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Evaluation of regeneration of spent three-way catalysts for catalytic oxidation of aromatic hydrocarbons

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ABSTRACT

The washing effects of using different acid solutions (0.1 N; CH_3COOH, $C_2H_2O_4$, and $C_6H_8O_7$) on the activity of spent three way catalysts (TWCs) in oxidizing volatile organic compounds (VOCs) were investigated to recycle the spent catalyst. The spent TWCs and acid solution treated TWCs are characterized by using X-ray diffraction (XRD), N_2 adsorption, transmission electron microscope (TEM) and an inductively coupled plasma (ICP) analysis. The spent TWCs are found to show a great potential for removal of organic compounds. Among the regeneration methods applied, the oxalic acid as a washing chemical agent proved to be quite effective in enhancing the catalytic activity of the spent TWCs compared to other acids (citric acid and acetic acid). Furthermore, the catalytic activities are clearly explained in terms of the remaining Pt/Al ratio of the TWCs, the ionization potential and dipolar moments of the organic compounds.

1. Introduction

Three way catalysts (TWCs) have been widely employed to remove a variety of toxic pollutants such as unburned hydrocarbons (HC), carbon monoxide (CO) and nitrogen oxides (NO_x). The TWCs typically consist of (1) cordierite or stainless steel based monolithic substrate having a honeycomb structure and (2) a high surface area carrier media (i.e., Al_2O_3) which is coated on the monolith. The wash-coat material is also composed of several components: (1) precious metals (Pt, Pd and Rh) which act as the active sites and (2) metal oxides (La, Ba, Si, Zr and Ce) which are mainly used as a stabilizer [1–3].

On the other hand, TWCs have been replaced by law with fresh catalyst when the catalytic performance of the TWCs is severely deteriorated below the emission limits. In general, the catalytic activity of TWCs is gradually deactivated during the real operation. This deactivation process is closely connected with the physico-chemical causes which can be classified into three types: chemical, thermal and mechanical causes [4–7]. In addition the TWCs have been also scrapped from the cars that have been totaled in accidents is posing a serious environmental hazard. Moreover, the quantities of spent TWCs had been expected to continuously increase because of the growing number of cars registered [8]. Thus, finding a proper solution for recovery, recycling and regeneration of TWCs is very valuable and meaningful task from both economical and environmental points of view.

The regeneration (or recycling) of the spent TWCs has been a major issue because it offers economical and environmental gain. A number of studies have focused on the recovery of the platinum group metals (PGMs) from the spent TWCs [8,9]. Although the metal recovery is an attractive procedure for cost effectiveness, this approach sometimes leads to environmental problems that surpass the economic gain. On the other hand, much attention has been paid to the regeneration (or reactivation) of the severely aged TWCs [10–16]. These methods can be largely classified into two main groups: (1) thermal gases (oxygen, hydrogen and chlorine containing gas) and (2) acids (chlorine coating reagents, $C_{10}H_{16}N_2O_8$, H_2SO_4, HNO_3, $C_2H_2O_4$, H_3PO_4, and $C_6H_8O_7$). However, this approach is limited to reapplication of the original process by regaining (or recovering) the lost activity (i.e., oxygen storage capacity and HC, CO and NO_x conversion) of the TWCs.

Recently, our group has demonstrated that the spent TWCs can be recycled or reutilized as a promising catalyst for removing volatile organic compounds (VOCs) [17] since spent TWCs contain several valuable elements such as Pt and Pd that are very efficient materials for treating toxic compounds. Thermal gases (air and hydrogen) and acids are successfully applied for regenerating the spent catalysts. Although the results of our previous work provide an insight into its alternative application, the possibility of spent TWC as a catalyst for VOCs abatement has not been sufficiently investigated. Thus, the purpose of this paper is to evaluate spent TWCs as potential catalysts for VOCs conversion and examine

