

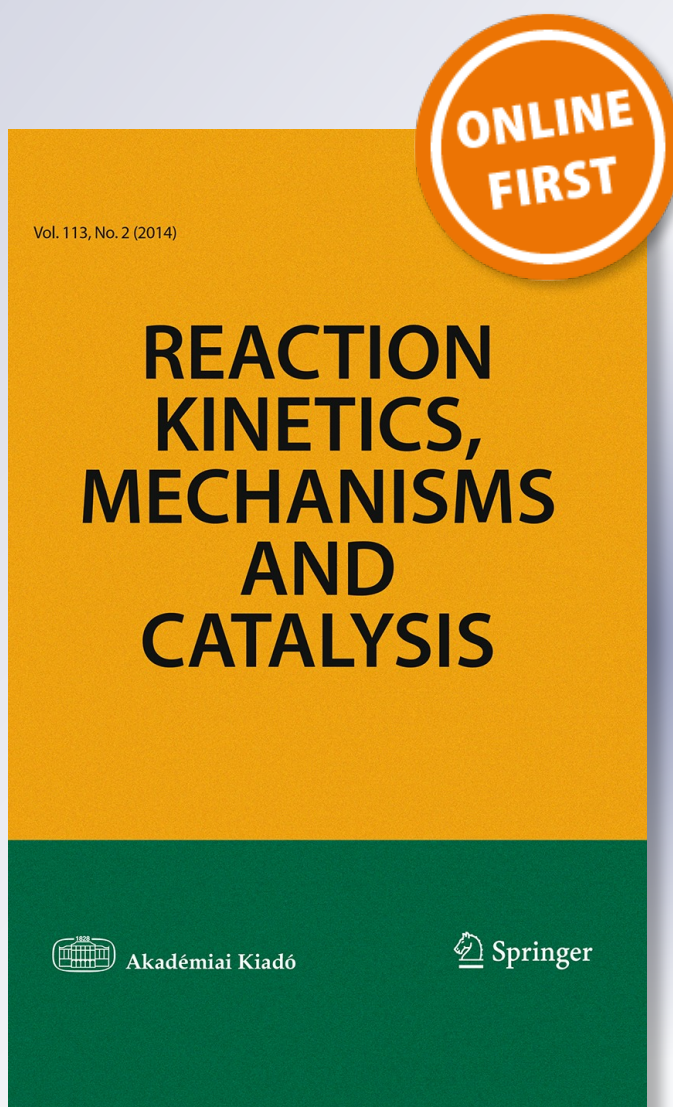
Catalytic removal of a mixture of volatile organic compounds present in indoor air at various work sites over Pt, MnO_x and Pt/MnO_x supported monoliths

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Catalytic removal of a mixture of volatile organic compounds present in indoor air at various work sites over Pt, MnO_x and Pt/MnO_x supported monoliths

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Abstract The elimination of volatile organic compounds (VOCs) was investigated placing a novel catalytic system in the chimney venting of a commercial air extractor, using Pt, Mn and Pt/Mn catalysts supported on ceramic monoliths. The VOCs oxidized were chloroform (CHCl₃), methyl ethyl ketone (MEK), toluene and xylene, alone, in a binary mixture and in a mixture of all of them. The conversion of VOCs is in general similar, regardless of whether they are in a mixture or alone. Pt/Mn monoliths exhibited the highest destruction activity for the abatement of VOCs alone and in mixtures, due to a synergetic effect between platinum and manganese. The results show that these catalytic systems can be coupled satisfactorily in the venting of the air extraction equipment still working at high flow rates.

Keywords Monoliths, bentonite · VOC mixtures · Mn · Pt · CHCl₃

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Introduction

Volatile organic compounds (VOCs) are considered as significant atmospheric pollutants not only for their toxicity and malodorous nature, but also because they are ozone and smog precursors [1]. Major VOC emissions are from industrial sites, such as chemicals manufacturing plants; paper, pharmaceutical and food coating processes; car, metallic and plastic surfaces, textiles, etc. VOC emissions may be classified into two categories: non-halogenated (ethane, propylene, benzene, toluene, ethanol, acetone, methyl ethyl ketone) and halogenated organic compounds (chlorinated ethanes, carbon tetrachloride, vinyl chlorides, chloroform).

