spirit molecular density was enough to make the reaction process most convenient. Thus, with 1g of the catalyst, the removal efficiency reached the highest value with an input concentration of 2500ppm. However, this concentration value (2500 ppm) to reach the removal efficiency of over 90% for CuO-MnO_x/OMS-2 is not as high as those

of some noble metal catalysts such as Pt/Al₂O₃ (Huang *et al.*, 2015) which required 4200 ppm of BTX to reach an efficiency of 90% at 230°C. However, it is quite high compared to other OMS-based catalysts such as OMS-70 for 2000 mg/m³ of benzene to reach an efficiency of 90% at 258°C (Hou *et al.*, 2013b), OMS-2 for 1410 ppmv of ethyl acetate and efficiency of 100% at 400°C (Gandhe *et al.*, 2007), and OMS-2(S) for 100 ppm of acetaldehyde and efficiency of 100% (Wang and Li, 2010).

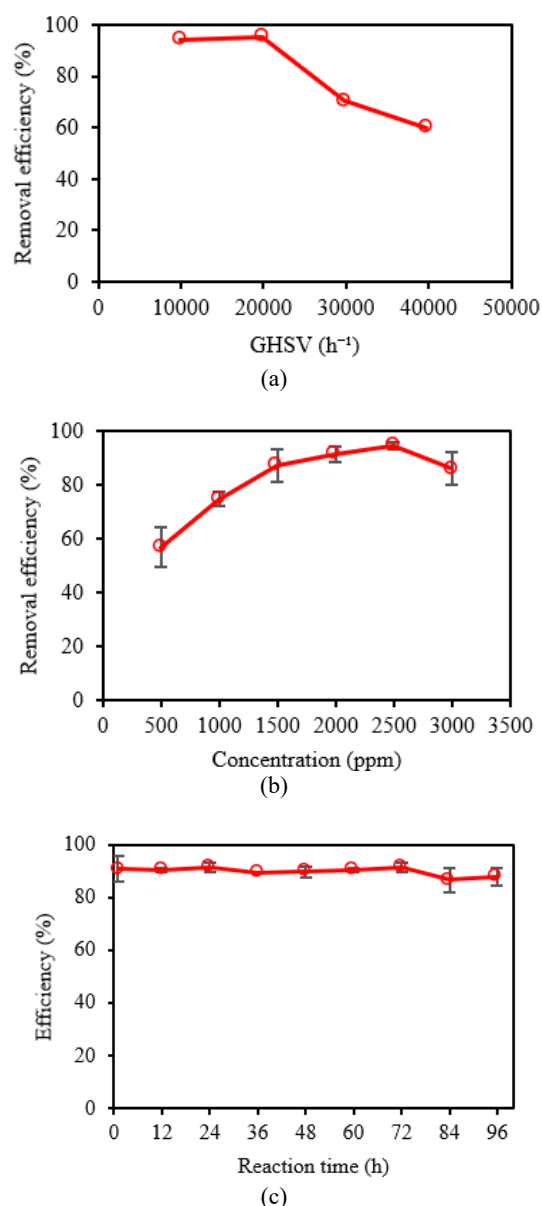


Figure 4. Effect of (a) GHSV and (b) input white spirit concentration on catalytic removal efficiency, and (c) long-time test of the catalytic reaction

Durability is one of the important parameters for the application of catalysts in environmental treatment. Therefore, this experiment was conducted by allowing the reaction process to continue for 4 days, with an inlet gas concentration of 2500 ppm and reaction temperature of 150°C. As shown in Figure 4(c), the removal efficiency of the catalyst was relatively stable in the range of 86.52–94.49%. This result aligns with other studies that investigated the removal of benzene by OMS-2 for 100 h (Hou *et al.*, 2013a; Hou *et al.*, 2013b). Overall, this study showed that CuO-MnO_x/OMS-2 prepared via the hydrothermal and impregnation method has good durability and the potential for practical application in removing VOCs in industry.

Conclusions

This study, CuO and MnO were impregnated on the support materials of OMS-2 and γ -Al₂O₃ which were then used as catalysts for white spirit vapor removal. Among the catalysts, CuO-MnO_x/OMS-2 had the highest removal efficiency due to the effective interaction between Cu and Mn in the Mars-van Krevelen mechanism. With 1 g of the CuO-MnO_x/OMS-2 catalyst, the removal efficiency reached the highest value of 94.49% at a white spirit concentration of 2500 ppm and the air flow rate of 1 L/min. The study also determined the material characteristics of CuO-MnO_x/OMS-2. It found that CuO-MnO_x/OMS-2 has good durability to remove white spirit, which is promising for practical application in the oxidation of VOCs in air.

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