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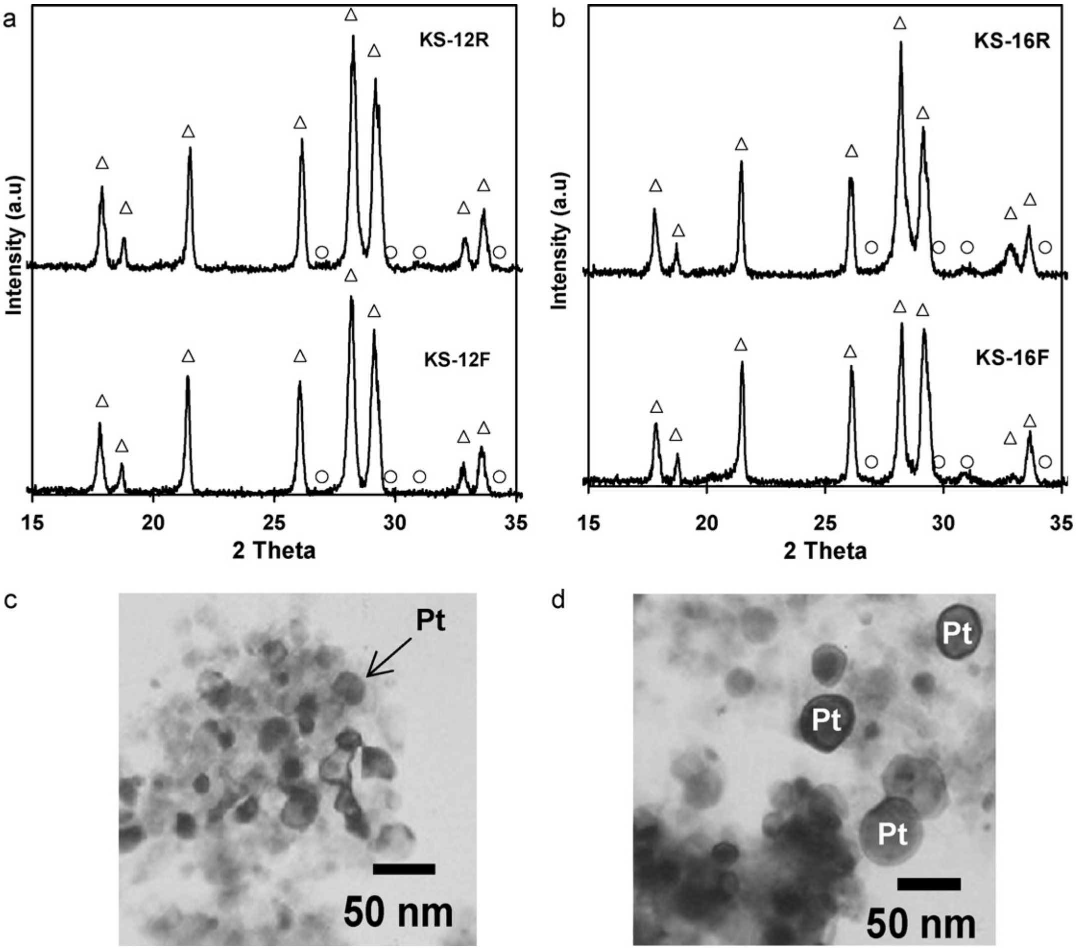

Fig. 1. XRD patterns of KS-12 (a) and KS-16 (b): $2MgO{\cdot}2Al_2O_3{\cdot}5SiO_2$ (△) and $CePO_4$ (○); TEM photographs of KS-12 (c) and KS-16 (d).

the effects of acid solution treatment on the catalytic behavior. Two spent TWCs were used as the model catalysts. They were obtained from the same model (Sepia, KIA) of gasoline driven automobile but with different mileages (120,000 km and 160,000 km). In particular, three organic acid solutions (CH_3COOH, $C_2H_2O_4$, and $C_6H_8O_7$) with different acidity were applied to removing the contaminants accumulated on the catalyst and reactivating of the spent TWCs. To characterize the spent TWCs and its acid solution treated TWCs, X-ray diffraction (XRD), Brunauer–Emmett–Teller (BET) surface area, transmission electron microscope (TEM) and inductively coupled plasma (ICP) analysis were employed. The catalytic performance of the spent TWCs and the regenerated (or reactivated) TWCs were examined in the low temperature catalytic oxidation of benzene, toluene, and xylene (BTX) to explore the possibilities of removing VOCs.

2. Experimental

2.1. Material and characterization techniques

The aged commercial catalytic converters were extracted from two automobiles (Sepia, KIA) which used approximately 120,000 km (KS-12) and 160,000 km (KS-16) mileage in South Korea. To investigate the degree of deactivation and apply the chemical regeneration procedures, the aged TWCs were cut into two sections. The small fragments having a cylindrical shape (diameter $d = 10$ mm and length $L = 30$ mm) were named as KS-12 (or 16) F (front) and KS-12 (or 16) R (rear), respectively.

In this study, three different acid solutions of CH_3COOH, $C_2H_2O_4$ and $C_6H_8O_7$ with 0.1 N were used to regenerate the aged TWCs. Inductively coupled plasma (ICP, PerkinElmer OPTIMA 4300DV) was employed to measure the concentrations of TWCs basic components and contaminants over the aged TWCs and its acid solution treated samples. The textural structure of the spent TWCs and its regenerated TWCs was investigated by nitrogen adsorption/desorption isotherms at 77 K using a Micromeritics ASAP 2020 apparatus. X-ray diffraction (XRD, Phillips PW3123 diffractometer) pattern of the samples was also obtained from 15° to 35°. The morphology and the particle size of the catalysts were examined using transmission electron microscopy (TEM, JEOL JEM-2000FAXII).