On the other hand, consideration of indoor exposures to air pollutants is critical because people spend a large fraction of their time indoors, where concentrations of many airborne pollutants often tend to exceed ambient levels [2, 3]. This occurs in part because there are numerous indoor sources of VOCs (carpets and adhesives, home and personal care products, cleaning and disinfecting chemicals, barbecue, cosmetics, paints, solvents, and varnishes) and because the relatively low rates of outdoor air ventilation prevent the rapid dispersal of airborne contaminants [4–11]. Common symptoms of exposure to VOCs include eye, nose and throat irritation, headaches, nausea and asthma, and a long-term exposure to high levels of VOCs increases the risk of cancer and central nervous system damage.

The more important VOC treatment techniques are incineration, catalytic oxidation, adsorption, biological treatment and condensation. Likewise, catalytic oxidation can be applied as a technology for the abatement of such emissions. In this sense, Centi et al. [12] said that “the development of innovative environmental catalysis is also a crucial factor towards the objective of developing new sustainable industries” and they proposed the reduction of indoor pollution as a new field in catalysis.

Generally, two kinds of catalysts are used for the destructive oxidation of VOCs, noble metals (Pt, Pd) and metal oxides (chromium oxides, manganese oxides, perovskites, basic zeolites) [13–18].

Catalytic oxidation of an individual VOC has been widely studied; however, there are few articles about their mixtures. It has been reported that the oxidation of a VOC in a mixture differs from its single oxidation due to the interaction of the different species with the catalyst. Such interaction may be inhibitory character, since each VOC competes for the same active site [19, 20] or it may have promoting effects, possibly due to the exothermic nature of the full oxidation reaction [21, 22], which generates an temperature increase in the catalyst surfaces.

The objectives of this paper are: (i) to determine a quantitative and qualitative monitoring of indoor air in different small enterprises (ii) to analyze the catalytic oxidation of gaseous effluents and (iii) to adapt known technology, such as commercial exhaust fans, by developing Pt and/or Mn catalysts supported on ceramic monoliths in the vent of the duct system. The purpose of this development is first to remove the VOCs in the indoor air through the exhaust, and then treat them off-site prior to their elimination.

Experimental

Indoor sampling of VOCs

The analyses of indoor VOCs were fully described in Ref [2]. The sampling of VOCs was carried out using activated charcoal tubes (coconut charcoal), a 0.150 L min^{-1} vacuum pump flow and 3 M passive diffusion monitoring devices. VOCs were sampled in small enterprises, namely an electromechanical repair and car painting center (ERCPC, five samples), sewing workrooms (SWR, three samples), takeaway food shops (TAFS, three samples), chemical analysis laboratories (CAL, ten samples) and a photocopy center (PC, three samples), placing sampling devices near the exhaust fans. Sampling was conducted over a period of 30 days, during which adequate sensitivity and integrated sample of exposure concentration was obtained for people who work in those places. The selected facilities are establishments with less than six employees. Moreover, it is worth mentioning that among the VOCs found in workplaces, some of them are carcinogenic or potentially carcinogenic, so it is necessary to have ventilation and air exchange in the workplace.

