



CONVERSION OF NITROGEN OXIDES AND AROMATIC VOLATILE ORGANIC COMPOUNDS ON SCR-DeNO_x COMMERCIAL CATALYST♦

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Abstract: The results of laboratory research work concerning the benzene and nitrogen oxides conversion on commercial catalyst SCR-DeNO_x (V₂O₅-WO₃/TiO₂, with 0.53 %wt. V₂O₅), which is specific to the process of selective catalytic reduction in high-dust system. Experiment was carried-out at temperatures of 573 – 623 K, for low concentration of benzene (50 – 100 ppm C₆H₆) in gas flux at a spatial velocity equal to 6400 h⁻¹. The influence of water vapours (5 % vol. H₂O) and of specific reactants for the SCR-DeNO_x (NO_x, NH₃) process upon benzene conversion in air stream, as well as the influence of benzene on nitrogen oxides conversion were studied. The catalytic oxidation of benzene is partial and occurs with conversion higher than 70 % at temperature of 623 K in the presence of water vapours, catalyst selectivity toward CO₂ formation being equal to 60 %. Presence of benzene had no significantly influence on conversion of nitrogen oxides in selective catalytic reduction reaction.

Keywords: VOC, benzene oxidation, NO_x reduction, V₂O₅-WO₃/TiO₂, SCR-DeNO_x catalyst

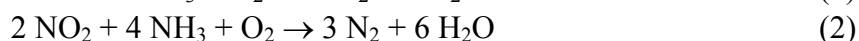
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INTRODUCTION

The volatile organic compounds (VOC) are an important class of environmental pollutants that may result from municipal and medical waste incineration alongside of major pollutants as NO_x, SO₂, HCl, HF, CO and dust. These compounds belong to the category of non-halogenated or halogenated aromatic hydrocarbons (benzene, benzo-pirene, dibenzo-antracene, penta-chloro-benzene, hexa-chloro-benzene, polychlorinated dibenzo-dioxins (PCDDs) and dibenzo-furans (PCDFs), polyhalogenated biphenyl's etc.). The dioxins and furans are very toxic, carcinogenic and environmentally persistent organic pollutants [1, 2].

As a result of strong pollutant behaviour and also to their significant impact on human health, European legislation specific to waste incineration [2], established a maximum admissible concentration in total organic carbon equal to 10 mg C/Nm³ in gas emission, while for dioxins and furans, a limit value of 0.1 ng I-TEQ/Nm³ (International Toxicity Equivalents) is imposed.

Generally, in the case of technological stationary sources, gases with nitrogen oxides content (approx. 95 % NO and 5 % NO₂ [3]) are purified through selective catalytic reduction (SCR) in the presence of ammonia, injected in treated gas that contains oxygen in excess [3 - 5]. Typical reactions for SCR-DeNO_x process are the following:



The used catalyst named commercial SCR-DeNO_x (V₂O₅-WO₃/TiO₂) catalyst consists in vanadium pentoxide (active component) and wolfram trioxide (promoter) deposited on titanium oxide, anatase form, support. Catalytic activity is due to the presence of Lewis (V⁵⁺=O) and Brönsted (V⁵⁺-OH) types acid centres [3, 6].

For a better efficiency of SCR process and implicit for not catalysing the oxidation reaction from SO₂ to SO₃ and the production of N₂O, the catalyst has a low content of active component (< 1 %wt. V₂O₅ [6]). At the same time, in order not to occur the thermal deactivation of the catalyst by support sintering (TiO₂ - anatase → TiO₂ - rutile), working temperature for the SCR-DeNO_x process is ranged between 573 and 673 K [5, 7].

Some authors demonstrated that, in the presence of V₂O₅-WO₃/TiO₂ catalysts, oxidation of volatile organic compounds [8 - 12] and even of dioxins and furans [13 - 17] may take place. In the case of catalysts with low content of active component (under 3 %wt. V₂O₅) and temperatures lower than 773 K, oxidation of volatile organic compounds may be partial, forming burning products (CO₂, H₂O, CO) and other organic derivatives (phenols, aldehydes, ketones, organic acids) that exhibit a higher or lower pollutant behaviour in relation with environment [8, 10, 15 - 17].

