

New Nanocatalyst for VOCs Removal from Recycled Plastics

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Graphene-based supports for catalysis are currently attracting great attention due to their unique and advantageous properties. However, their application in gas-phase thermal catalysis including catalytic oxidation of volatile organic compounds (VOCs) remains a theoretical research stage. Here, a new use of a graphene-based material as a catalyst support for 2-ethyl-1-hexanol (2-EH) oxidation was developed. The design of CuO/Ag catalyst incorporated into the porous structure of reduced graphene oxide aerogel (rGOA) exposes more active surface for catalysis. Therefore, this hybrid design enables great performance enhancements in 2-EH oxidation, displaying a ~ 99% removal at a temperature of 350 °C. Highly stable performance and excellent recycling ability are observed over CuO/Ag-rGOA dual function adsorbent-catalyst material. The high activity and good stability of CuO/Ag-rGOA make it an excellent candidate for the development of 2-EH oxidation catalysis.

1. Introduction

Volatile organic compounds (VOCs) are defined as “any compound of carbon, excluding carbon monoxide, carbon dioxide, carbonic acid, metallic carbides or carbonates, and ammonium carbonate, which participates in atmospheric photochemical reactions” by U.S. Environmental Protection Agency (Environmental Protection Agency, 1990). Therefore, they are a large group of carbon-based chemicals that easily evaporate at room temperature (Ojala et al., 2011). VOCs severely threaten human health and ecological environment because most of them are toxic, mutagenic, and carcinogenic (Peng and Wang, 2007). Rapid urbanization and industrialization contributed to the growing emissions of VOCs into the environment. Emissions of VOCs can be from a wide range of outdoor (chemical industries, paper production, food processing, petroleum refineries, etc.) and indoor sources (household production, pressed woods, heat-exchanger systems, office supplies, printers, etc.) (Liotta et al., 2010). Type and nature of VOCs depend on the source of emission. In particular, nowadays, emission during processing of strongly odorous VOCs is one of the major problems related to the usage of recycled plastics. The most common VOCs are halogenated compounds, aldehydes, alcohols, ketones, aromatic compounds, and ethers (Ojala et al., 2011; Olsen and Nielsen, 2001).

As a consequence of the persistent increase of VOCs and their harmful impact on human health and ecological environment, a stringent emission regulation has been proposed according to the Gothenburg protocol (Gothenburg Protocol to reduce transboundary air pollution, 1999), which stipulates that the reduction of VOCs emission by 2020 should be half of the amount released in 2000 (Huang et al., 2015). Consequently, the reduction of VOC emissions is becoming imperative, thus the development of effective VOCs elimination techniques is of great importance and crucial. The VOCs control methods can be generally divided into: recovery methods (adsorption, absorption, membrane separation and condensation, etc.) (Kujawa et al., 2015) and destruction methods (plasma catalysis, biological degradation, photocatalytic oxidation, thermal oxidation, catalytic oxidation, etc.) (Moretti, 2002). Among these methods, heterogeneous catalytic oxidation is regarded as the most favourable technology to control VOCs emission with low energy consumption and with selective conversion into harmless molecules (Quiroz Torres et al., 2013). During the catalytic oxidation process, pollutants, which are usually contained in a gaseous stream, are oxidized in the presence of suitable catalysts at much lower temperature (250 ÷ 500 °C) that thermal oxidation process (~ 1200 °C), resulting in a fuel saving of 40 - 60% and in a lower operation cost (Carabineiro et al., 2015; Chen et al., 2014). The catalytic oxidation aims at the complete destruction of VOCs rather than transferring it to another phase as in