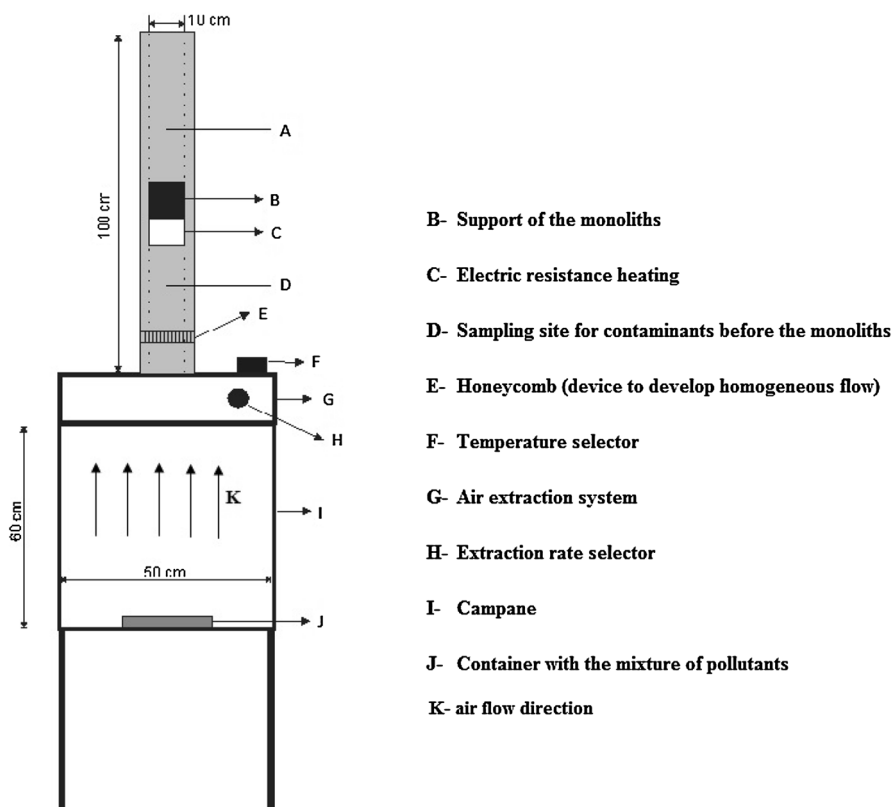


Fig. 1 Schematic of the device used for the study of the destruction of VOCs

Catalyst synthesis and characterization

Monoliths and supported catalyst preparation and characterization are explained in detail in a previous paper [23]. Briefly, ceramic monoliths made of natural clays and gibbsite (denoted B), were impregnated with platinum, manganese and platinum/manganese (named Pt/B, Mn/B and Pt/Mn/B, respectively).

Catalytic oxidation

As shown in Fig. 1, a commercial air purifier was adapted by placing a catalyst system heated by electrical resistances in an exhaust outlet duct. The circulation flow of gases entering the extraction system was calculated according to the American Society for Testing and Materials Standards (ASTM D3687-89; ASTM D3686-89; ASTM D3464-91). The catalytic system was composed of eight monoliths placed in an aluminum cylindrical device (Fig. 2) with a gas flow rate (Q) of $79,000 \text{ L h}^{-1}$.

From the samples for indoor air analyses in small enterprises (Table 1), a mix of commercial solvents that represents the main compounds found in the sites studied was selected. The VOC gas stream is generated from the evaporation of a liquid mixture of VOCs at constant temperature ($28 \pm 1^\circ \text{C}$), in order to reproduce the observed mean temperature inside the enterprises. The oxidant was the O_2 present in the air (which is extracted by the commercial air purifier).

The liquid mixture used to analyze the catalytic activity was composed of methyl ethyl ketone (MEK), chloroform (CHCl_3), toluene and (m + p + o)-xylene. The concentration in the liquid phase was 41.5, 26.0 and 32.5 % of H, Cl and C, in order, which represents a concentration of 38 mgH m^{-3} , 24 mgCl m^{-3} and 30 mgC m^{-3} in standard conditions for temperature and pressure (STP). The composition of the liquid mixture to evaporate and the composition of the gas mixture that enters the catalytic system are listed in Table 2. To analyze possible interference between different VOCs, the effect of individual components and binary mixtures of components was analyzed. For testing the catalytic activity of both individual

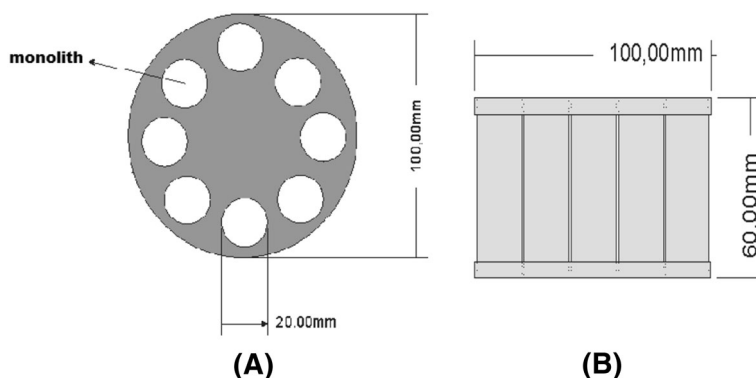


Fig. 2 Top (a) and side (b) view of the device used for support eight monoliths

Table 1 Levels of VOCs in the different small enterprises (in $\mu\text{g m}^{-3}$)

