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Evaluation of wire-mesh honeycomb containing porous Al/Al$_2$O$_3$ layer for catalytic combustion of ethyl acetate in air

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Abstract

Wire-mesh honeycomb (WMH) catalyst is a monolith made from layers of flat and corrugated wire-meshes. To obtain an attrition-resistant washcoat on mesh surface, the mesh substrate was pre-coated with aluminum powder by electrophoretic deposition. A well-adhered aluminum layer was formed on wire-mesh surface after thermal treatment at 800 °C. Then, additional tuned calcination results in the formation of a thin Al$_2$O$_3$ layer in the outer surface of deposited aluminum particles. The Al/Al$_2$O$_3$-WMH was evaluated for catalytic combustion of ethyl acetate, a typical refractory volatile organic compound (VOC), after washcoating Pt/TiO$_2$ catalyst on it. The conversion over Al/Al$_2$O$_3$-WMH did not decrease during the long-term operation, since the primer layer of Al/Al$_2$O$_3$ enhanced the catalyst adhesion. Results were compared with conventional ceramic honeycomb (CH) containing the same cell density and catalyst loading. The activity of WMH was much higher than that of CH, especially at the high-temperature ranges. It was due to the difference in external mass transfer rate between two modules. The fact was confirmed by the calculation of the external mass transfer coefficient of WMH and CH.

Keywords: Catalytic combustion; Honeycomb reactor; VOC; Ethyl acetate; Wire-mesh; Mass transfer; Electrophoretic deposition

1. Introduction

Typical reactor type for catalytic combustion of volatile organic compounds (VOCs) is monolithic support that is characterized by very low pressure drop even at high flow rate. Monoliths are normally shaped as honeycombs or layered plates, and commonly composed of ceramic or metal materials [1]. The main drawbacks of ceramic monoliths are their susceptibility to thermal and mechanical shock and high manufacturing cost. As a reason, application of metal substrates as catalyst supports is becoming more common recently. The main advantages of metal substrates can be summarized as follows; high-thermal conductivity, high surface to volume ratio, low pressure drop, and low manufacturing cost. However, the metal substrates have low surface area to be used as catalyst supports. In order to achieve a high surface area, there have been many attempts to form an inorganic oxide layer on the surface of metal substrate. Conventional washcoating methods are commonly used for catalyst coating on monoliths [3], however, cannot be suited to metallic monoliths because of poor adhesion and non-uniformity of coatings. This is the main reason for the development of novel preparation techniques for use in metallic monoliths.

Plasma spray deposition was widely studied because it was able to offer better mechanical strength and thermal stability of coated layer [4–6]. Ahlström-Silversand and Odenbrand [4] applied a layer of porous alumina powder on substrate of the stainless steel meshes by thermal spray method; the coat was then impregnated with catalytic components. To attain a satisfactory conversion for CO and hydrocarbon combustion, up to five layers of wire-mesh were stacked in series in their tests. In the preparation of zeolite membranes, in situ crystallization in an autoclave was used to deposit a zeolite layer on a macroporous metallic substrate [7]. The electrophoretic deposition (EPD) method has been considered as another technique, since it

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requires a short deposition time and simple apparatus [8–10]. Vorob'eva et al. [10] tried to prepare wire-mesh coated with active alumina using EPD method. They used alumina sol for particle suspension during electrophoretic deposition.

In previous work, we first investigated applying EPD method to deposit aluminum as catalyst support material on the wire-mesh substrate [11]. Thin Al_2O_3 layer was formed on the outer surface of the Al particles, completely encapsulating Al particles. The surface of each wire-mesh was pre-coated with Al_2O_3 particles to form a certain thickness of sintered but somewhat porous layer. With these wire-meshes containing porous Al/Al_2O_3 primer layer, a honeycomb type module was manufactured by stacking alternately the corrugated and flat type of the wire-mesh sheets within a frame as proposed by Chung et al. [12]. The construction of the wire-mesh honeycomb (WMH) from mesh screen, in which radial mixing of the gas flow can readily occur through the mesh pores, should enhance the external mass transfer rate and give a more uniform distribution of fluids across the entire bed diameter. In addition, reactions limited by interphase mass transfer rates should benefit from the higher efficiency and substantial reduction in reactor size offered by the WMH.