2.2. Catalytic oxidation of hydrocarbons

The catalytic activity measurements were carried out in a conventional fixed bed flow reactor at atmospheric pressure. About 1.0 g of catalyst sample was loaded in the middle of the reactor with quartz support and total flow rate of the gas mixture through the reactor was $100\ cm^3\ min^{-1}$(=equivalent space velocity of $8000\ h^{-1}$). Benzene, toluene and xylene (BTX) were purchased from Fisher chemical and used without any further treatment. The concentration of each VOCs was fixed as 1000 ppm, which was controlled by the temperature of the saturator as well as the additional air stream. For ensuring no adsorption, condensation and reaction of reactant and product in the tubes, all the lines were sufficiently preheated to a temperature of 120 °C. A gas chromatograph (GC, Shimadzu GC-14A) equipped with a thermal conductivity detector with a porapak Q column (50–80 mesh, $3\ mm\ \Phi \times 3\ m$) and

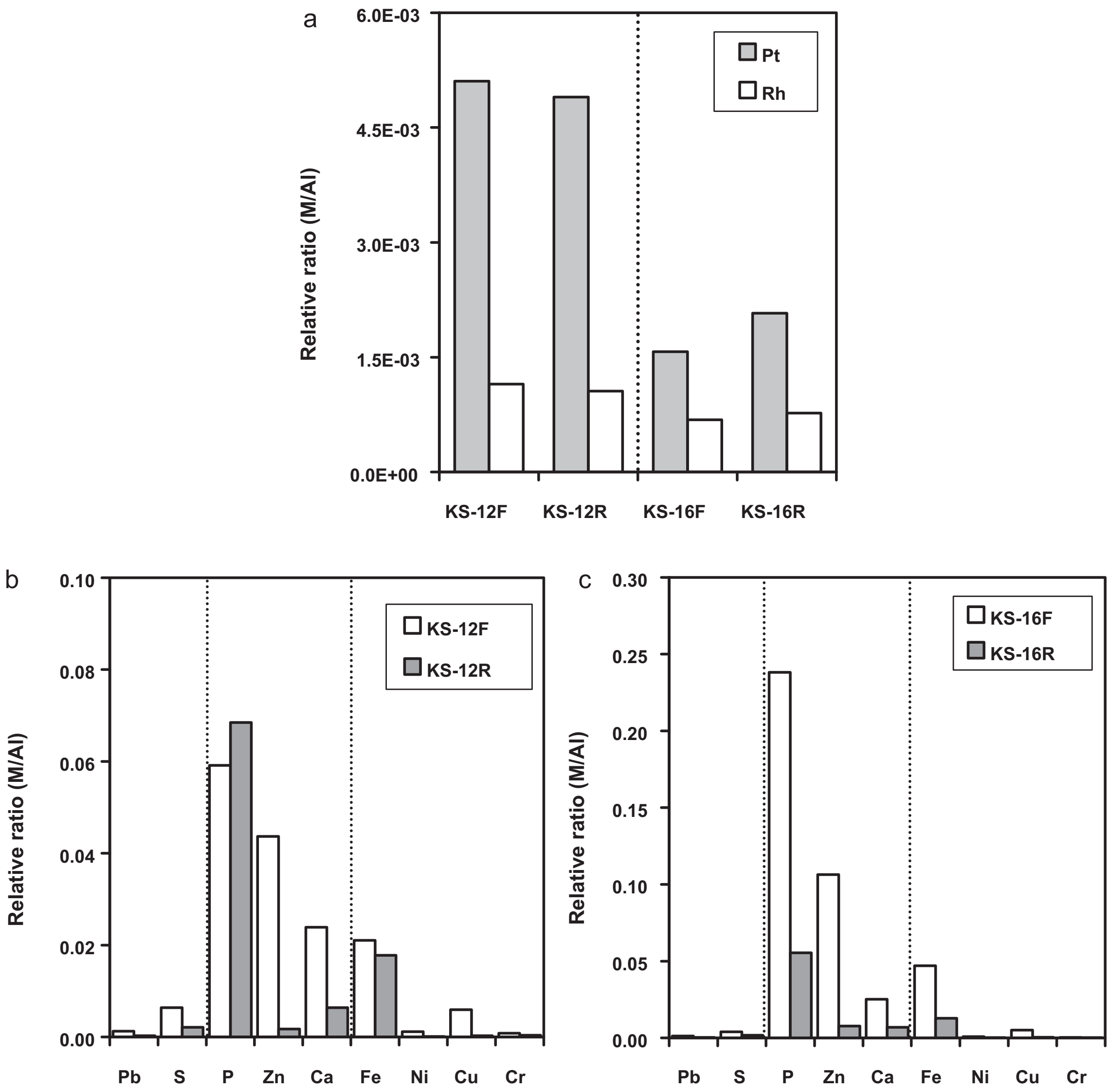

Fig. 2. Mass atomic ratios of basic (M = Pt and Rh) (a) and contaminants (M = Pb, S, P, Zn, Ca, Fe, Ni, Cu and Cr) (b and c).

a chromatographic column composed of a 5% bentone-34 and 5%DNP/simalite (60–80 mesh, 3 mm $\Phi \times$ 3 m) was used to analyze the inlet and exit gas streams of the VOCs and CO_2. A blank test, conducted in absence of catalyst, showed completely negligible (≈0%) conversion below 600 °C. In addition, CO_2 and H_2O were the only products detected under experimental conditions. Thus, the conversion (X) of VOCs (BTX) was calculated using the following equation:

$$\text{Conversion}\,(X) = \frac{\text{moles of product}}{\text{moles of feed}} \times 100(\%) \qquad (1)$$