Compound	ERCPC			CAL			TAFS			PC			SWR		
	Median	Mean	Max	Median	Mean	Max	Median	Mean	Max	Median	Mean	Max	Median	Mean	Max
<i>n</i> -Hexane	50	172	546	6	17	208	ND	ND	ND	ND	ND	ND	4	20	55
Methyl ethyl ketone	24	155	609	1	1	1	ND	ND	ND	ND	ND	ND	ND	ND	ND
Chloroform	48	175	472	63	80	223	ND	ND	ND	ND	ND	ND	ND	ND	ND
Cyclohexane	ND	ND	ND	ND	ND	ND	2	2	3	ND	ND	ND	ND	ND	ND
Carbon tetrachloride	437	436	516	21	96	382	ND	ND	ND	ND	ND	ND	ND	ND	ND
Benzene	29	59	207	5	7	23	ND	ND	ND	ND	ND	ND	ND	ND	ND
Trichloroethylene	109	109	179	2	1	2	ND	ND	ND	ND	ND	ND	ND	ND	ND
Methylcyclohexane	71	44	182	1	2	14	1	1	2	1	1	2	ND	ND	ND
Methylisobutylketone	328	296	744	1	1	1	ND	ND	ND	ND	ND	ND	ND	ND	ND
Toluene	601	243	1,779	9	8	24	1	2	3	3	3	4	1	6	16
Ethylbenzene + <i>m</i> -Xylene	385	379	725	3	6	20	ND	ND	ND	ND	ND	ND	5	3	9
<i>p</i> -Xylene	836	722	1,680	2	12	124	1	1	ND	2	2	3	ND	ND	ND
<i>o</i> -Xylene	960	258	3,525	9	16	60	ND	ND	ND	ND	ND	ND	1	5	14
Styren	160	168	303	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
<i>n</i> -Decane	ND	ND	ND	ND	ND	ND	2	2	2	1	1	2	3	2	3
Limonene	ND	ND	ND	ND	ND	ND	2	2	2	ND	ND	ND	2	1	3
<i>n</i> -Dodecane	ND	ND	ND	ND	ND	ND	1	1	1	1	1	1	2	1	2

ND not detectable

pollutants and binary mixtures, the amount of each pollutant to be evaporated remained as in the original mixture. It should be noted that, in all the analyses, the initial concentration of Cl was greater than that found in the interior of the factories/inside the facilities in order to test whether the catalysts were able to remove Cl and meet the rules for emission limit values. The activity studies were performed with a water level in the gas stream between 14,900 and 16,000 ppmv (55–60 % relative humidity at room temperature) [24]. The reaction temperatures studied were 160, 230 and 295 °C. After each day of sampling, monoliths were treated in air at 425 °C for 90 min.

The concentration of VOC at the inlet and exit gas streams was determined by gas chromatography (GC), Agilent 6890 N GC equipped with a FID detector, with a ZB-624 column (30 m × 320 µm × 1.80 µm). Prior to GC analyses, pollutants were sampled using activated charcoal tubes at a rate of 0.150 L min⁻¹ for approximately 40–45 ms. Then, VOCs were desorbed using 1 mL of GC grade dichloromethane and were subjected to sonic action for 15 ms prior to the GC analysis. CO₂ was monitored with a Telaire IR CO₂ detector, placed at the exit of the outlet duct.

For the sampling of chlorine and HCl, the output gas stream was bubbled through an air sampling pump (0.700 L min⁻¹), containing 15 mL of 0.1 N NaOH solutions, simultaneously with the sampling of VOCs. Chlorine was analyzed by the *N,N*-diethyl-*p*-phenylenediamine colorimetric method [25] and HCl was determined by argentometric titration [26].