The aim of the present work was evaluation of Al/Al_2O_3-WMH for catalytic combustion of ethyl acetate (EAc) that was recognized as the most difficult to oxidize compound largely produced. The Al/Al_2O_3-WMH was loaded with Pt(1.0 wt.%)/TiO_2 catalyst and examined at various working conditions to understand its mass and heat-transfer characteristics. For the comparison, a conventional ceramic honeycomb (CH) module with the same cell density and catalyst loading was also examined under the same working conditions. Finally, the effect of Al/Al_2O_3 primer layer on catalyst activity and stability was investigated.

2. Experimental

2.1. Preparation of Al/Al_2O_3–coated wire-meshes and honeycomb reactor

As the bare surface of metal wire can hardly attach catalyst powder, the wire-mesh sheets (both flat and corrugated one) were coated first with aluminum powder by using electrophoretic deposition method. The details of electrophoretic deposition of aluminum particles were well described elsewhere [11]. Aluminum powder (spherical, Angang steel Co.) was used as a deposition material, with a mean particle size of 4.5 μm and a specific surface area of 1.83 m^2/g. Polyacrylic acid and triethylamine (Aldrich) were used as additives, which were expected to improve the adhesion of aluminum particles and control the suspension conductivity, respectively. Commercial wire-mesh (SUS 316L, 20 mesh screen) has a size of hole opening around 0.9 mm, a wire diameter (d_w) of 0.27 mm, and a mesh weight about 0.0556 g/cm^2 in average. The surface of the wire-mesh substrate was treated with 10 wt.% H_2SO_4 solution for 2 min to remove dirt and grease.

First, suitable amount of additives ($1.0–5.0 \times 10^{-3}$ M) were added to ethanol (99.9%, Aldrich) under stirring, and then aluminum powder (less than 0.5 wt.%) was added to the suspension. The slurry solution was well mixed using an ultrasonic vibrator for 3 min. For particle deposition at the anode, the wire-mesh was cut into pieces (3.5 cm × 10 cm). The stainless steel plate was used as a cathode and the size is the same as that of the anode. The distance between the two electrodes was kept at 1.0 cm and the dc voltage was changed from 10 to 300 V. The aluminum-coated samples were dried at room temperature for 12 h, sintered 900 °C for 3 h with He, and then calcined at 500 °C for 5 h. The final calcination step was intended for forming a shallow passivated layer of Al_2O_3 on the surface of individual aluminum particles.

For making honeycomb-type catalyst module, two types of above Al/Al_2O_3-coated wire-mesh sheets were used; flat sheets and corrugated (triangle shaped with side length 4 mm) ones. The two sheets were packed alternately to a final thickness of about 3.5 cm. The wire-mesh honeycomb module, manufactured this way, has rectangular shape with a size of 3.5 cm × 3.5 cm in width and 5.0 cm in length. The parallel passage channels were shaped as equilateral triangles with a side length of around 4 mm. The above WMH module contains 50 cells per square inch (CPSI). For comparison of catalytic activity, a traditional ceramic (cordierite) honeycomb was also prepared, where the size and cell density were same as wire-mesh honeycomb. The channels were of square cross-section 3 mm × 3 mm with walls of thickness 0.5 mm.

2.2. Reactor system

As active catalyst in the combustion of ethyl acetate, we selected Pt/TiO_2 as an active component for the reaction. Particles of TiO_2 (anatase, Samchun Co., BET surface area 55 m^2/g) were impregnated with an aqueous solution of H_2PtCl_6 (Aldrich) to the incipient wetness. The impregnated sample was dried at 110 °C for 12 h and reduced at 450 °C for 8 h. For the washcoating of Pt/TiO_2 catalyst to the WMH, a slurry solution was made by mixing the catalyst powder with an appropriate amount of water, along with an inorganic binder such as colloidal silica (less than 10 wt.%). The honeycomb as dipped into the slurry solution, and then dried 120 °C for 12 h and calcined at 500 °C for 3 h. Similarly, the same amount of Pt/TiO_2 catalyst was loaded to the ceramic honeycomb.

