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The efficiency of catalytic ozonation using carbosieve in xylene removal from waste air stream

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ABSTRACT

Catalytic ozonation process (COP) is a new method of single ozonation process that has recently received considerable attention in air pollution control industry. The present study aimed to evaluate the efficiency of catalytic ozonation using carbosieve in xylene removal from the waste air stream. An experimental-laboratory study was conducted at the pilot scale, and the performance of carbosieve adsorbent system, simple ozonation, and catalytic ozonation was studied. A lab-scale pilot was designed and launched at the chemical laboratory of the Faculty of Health, Hamadan. Xylene liquid with purity of 99.5% was prepared from Merck (German) and carbosieve from Aldrich Co. (USA). The specific surface rate and the used catalyst size in this study were 972 m^2/g and 1.8–2.3 mm, respectively. Results showed that by increasing xylene concentration in the beginning of the system, the time of bed saturation and adsorbent breakpoint decreased, and removal efficiency by ozone gas declined by increasing xylene concentrations in the reactor inlet from 20 to 200 ppm. The findings showed that the carbosieve breakpoint and saturation in catalytic ozonation can happen later from the absorbent carbosieve system alone. It seems that the catalytic ozonation was more efficient in xylene removal compared with absorbent carbosieve system and simple ozonation. The COP by carbosieve could be a promising alternative to conventional volatile organic compounds; removal systems are particularly toxic compounds from waste air stream.

Keywords: Catalytic ozonation; Carbosieve; Xylene; Air pollution; Air stream

1. Introduction

Volatile organic compounds (VOCs) are a large group of air pollutants emitted into the atmosphere from variety of sources. Nowadays, the biggest challenges and environmental concerns such as ozone depletion layer, the greenhouse effect, photochemical oxidants production, air quality deterioration, and potential health hazards are the results of the presence of these compounds in the atmosphere [1–3]. Mono aromatic hydrocarbon compounds such as benzene, toluene, ethyl benzene, and xylene (BTEX) are the most common VOCs and are very important because of having the highest toxicity, reactivity, and potential of photochemical oxidants production and endangering public health and welfare, so there are a lot of them in the air around cities and industrial areas [4–6]. Among these compounds, xylene (benzene dimethyl) is an important chemical material that in recent years is widely used in various industries due to the relatively safe substitute for benzene. Xylene is used as a solvent in the printing industry, rubber, leather, paint and wax polishes, pathology, histology laboratories and in products such as synthetic fibers, plastics,

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insecticides and pesticides, glues, and lacquers [7,8]. Xylene is also one of the major and important fundamental compounds of the gasoline used as fuel for cars [9]. Xylene has fewer side effects than benzene due to having side branches of the blood effects. Thus, the International Agency for Research on Cancer (IARC) [10] has placed it in Group 3 as carcinogenic chemicals (A4 group of American Conference of Governmental Industrial Hygienists). The dermal and inhalation exposure to xylene can cause hepatotoxin, kidney damage, and above all, disturbances and damage to the central nervous system [11,12]. In addition, xylene is one of the 189 hazardous air pollutants and a component of priority pollutants listed in the Hazardous and Toxic Atmospheric Contaminants under Clean Air Act Amendments of 1990 (USA). Based on the existing standards, permissible exposure, which limits with xylene, is TLV-TWA = 100 ppm and TLV-STEL = 150 ppm [13].