The possibility to oxidise the aromatic volatile organic compounds on typical SCR catalysts constitutes the starting point for the study presented in this paper.


Considering benzene as model molecule for aromatic VOC, the experimental data regarding its conversion on a SCR-DeNO_x commercial catalyst with low content in active component (0.53 %wt. V₂O₅) are presented. Two aspects were analysed in this study: a) influence of gas composition (air, water vapours, NO_x, NH₃) on benzene catalytic conversion; b) influence of presence of benzene on NO_x catalytic conversion.

EXPERIMENTAL

The laboratory tests regarding the benzene and nitrogen oxides conversion were carried out in the presence of V_2O_5 - WO_3 / TiO_2 (0.53 %wt. V_2O_5), which is specific to the process of selective catalytic reduction SCR in high-dust system. The high-dust system can be applied when the dust and SO_2 content in the waste gas are sufficiently reduced to allow the direct feed of the waste gas into the SCR-DeNO_x reactor [17].

The SCR-DeNO_x catalyst was used in its commercial monolith form (IHI – Japan), prepared by co-precipitation and possessing straight channels by square section. The main characteristics of the catalyst are presented in table 1. The catalytic element used in experiments consisted in 3 x 3 channels, having a length of 11 cm and a volume equal to 27.634 cm³.

Table 1. Characteristics of the commercial catalyst SCR-DeNO_x (V_2O_5 - WO_3 / TiO_2)

Catalyst commercial characteristics	Value	Monolith form
V_2O_5 , %wt.	0.53	
WO_3 , %wt.	7.7	
CaO, %wt.	1.4	
$Na_2O + K_2O$, %wt.	0.09	
Pore specific volume, cm ³ /g	0.3 ± 0.05	
BET surface area, m ² /g	75 ± 15	
Specific geometric surface area, m ² /m ³	555.5	
Pitch, mm	4.95	
Wall thickness, mm	1 – 1.3	
Maximum temperature for catalyst use, K	693	

The catalytic oxidation reaction of benzene was conducted in air stream, both in the presence and absence of water vapours (5 %vol. H_2O) at temperatures of 573 – 673 K, for low concentration of this one (50 – 100 ppm C_6H_6) in the gas flux. A ratio of specific reactants for the SCR-DeNO_x process (NH_3/NO_x) in the range 0.75 – 1.125 (450 ppm NH_3 /600 ppm NO_x and 450 ppm NH_3 /400 ppm NO_x , respectively) was used. The gas spatial velocity has a value of 6400 h⁻¹, corresponding to an initial volume flow equal to 0.177 Nm³/h.

Experimental data concerning gas concentration before and after the catalytic reaction were achieved on a laboratory set-up operated in continuous regime. Working procedure for achievement of experimental data is presented in figure 1.

Catalytic reactor consists in two concentric quartz tubes (diameters of 40 x 1.5 mm, 32 x 1.5 mm, respectively), placed into an electrically heated oven. The incoming simulated gas flue gas was heated in the outer ring cylinder to the required reaction temperature before its flow was reversed and returned in the inner cylinder of the reactor, where is inserted the honey comb shaped SCR-DeNO_x catalysts test element. Inside the monolith catalyst from the reactor, a thermo-element (Ni-Cr-Ni) was inserted, which was connected to a computer, the reaction temperature being, thus, monitored on-line.