3. Results and discussion

Fig. 1(a) and (b) presents the X-ray diffraction results in 2 theta ranges of 15–35° for KS-12 and KS-16, respectively. These figures largely display the intense reflections of the cordierite ($2MgO{\cdot}2Al_2O_3{\cdot}5SiO_2$, △) that is the main material of the monolith. In addition, the diffraction peaks at 26.96, 29.80, 31.90 and 34.30 labeled as (○) represent the Ce(III) orthophosphate ($CePO_4$), which is mainly formed under real driving conditions and closely connected with the chemical deactivation of TWCs through the loss of the oxygen storage capacity. However, platinum group metals (PGMs) were not detected in the spent TWCs employed, which may be due to the fact that the loading amount of PGMs was low and also dispersed relatively uniformly over the ceramic monolith.

Fig. 1(c) and (d) shows the representative TEM images of the KS-12F and the KS-16F. These images clearly revealed that the morphology of the spent TWCs has various appearances: round, oval, and rugged-shape particles ranging (from 5 to 40 for KS-12F and 7 to 55 for KS-16F) nm in size. On the whole, the particle size was enlarged as the catalyst mileage increased. These images also showed the agglomerated areas of noble metals (i.e., Pt and Rh) and wash-coat particles (i.e., Mg, Al, Ce and Zr).

Table 1
BET surface area, average pore width and T_{50} of K-S-12 and K-S-16.

Catalyst	BET surface area, S_{BET}[a] (m^2/g)	Average pore width, D_{APW}[b] (Å)	T_{50} (°C)
K-S-12-F	6.2	103.6	186
K-S-12-R	8.4	121.1	200
K-S-16-F	5.7	102.1	228
K-S-16-R	8.1	116.4	204

[a] S_{BET}, $P/P_0 = 0.05–0.30$.
[b] $D_{APW} = 4V_t/S_{BET}$, V_t: total pore volume.

To quantitatively examine the degree of contamination and active metals found in the spent TWCs, an elemental analysis (ICP) was carried out. As discussed in reporting the XRD measurement, Al, Mg and Si are the main components of cordierite. In addition, platinum group metals and Al, Ce and Zr are constituent elements of the wash-coat. Previous works have shown that Al concentration did not severely change with mileage [15–18]. Thus, in this work, Al concentration was selected for evaluating the related information. Fig. 2 shows the relative concentration ratios of the basic noble metals and the contaminants found in the spent TWCs. As shown in Fig. 2(a), the KS-12 and KS-16 only consist of Pt and Rh, which are extensively used for oxidation of hydrocarbon and carbon monoxide and the reduction of nitrogen monoxide to nitrogen [19–21]. However, no distinctive evidence of Pd was found in the spent TWCs employed. On the whole, the relative Pt/Al and Rh/Al ratios decreased with the mileage, but there is no clear correlation between the relative ratio and the position extracted. Contrary to our expectation, in the case of KS-12 the front part (KS-12F) has relatively higher Pt and Rh ratios than that of the rear part (KS-12R). However, a reverse tendency was observed for the case of KS-16. On the other hand, as seen in Fig. 2(b) and (c), the general order of the contaminants deposited on the spent TWCs was P > Zn > Ca > Fe > Cu > S, which agrees well with the previous reports [5]. In addition, among the various contaminants examined, gasoline and lubricant oil additives (P, Zn and Ca) and metallic compounds (Fe and Cu) were the main components deposited on the KS-12 and the KS-16, whereas fuel additives (Pb and S) constituted minor contaminants. It is also evident that the remaining concentration ratio of TWCs contaminants greatly increased with mileage. However, the ratio determined was not well correlated with the extracted locations of the TWCs.

To examine the difference in textural properties for the spent TWCs between front part and rear part, nitrogen adsorption measurements were carried out. Table 1 lists the specific surface area (S_{BET}) and average pore width (D_{APW}) of KS-12 and KS-16 catalysts. The S_{BET} and D_{APW} of the rear parts (KS-12-R and KS-16-R) are considerably larger (or greater) than that of the front parts (KS-12-F and KS-16-F). This difference can be explained by the uneven distribution (or deposition) of TWC contaminants. In addition, the S_{BET} and D_{APW} slightly decreased with mileage, but a small difference between mileages is observed.