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other techniques, such as in condensation and adsorption. The desired reaction is the total oxidation to H₂O and CO₂ without formation of by-products. As catalytic combustion takes place at lower operating temperatures, its start-up fuel requirement is lower. Therefore, moderate volumes of contaminated air can be treated at much lower fuel costs (Peng and Wang, 2007). Although catalytic oxidation can be applied effectively to treat waste gas streams with varying concentrations and flow rates of VOCs, it is most suitable for moderate flow rates and a low concentration of VOCs (Ojala et al., 2011). Other advantages of catalytic destruction are that this technique is relatively more environmentally friendly due to its low temperature operation and to its potential for excellent selectivity towards the formation of dioxins and non-hazardous reaction products (Jamalzadeh et al., 2013). One of the main challenges of catalytic destruction of VOCs is the selection of the proper catalysts from a large number of available catalysts. Due to their wide variety and nature of the range of mixtures of VOCs, it is most often difficult to identify the best possible catalysts. Noble metals are preferred as catalysts because of their high specific activity, strong resistance to deactivation and ability to be regenerated. Moreover, their industrial application is limited by their high cost, susceptibility to poisoning by chlorine/chloride compounds. In addition, if used alone (e.g. in the absence of oxides,..) they are not selective enough. However, catalysts based on transition metal (Ni, Co, Fe, Mn, Cu, etc...), transition metal oxides and/or mixture of them exhibit several advantages, which make them an effective alternative to the noble metals catalysts. In particular, they are not expensive and have a long life, tolerance, regeneration capacity and availability of a range of metal oxides of different sizes and shapes. The catalytic activity of catalysts based on transition metal is generally lower than noble metals, but the combination of the former ones would enable to achieve a catalytic efficiency comparable to the latter ones due to the synergistic effect of two or more transition metals, transition metal oxides and/or mix of them. Dual function adsorbent-catalyst with both support adsorption and catalyst function was recently suggested as an appropriate control method for treating low concentration levels of VOCs in waste gases as adsorption is performed at low temperature (Atwood et al., 1998). In particular, it appears to be a cost-effective alternative to other classes of catalysts, which could be used to treat VOCs in two steps. Silver (Ag) has been well known for its epoxidation activity and the deep oxidation of CO and methane (Kundakovic et al., 1999). Furthermore, in recent years, many researchers have shown that metal oxide catalyst, with the highest catalyst activity, are copper based metal oxide catalyst (CuO) (Li et al., 2015). Recently, graphene oxide (GO) has drawn increasing attention as an ideal support material due to its special layered structure and surface properties. GO contains abundant oxygen functional groups (hydroxyl, epoxy, carbonyl, carboxyl groups) (Santra et al., 2013) on its surface. These functional groups can also serve as anchoring sites to decorate metal or metal oxide nanoparticles onto the GO surface, thus guaranteeing fertile opportunities to synthesize novel functional catalysts. Furthermore, by loading a catalyst on a GO support, the exposed active sites of the resulting system are far more compared to what would be expected from simple catalyst nanoparticles, thus achieving the maximum efficiency in the use of active sites (Albero et al., 2015). Typically, aerogels are regarded as suitable candidates to be adopted as catalyst supports thanks to their fascinating textural and structural properties, and to the versatility of their synthesis route, which allows a flexible control of the texture, composition, homogeneity and structural features of solids at a molecular level (Zhang et al., 2017). On the other hand, carbon aerogel, in particular graphene aerogel (GA), has shown high catalytic performances as catalyst support for VOCs combustion due to two major reasons: (i) its hydrophobic character allows performing VOC combustion at relatively low temperatures with minimum water vapor chemisorption on the catalyst surface; (ii) its tunable textural properties enable a homogeneous and high dispersion of the active metal phase on it, along with a good adsorption performance toward VOCs (Roland et al., 2014; Morales-Torres et al., 2015; Tomatis et al., 2016). Among VOCs, 2-ethyl-1-hexanol (2-EH) can be naturally present in food and it is typically produced through petrochemical synthesis and predominately used for the production of plastics. The presence of 2-EH in the atmosphere can cause severe health problems. In this work, the removal of 2-EH from a VOCs stream emitted from recycled plastics, by means of catalytic oxidation through a novel dual function adsorbent-catalyst material, made of snowman-like nanoparticles of CuO and Ag embedded in a graphene oxide aerogel (GOA) porous matrix was proposed. The combination of the highly active phases of CuO/Ag and the adsorption ability of graphene, which favours the reaction and stabilizes the nanoparticles, leads to the quasi-complete abatement of 2-EH (>98 %).

2. Experimental

2.1 Preparation of graphene oxide

The preparation of GO was carried out by using commercially available reagents. In particular, graphite powder (synthetic, 99.9%), sulfuric acid (H₂SO₄, 95-97%), potassium permanganate (KMnO₄, ≥ 99%), hydrogen peroxide (H₂O₂, 30%), were purchased from Aldrich Chemical Co and L-Phenylalanine (Ultra ≥

99%), was purchased from Fluka, and used as received. The graphene oxide (GO) was prepared from pristine graphite through a modified Hummers' method and subsequent sonication, as previously reported (Casa et al., 2018).