A detailed substance specific quantitative and qualitative analysis of the hydrocarbons in the gas stream was obtained by gas chromatography analysis. Gas samples were

taken at temperature of 322 K with a flow of 0.2 L/min, after a previous separation of water vapours in adsorber filled with NaOH pellets (from Merck/Germany). About 0.2 L/min of the sample cleaned through the above mentioned procedure gas were fed to a Carbotrap 349 (Supelco/USA) adsorption tube kept at 322 K to adsorb the hydrocarbons. The adsorbed hydrocarbons have been desorbed in a Turbo Matrix ATD (Perkin Elmer/USA) thermo-desorber and fed to an Auto System XL (Perkin Elmer/USA) gas chromatograph equipped with flame ionization detector (FID, 450 mL/min synthetic air and 45 mL/min H₂), and a electron capture detector (ECD, 40 mL/min N₂). The separation was achieved in a 60 m, 0.32 mm inner diameter capillary column with a 5 µm PE-1 film, helium carrier gas and the temperature programme of 1 min/323 K, 293 K/min to 523 K and 19 min/523 K.

Calibration standards have been prepared by dissolving the hydrocarbons in 1-heptanole and injecting an appropriate µL volume into a Carbotrap tube and submission of these tubes to the thermo desorption gas chromatographic analytic procedure. The specific reactivities necessary for gas chromatography calibration and for conducting the experiments were provided by Merck/Germany.

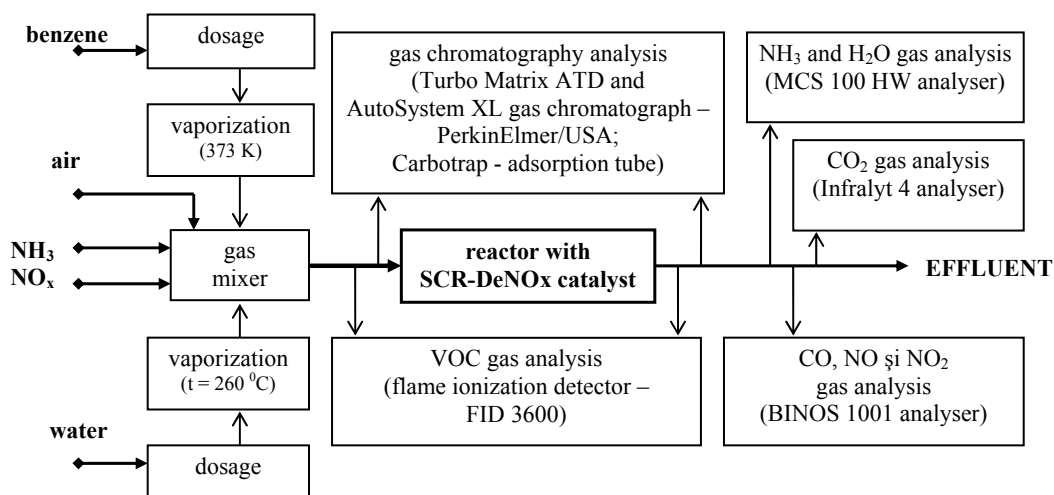


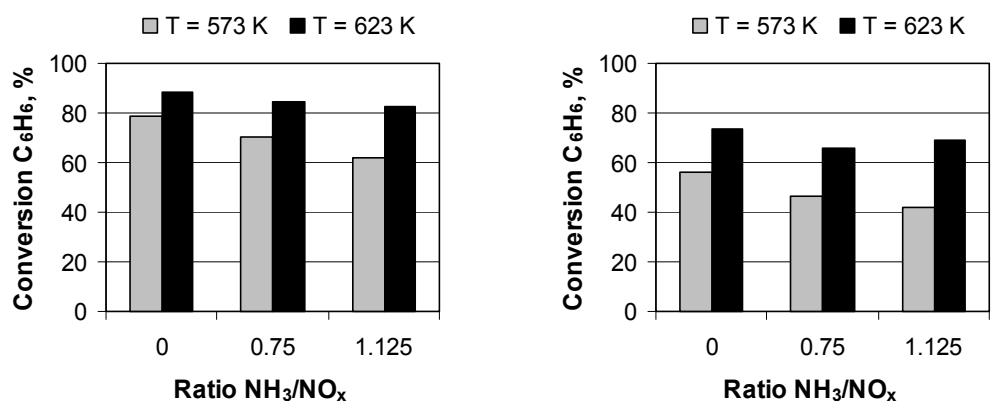
Figure 1. Working procedures for achievement of experimental data

