

## The Reactivity of $V_2O_5$ - $WO_3$ - $TiO_2$ Catalyst Supported on a Ceramic Filter Candle for Selective Reduction of NO

Joo-Hong Choi<sup>†</sup>, Sung-Kyoung Kim and Young-Cheol Bak

Dept. of Chem. Eng./Eng. Resear. Inst., Gyeongsang National University, Jinju 660-701, Korea

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**Abstract**—For realizing the environmental issues and constituting an economical treatment system, a catalytic filter based on  $V_2O_5/TiO_2$  supported on tubular filter elements has many advantages by removing  $NO_x$  and particulate simultaneously from flue gas. In order to improve the activity of a catalytic filter based on  $V_2O_5/TiO_2$  supported on a commercial high temperature filter element (PRD-66), the promoting effects of  $WO_3$  were investigated in an experimental unit. PRD-66 presented very good properties for SCR catalyst carrier since it contains much active material such as  $Al_2O_3$ ,  $SiO_2$ , and  $MgO$  whose contributions were remarkable. For additional catalyst carrier,  $TiO_2$  particles were coated in the pores of PRD-66 with relatively good distribution of the particle size less than  $1\ \mu m$ , by a coating process applying centrifugal force.  $WO_3$ , in the  $V_2O_5$ - $WO_3$ - $TiO_2$ /PRD-66 catalytic filter system, increased the SCR activity significantly and broadened the optimum temperature window. The catalytic filter shows the maximum NO conversion of more than 95% for NO concentration of 700 ppmv at face velocity of 0.02 m/sec, which is comparable to the current commercial catalytic filters of plate form.

Key words: Promotion Effect,  $WO_3$ , Catalytic Filter, NO Reduction, Ammonia,  $V_2O_5/TiO_2$

### INTRODUCTION

Catalysts for selective catalytic reduction (SCR) of  $NO_x$  have been widely developed in their forms and compositions [Busca et al., 1998; Che et al., 2000; Choi et al., 1999a, 1994; Nam et al., 1995; Părvulescu et al., 1998; Ham et al., 2000]. Among them, catalysts based on vanadia-titania are effectively commercialized for the reduction of  $NO_x$  with ammonia in  $SO_2$ -containing flue gases [Amiridis et al., 1996; Bosch and Janssen, 1988; Forzatti, 1966; Terabe et al., 1994]. Vanadium oxide has been known as the active site not only for SCR but also for the undesired oxidation of  $SO_2$ . Thus, it is present in very small amounts ( $<1\%$  w/w) in commercial catalysts. The catalysts contain a large amount ( $<10\%$  w/w) of  $WO_3$  or  $MoO_3$  over the  $TiO_2$ -anatase carrier [Alemany et al., 1996].  $WO_3$  has been known as providing much less activity for SCR but presenting an inhibiting effect against the oxidation of  $SO_2$  by vanadium oxide as well as stabilizing the structural and morphological characteristics of  $V_2O_5$  anatase [Alemany et al., 1995].  $WO_3$  also increases the acidity of  $TiO_2$  [Sohn and Bae, 2000]. One of the recent key issues on the commercial SCR catalyst is to limit the possible oxidation of  $SO_2$  to  $SO_3$  as much as because  $SO_3$  forms alkali sulphates. Although sulphate species of monolayer on vanadia stabilizes the anatase phase of vanadia inhibiting the phase transition [Alemany et al., 1996], they inhibit the SCR activity by plugging the monolith gap. Such a plugging problem in the monolith catalyst also causes a serious operational problem of this catalyst, especially at high dust concentration. Therefore, the role of W atom in the  $V_2O_5/TiO_2$  catalytic system is very important.