Due to various environmental and health effects and the need to eliminate or reduce these compounds in the contaminant emission streams, different and conventional methods are utilized such as adsorption, absorption, thermal and catalytic oxidation, ozonation, biofilters, and condensation for their refinement from the waste air stream [14]. Compared with conventional methods, advanced oxidation processes (AOPs), which involve various options such as O_3/H_2O_2, O_3/UV and UV/some semiconductor (TiO_2), are very suitable methods for the degradation of toxic air pollutants that can enhance the process of removing these compounds. Pollutants degradation by AOPs is based on the production and participation of free radicals that have high oxidation power and are able to convert the organic compounds into inorganic and harmless materials. These free radicals convert them into inorganic materials through one or more stages by attacking organic matter molecules and obtaining the hydrogen ions. Catalytic ozonation process (COP) is a new method of single ozonation process (SOP), which has recently received considerable attention in air pollution control industry. In this process, for improving the performance of SOP, accelerating the decomposition process, increasing of reaction time, and also the presence of residual ozone in the output air, various materials as catalyst are used [15–18]. Catalysts such as activated carbon, silica gel, metal oxides, and various resins have been used in the COP for removing VOCs from waste air streams [15–17]. Therefore, with regard to the application and functionality of various carbon absorbents in the treatment of air pollutants and confirming the role of activated carbon in ozone deformation and converting to free activated radicals, carbosieve that is a type of carbon absorbent was used. Carbosieve is produced as a result of pyrolysis of synthetic polymers or some petroleum compounds with high porosity and surface. Carbosieve is applied as a catalyst and non-polar adsorbent in COP and specifically used to absorb volatile hydrocarbons [19,20]. The aim of this study was to evaluate the efficiency of catalytic ozonation using carbosieve in xylene removal from the waste air stream.

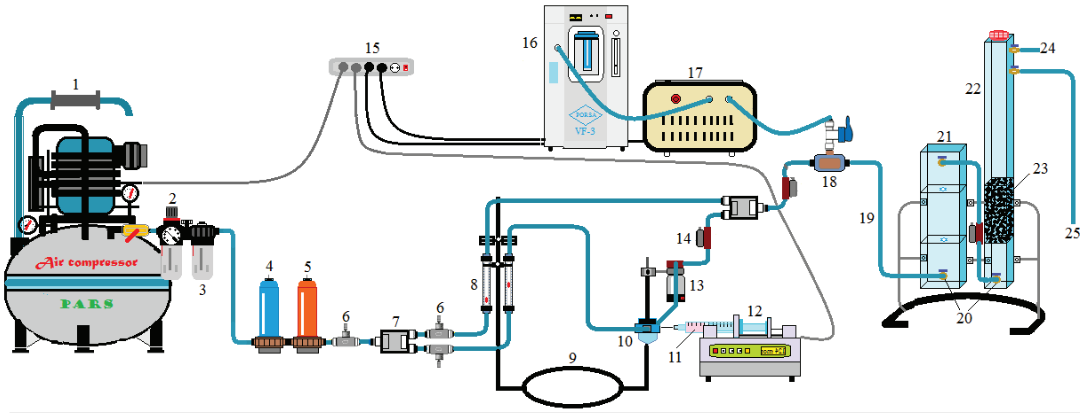
2. Materials and methods

An experimental-laboratory study was conducted at the pilot scale, and performance of carbosieve adsorbent system, simple ozonation, and catalytic ozonation was studied. For this purpose, a lab-scale pilot was designed and launched at the chemical laboratory of the Faculty of Health, Hamadan. The system used in this study is shown in Fig. 1.

Xylene liquid with purity of 99.5% was prepared from Merck (German) and carbosieve from Aldrich Co. The specific surface rate and the used catalyst size were 972 m^2/g and 1.8–2.3 mm, respectively. For preparing the specified density of the polluted gas, an atmospheric standard was used by dynamics method. Air-containing polluted gas and ozone gas continuously entered from the bottom of the reactor.

The reactor was made of glass and a height of 0.7 m installed at 0.2 m from its floor, lattice plane for orientation of the adsorbent (catalyst). Required air was supplied by a compressor equipped with oil gear and, after pressure adjustment for drying and removing any possible contamination of organic compounds, was passed through the combined column of silica gel and activated char.

For preparing air-containing polluted gas in the desired concentrations, a syringe pump was used for injection of



1- Air compressor 2- Pressure regulator 3- Oil trap and prefilter 4- dryer (silicagel column) 5- Activated carbon filter 6- flow control valve 7- Connect tee fitting 8- Flow rate control 9- Bracket 10- Xylene injector vesel 11- Xylene reservoir 12- Auto programmed injector 13- Evaporator & mixing chamber 14-Sampling port 15- Power supply 16- Oxygen making machine 17- Ozone generator 18- Ozone injector 19- Connector pipes 20- Needle valve 21- Baffle mixing chamber 22- Processing reactor 23- Catalyst (carbosieve) 24- Gas outlet 25- sampling pump & Gas chromatograph.