The honeycomb modules (WMH and CH) were tested in a square-shaped (3.7 cm × 3.7 cm) reactor with a total bed length of 1.0 m. Fig. 1 shows the schematic diagram of the experimental apparatus. The stream of pollutant was generated by bubbling a flow of air through a saturator that was fixed at a certain temperature. An online gas chromatography (GC) device equipped with an HP-5

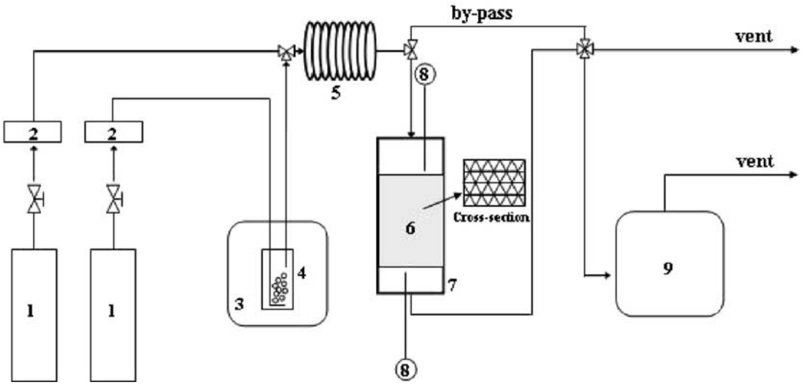

Fig. 1. A schematic diagram of catalytic combustion reactor. (1) Gas cylinder (air); (2) mass flow controller (MFC); (3) water bath; (4) saturator; (5) mixing chamber; (6) catalyst module; (7) reactor; (8) thermocouple; and (9) gas chromatography (GC).

column and FID detector was used for instant analysis of the ethyl acetate concentration in the bypassed feeding gas or exit gas of the reactor.

2.3. Catalyst characterization

The specific surface area was determined by nitrogen adsorption in a constant volume adsorption apparatus (Micrometrics, ASAP 2021C). The surface structure and the cross-section of coated wire were studied by means of X-ray diffraction (XRD, Mac Science Co., M18XHF) and scanning electron microscopy (SEM, Hitachi, S-2460N) equipped with an EDS system. X-ray photoelectron spectroscopy (XPS, VG Scientific, ESCA Lab. 220XL) was used for determining the surface structure and the phase of the coated sample. Hydrogen chemisorption was carried out for measuring the platinum dispersion in a static volumetric adsorption apparatus, equipped with a high-vacuum pump.

3. Results and discussion

3.1. Primer layer of Al/Al_2O_3 and surface properties

It is revealed that the aluminum particles are negatively charged in the medium and move towards the anode under the applied field. In the absence of suitable additives, no deposition is observed with the suspensions in either methanol or ethanol at any concentration of aluminum powder. Deposition begins to occur as the concentrations of TEA are increased to about 4.0×10^{-3} M. The adherence of the deposit was adequate for the concentrations of PAA below 5.5×10^{-4} wt.% and TEA from 6.0×10^{-3} M to 9.0×10^{-3} M. When the concentrations of additives are out of the range, the adherence of the deposit becomes poor or non-uniform.

After deposition, the Al-coated wire-meshes were thermally treated with He gas purge for particle sintering. The adherence of the Al coatings has been evaluated according to the ultrasound method, described in the literature [13]. The method was based on the measurement of the weight loss caused by exposure to ultrasounds. The coated wire-meshes were immersed in petroleum ether inside a sealed beaker, and then treated in an ultrasound bath for 0.5–2.0 h. Al coatings are easily detached from the metallic substrate when the sintering temperature is less than 600 °C, since it is difficult for aluminum particles to be sintered each other at the temperatures. It is shown that the detached amount of the Al coatings is below 2 wt.% over 750 °C. At 900 °C, the Al coatings are hardly detached from the metallic substrate even after ultrasound vibration. However, the Al coating layer might be highly sintered each other at the temperature, so it had non-porous surface structure that was inadequate for catalyst monolith. The suitable temperature range, therefore, is around 800 °C during thermal treatment. S.E.M. pictures of the typical coated sample after sintering at 800 °C is shown in Fig. 2. It can be seen that the Al-coated wire-mesh has highly rough and porous surface (a), moreover the deposited aluminum particles are well packed each other (b). The thickness of the aluminum layer was measured around 100 μm.