The advantage of the catalytic filter in the form of rigid filter elements like ceramic filter candle is solving the plugging problem while

providing the lower pressure drop system as well as its dual function for the control of particulates and nitric oxides. The advanced ceramic filter elements for application at high temperature are composed of two layers: interior support and exterior membrane layer [Choi et al., 1999b]. Most of the filtration is carried out through the membrane layer whose thickness is about  $100\ \mu m$ . The support layer is useful not for the function of the filtration but for a rigid supporter [Choi et al., 1999c]. The catalytic filter utilizes these large pores of the support layer whose pore size is about  $100\ \mu m$  for the room of the catalyst support. However, the potential task for applying the catalytic filter in an advanced system is how to increase the catalytic activity by increasing the catalyst dispersion. Recently, the technology to support  $TiO_2$  particles in the pores of PRD-66 has been successfully developed by using the sol-gel centrifugal coating process [Choi et al., 2000]. The  $V_2O_5$ - $TiO_2$  based-catalytic filter prepared by the method presents NO conversion more than 90% for the NO concentration of 500 ppmv at the face velocity of 0.02 m/sec. The result satisfactorily meets the regulation of NO from the stationary process. However, in order to meet the ammonia slip (less than 10 ppmv) recommended for the commercialization of SCR unit using ammonia, the catalytic activity still needs to improve. So the aim of this work is to improve the catalytic activity by using  $WO_3$ . The promoting effect of  $WO_3$  was discussed from the experimental results obtained by using simulated gas.

### EXPERIMENTAL

#### 1. Preparation of Catalytic Filter

The catalytic filters were prepared with the methods of the centrifugal coating of  $TiO_2$  sol-gel prepared using tetra isopropyl orthotitanate (TIPOT) [Choi et al., 2000]. The commercial ceramic filter element, PRD-66 (PRD), from AlliedSignal was utilized as received form and compositions. The PRD, which is prepared by spin-weav-

<sup>†</sup>To whom correspondence should be addressed.

E-mail: jhchoi@nongae.gsnu.ac.kr

ing the textile glass yarn, is a composite of corundum, cordierite, and mullite. It has high porosity of 60% with its pore size of the support layer larger than 100  $\mu\text{m}$ . It has an effective membrane layer of median pore size about 10.5  $\mu\text{m}$ . The original dimension of the filter candles is 1.5 m length, 0.04 and 0.06 m of inner and outer diameter, respectively. It was cut in the tubular form of 0.02 m length in order to fix in a small experimental reactor. The filter tube was washed in an ultrasonic water bath and treated in 0.05 M NaOH aqueous solution and dried for 2 hr at 110  $^{\circ}\text{C}$  before being used.

The  $\text{TiO}_2$  particles were first supported on the PRD by letting  $\text{TiO}_2$  sol-gel solution flow across the filter element by applying centrifugal force [Choi et al., 2000] in order to enhance for the  $\text{TiO}_2$  particles to penetrate into the inner layer of the filter element. The rotation speed was controlled at 580 rpm and measured by a tachometer. Colloidal  $\text{TiO}_2$  was prepared with a sol-gel method [Terabe et al., 1994; Choi et al., 1999a]. The  $\text{TiO}_2$ -loaded filter tube was dried overnight at room temperature and 1 hr at 110  $^{\circ}\text{C}$  and then calcined in the air stream at 450  $^{\circ}\text{C}$  for 2 hr. In order to impregnate the catalysts,  $\text{TiO}_2$ -loaded PRD was immersed in the oxalic acid solution of ammonium metavanadate and/or ammonium paratungstate. The solution was slowly evaporated at low temperature until the filter element absorbed all of the solution (called dry impregnation), followed by drying overnight and calcining at 450  $^{\circ}\text{C}$  for 2 hr. In the dip impregnation process,  $\text{TiO}_2$ -loaded PRD was immersed in the excess solution for 12 hr, followed by drying overnight and calcining at 450  $^{\circ}\text{C}$  for 2 hr. The catalytic filters fabricated in the several compositions and different procedures are described in Table 1. The composition of catalysts in Table 1 denotes the weight percent over the PRD. Catalytic filters of VWT series denote  $\text{V}_2\text{O}_5$ - $\text{WO}_3$ - $\text{TiO}_2$ /PRD. VWT2 and VWT4 represent the catalytic filters fabricated by the procedure that V atoms were impregnated on the previously prepared  $\text{WO}_3$ - $\text{TiO}_2$ /PRD. VOT, VWO and OWT catalytic filters present  $\text{V}_2\text{O}_5$ - $\text{TiO}_2$ /PRD,  $\text{V}_2\text{O}_5$ - $\text{WO}_3$ /PRD, and  $\text{WO}_3$ - $\text{TiO}_2$ /PRD, respectively. The morphology and elemental composition of the catalytic filter were analyzed with SEM and SEM-EDS, respectively.