Fig. 1. Schematic diagram of the experimental setup.

liquid xylene. Ozone was prepared by an ARDA ozone generator, COG-0M model. In this generator, ozone is produced through creating an electrical discharge with an AC in a discharge valve and in the presence of oxygen. Required oxygen of ozone generator was supplied from an oxygen machine, PORSA VF-3 model with high purity and ability to adjust the injection of oxygen. In ozonation unit, ozone gas was entered into the contaminated gas stream with xylene, transversely.

In order to prevent and reduce the possible fluctuations and for integration of the equilibrium and constant stream containing of contaminated gas, before entering to the reactor containing a bed of adsorbent, a baffled mixing chamber was used; the stream after going out of the mixing chamber entered the reactor containing carbosieve as the catalyst and non-polar adsorbent bed. The carbosieve was placed for 2 h at 400°C before testing for removing moisture and any possible contamination and was dried in a desiccator.

Sampling was performed using charcoal tube manufactured by SKC Co., and xylene concentrations were measured before and after testing in the air stream using the 1501 NIOSH Manual of Analytical Method and using gas chromatograph Shimadzu, Japan, 2010 model equipped with a flame ionization detector, which is the selecting detector for identifying organic compounds with a column length of 30 m, columns internal diameter of 0.25 mm, film thickness of 0.25 μm and temperature range of 50°C–200°C.

Optimal decomposition conditions of gas chromatography with site injection temperature of 200°C and the detector temperature of 250°C, the carrier gas at about 30 mL/min, the stream of hydrogen gas of 30 mL/min and air stream around 300 mL/min were provided [21,22]. Experiments at ambient room temperature (27°C ± 2°C) and atmospheric pressure and suitable ventilation were performed. In order to assess the accuracy of the decomposition method, each sample was injected three times in the capillary column chromatographic, and their mean values were reported as the value of the parameter.

3. Results and discussion

3.1. The efficiency of the carbosieve adsorbent bed in xylene removal from contaminated air stream

At this stage, the parameters affecting system performance, including the inlet air stream rate into reactor 3 L/min, amount of adsorbent bed 15 g, and the relative humidity 5%, were kept constant, and only the performance of system on various xylene concentrations was studied and tested. The results showed that by increasing xylene concentration in the beginning of the system, the time of bed saturation and adsorbent breakpoint decreased. The efficiency results of carbosieve in xylene removal are shown in Fig. 2.

3.2. The ozone efficiency in xylene removal from waste air stream

Fig. 3 shows the efficiency of ozone gas (simple azonation) individually and absence of carbosieve in xylene removal from waste air stream. To measure the concentration of ozone gas generated by the ozone generator, iodometry method was used. In this method, the output of the ozone generator was determined by passing the gas through two sets of containers containing potassium iodide (KI) for 10 min [23,24]. Ozone gas concentration was calculated through fixed input 3 L/min pure oxygen into ozone generator machine in this system, 0.43 mg/L, and other variables affecting the performance of the system include the inlet air stream rate of 3 L/min into reactor and fixed relative humidity of 5%. At this stage, removal efficiency by ozone gas decreased by increasing xylene concentrations in the reactor inlet from 20 to 200 ppm.

3.3. Determining the efficiency of catalytic ozonation for xylene removal from waste air stream

The results of catalytic ozonation efficiency are shown in Fig. 4. At this stage, to determine the efficacy of the catalytic ozonation, combination of the two methods was used:

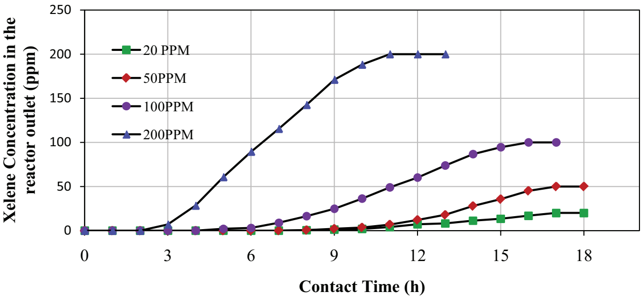


Fig. 2. The breakpoint and saturation time of the carbosieve adsorbent bed in xylene removal from waste air stream.