The flame ionization detector FID Modell 3600 from Bernath Atomic/Germany was used to determine the concentration of organic compounds in propane-carbon equivalents in the influent and effluent gases in the flow reactor. A continuous non-dispersive Binos 1001 infrared analyser (Rosemount/Germany) to determine the CO, NO, NO₂ and an Infralyt 4 (VEB Junkalor/Germany) for determining the CO₂ concentration of the gases were used. The gas sample stream was dried in a gas cooler at 277 K before measurement in the continuous analysers. Appropriate standard gases were used to calibrate the instruments. For calibration purposes reference gases with 774 ppm CO₂ (±2%) in nitrogen and 400 ppm CO (±2%) in nitrogen, 628 ppm NO nitrogen and 89.1 ppm NO₂, respectively, in synthetic air from Linde/Germany were used during the investigation. The photo-spectrometer MCS 100 HW analyser (Perkin Elmer/USA) was used to determine the NH₃ and H₂O vapours.

RESULTS AND DISCUSSION

The influences of temperature and gas composition on benzene and nitrogen oxides conversion within the reduction reaction in the presence of ammonia, using SCR-DeNO_x (0.53 % wt. V₂O₅) commercial catalyst were investigated. In another study [18] was demonstrated that variation of benzene concentration within the range 10 – 140 ppm C₆H₆, does not influence the catalytic conversion. Thus, in the case of benzene catalytic oxidation in humid air stream (5 % vol. H₂O), at a temperature of 523 K, catalytic conversion was maintained around value of 40 %, for 100 hours. Conversion is defined by the ratio between the amount of transformed reactant and initial amount of reactant subjected to catalytic transformation.

Benzene conversion in oxidation catalytic reaction increases with temperature and is influenced by gas composition (figure 2).



a) $\text{air} + \text{C}_6\text{H}_6 + \text{NH}_3 + \text{NO}_x$

b) $\text{air} + \text{C}_6\text{H}_6 + \text{NH}_3 + \text{NO}_x + \text{H}_2\text{O}$

Figure 2. Benzene conversion in oxidation reaction on SCR-DeNO_x (0.53 % wt. V₂O₅) commercial catalyst as function of gas composition (air, 5 % vol. H₂O; 450 ppm NH₃/600 ppm NO; 450 ppm NH₃/400 ppm NO)

Benzene catalytic oxidation in air stream, in the absence of humidity occurs with conversions above 75 %, at temperature of 573 K, reaching almost 90 % at temperature of 623 K. Presence of water vapours in a concentration of 5 % vol. H₂O into the gas flux has a negative influence on benzene conversion, which is diminished with 15 % (at 623 K) up to 25 % (573 K) toward the value obtained in the case of oxidation in the absence of these ones.

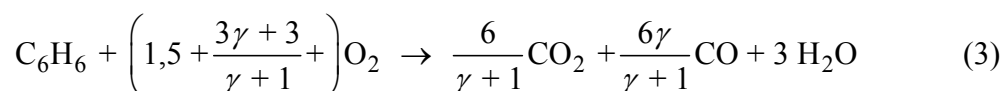
Benzene catalytic oxidation in the presence of SCR-DeNO_x (0.53 % wt. V₂O₅) commercial catalyst is incomplete, apart of carbon dioxide, carbon monoxide being formed. Analysis of gas composition after benzene catalytic oxidation reaction in air stream in the absence and presence of water vapours (5 % vol. H₂O), respectively, allowed the assessment of catalyst selectivity toward formation of carbon dioxide and degree of benzene transformation in burning products CO and CO₂, respectively. In table 2 the influence of water vapours on efficiency of benzene oxidation process on SCR-DeNO_x commercial catalyst, at temperature of 573 K, is presented.