The activity of the spent TWCs for VOC oxidation was measured in a gas mixture of toluene and oxygen. In this work, the light-off curves were used to evaluate and compare the activity of the spent TWCs employed. Fig. 3(a) and (b) shows the light-off curves for toluene oxidation over the spent KS-12 and the spent KS-16 as a function of reaction temperature. As shown in these figures, toluene was completely oxidized over all spent TWCs studied below 250 °C. Thus, we confirmed that the spent TWCs could be reused without any problems as a potential catalyst for the removal of toxic compounds. According to these figures, the catalytic activity curve for the KS-12 is slightly steeper than that of the KS-16. In addition, it is evident that the KS-12 is more active than the KS-16. For example, the KS-12 showed the complete toluene conversion to carbon dioxide in the 210–230 °C range, whereas KS-16 reached the same conversion in the 220–250 °C range. Table 1 also presents T_{50}, which is the temperature that approached 50% conversion, to compare the catalytic activity of the spent TWCs. The catalytic activity of the spent TWCs, which was employed based on T_{50}, decreased in the order of KS-12-F (186 °C) > KS-12-R (200 °C) > KS-16-R (204 °C) > KS-16-F (228 °C). This is the same order of the relative precious metal (Pt and Rh)/Al ratios as detected in the spent TWCs. Thus, it is reasonable to suggest that the catalytic activity of the spent TWCs for VOCs oxidation is greatly dependent on the remaining precious metal ratio rather than the contaminant ratio deposited on the spent TWCs and the textural properties (i.e., S_{BET} and D_{APW}) of the catalyst.

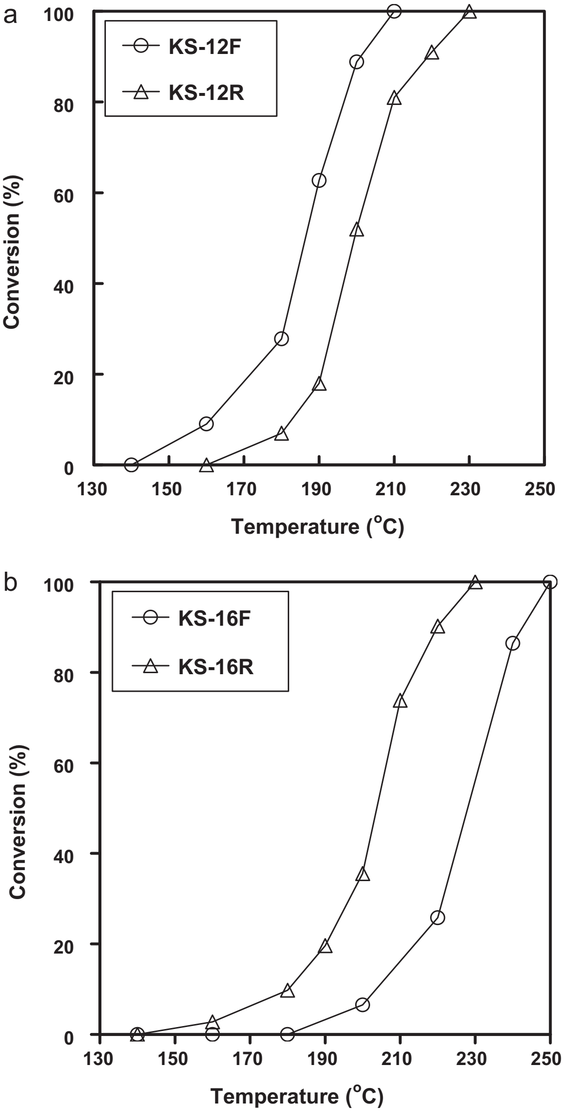

Fig. 3. Conversion profiles of toluene oxidation over KS-12 (a) and KS-16 (b) (reaction condition: catalyst weight = 1.0 g; toluene concentration = 1000 ppm in air; GHSV = 8000 h^{-1}).

On the basis of the findings reported above, the KS-12F and KS-16F were chosen to further investigate the effects of acid aqueous pretreatment on catalytic performance. The effects of acid aqueous pretreatment on the textural properties of the spent TWCs were evaluated by using nitrogen sorption measurement, and they are listed in Table 2. The results indicate that the acid aqueous pretreated TWCs have a relatively higher S_{BET} and wider D_{APW} than those of the spent TWCs (KS-12-F and KS-16-F). These results are

Table 2
Textural properties of oxalic, citric and acetic acid solution treated KS-12F and oxalic, citric and acetic acid solution treated KS-16F.

Pretreatment	pK_a or pK_{a1}	BET surface area, S_{BET} (m^2/g)		Average pore width, D_{APW} (Å)	
		KS-12F	KS-16F	KS-12F	KS-16F
$C_2H_2O_4$	1.27	16.1	13.2	114.5	118.7
$C_6H_8O_7$	3.15	7.7	7.1	106.8	106.7
CH_3COOH	4.76	6.4	7.2	107.1	105.3