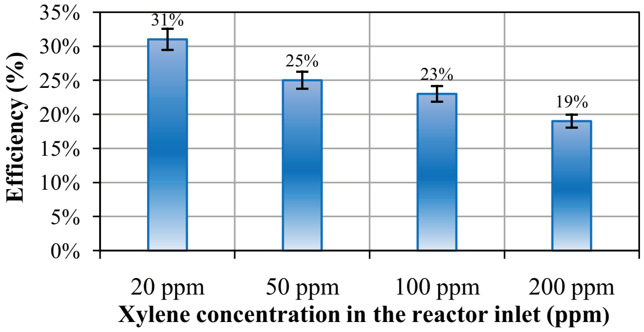


Fig. 3. The ozonation efficiency in xylene removal from waste air stream.

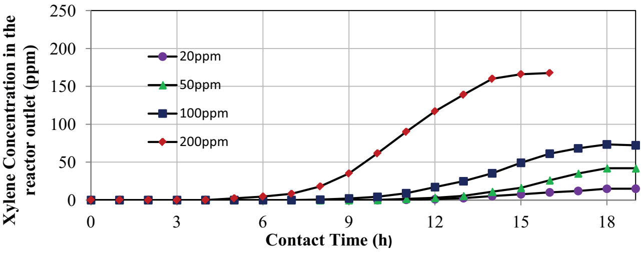


Fig. 4. The breakpoint and saturation time in the catalytic ozonation process in xylene removal from waste air stream.

adsorbent bed carbosieve as catalyst and simple ozonation considering experimental conditions and affective variables consisting the inlet air stream rate of 3 L /min into the reactor, the adsorbent bed of 15 g, the amount of ozone input of 0.43 mg/min, and relative humidity of 5%, and the efficiency of catalytic ozonation was investigated in terms of the concentration of xylene output from system and the breakpoint of the adsorbent. The findings showed that the carbosieve breakpoint and saturation in catalytic ozonation can happen later from the absorbent carbosieve system alone.

3.4. Determining the xylene adsorption capacity in the adsorbent carbosieve system and catalytic ozonation process

At this stage, the maximum adsorption capacity (capacity at saturation point) in mg adsorbed xylene per g carbosieve on two adsorbent carbosieve systems, and catalytic ozonation was investigated. Eq. (1) was used for calculating mg adsorbed xylene onto the g adsorbent.

$$q_e = (C_0 - C_t) \times \frac{v}{m} \tag{1}$$

In this equation, q_e is the absorptive capacity (mg/g); C_e is the initial concentration (mg/L); C_t is the adsorbed concentration (mg/L); v: volume (L); and m is the weight of adsorbent (g). The results of xylene absorptive capacity in input concentrations using carbosieve in relative humidity of 5% in the catalytic process show the significant increase of xylene absorption compared with the carbosieve sorbent alone (Fig. 5).

For better displaying the efficiency of the COP, the xylene removal in the carbosieve adsorbent bed system and simple ozonation process were studied. The results of the carbosieve adsorbent bed efficiency in the removal of various concentrations of xylene from the waste air stream showed that the absorbent breakpoint in the reactor outlet decreased from 7.5 to 2 h, using carbosieve absorbent individually by increasing xylene concentrations from 200 to 20 ppm.

Thus, by increasing xylene concentrations in inlet air to the reactor, the removal efficiency decreased through carbosieve. Accordingly, carbosieve is not suitable for removal of high concentrations of xylene because in a short period of time it saturates by contaminants and needs frequent regeneration. In addition, carbon adsorbents such as carbosieve by physical absorption of contaminant, which is a specific and reversible process gather contaminant molecules on its surface in several layers by Van der Waals forces and without changing the nature of the absorbed contaminant, and only with changing the phase and its adsorption from inlet air on absorbed body causes its elimination [25,26].