Results and discussions

Analysis of indoor VOCs

In the qualitative analysis of VOCs in the indoor air of electromechanical and textile plants, the presence of *n*-hexane, *n*-decane, MEK, CHCl₃, toluene, tri- and perchloroethylene, *o*-, *m*- and *p*-xylene, benzene and CCl₄ was mainly detected. Carbon tetrachloride, which appeared only in one sample, was associated with the

Table 2 Concentration of VOCs measured at the inlet and output of the catalytic system at 295 °C

Compound	Liquid composition (%)	Gas inlet (mg m ⁻³)	Mn/B outlet (mg m ⁻³)	Pt/B outlet (mg m ⁻³)	Pt/Mn/B outlet (mg m ⁻³)
MEK	13	6	1	1	1
CHCl ₃	43	143	19	6	12
Toluene	22	15	13	6	1
Xylenes	22	6	4	2	1
Sum of VOCs		166	37	14	14
C total		36	18	8	3
Cl total		126	17	6	11

use of a special solvent for painting a car, (Table 1). The composition of the gas stream in the electromechanical plant was $0.589 \text{ mgCl m}^{-3}$ and 2.09 mgC m^{-3} (STP); in the chemical analysis laboratory, $0.050 \text{ mgCl m}^{-3}$ and 0.046 mgC m^{-3} (STP); and in the garment sewing, tailoring and dyeing facility, 0.067 mgC m^{-3} (STP). Taking into account these results, MEK, CHCl_3 , toluene and xylenes were selected to prepare a synthetic mixture that was representative of VOCs found in indoor workplaces.

Catalytic activity

Catalytic oxidation of individual compounds

The catalytic activity of supported Pt, Mn and Pt/Mn ceramic monoliths towards the oxidation of each VOC is presented in Table 3.

According to the previous TPR results [23], the reduction of the Mn/B catalyst occurs at higher temperatures than that of Pt/Mn/B, and consequently the oxidation of VOCs on the Mn/B catalyst was more difficult than that on the Pt/Mn/B catalyst, in agreement with Sanz et al. [27], wherein the addition of platinum to manganese oxides markedly improves activity against toluene, due to the higher mobility of oxygens.

For the oxidation of MEK, xylenes and toluene, the activity decreases in the order

$$\text{Pt/Mn/B} > \text{Pt/B} > \text{Mn/B}$$

It may be assumed that the higher activity of the Pt/Mn catalysts is due to some favorable synergetic effects between Pt and Mn [23]. On the other hand, regardless of the catalysts, the order of VOC destructibility among MEK, xylenes and toluene in general was:

$$\text{MEK} > \text{Xylenes} > \text{Toluene}$$

Nevertheless, the conditions required to achieve a specified level of destruction depend on the catalyst composition. Pt/B is slightly more active than Mn/B in the

Table 3 Catalytic oxidation of individual compounds

Compound	Mn/B			Pt/B			Pt/Mn/B		
	Conversion (%)			Conversion (%)			Conversion (%)		
	160 °C	230 °C	295 °C	160 °C	230 °C	295 °C	160 °C	230 °C	295 °C
CHCl_3	13	31	82	11	36	88	17	41	75
MEK	18	28	85	28	42	95	66	88	98
Toluene	1	1	2	11	27	55	66	74	79
Xylenes	10	17	23	14	26	64	81	86	90

$$\text{Conversion (\%)} = \{1 - [(\mu\text{g m}^{-3})_{\text{OUT}}/(\mu\text{g m}^{-3})_{\text{IN}}] \times 100$$

oxidation of chloroform and MEK, but it is much more active in the oxidation of the aromatic compounds.

Considering the dipole moments (μ) of MEK (2.08 D), xylene (1.3 D) and toluene (0.4 D), the conversion may be associated with the polarity of the reactant molecule. However, the size of the adsorbed molecule on the surface catalyst could also be evaluated according to the works of Kim et al. [28, 29].