## 2. The Performance Test

The activity of the catalytic filter was tested in an experimental unit using a gas mixture of NO,  $\text{NH}_3$ ,  $\text{O}_2$ , and  $\text{N}_2$  [Choi and Ahn, 1999a]. 0.2%  $\text{NH}_3$  in  $\text{N}_2$ , 0.15% NO in  $\text{N}_2$ , and air were used as the source of the gas mixture. In a normal test run, the concentrations of NO,  $\text{NH}_3$ , and  $\text{O}_2$  were 500, 500 ppm, and 2 vol% in  $\text{N}_2$  bal-

anced, respectively. The total gas flow rate was exactly controlled with a mass flow controller at the actual face velocity of 0.02 m/sec. The catalytic filter was mounted using flanges. It was first fixed between top and middle flanges with long bolts. The reaction part was sealed with the top and bottom flanges. The reaction gases were introduced from bottom inlet of the reactor and passed out across the catalytic filter. Thus, the direction of gas pass (inward from the outer surface) is the same as that of industrial filtration mode using ceramic filter element. After the impregnation step, the catalytic filter was dried and kept in a low humidity chamber. And it was calcined on-site at the reaction unit before being used for the test of catalytic performance. NO conversion over the catalytic filter was measured at steady state of each temperature.

The concentrations of NO,  $\text{NO}_2$ , and  $\text{NH}_3$  were continuously analyzed by using a chemiluminescence method (TEI model 17, Thermo Environmental Instruments Inc.). Oxygen concentration was analyzed by an off-line GC with TCD using GS-Q capillary column.

## RESULTS AND DISCUSSIONS

### 1. Characteristics of the Catalytic Filters

The particle size of  $\text{TiO}_2$  sol-gel is effectively controlled by the change of HCl/TIPOT mole ratio (H/T) and increases with the increase of HCl concentration [Choi et al., 1999a]. H/T was adjusted to 3 in this study in order to control the particle size of  $\text{TiO}_2$  about 400-500 nm. About 50 ml of the sol-gel solution was circulated 10 times across the filter element with a rotation speed of 580 rpm. Fig. 1a represents the SEM photograph of the fresh PRD showing that the external membrane layer has very fine pores and the interior supporting one has large pores of more than 100  $\mu\text{m}$ .  $\text{TiO}_2$  particles less than 1  $\mu\text{m}$  were coated with good distribution in the interior pores of PRD as shown at Fig. 1b. Thus it is certain that the coating method applying a centrifugal force is very effective to load the  $\text{TiO}_2$  particles into the inner pore of the support layer of the filter element. In case of VWT3 composed of  $\text{V}_2\text{O}_5$ - $\text{WO}_3$ - $\text{TiO}_2$ /PRD, V and W atoms were also distributed over the entire surfaces of both the PRD and  $\text{TiO}_2$ . It is certain then that the PRD has a remarkable surface area for the catalyst carrier. The elemental analysis using SEM-EDS shown in Table 2 presents the amount of surface element for catalytic filter.  $\text{TiO}_2$  covers about 65% of total surface of catalytic filter. And total coverages of V and W atoms are 2.7 and 9.2% w/w, respectively, with respect to  $\text{TiO}_2$ . Parts of