Although the water vapours decrease the benzene conversion, their presence into the system is beneficial as a result of the fact that they enhance the catalyst selectivity toward the oxidation with CO₂ forming. In this case, the selectivity reaches approximately 60 %.

Table 2. Mass balance for benzene oxidation reaction on SCR-DeNO_x (0.53 %wt. V₂O₅) commercial catalyst, at temperature of 573 K

Oxidation conditions	$c_{C_6H_6}^0$ ppm	$x_{C_6H_6}$ %	$S_{CO_2/CO}$ %	$\frac{c_{CO_2}}{c_{C_6H_6}^0}$ mol/mol	$\frac{c_{CO}}{c_{C_6H_6}^0}$ mol/mol	$\eta_{C_6H_6}$ %	CO+CO ₂ in gas %
air	89.1	77.5	52	1.89	1.72	60.1	77.5
air + H ₂ O	94.5	56.3	59	1.52	1.06	42.9	76.2

From mass balance one may observe that carbon oxides (CO + CO₂) sum represents above 75 % from the total of products resulted in benzene catalytic oxidation reaction, which may be written in the global form:



where γ is the molar ratio between CO and CO₂. Concordant to this one, the molar balance achieved in the basis of the data from table 2 presents an error of 2 – 4 % in the case of oxygen, in accordance with experimental results. Oxidation reaction occurs with heat release, but the system is maintained in autothermal regime. In the case of occurrence of benzene total oxidation reaction in adiabatic regime, at a concentration of 50 ppm, temperature variation is equal to 2.6 degrees, conversion having a value of 50 %, at temperature of 523 K.

Presence of reactants specific to SCR-DeNO_x (NO_x and NH₃) process negatively influences benzene conversion, regardless of the fact that exist water vapours or not into the system. However, in these conditions, benzene conversion is higher than 60 %. For a ratio NH₃/NO_x = 0.75 a 5 % up to 10 % decrease of benzene conversion toward the value obtained in the absence of reactants specific to SCR-DeNO_x process, was observed. Utilization of a ratio NH₃/NO_x above one, determines a more accentuated decrease of benzene conversion: from 5 % (623 K) up to 17 % (573 K).

In order to explain the decrease of benzene conversion in the presence of reactants specific to SCR-DeNO_x process, their influence was studied separately. In figure 3 is presented the influence of nitrogen oxides and ammonia on benzene conversion in catalytic oxidation reaction in humid air stream. Through reversible tests concerning the benzene oxidation reaction, both in the absence and presence of nitrogen oxides or ammonia, returning to initial value of conversion obtained in absence of these ones was observed.

If the presence of nitrogen oxides does not influence benzene conversion, ammonia, together with water vapours, have significant influence on it. In this case, at temperature of 573, benzene conversion reaches up to 10 %. This behaviour is due to ammonia adsorption on active centres, which, in the absence of NO_x is not followed by desorption stage, affecting, thus, the normal regeneration cycle of active centres in

accordance to SCR-DeNOx process cycle, proposed by Topsøe [3]. Accordingly, in this mechanism, the catalytic activity is found to be related to the ammonia adsorbed on the Brönsted acid site associated with V^{5+} -OH sites. $V^{5+}=O$ groups are also involved in the reaction and specifically in the activation of adsorbed ammonia. This activation process involves the transfer or partial transfer of hydrogen from the NH_3 molecule and sites accordingly, reduced V^{4+} -OH sites are produced. Once ammonia has been activated NO from the gas-phase reacts with the activated ammonia complex leading to the formation of an intermediate that further decomposes to nitrogen and water. Regeneration of the active sites (oxidation of the reduced V^{4+} -OH sites to $V^{5+}=O$ groups) occurs by gas-phase oxygen.