Several authors have reported that the order of destructibility of VOCs is related to the strength of the weakest C–H bond in the substrate [30, 31]. The energy to break the weakest C–H bond in toluene (360 kJ mol^{-1}) is lower than that in MEK (386 kJ mol^{-1}). MEK is more easily oxidized than toluene, indicating that factors other than the breakage of the weakest C–H bond are involved in the oxidation of VOCs. Adsorption may also become important in determining VOC reactivity. The higher conversion of MEK compared to toluene can be explained by the different reactivity of the two reactants with the lattice oxygen of the same catalysts; Peluso et al. [32] demonstrated that manganese oxide catalysts present two different sites of adsorption against oxygenated organic compounds, enhancing their reactivity.

The conversion of xylenes is slightly higher than that of toluene over Pt/Mn/B and Pt/B monoliths, maybe due to the increase in the number of methyl groups [33] and to the different adsorption of the aromatic compounds [34]. On the Mn/B monoliths, toluene conversion is negligible and xylene conversion reaches only 20 % at 295 °C.

On the other hand, no partial oxidation products were detected in the aromatic VOCs examined. Only traces of acetaldehyde were detected in MEK oxidation at low levels of conversion.

Chloroform oxidation gives completely different results. In this case, the incorporation of Pt to Mn/B does not improve the catalytic performance.

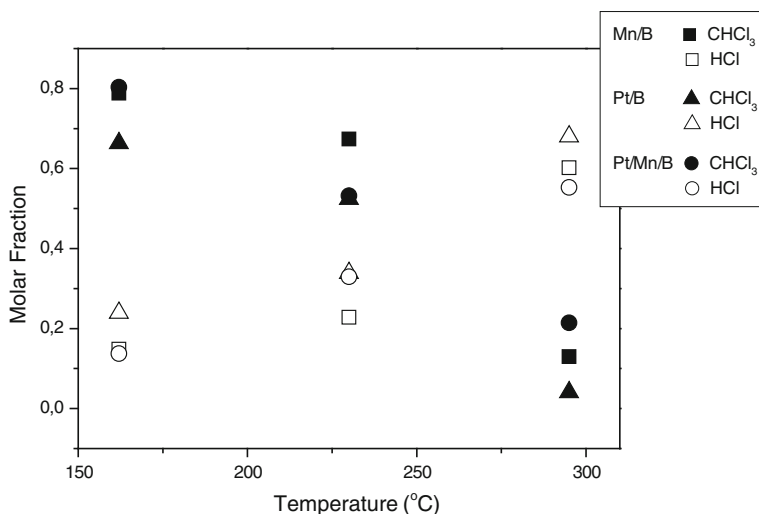


Fig. 3 Molar fraction of CHCl_3 and HCl at 160, 230 and 295 °C, formed over Mn/B, Pt/B and Pt/Mn/B catalysts

The reaction products detected in the combustion of chloroform were HCl, CO₂ and H₂O.

Fig. 3 shows the molar fraction of CHCl₃ and HCl formed on catalysts at 160, 230 and 295 °C. The presence of HCl observed in this study agrees with what several authors have reported [35, 36] who pointed out that the formation of Cl₂ via the oxidation of halogenated organic molecules can be minimized in favor of HCl by adding water vapor to the feed stream.

Catalytic oxidation of binary mixtures of VOCs

The activity of the catalysts was also investigated for all the possible combinations of binary mixtures of the VOCs under study (considering the mixtures of xylene isomers as one component). Results are summarized in Table 4 for binary mixtures of chloroform-MEK; chloroform-toluene; chloroform-xylenes; MEK-toluene; MEK-xylenes; and toluene-xylenes.

For the Mn/B monoliths, the conversion of every VOC in the different binary mixtures does not differ from its single oxidation, having almost constant conversion values at the different temperatures studied.

In the case of Pt/B monoliths, the results showed that the conversion of VOCs in the binary mixture is similar to that of every compound individually, except in the MEK-chloroform mixture. There is a slight decrease in MEK conversion in the mixture with chloroform. On the other hand, chloroform conversion is slightly enhanced in the mixture with MEK.