Above Al-coated wire-meshes were calcined in order to form a thin oxide layer on Al layer. As a result, thin Al_2O_3 layer was formed in the outer surface of the Al particles after tuned calcination (500 °C, 3 h), completely encapsulating each Al particles. Table 1 summarizes the physical properties, i.e. BET surface area, average pore diameter, Al coating thickness, and Al_2O_3 thickness of Al/Al_2O_3-coated wire-mesh. In contrast to the fresh wire that has non-porous and relatively smooth surface structure, the Al/Al_2O_3-coated wire-mesh has highly rough and porous surface. It is, therefore, considered that the surface properties of the Al-coated wire-mesh honeycomb such as surface area, roughness, and porosity are as good as those of the conventional ceramic honeycomb for washcoating of catalyst powder.

Pt/TiO_2 powder that was well-known as catalyst for combustion of VOCs, washcoated on the above Al/Al_2O_3-coated wire-mesh. For the comparison, the same amount of

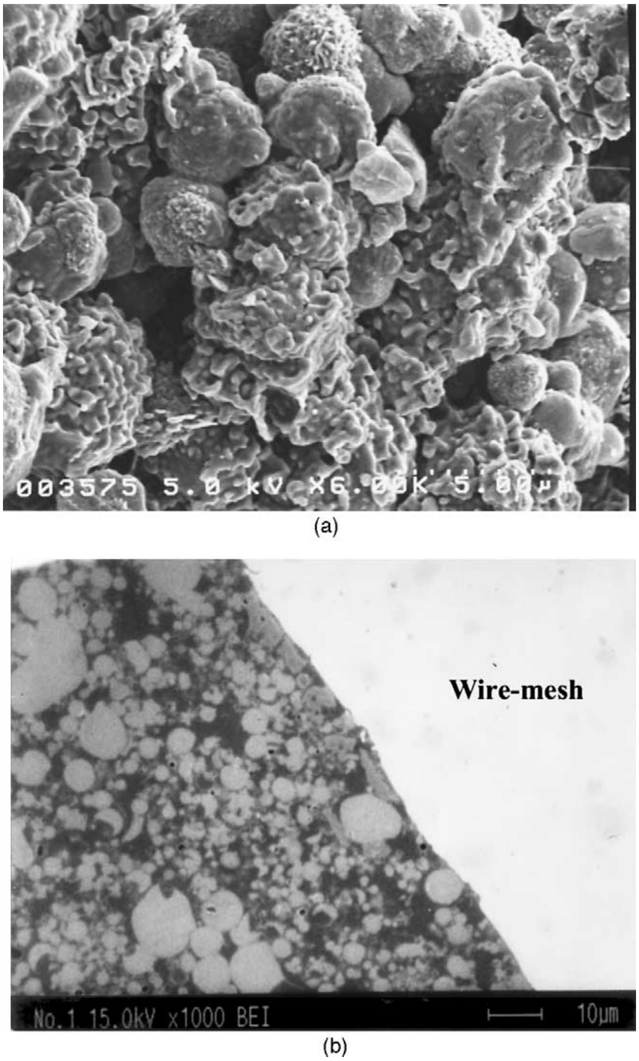

Fig. 2. S.E.M. pictures of (a) the surface and (b) the cross-sectional area of the Al/Al_2O_3-coated wire-mesh.