**Table 1. The description of catalytic filter prepared by sol-gel centrifugal method**

Name	$\text{V}_2\text{O}_5$ loading (% w/w)	$\text{WO}_3$ loading (% w/w)	$\text{TiO}_2$ loading (% w/w)	Impregnation method	Solvent	BET surface area ( $\text{m}^2/\text{g}$ )
VWT1	0.04	0.3	3.2	Dip co-impregnation	Oxalic acid	1.67
VWT2	0.06	0.5	3.0	Dip V impreg. on $\text{WO}_3$ / $\text{TiO}_2$ /filter	Oxalic acid	2.42
VWT3	0.46	3.7	3.1	Dry co-impreg.	Oxalic acid	1.33
VWT4	0.47	3.8	3.1	Dry V impreg. on $\text{WO}_3$ / $\text{TiO}_2$ /filter	Oxalic acid	1.16
VOT	1.62	-	2.9	Dry impreg.	Oxalic acid	0.19
OWT	-	1.1	4.9	Dry impregnation	Oxalic acid	3.49
VWO	0.49	3.9	-	Dry impregnation	Oxalic acid	0.57
OOO	-	-	-	Fresh filter	-	0.10
OOT	-	-	3.1	$\text{TiO}_2$ /filter	-	1.38

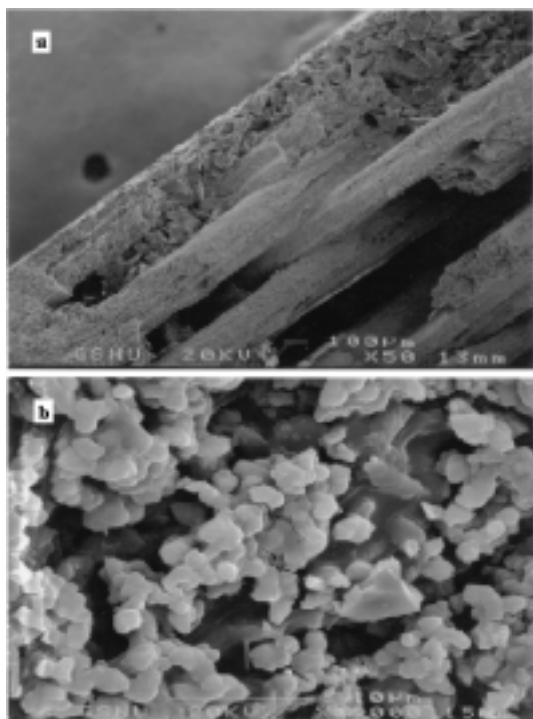


Fig. 1. SEM images of fresh PRD-66 (a) and  $TiO_2$  supported on PRD-66 (b).

Table 2. Elemental analysis of catalytic filter by SEM-EDS

Element	Fresh PRD-66		VWT3	
	Atomic (%)	Elemental (%)	Atomic (%)	Elemental (%)
Mg(MgO)	3.68	3.28	-	-
Al( $Al_2O_3$ )	61.03	60.38	24.00	12.59
Si( $SiO_2$ )	35.29	36.35	-	-
Ti( $TiO_2$ )			67.94	63.28
V( $V_2O_5$ )			1.81	1.79
W( $WO_3$ )			6.25	22.34

these atoms are also loaded on the  $TiO_2$ -free surface of PRD.

The BET surface area of the PRD is about  $0.1 \text{ m}^2/\text{g}$ , according to the data presented by maker [AlliedSignal, 1999]. And the value measured in this study was  $0.96 \text{ m}^2/\text{g}$  in the form of fragment about 1 mm. The different value in BET surface area for the fragment form is due to the increase of surface by reducing the particle size. The BET surface areas shown in Table 1 are corrected by eliminating the difference ( $0.86 \text{ m}^2/\text{g}$ ) from the measured one. The surface area of catalytic filter increased additionally  $1.28 \text{ m}^2/\text{g}$  by supporting  $TiO_2$ . However, it was markedly decreased from 1.38 (for OOT) to  $0.19 \text{ m}^2/\text{g}$  (for VOT) by the loading of V atoms. However, it increased to 3.49 (for OWT) from  $1.38 \text{ m}^2/\text{g}$  (for OOT) by the loading of W atoms. The surface area of VWT3, containing both V and W oxide was  $1.33 \text{ m}^2/\text{g}$ , which means that W atoms increase the surface area of  $V_2O_5$ - $TiO_2$ /PRD. BET data in Table 1 reveal a general pattern that V loading reduces the surface area while W loading increases it. This result is similar to the case of  $V_2O_5$ - $WO_3$ / $TiO_2$  ternary system reported by Alemany et al. [1995]. They reported that the sur-