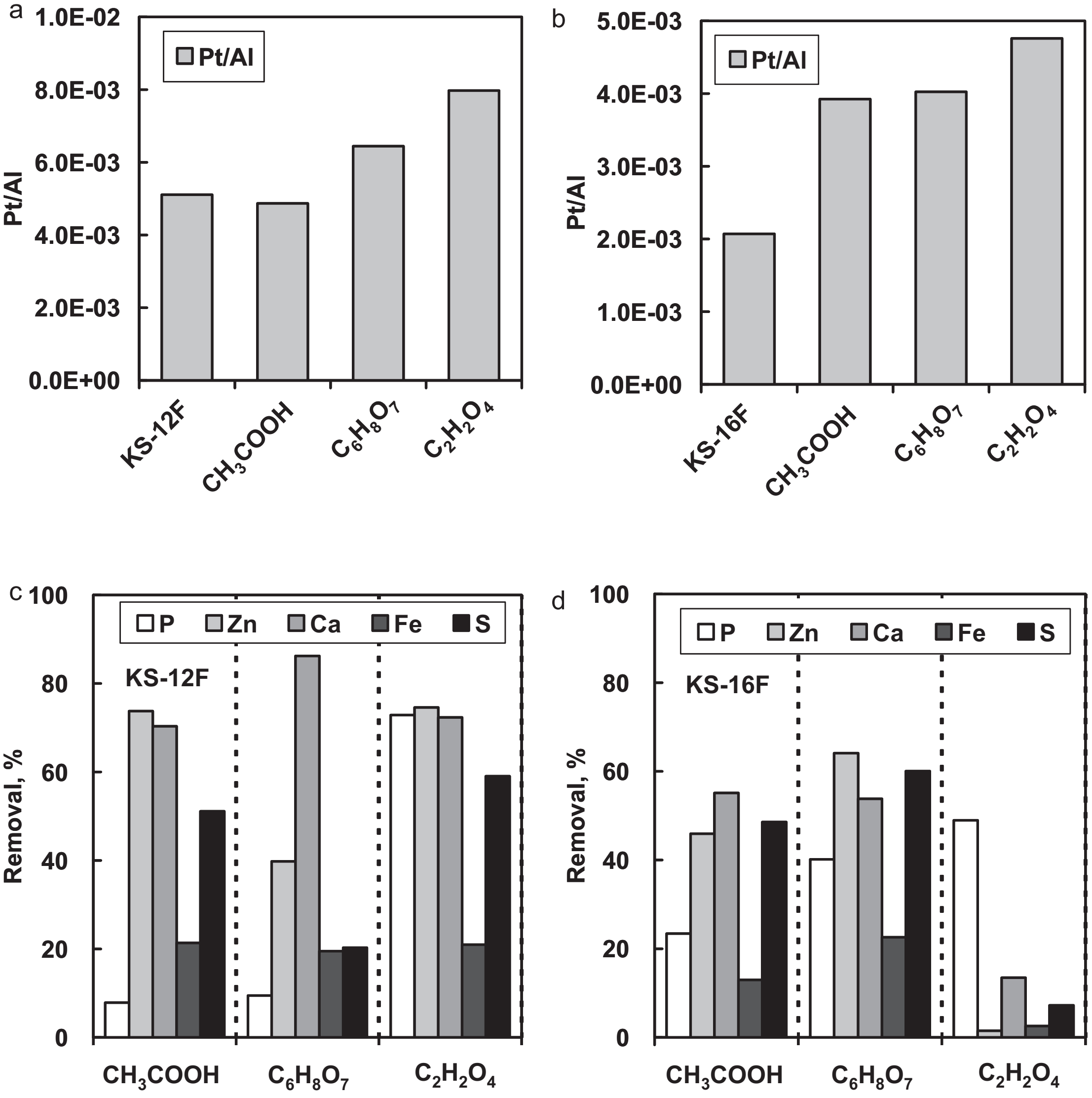

Fig. 4. Pt/Al ratio of acid solution treated TWCs ((a) KS-12F and (b) KS-16F); percent removal of P, Zn, Ca, Fe, Cu, Pb and S obtained by using acid solution treatment procedures over KS-12F (c) and KS-16F (d).

closely related to the removal of physic–chemical contaminants accumulated both on the surface and the inside of the pore networks of the spent TWCs. In this work, the oxalic acid solution was found to be more efficient than other organic acids (citric and acetic) in treating the spent TWCs. It is also interesting to note that the textural properties of the acid solution treated TWCs are inversely dependent on the acidity of the acid. In other words, the lower the pK_a (or pK_{a1}), the larger and wider the S_{BET} and the D_{APW}.

Fig. 4(a) and (b) shows the remaining Pt/Al ratio determined after using three different acid solution treatments for the regeneration of the KS-12-F and the KS-16-F. We observe that the Pt/Al ratio has greatly increased after acid solution pretreatment except for the acetic acid solution treated KS-12F which has a slightly lower ratio than the spent TWCs. Moreover, the pretreatment with oxalic acid aqueous led to the highest increase of the remaining Pt/Al ratio compared to other pretreatment methods as shown in Fig. 4(a) and (b). The Pt/Al ratios determined follow the order: (1) KS-12F: $C_2H_2O_4 > C_6H_8O_7 >$ parent > CH_3COOH and (2) KS-16F: $C_2H_2O_4 > C_6H_8O_7 > CH_3COOH >$ parent. This trend is also consistent with that of the textural properties of the acid solution treated TWCs.