Pt/TiO_2 was also washcoated on the wire-mesh that was pretreated with acid/thermal treatment [12]. Similarly, the adherence abilities of those catalyst-coated wire-meshes were assessed by ultrasound method, as shown in Table 2. There was an obvious weight loss in the conventional washcoated samples, i.e. approximately 21 wt.% loss after 30 min ultrasound vibration. In conventional method, the catalyst coatings were easily detached from the wire-mesh under the influence of mechanical shock, since metal oxide could hardly adhere to metal surface in natural. On the other hand, the developed two-step coating wire-mesh was excellent resistant ability to mechanical shock. It reveals that the primer layer of Al/Al_2O_3 plays an important role in coating adherence of catalyst onto metallic substrates. Sintered Al particles made the coatings stick to metallic substrate strongly and Al_2O_3 layer be profitable for catalyst washcoating. Moreover, it can be expected that thin Al_2O_3 layer that encapsulated the Al particles prevent the oxygen gas from penetrating into metallic substrate through the catalytic oxidation of VOCs.

Table 1
Physical properties of Al/Al_2O_3-coated wire-mesh

Properties	Al/Al_2O_3-coated wire-mesh
BET surface area	12.23 m^2/g
Average pore diameter	110 Å
Average thickness of Al measured by SEM	100 µm
Average thickness of Al_2O_3 onto Al particles after calcination measured by XPS	30 nm

3.2. Catalytic combustion of ethyl acetate over WMH and CH

Honeycomb-type catalyst module was made by packing two types of Al/Al_2O_3-coated wire-meshes; flat sheets and corrugated (triangle shaped with side length approximately 4 mm) ones. The two sheets were packed alternately to a final thickness of about 3.5 cm. In order to test the Al/Al_2O_3-coated wire-mesh honeycomb as catalytic support, catalytic combustion of ethyl acetate was performed after modules of the WMH were doped with Pt(1.0 wt.%)/TiO_2 catalyst. It is known that EAc is more difficult to be catalytic decomposed than aromatic compounds [14]. In practice, the pollutants are normally mixtures of various VOCs. The combustion curve of a pollutant in mixture may change greatly from the individually measured one. For example, in the combustion of benzene and EAc mixture [15], the combustion curve of benzene in the mixture was the same as EAc combustion, which was about 50 °C higher than that of benzene alone. On the other hand, the combustion of EAc in the mixture was not influenced by the presence of benzene. It was concluded in [15] that for noble metal catalyst, the most difficultly oxidized compound very often controls the overall oxidation, and the complete conversion of all components is achieved at temperatures a little higher than that corresponding to the most refractory VOC measured alone. Therefore, ethyl acetate, one of the most refractory VOCs, was selected for further investigation of VOC combustion.

First, WMH modules were loaded with 8.0, 12.0, 22.5, and 30.0 wt.% of Pt/TiO_2 powder to find a proper loading amount of the catalyst on WMH. Gas hourly space velocity (GHSV) and EAc concentration in air were maintained at 2000 h^{-1} and 1000 ppm, respectively. There exists a proper loading amount of powder, too much will accumulate in the cross-points of wires or block the mesh holes and detach easily, too thin coating will be difficult to ensure a uniformly covering of all the geometric surface. The results in Fig. 3 show that the catalytic activity increases sharply up to a loading of 22.5 wt.%. It was observed that the catalyst was more or less accumulated at the cross-points in the WMH as the loading increased above 22.5 wt.% and some openings in the wire-meshes began to be blocked with a heterogeneous color appearance. It is enough around 22.5 wt.% loading of catalyst for covering all the wire surfaces, as evidenced by

Table 2
The adherence abilities of the catalyst coatings assessed by ultrasound vibration

Substrate	Al/Al_2O_3-coated wire-mesh	Whisker-like wire-mesh	Fresh wire-mesh
Catalyst coating amount (g)	8.1	8.0	8.0
Pretreatment	Al/Al_2O_3 pre-coating layer by EPD	Acid/thermal	None
Scaling off amount (wt.%) after 0.5 h	0.26	7.4	20.9
Scaling off amount (wt.%) after 2 h	0.46	11.7	24.2

the uniform color of the WMH after calcination. At this catalyst loading, the average thickness of catalyst layer was measured around 30 μm by S.E.M.