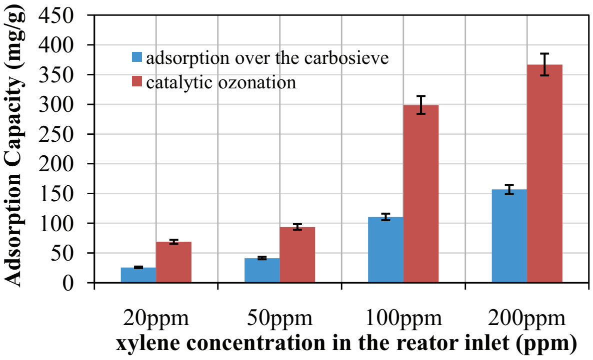


Fig. 5. The compare xylene adsorption capacity in carbosieve adsorbent system and catalytic ozonation.

Therefore, when the contaminant is dangerous or pollutes the environment, using absorbents alone would be considerable and cannot be a complete process for removing fly contaminants from waste air stream. The main limitation of surface adsorbents is reduction of efficiency when they saturate or reach equilibrium with contaminants [25–27]. Dwivedi et al. [27] in a comparative study on removal of VOCs by cryogenic condensation and adsorption by activated carbon fiber reported that in adsorption onto activated carbon, adsorbent breakpoint decreased considerably by increasing contaminant concentration. They proposed that using the absorption method for the control of VOCs from the outlet waste air stream about ppm and less than ppm is suitable.

The presence of moisture in the inlet air stream reduced the efficiency of absorbed contaminant removal by carbon absorbents. Although this study was performed with low relative humidity conditions (5%), but in conditions of high relative humidity, the water molecules are collected in the hydrophilic sites of adsorbent bed, followed by filling of the existing micropores on the carbosieve surface by capillary effect. This will reduce the carbosieve absorption capacity by preventing the entering xylene to absorption sites [28–30].

Rodríguez-Mirasol et al. [30] studied the influence of water vapor on the VOCs adsorption on lignin-based activated carbons and showed that by increasing the amount of water vapor in the inlet air on the absorbent, the absorbed VOC severely decreased. This reduction of adsorption efficiency for higher moisture values may be due to competing of water molecules with VOC molecules in adsorption on the activated carbon surface and its saturation. Nikpay et al. [31] investigated methyl iodide gas removal from the air by activated carbon impregnated with amine salts and stated that the low uptake of methyl iodide onto activated carbon is possible only in conditions of low relative humidity and cannot be a reliable adsorbent in treatment of waste air streams to methyl iodide in high relative humidity conditions. By increasing relative humidity to 65%, the adsorption capacity onto activated carbon decreased about 92%.

The results of assessing SOP efficiency in the xylene removal from waste air stream showed that in the studied concentrations, the average removal efficiency is <25%. By increasing concentration from 20 ppm at the beginning of the system to 200 ppm, the removal efficiency decreased from 31% to 19%. The main reason for efficiency reduction in the use of ozone gas alone is capability of low oxidation of aromatic compounds and alkenes by ozone and low detention time required for complete reaction between ozone and xylene as a result of the system continuity [32], corresponding with Chao et al.'s study [33]. They found that in evaluating the potential use of combined ozone and zeolite system for gaseous toluene elimination, toluene removal efficiency by ozone alone was about 8% and explained that the reaction time between ozone and toluene is very short.

For overlapping and eliminating the limitations of ozonation method and carbosieve absorbent alone and improving their performance in the refinement of VOCs from waste air stream, a combination of these two methods as the catalytic ozonation was used. The results of this survey indicated that catalytic ozonation has a higher significant efficiency compared with discrete absorption method on carbosieve and simple ozonation. The adsorbent breakpoint and exiting xylene from combined reactor decreased from 12 to 5 h with increasing concentration from 20 to 200 ppm. This time for the absorption system on the carbosieve was between 7.5 and 2 h. Thus, it was observed that appearance time of xylene in the outlet of combined reactor was greater in the same laboratory conditions, and average efficiency of 48% was observed in all concentrations.