To examine the correlation between the catalytic activity and the removal of contaminants, the TWCs pretreated with acetic, citric and oxalic acid solution were analyzed for four different contaminants (e.g., P, Zn, Ca, Fe and S) which are the highest components accumulated on the spent TWCs. Fig. 4(c) and (d) compares the percent (%) removal of those contaminants. As shown in these figures, the percentage removal of phosphorous is $C_2H_2O_4 > C_6H_8O_7 > CH_3COOH$. This trend well matches the previous reports [17], which is closely connected with the order of acidity. However, there is no clear correlation between the percentage removal of contaminants and the acidity (or pK_a and pK_{a1}) of acids, although those acids applied are to some extent useful for removing the contaminants.

Fig. 5(a) and (b) shows the effect of the acid solution treatment on the toluene removal activity. For comparison, data on the spent KS-12F and the spent KS-16F are also included. The catalytic activity of the catalyst was greatly dependent on acid solution treatment conditions. On the whole, a clear improvement was observed in

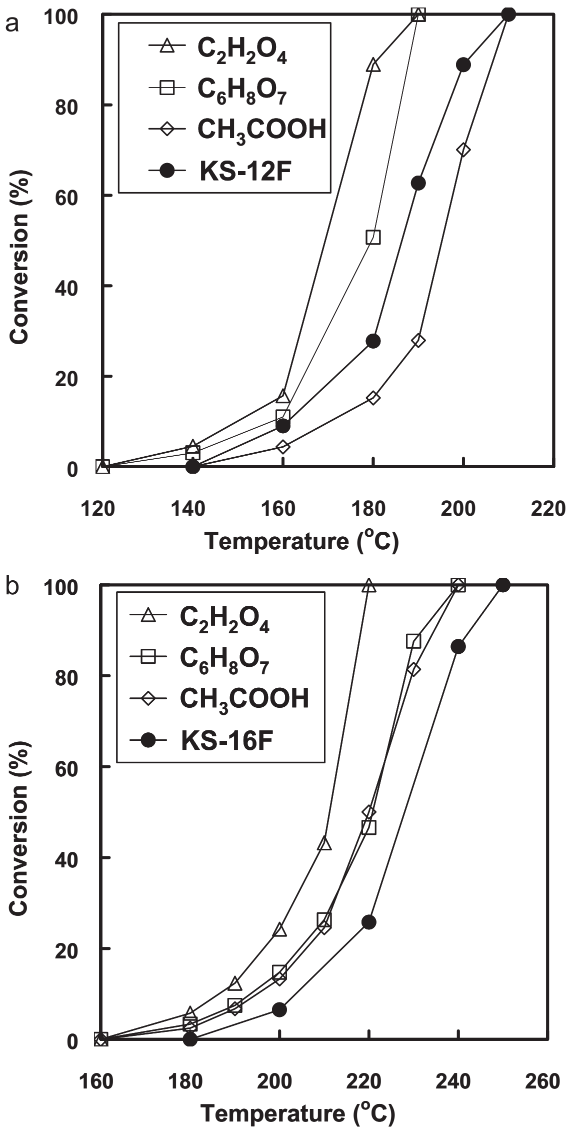

Fig. 5. Conversion profiles of toluene oxidation over different acid solution treated KS-12F (a) and different acid solution treated KS-16F (b) (reaction condition: catalyst weight = 1.0 g; toluene concentration = 1000 ppm in air; GHSV = 8000 h^{-1}).