The combustion of EAc over conventional ceramic honeycomb was also performed for the activity comparison with WMH. The conversion of EAc in WMH and CH at 210 and 250 °C was demonstrated in Fig. 4. The amount of Pt(1.0 wt.%)/TiO_2 catalyst loading, Pt dispersion, and reaction conditions, were all same. The differences in geometric surface area were very small between these two modules. The conversion in WMH is always higher over that in ceramic honeycomb, especially at the high temperature and lower gas flow rate, where the role of mass transfer is more important. WMH and CH were then scaled up (15 cm × 15 cm × 15 cm; GHSV = 10,000 ~ 30,000 h^{-1}; 150 CPSI) and tested for catalytic combustion of ethyl acetate in air (Fig. 5). It was shown that the conversion in WMH was also higher than that in CH even at high GHSV 30,000 h^{-1}. The differences in catalytic activity between WMH and CH may be caused by the external mass transfer, later mentioned in detail.

Catalytic stability tests were performed with two types of WMHs; acid/thermal pre-treated one and Al/Al_2O_3 pre-coated one. They were loaded with the same amount of catalyst and run for 120 days under conditions of 1000 ppm, GHSV = 4000 h^{-1}, and 300 °C. The detached catalyst was collected from the bottom of the reactor and weighed during the operation. As indicated in Fig. 6, the conversion achieved by the acid/thermal pre-treated WMH mainly fell under 80%, since the coated catalyst was scaled off during the long-term operation. On the other hand, the ethyl acetate conversion of Al/Al_2O_3-WMH was remained over 95%, indicating no catalyst loss after this period.

3.3. *Influence of interphase mass transfer over WMH and CH*

To probe the importance of interphase mass transfer in WMH, we changed the channel size of WMH and the linear velocity of the reactant stream. The bigger channel size means the longer interphase mass transfer path length for reactant, therefore the lower activity especially at the conditions where the interphase mass transfer rate plays an important role. Fig. 7 shows the EAc conversion of two kinds of WMHs containing different channel size, 25 and 50 cells per square inch. The WMH with smaller channels (50 CPSI) shows higher activity than the WMH with bigger channels (25 CPSI). The difference in temperature for the same conversion is large in the high-temperature range that is more severely limited by interphase mass transfer rate.

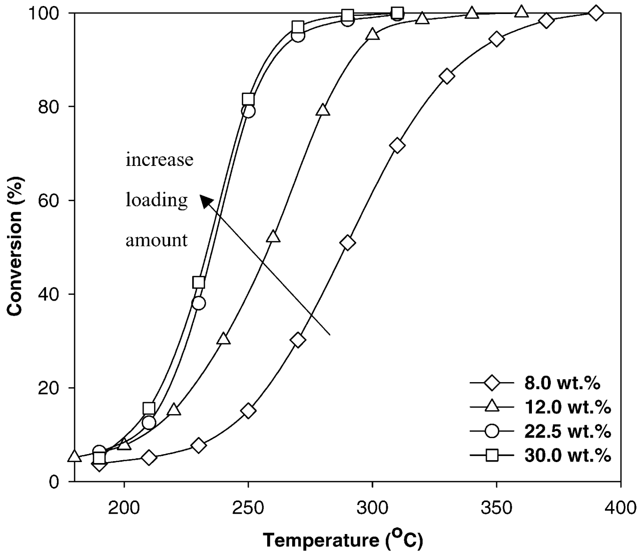

Fig. 3. Influence of Pt(1.0 wt.%)/TiO_2 catalyst loading on the conversion of ethyl acetate in wire-mesh honeycomb. GHSV = 2000 h^{-1}, 1000 ppm EAc in air.

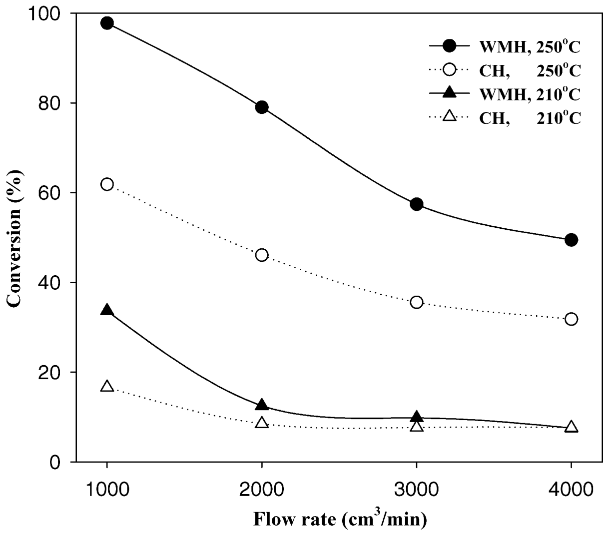

Fig. 4. Activity comparison between WMH and CH with the same catalyst loading; 25 wt.% of Pt(1.0 wt.%)/TiO_2 loading, 1000 ppm EAc in air.