2.2 Preparation of graphene oxide aerogel

The GOA was prepared by in situ chemical reduction-induced assembly using L-phenylalanine as a reducing agent, as described elsewhere (Sarno and Cirillo, 2018).

2.3 Preparation of CuO/Ag-rGOA

The preparation of the CuO/Ag-GOA catalyst (Figure 1a) was carried out by means of commercially available chemicals. Benzyl ether (99%), 1,2-hexadecanediol (97%), oleic acid (90%), oleylamine (>70%), copper (II) acetylacetonate, silver nitrate ($\geq 99.0\%$) were purchased from Aldrich Chemical Co. In particular, $\text{Cu}(\text{C}_5\text{H}_7\text{O}_2)_2$ (2 mmol), 1,2-hexadecanediol (10 mmol), oleic acid (6 mmol), oleylamine (6 mmol), 0.035 g of silver nitrate were mixed and magnetically stirred in presence of 0.5 g of graphene oxide aerogel under a nitrogen flow, following standard airless procedures (Sarno et al., 2016).

2.4 Catalyst activity

The activity of the as-prepared catalyst for 2-EH oxidation was evaluated by a laboratory apparatus (Figure 1b) consisting of a fixed-bed reactor under atmospheric pressure. A total amount of 100 mg of catalyst was loaded into a quartz tube reactor (length = 300 mm, inner diameter = 16 mm; external diameter = 35 mm). Gaseous 2-EH was generated by blow a purified air flow on a 2-EH solution in an incubator kept at 0°C . The total flow rate was 50 mL min^{-1} with 130 ppm of 2-EH. The composition of gas from the catalytic unit was monitored with a gas chromatograph GC-MS, after passing in the catalytic unit for methanation (catalyst based on Ni under an hydrogen flow for carbon oxides conversion). The 2-EH conversion was determined adopting the following equation:

$$\text{2-EH Conversion (\%)} = \text{CO}_2 \text{ yield (\%)} = \frac{[\text{CO}_2]_{\text{out}}}{[\text{2-EH}]_{\text{in}}} \times 100\% \quad (1)$$

where $[\text{CO}_2]$ is CO_2 concentration in the effluent, and $[\text{2-EH}]$ is the 2-EH concentration in the feed gas.

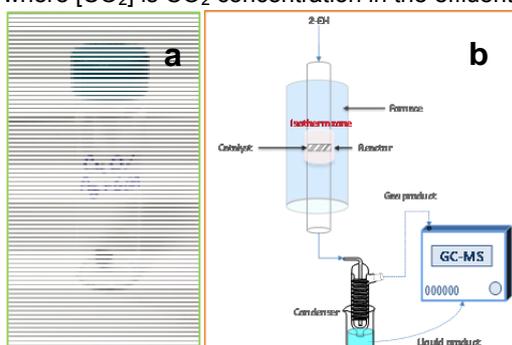


Figure 1: A photo of catalysts based on reduced graphene oxide (a). Schematic representation of the experimental units used for the catalytic oxidation of 2-EH (b)

2.5 Materials characterization

The characterization was performed by using several techniques. Transmission electron microscopy (TEM) images were obtained with a FEI Tecnai electron microscope operating at 200 kV with a LaB_6 filament as the source of electrons. The preparation of samples for TEM analysis involved sonication in ethanol for 2–5 min and deposition on a carbon grid. Scanning electron microscopy (SEM) images were obtained by the use of a LEO 1525 electron microscope. Raman spectra were obtained at room temperature with a micro-Raman spectrometer Renishaw inVia with a 514 nm excitation wavelength (laser power 30 mW) in the range $100\text{--}3000 \text{ cm}^{-1}$. XRD measurements were performed with a Bruker D8 X-ray diffractometer using $\text{CuK}\alpha$ radiation. The reaction products were determined using a gas chromatography mass spectrometry (GC-MS) (Thermo Fischer Scientific) by using HP-5 capillary column ($0.25\mu\text{m}\text{--}0.25\text{mm}\times 30\text{m}$). Temperature setting was as follows: injection port = 100°C , column = 100°C , detector = 120°C . The chromatogram was calibrated with gases at known concentrations as standard mixtures. Helium was used as the carrier gas.