Pt/Mn/B monoliths present a different behavior in binary mixtures. The conversion of MEK, toluene and xylenes was not affected by the presence of

Table 4 Catalytic oxidation of binary mixtures of VOCs

	Mn/B			Pt/B			Pt/Mn/B		
	Conversion (%)			Conversion (%)			Conversion (%)		
	160 °C	230 °C	295 °C	160 °C	230 °C	295 °C	160 °C	230 °C	295 °C
CHCl ₃	18	24	83	23	40	90	27	38	56
MEK	12	31	81	10	33	87	85	92	94
CHCl ₃	18	26	85	26	41	96	20	47	61
Toluene	1	1	2	10	27	54	60	72	81
CHCl ₃	18	24	82	21	37	90	30	37	59
Xylenes	10	16	22	14	25	63	84	86	85
Toluene	1	1	2	12	30	55	55	59	80
MEK	13	32	82	12	35	90	35	64	75
MEK	12	32	82	12	34	89	41	66	80
Xylenes	10	15	20	11	27	66	64	75	86
Toluene	1	1	2	12	28	56	52	69	87
Xylenes	10	17	24	17	28	69	48	62	84

$$\% C = \% \text{ conversion} = \{1 - [(\mu\text{g m}^{-3}) \text{ OUT}/(\mu\text{g m}^{-3}) \text{ IN}] \times 100$$

chloroform. In contrast, chloroform conversion decreased at 295 °C in the mixture with MEK and aromatic compounds (toluene and xylenes). The Mn^{n+} sites where chloroform adsorbs are blocked by MEK perhaps by a metal-O interaction. Aromatics compounds also block the chloroform adsorption sites, maybe by a steric effect (parallel adsorption).

In the mixture MEK-toluene, there was a mutual inhibition at 160 and 230 °C for the monolith Pt/Mn/B. The mixture MEK-xylenes behaved similarly to the MEK-toluene mixture.

In the mixture of aromatic VOCs, no significant changes in the conversion of both molecules, toluene and xylene, are observed at the maximum temperature studied (295 °C). However, other authors like Lee [34] indicates that the presence of methyl groups on the aromatic ring would favor the oxidation due to the stability of the π -complex formed.

Catalytic oxidation of a mixture of several VOCs

The performances of the catalysts in the destruction of a mixture of chloroform, MEK, toluene and xylenes at 160, 230 and 295 °C are shown in Fig. 4. As can be seen, for Mn/B and Pt/B catalysts, VOCs in general keep their conversions

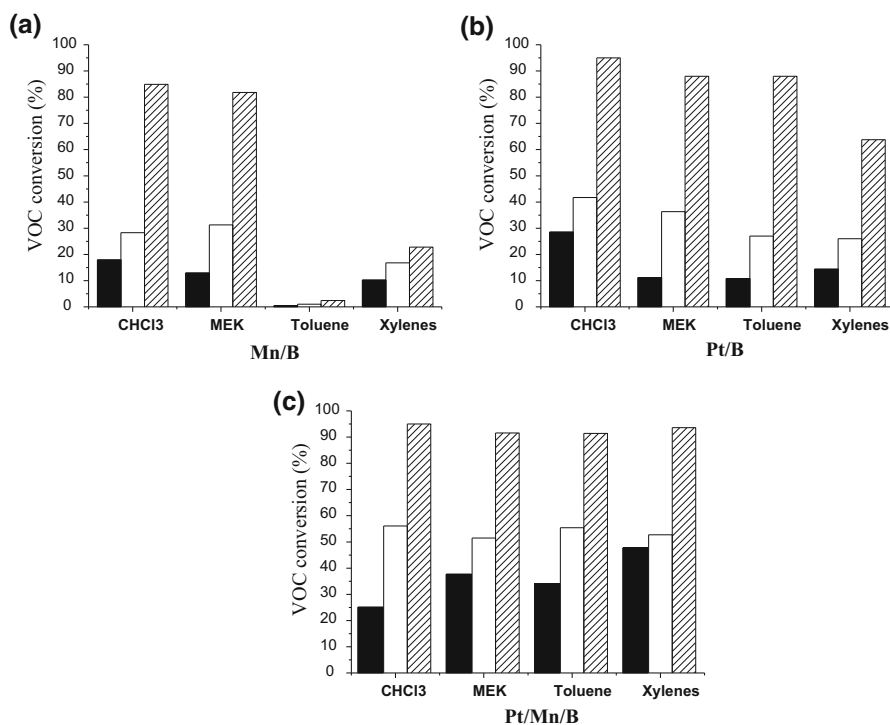


Fig. 4 Catalytic oxidation of a mixture of VOCs at 160 , 230 and 295 °C over: (a) Mn/B; (b) Pt/B and (c) Pt/Mn/B