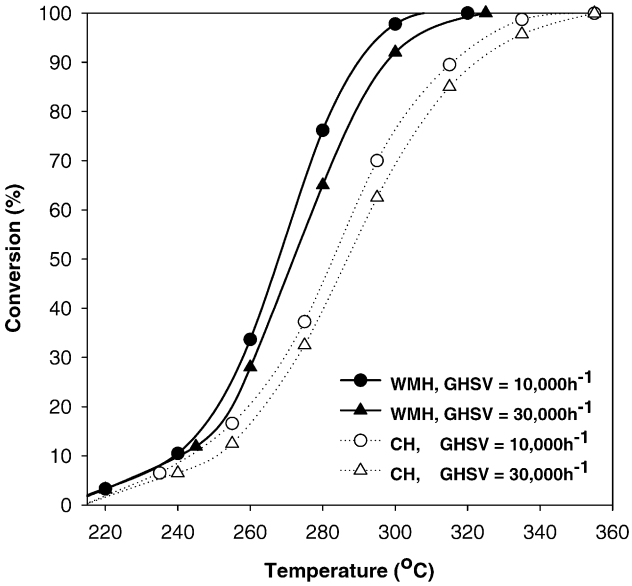

Fig. 5. The conversion of ethyl acetate in WMH and CH with 150 CPSI; 25 wt.% of Pt(1.0 wt.%)/TiO_2 loading, 1000 ppm EAc in air.

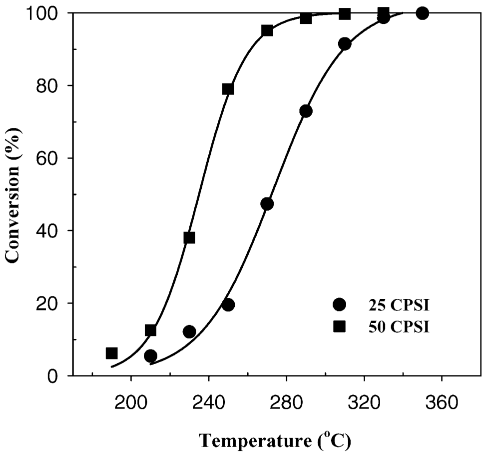

Fig. 7. The effect of channel size in WMH; 25 wt.% of Pt(1.0 wt.%)/TiO_2 loading, GHSV = 2000 h^{-1}, 1000 ppm EAc in air.

The influence of channel size is exaggerated greatly by the difference in catalyst activity, which decides the reaction rate at the low-temperature range. The influence of interphase mass transfer is more reflected more realistically in the experiment with modules of similar activities and same GHSV, but at different gas flow rates.

EAc conversion as a change of linear velocity was also examined. Two WMHs with the same size and catalyst loading were packed in series and the reactant flow rate was double to maintain the same GHSV. The conversion of the one-module and two-module reactors was about the same at the low-temperature range at the same GHSV, but the double module reactor showed higher conversion after 270 °C and the deviation increased with temperature.