3. Results and discussion

The structural characteristic of CuO/Ag-rGOA was examined by Scanning and Transmission Electron Microscopy. SEM image, at 30,00 KX of magnification (Figure 2a), of CuO/Ag-rGOA displays a porous morphology, showing that the material possesses well-defined macropores and mesopores. In particular, the SEM image reveals the presence of quasi-spherical aggregates of CuO/Ag nanoparticles embedded in the aerogel. In addition, the morphology of CuO/Ag-rGOA catalyst was observed by TEM analysis (Figure 2b). TEM image at higher magnification clearly reveals that the as synthesized catalyst consists of snowman-like CuO/Ag NPs, characterized by quasi-spherical CuO NPs (about 8 nm diameter) supporting faceted Ag NPs (< 2 nm) dispersed in the rGO aerogel.

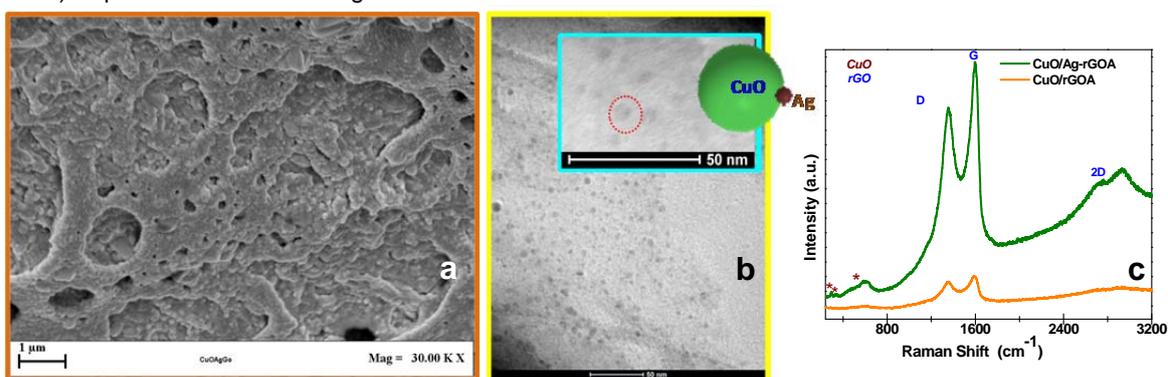


Figure 2: SEM image (a) and TEM images of CuO/Ag-rGOA catalyst (b); Comparison of Raman Spectra of CuO/Ag-rGOA and CuO/rGOA (c)

In Figure 2c, Raman spectra of CuO/Ag-rGOA and CuO/rGOA, in the wavenumber range 240–2900 cm⁻¹, are reported. The main Raman signals of CuO are found at 295, 335 and 610 cm⁻¹ (Tsai et al., 2018). The typical graphene Raman bands are also present. In particular, the D band is at around 1355 cm⁻¹. It is a breathing mode of k-point photons of A_{1g} and it corresponds to the imperfections and disorders of graphene, particularly the defects located at the edge of it. The G band is at approximately 1587 cm⁻¹, which represents the E_{2g} in-plane vibrational mode of sp² hybridized C atoms (Sarno et al., 2017a).

The 2D band, located at around 2700 cm⁻¹, is due to a two photon double resonance Raman process. Its presence confirms the reduction of graphene oxide. Moreover, the appearance of a peak at about 2700 cm⁻¹ is indicative of the GO reduction. This happened during the synthesis procedure for both chemical and thermal phenomena, leading to a reduced graphene oxide (rGO) (Sarno et al., 2017b).

On the other hand, it is worth noticing that the intensities of the D and G bands of CuO/Ag-rGOA were increased after the decoration with Ag NPs. This is due to the Ag NPs enable Raman signals increases, through the Surface-Enhanced Raman Spectroscopy (SERS) effect (Sarno and Casa, 2018). The Raman spectrum of GOA (Sarno and Cirillo, 2018) shown that the D and G bands of GOA were located at 1345 and 1601 cm⁻¹, respectively.