face area of  $V_2O_5(x)$ - $WO_3(9)$ / $TiO_2$  gradually decreases as the  $V_2O_5$  loading (x) increases, while  $WO_3$  loading increases BET surface area at a given  $V_2O_5$  loading or bare  $TiO_2$ .

## 2. Performance of the Catalytic Filter

In order to investigate the effect of  $WO_3$  on NO reduction activity of SCR catalytic filter, NO conversion over catalytic filters, OWT ( $WO_3$ - $TiO_2$ /PRD), VOT ( $V_2O_5$ - $TiO_2$ /PRD) and VWT3 ( $V_2O_5$ - $WO_3$ - $TiO_2$ /PRD), were compared. Fig. 3 represents NO conversion when NO concentration in the reactant is 500 ppm at the face velocity of 0.02 m/sec. VWT3 shows the maximum NO conversion of 95%, while VOT and OWT show 68 and 75%, respectively. NO conversion over OWT is less than 40% in the optimum temperature range of 320–350 °C for VOT. And the temperature window presenting NO conversion of more than 95% of its maximum is broadened from 30 °C (320–350 °C) for VOT to 60 °C (290–350 °C) for VWT3. The results indicate that  $WO_3$  significantly promotes catalytic activity at lower temperature and broadens the temperature window. However, OWT containing only  $WO_3$  reveals low activity at high temperature (400 °C) with a narrow temperature window. This result is very similar with that over the ternary ( $V_2O_5$ - $WO_3$ - $TiO_2$ ) catalyst reported by Alemany et al. [1995]. They reported that  $WO_3$  in the catalytic system promoted SCR activity as well as increased the selectivity to  $N_2$ . In the presence of  $WO_3$  (9% w/w), the temperature required for 50% conversion of NO was lowered from 300 to 270 °C for  $V_2O_5$  (0.78% w/w), and to 210 °C for  $V_2O_5$  (1.4% w/w). And the temperature window for maximum NO conversion was greatly broadened in the ternary catalyst from 15 to 70 °C for  $V_2O_5$  (1.4)- $WO_3$  (9)/ $TiO_2$  since the catalyst has high activity at lower temperature and high selectivity to  $N_2$  at higher temperature. The role

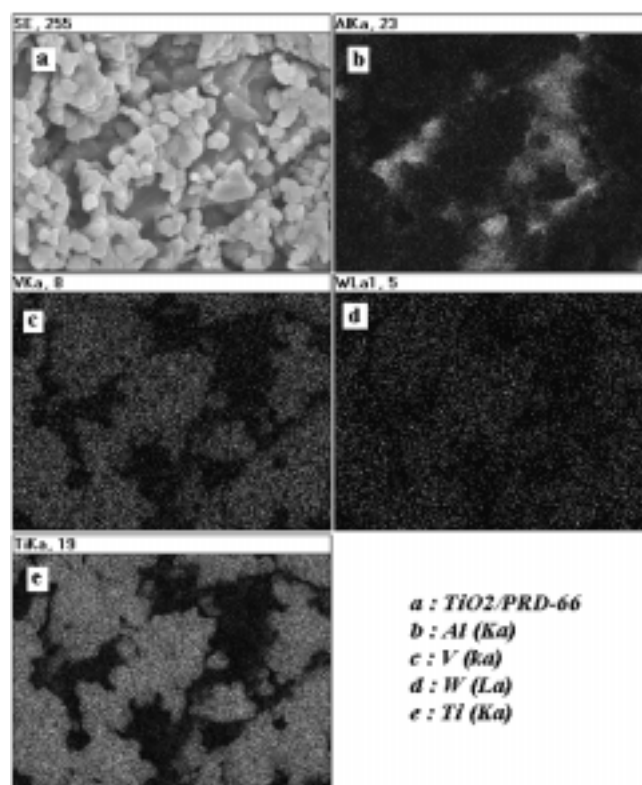


Fig. 2. EDS for elements mapping of VWT3 catalytic filter.