In carbosieve system, the outlet xylene concentration was equal to the inlet concentration after saturation of the bed in the outlet reactor, while in catalytic ozonation system, the outlet xylene concentration after saturation was less than the inlet concentration. Also, the comparison of the results of the maximum capacity of xylene adsorption in two adsorption systems on carbosieve and catalytic ozonation showed that in similar conditions, the maximum capacity of xylene adsorption per gram of adsorbent dose in the catalytic ozonation system, especially at higher doses, is more than twofold of the adsorption capacity in the carbosieve system, confirming the higher efficiency of catalytic ozonation. Thus, the synergistic effect and combination of ozone catalyst (carbosieve) in the COP for removing xylene were recognizable, due to the dominance of the catalytic reactions with regard to the oxidation reactions and adsorption. It seems that the catalyst surface properties and surface active sites (Lewis acid sites) are the most important factors in the ozone decomposition into active radicals (Fig. 6).

It is reported that in the reaction between ozone and Lewis acid sites in surface adsorbents, the ozone is decomposed to active oxygen atoms and active oxygen atoms by attacking single joint bonds between carbon atoms (C–C) or joint bonds between carbon hydrogen atoms (C–H), causing the decomposition of xylene. Also, the surface adsorbent with adsorption of ozone and xylene increased the required detention time for the reaction between ozone and xylene and improved the removal efficiency. Furthermore, the oxygen molecules from ozone decomposition react with existing water vapor in the system, leading to production of activated hydroxyl radicals, which participate in indirect decomposition and oxidation of xylene and increase removal efficiency (Fig. 7) [31–36].

The results of similar researches have also shown that in COP, addition of ozone ($E° = 2.08$ V) O_3, active oxygen molecules ($E° = 2.42$ V) O•, hydroxyl radicals ($E° = 2.80$ V) OH• and oxygen molecule ($E° = 1.32$ V) O_2 has a significant role in the decomposition of xylene, which increases the rate of ozone decomposition for xylene oxidation. This molecule also oxidizes the radical intermediate compounds, which are the main reasons for the deactivation of the catalyst in autoxidation processes [32–35].

In the study of Einaga and Ogata [37] on benzene oxidation with ozone over covered catalysts with manganese oxide catalysis, the catalyst surface area has been introduced as one of the most important factors in the reaction related with the ozone decomposition rate and benzene oxidation. Kwong et al. [15] studied VOCs' removal from batch spaces by ozonation on various porous materials and reported that ozone on Lewis acid sites of the porous adsorbent was decomposed to oxygen molecule and active oxygen atom.

Several possible mechanisms have been proposed for VOCs' catalytic ozonation. The first possible mechanism is that both ozone and VOC are simultaneously adsorbed onto the adsorbent, and then adsorbing ozone undergoes a decomposition process and is converted into active radicals and secondary reactions with the VOC. Another possible mechanism is that VOC is adsorbed onto the adsorbent and directly reacts with the ozone gas phase. VOC reaction rates depend on the concentration of ozone in the gas phase (Fig. 8). The removal rate of toluene in COP with regard to zeolite adsorption process and MCM-41 increased from 20% to 40%, which is in line with the results of this section [15].

Einaga and Futamura [38] investigated catalytic oxidation of benzene with ozone over alumina-supported manganese oxides and stated that benzene oxidation reactions by ozone gas depend on the decomposition of ozone. Their findings indicate that the activity of various species of active oxygen

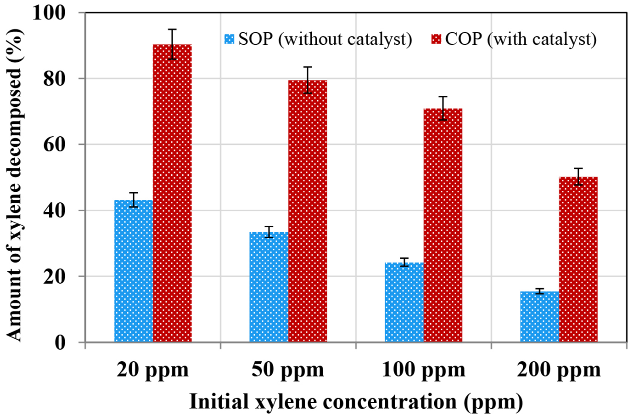


Fig. 6. The results of xylene decomposition in ozonation and catalytic ozonation processes.