regardless of whether they are in a mixture or pure state. There is an increment in the activity of MEK and CHCl_3 at low temperatures.

In the case of Pt/Mn/B, chloroform conversion is higher in the mixture than in the pure state. This can be explained by a local temperature rise on the surface of the catalyst, caused by the exothermic character of the oxidation of the rest of VOCs present in the mixture. At 160 °C, the conversion of MEK, toluene and xylenes in the mixture was lower than VOC conversion alone. At 295 °C, the conversion of MEK, toluene and xylenes was in general similar to or higher than that of the VOCs on their own, reaching conversion levels higher than 90 % for all the four VOCs studied.

The incorporation of platinum over Mn/B monoliths increases the VOCs conversion in the mixture of all of them. The increment could be associated on the one hand to a local heating effect. On the other hand, as we showed in a previous paper [23], there is a synergetic effect between manganese and platinum, along with an increment in the surface acidity of the catalyst.

The concentration of each individual compound and the total VOC concentration (the sum of the concentration of each individually VOC) expressed in mg VOC m^{-3} (STP), at the output of the catalytic system at 295 °C is listed in Table 2, for the three catalysts studied. The VOC emission values for Pt/Mn/B and Pt/B are below the EPA VOCs emission limit values in waste gases [37], which for activities such as heatset web offset printing, surface cleaning and manufacturing of pharmaceutical products are 20 mg C m^{-3} (STP).

According to these results, from the point of view of emissions it can be seen that the solid Pt/Mn/B is a promising catalyst for this kind of solvent mixtures. The system studied, which consisted of an exhaust air-catalyst system, has demonstrated a potential use as a test for small businesses/enterprises that have air purification systems of this type, thus avoiding emissions of VOCs to the environment. This type of catalytic system can be used to reduce organic pollutants in indoor air, before they are released to the atmosphere.

Conclusions

At present, the effect of volatile organic compounds on air contamination and on human health is recognized. Indoor ambient monitoring revealed that the high levels of VOCs are associated with places where solvents are frequently used, either for paint dilution or cleaning mechanical parts (electromechanical repair and car painting center), or as solvents of the ink and/or toner used in printing (photocopy centers), and also in chemical process (chloroform for laboratories).

These levels of VOCs found in the different indoors environments show the importance of the use of abatement technologies for the complete reduction of the levels of pollution to mitigate their impact on worker's health and their venting to the atmosphere.

From simple modifications of a commercial air pump, placing the vent stack of the catalytic device, the destruction of a solvent mixture consisting of MEK, chloroform, toluene and xylenes was analyzed.

The activity order in the combustion of MEK, toluene and xylenes decreases in the order:

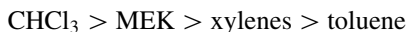
$$\text{Pt/Mn/B} > \text{Pt/B} > \text{Mn/B}$$

In contrast, in the combustion of chloroform, the activity decreases in the order

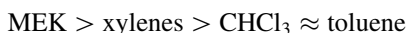
$$\text{Pt/B} > \text{Mn/B} \approx \text{Pt/Mn/B}$$

Nevertheless, all the catalysts are able to reduce emissions of chlorine into the atmosphere.

For Pt/B and Mn/B catalysts, the order of destructibility of VOCs, alone or in the mixtures of them, was:



Whereas in the Pt/Mn/B monolith the order was:



The great activity of the Pt/Mn/B monoliths could be due to a favorable synergetic effect between platinum and manganese.

The catalytic system developed in this work is a promising technology for reducing indoor pollution in small enterprises.

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