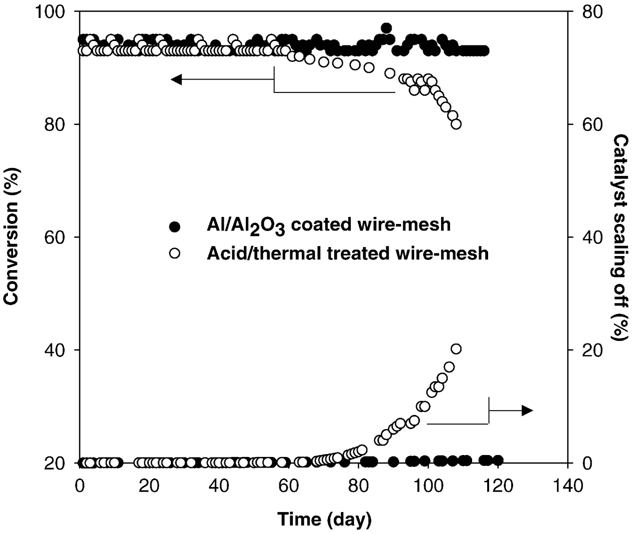

Fig. 6. Long-term tests of Al/Al_2O_3 pre-coated WMH and acid/thermal-treated WMH with the same catalyst loading; 25 wt.% of Pt(1.0 wt.%)/TiO_2 loading, 1000 ppm EAc in air, GHSV = 4000 h^{-1}, T = 300 °C.

The mass transfer coefficients (k_m) of WMH and CH were calculated from the previous results of EAc conversion with different temperatures and flow rates. Details of the calculation equation are given in elsewhere [2]. The data points for different temperatures would not form a straight line, since external diffusion limitation is important at high temperatures. The distance between the data plots and straight line enlarges with the increase of gas flow rate, which implies a more effect on mass transfer limitation. When both diffusion and reaction resistance are significant, and for a first order reaction, the reaction rate constant (k) is a term that depends on the surface reaction and external mass transfer resistances. It was supposed that the internal mass transfer resistance of the catalyst is neglected for WMH and CH, since the thickness of the catalyst layer is very thin. Indeed, it was confirmed from the calculation of effectiveness factor (η) in the range of 0.98–0.99 [16].

We then measured the intrinsic rate constant, k_r at low-temperature region where the external mass transfer can possibly be avoided. It was found that at temperatures less than 480 K, the measured value of k_r remained more or less constant regardless of the flow rate change. The k_r values measured at this mass transfer eliminated region were used

Table 3
Calculated external mass transfers (k_m) of WMH and CH with different flow rates

Module	Flow rate (cm^3/min)	External mass transfer, k_m (cm/s)
WMH	1000	1.79 ± 0.03
	2000	2.92 ± 0.06
	3000	4.21 ± 0.07
CH	1000	0.97 ± 0.09
	2000	2.01 ± 0.10
	3000	3.11 ± 0.06

for regression analysis to estimate the values of E_a and ln k_{r0}. As a result, activation energy E and frequency factor of the reaction are 23.3 kcal/mol and $4.827 \times 10^{10}\ s^{-1}$, respectively.

The mass transfer coefficients of WMH and CH could be calculated by substitution of overall and intrinsic kinetic rate constants, where the external surface areas (a_m) were 11.0 cm^2/cm^3 for WMH and 8.9 cm^2/cm^3 for CH with the same cell density. The external mass transfer coefficients of EAc oxidation over WMH and CH were listed in Table 3. It is revealed that the resistance of mass transfer is much smaller in WMH than in CH at all the flow rates. This result exactly corresponds to the EAc conversion of WMH and CH with different linear velocity as previously described in Fig. 4.

4. Conclusions

A novel metallic wire-mesh honeycomb containing the primer layer of Al/Al_2O_3 was prepared. The primer layer of Al/Al_2O_3 was strongly attached onto the wire-mesh surface due to partial sintering of deposited Al particles. Thin Al_2O_3 layer was formed in the outer surface of the Al particles after calcination, completely encapsulating each Al particles. It has shown to be porous structure with a large surface area, as a result. After washcoating Pt/TiO_2 catalyst on WMH, it was evaluated for catalytic combustion of ethyl acetate, a typical refractory volatile organic compound. Its benefits over traditional ceramic honeycomb catalyst were also proved by the activity comparison. As wire-mesh honeycomb has channels whose walls are interconnected each other through the holes in the wire-mesh sheets, the gas feed that enters through the channels of WMH can have flows to the three dimensional directions; inducing turbulent effect to facilitate mass transfer rate of molecules inside channels. It was shown that the resistance of mass transfer was much smaller in WMH by the calculation of mass transfer coefficients.

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