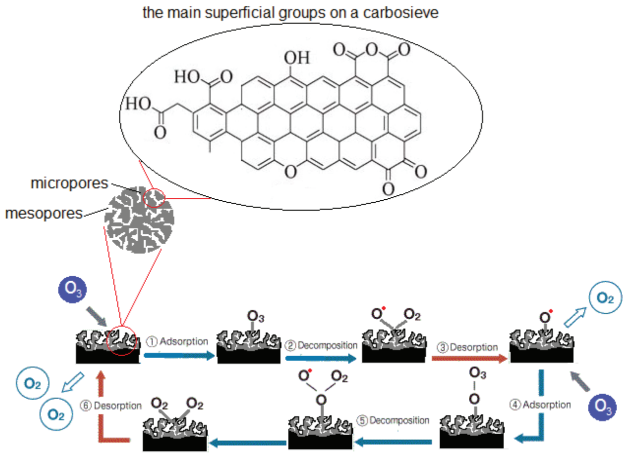


Fig. 7. The role of surface active sites of carbosieve in ozone decomposition.

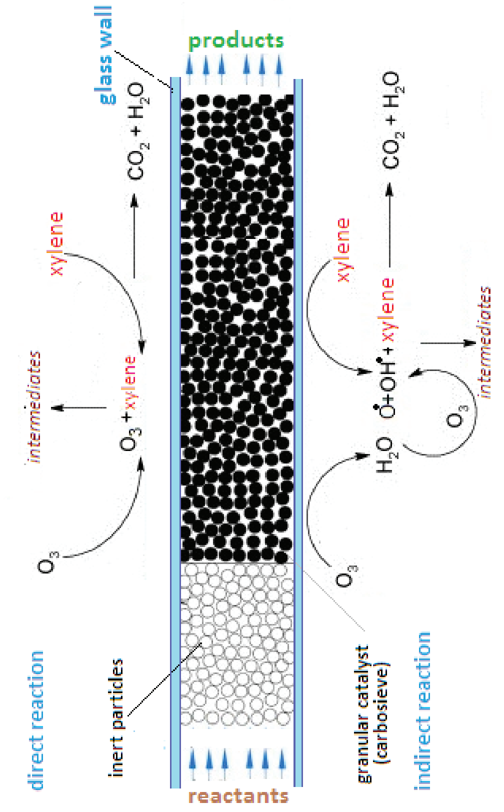


Fig. 8. Direct and indirect reactions of xylene decomposition.

formed by ozone destruction on the catalyst is responsible for the decomposition and oxidation of benzene.

Chiang et al. [39] evaluated the ozonation of activated carbon and its effect on the adsorption of VOCs exemplified by methyl ethyl ketone and benzene and reported that ozonation of activated carbon can increase the special surface and oxygen-containing functional groups of activated carbon. Ozone reactions on activated carbon change the physical structure of activated carbon through enlarging the whole volume in the micropore surface, and as a result, active sites and functional groups, especially hydroxyl and carboxyl, increase.

4. Conclusion

The findings of the present study revealed that the catalytic ozonation had more efficiency in xylene removal compared with absorbent carbosieve system and simple ozonation. Therefore, the COP eliminates much higher concentrations of xylene over time. According to the results obtained from this study, the COP with carbosieve could be a promising alternative to conventional systems of VOCs removal, particularly toxic compounds from waste air stream.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

SAB was the main investigator and collected the data; MRS and GHA supervised the study. FGH was advisor of the study. MS and TJ performed the statistical analysis, and ED drafted the manuscript. All authors read and approved the final manuscript.

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