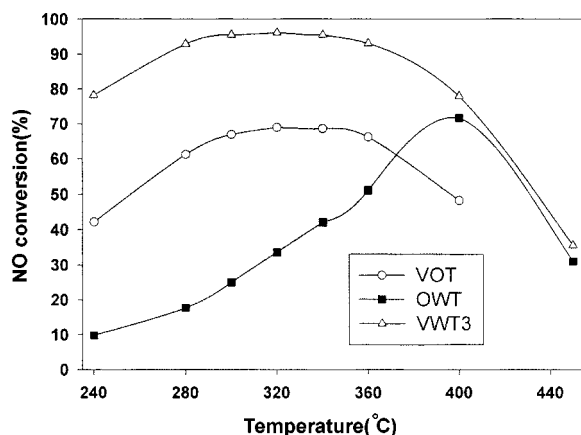


Fig. 3. The effect of  $\text{WO}_3$  on the catalytic activity of  $\text{V}_2\text{O}_5\text{-TiO}_2/\text{PRD}$ .

of  $\text{WO}_3$  in the ternary catalyst has been understood as increasing the surface acidity of  $\text{V}_2\text{O}_5/\text{TiO}_2$  [Shin et al., 1994]. And the higher reactivity is related to their superior redox properties owing to interaction between the monomeric V and W oxides over  $\text{TiO}_2$  [Alemany et al., 1995]. In case of catalytic filter fabricated with PRD filter element, the PRD itself has several kinds of oxide support. According to the manufacturer's report [AlliedSignal, 1999], the PRD is composed of corundum (alumina), cordierite, and mullite after fabricating from the oxide mixture of  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ , and  $\text{MgO}$ .  $\text{V}_2\text{O}_5/\text{Al}_2\text{O}_3$  is a very good SCR catalyst [Saracco et al., 1995].  $\text{SiO}_2$  and  $\text{MgO}$  show relevant properties for catalyst carrier [Pârăulescu et al., 1998]. Although vanadia/alumina and vanadia/silica have a lower activity for SCR than vanadia/titania, titania grafted on silica or alumina shows a remarkable activity. Thus, it is estimated that  $\text{V}_2\text{O}_5\text{-WO}_3\text{-TiO}_2/\text{PRD}$  has plenty of active surface composed of complicated active sites formed on  $\text{TiO}_2$  particles as well as the PRD itself. This assumption is evident from the analysis of EDS of VWT3 shown in Fig. 2. According to the result,  $\text{TiO}_2$  particles form the isolated particles in the pore of the PRD while  $\text{V}_2\text{O}_5$  and  $\text{WO}_3$  are distributed all over the surfaces (bare PRD and  $\text{TiO}_2$  particles) exposed. The results shown at Fig. 4 also support the assumption since VWO containing no  $\text{TiO}_2$  particles has a good SCR activity, which means PRD has remarkable surface areas for SCR catalyst of  $\text{V}_2\text{O}_5\text{-}$

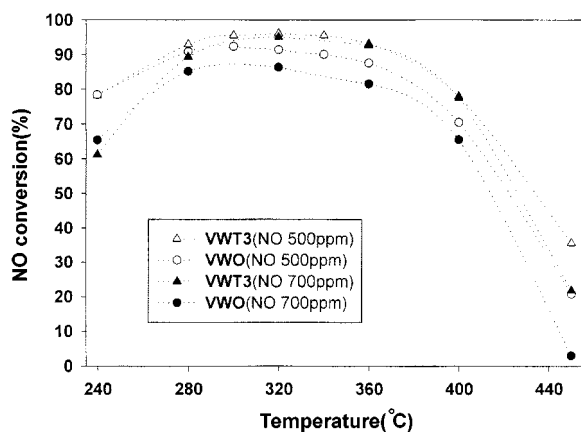


Fig. 4. NO conversion over VWT3 and VWO at different NO concentration.