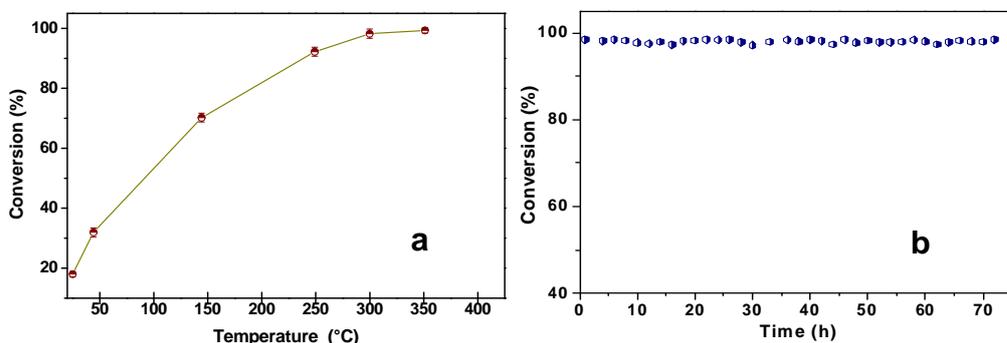


Figure 3: Catalytic activity (a) and stability test of CuO/Ag-rGOA at 350 °C (b). Reaction conditions: GHSV = 30000 mL · (g_{cat} · h)⁻¹; total flow rate = 50 mL · min⁻¹; 100 mg of catalyst

The experimental results revealed that the CuO/Ag-rGOA catalyst exhibited a very good catalytic activity for 2-EH oxidation. The catalytic activity tests of 2-EH oxidation are reported in Figure 3. In particular, Figure 3a shows the 2-EH conversion at different reaction temperatures at a low concentration of 2-EH. Our catalyst exhibits a conversion of almost 99 % at a temperature of 350 °C.

Generally, the activity of a given catalyst can be mainly characterized by the temperature and by the conversion efficiency. These parameters, from previously published papers (Papaefthimiou et al., 1997; Minicò et al., 2001; Brazlauskas and Kitrys, 2008) on the oxidation of alcohols with different catalysts, are reported in Table 1. Our catalyst, based mainly on inexpensive components, is the first example of successful 2-EH abatement.

Table 1. Reported catalyst systems for the catalytic oxidation of alcohols

Alcohols	Catalysts	T (°C)	Conversion (%)	References
n-butanol	Pt/Al ₂ O ₃	340 ^(a)	Complete	Papaefthimiou et al., 1997
2-propanol ethanol	Au/Fe ₂ O ₃	350 ^(b)	100	Minicò et al., 2001
1-propanol	CuO/CaX	350 ^(c)	Total	Brazlauskas and Kitrys, 2008
2-ethyl-1-hexanol	CuO/Ag-rGOA	350	~99	<i>This work</i>

^(a) 0.3% Pt/Al₂O₃ were prepared by calcination at 550-800°C for 2 h; ^(b) Au/Fe₂O₃ catalyst was subject to calcination pretreatments by flowing a 10% O₂ stream diluted in He at the chosen temperature (200, 300 or 450°C) for 1 h.; ^(c) CuO/zeolite catalyst was calcined in air flow at 450°C for 6h.

Stability is a critical aspect in the development of catalysts. As shown in Figure 3b, the catalytic activity was almost maintained for 72 hours of tests, evidencing the stability of the catalyst during operation at the best. Catalytic tests to evaluate the specific role of the different nanocatalyst components are necessary and have been programmed.

4. Conclusions

Catalytic tests for the oxidation of gaseous 2-EH were conducted. In particular, an innovative catalyst, which allows both the adsorption and the conversion/removal of molecules responsible for bad smells, has been synthesized. The synthesized catalyst, CuO/Ag-rGOA, consists of a porous graphene oxide aerogel with high adsorption properties, loaded with catalytic CuO/Ag nanoparticles. CuO/Ag-rGOA exhibited high catalytic performance and excellent oxidation stability toward 2-EH oxidation. 2-EH conversion was almost 99% and it was carried out at 350 °C. Its catalytic activity was maintained for 72 hours of tests. The high activity and good stability of CuO/Ag-rGOA nanocatalyst make it an excellent candidate for the development of catalytic oxidation of 2-EH. The CuO/Ag-rGOA nanocatalyst offers great potential to be explored for developing efficient noble metal-free catalysts to removal 2-EH and others VOC at low temperature. The study of this catalyst for the abatement of other volatile compounds, also at different concentrations, is already in progress.

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