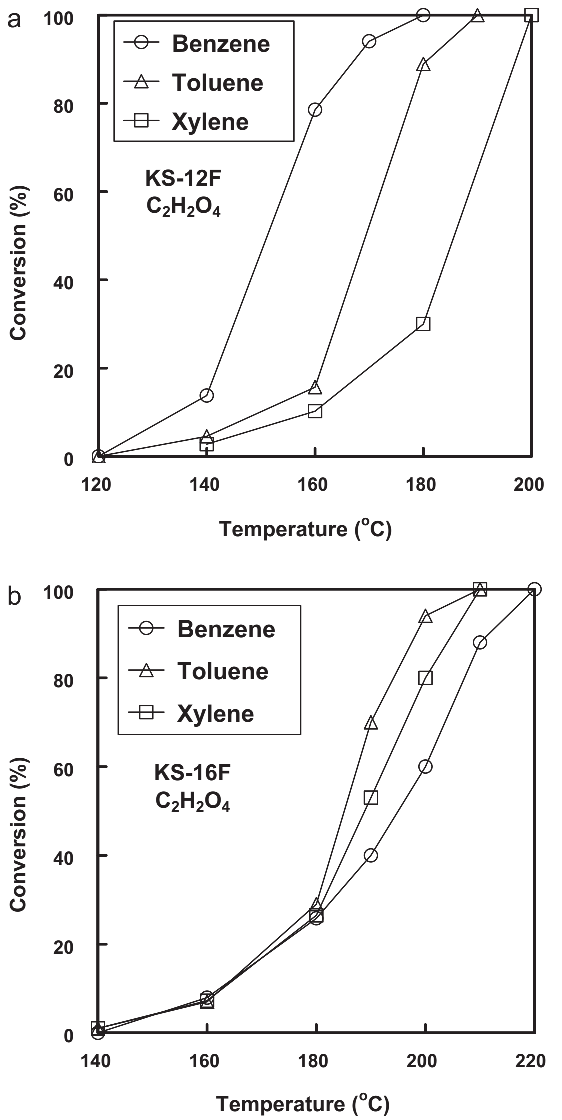

Fig. 6. Conversion profiles of BTX oxidation over oxalic acid solution treated KS-12F (a) and oxalic acid solution treated KS-16F (b) (reaction condition: catalyst weight = 1.0 g; BTX concentration = 1000 ppm in air; GHSV = 8000 h^{-1}).

toluene oxidation after the acid solution treatment, except for the acetic acid solution treatment (KS-12F). For example, toluene conversions of the acid solution treated TWCs are greatly higher in the 140–200 °C (for KS-12F) range and in the 180–240 °C (for KS-16F) range compared with the results of the spent TWCs. The catalytic activity of acid solution treated TWCs decreases in the following order: $C_2H_2O_4 > C_6H_8O_7 >$ parent $> CH_3COOH$ for KS-12F and $C_2H_2O_4 > C_6H_8O_7 \approx CH_3COOH >$ parent for KS-16F, which is identical to those of the remaining Pt/Al ratio and the acidity of organic acids. These results definitely prove that the oxalic acid solution treatment can be one of the most efficient methods for enhancing the catalytic activity.

Since the oxalic acid solution treatment proved to be the most effective way for improving the catalytic activity of the spent TWCs, we further examined the activity of the oxalic acid solution treated TWCs (KS-12F and KS-16F) with two different aromatic molecules (i.e., benzene and xylene, BTX). Fig. 6(a) and (b) shows the light-off curves for the oxidation of benzene, toluene and xylene over both oxalic solution treated KS-12F and KS-16F. In addition, the reaction temperatures of 20% (T_{20}), 50% (T_{50}) and 90% (T_{90}) for BTX conversion over the oxalic acid solution treated KS-12F and KS-16F are compared in Table 3. As can be seen in Fig. 6 and Table 3, the experimental results reported here proved that the oxalic acid solution treated TWCs also presented a higher activity on the methyl derivative aromatic compounds, but no clear trend between the oxalic solution treatment and the order of BTX activity was found in the activity tests. In the case of KS-12F treated with oxalic acid solutions, removal of each aromatic compound below 200 °C with xylene was completely oxidized at the highest temperature (200 °C), toluene at the next highest (190 °C) and benzene at the lowest (180 °C). This result is in good agreement

Table 3
T_{20}, T_{50} and T_{90} of oxalic acid solution treated KS-12F and oxalic acid solution treated KS-16F.

Catalyst	Compound	T_{20} (°C)	T_{50} (°C)	T_{90} (°C)
Oxalic acid treated KS-12-F	Benzene	142	151	168
	Toluene	161	169	181
	Xylene	170	186	197
Oxalic acid treated KS-16-F	Benzene	174	190	202
	Toluene	176	189	199
	Xylene	174	193	212

with those obtained in previous works for platinum based catalysts where the catalytic activity of aromatic hydrocarbons depends, to a great extent, on the order of adsorption strength and ionization potential (or dipolar moments) of the aromatic compounds [22–24]. However, in the case of oxalic acid aqueous treated KS-16F, the order of reactivity is appreciably different from that of oxalic acid aqueous treated KS-12F. The characteristics of BTX oxidation are almost identical below 180 °C, but the order of reaction greatly changed above this temperature. The following order of reaction was observed: toluene $\geq$ xylene > benzene, which is also associated with the trend of ionization potential of the aromatic compounds.

4. Conclusions

In this work we demonstrated that the spent TWCs employed are a promising candidate material in the catalytic oxidation of VOCs. The catalytic activity of the spent TWCs is mainly correlated with the containing Pt/Al ratio rather than the containing contaminants and the extracted position of the TWCs. On the other hand, the surface characteristics and catalytic activities are substantially different according to the regeneration methods. Pretreatment with different acids changed the textural properties of the spent TWCs. A higher acidity (or lower pK_a and pK_{a1}) led to a larger BET surface area and average pore width. On the whole, the acid solution treated TWCs were more active than the spent TWCs. The oxalic acid solution treatment was also found to be the most appropriate treatment compared with acetic and citric acid solution treatments in improving the catalytic activity of the spent TWCs. Furthermore, the containing Pt/Al ratio, which is correlated with the acidity, plays a much more important role in the toluene oxidation regardless of the removal degree of the contaminants (i.e., P, Zn, Ca, Fe and S) and the alteration degree of surface characteristics. We also found that the catalytic activity for BTX oxidation can be partially related to the ionization potential (or dipolar moment) of the aromatic compounds

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