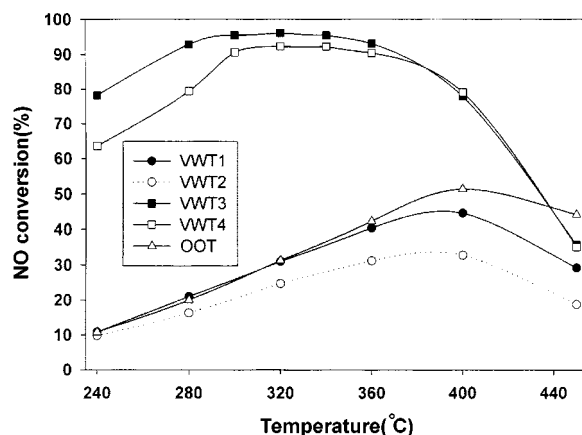


Fig. 5. The effect of V loading on NO conversion over VWT catalytic filters.

$\text{WO}_3$ . However, VWO catalytic filter has a limited activity that is not satisfactory when NO concentration is high as much as 700 ppmv. And the selectivity to  $\text{N}_2$  is relatively low at the high temperature region. Consequently, the PRD-66 filter element additionally supplies the sites for the catalyst supporter.

As the same aspects reported previously about ternary catalyst [Alemany et al., 1995; Topsøe et al., 1995], the pattern of the reaction over the catalytic filter was considerably affected by the V loading. Fig. 5 shows that VWT1 and VWT2 with the low load of V and W atoms present very low NO conversion that is lower than that of OOT. The loadings of V and W atoms in these two catalysts are less than 1.5% w/w with respect to  $\text{TiO}_2$  and less than 0.01% w/w with respect to the PRD. Topsøe et al. [1995] reported that more than 2% w/w of  $\text{V}_2\text{O}_5$  over  $\text{TiO}_2$  is required for the monolayer coverage of  $\text{V}_2\text{O}_5$  on  $\text{TiO}_2$  (BET surface area is  $90 \text{ m}^2/\text{g}$ ). Alemany et al. [1995] also reported that V and W cover 23 and 69% only for the high load catalyst of  $\text{V}_2\text{O}_5(2.56)\text{-WO}_3(9)/\text{TiO}_2$  (BET surface area is  $90 \text{ m}^2/\text{g}$ ). They reported that pure  $\text{TiO}_2$  represents the Lewis acid sites ( $\text{Ti-OH}$ ) and  $\text{V}_2\text{O}_5$  generates Brønsted acid sites ( $\text{V-OH}$ ) known as the active sites for NO reduction. The reason why catalysts prepared with dry impregnation show higher NO reduction than those prepared with wet impregnation is high catalyst loading. Fig. 5 also shows that VWT2 and VWT4 have lower activity than VWT1 and VWT3 fabricated with dry co-impregnation method, containing a corresponding amount of V and W, respectively. VWT2 and VWT4 were prepared by V impregnation over the  $\text{WO}_3\text{-TiO}_2/\text{PRD}$  preliminarily prepared by the impregnation with ammonium paratungstate oxalic solution. Several catalytic filters fabricated by this procedure, not mentioned in this paper, always showed lower activity than a co-impregnated one. This result implies that parts of active sites of  $\text{WO}_3$  preliminarily exposed are lost by the coverage by post-loaded  $\text{V}_2\text{O}_5$ . So the assumption that active sites ( $\text{V}_2\text{O}_5$ ) are formed on the W-modified  $\text{TiO}_2$  surface is not reasonable. Otherwise, the experimental results support the assumption mentioned by Alemany et al. [1996] that the active sites are monomeric isolated V and W oxide centers that strongly interact with each other through the  $\text{TiO}_2$  semiconductor support.

Fig. 6 presents the effect of face velocity on the catalytic activity of VWT3. The face velocity here denotes the total gas flow rate divided by the out layer surface area of the filter element. And it is

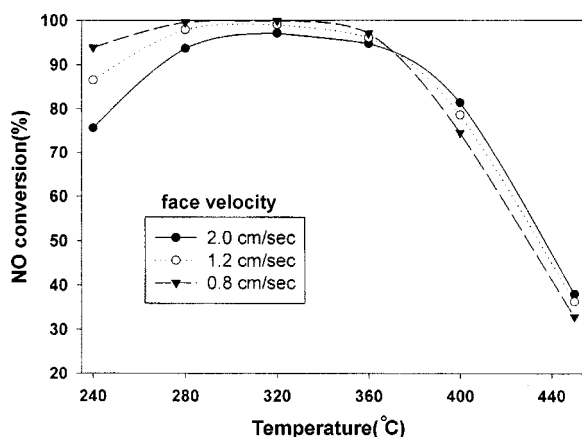


Fig. 6. The effect of face velocity on NO conversion over VWT3 catalytic filter.

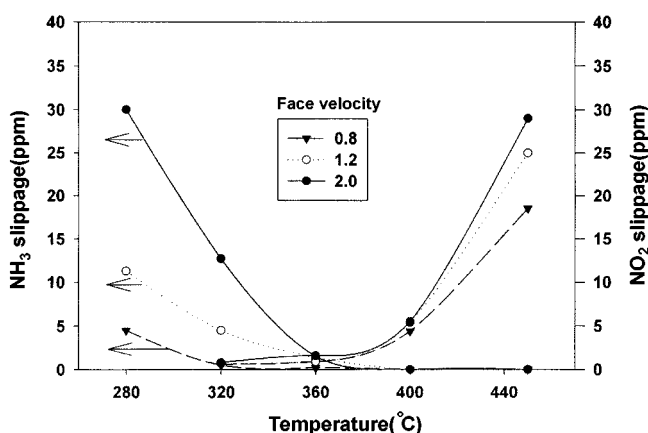


Fig. 7. The effect of face velocity on  $NH_3$  and  $NO_2$  slippage.

a very useful factor to evaluate the dimension of the filter unit. For general commercial filtration using a high temperature filter unit such as the integrated gasification combined cycle (IGCC), it is designed at the value of 0.01-0.03 m/sec, usually 0.012 m/sec. NO conversion over VWT3 reaches near to 100% when the face velocity is lower than 0.012 m/sec. And, in this condition,  $NH_3$  and  $NO_2$  slippages are less than 10 ppmv and 2 ppmv in the temperature range of 290-350 °C, respectively, as shown at Fig. 7. The catalytic filter also presents high activity of more than 95% of NO conversion when NO concentration is 700 ppm as shown at Fig. 4. Considering that NO conversion of currently operating SCR of plate-formed catalyst is about 80%, it is a very encouraging result that catalytic filters show such a high NO conversion.

## CONCLUSIONS

In order to develop an effective catalytic filter based on  $V_2O_5$ / $TiO_2$  supported on a commercial high temperature filter element (PRD-66), the promoting effect by  $WO_3$  was investigated.

PRD-66, known as a good filter element at high temperature, presented good properties for SCR catalyst carrier since it contains a great deal of active surface material such as  $Al_2O_3$ ,  $SiO_2$ , and  $MgO$  whose contribution is remarkable. For additional catalyst supporter,  $TiO_2$  particles were well distributed in the pores of PRD-66 with

particle size of less than 1  $\mu m$  and increased the surface areas to 1.38 from 0.1  $m^2/g$ .

$WO_3$ , in the  $V_2O_5$ - $WO_3$ - $TiO_2$ /PRD-66 catalytic filter system, increased the SCR activity significantly and broadened the optimum temperature window (from 30 to 60 °C) as the similar results reported for  $V_2O_5$ - $WO_3$ - $TiO_2$  ternary catalytic system. The catalytic filter based on the PRD-66 shows the maximum NO conversion of more than 95% for NO concentration of 700 ppm at face velocity of 0.02 m/sec in the temperature range between 290 and 350 °C, which is very promising for the commercialization of this catalytic filter system.

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