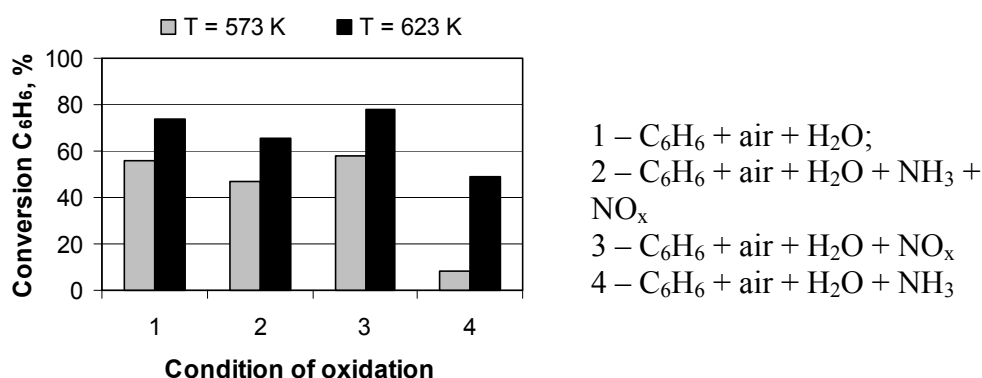


Figure 3. Influence of NO_x (600 ppm) and NH_3 (450 ppm) on benzene conversion in oxidation reaction in humid air stream (5 % vol. H_2O) in the presence of SCR-DeNOx (0.53 % wt. V_2O_5) commercial catalyst

Study regarding the behaviour of nitrogen oxides in selective reduction reaction, in the presence of ammonia, on SCR-DeNOx (0.53 % wt. V_2O_5) commercial catalyst resulted in conclusions that the value of NO_x conversion depends on the ratio NH_3/NO_x and is not significantly influenced by variation of temperature and the presence of water vapours or benzene (figure 4). Consequently, may be considered that, in the presence of SCR-DeNOx commercial catalyst, benzene oxidation occurs in parallel with the process of selective reduction reaction of nitrogen oxides.

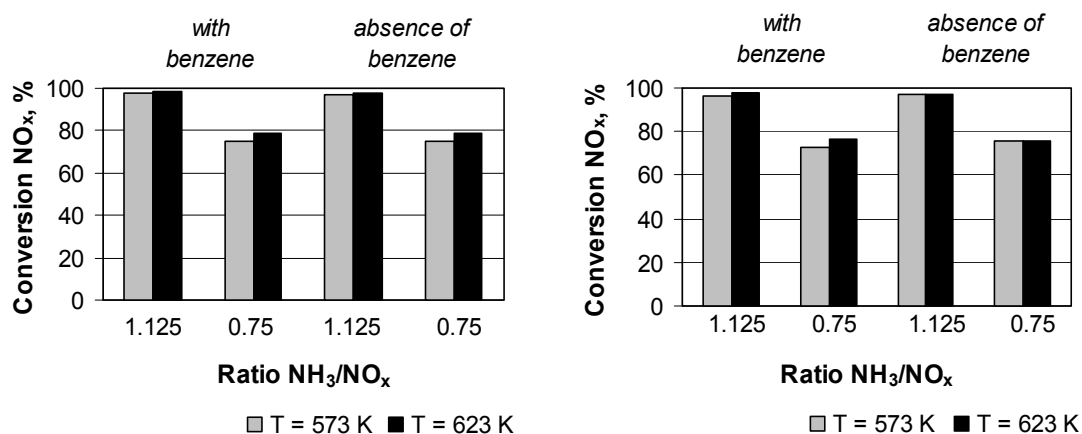
Decrease of benzene conversion in oxidation reaction when reactants specific to SCR-DeNOx process exist can be explained on the basis of competition between the two processes that may take place in the presence of active centres of the catalyst: NO_x reduction and benzene oxidation. In the absence of nitrogen oxides, ammonia adsorbed on Lewis ($V^{5+}=O$) acid centres leads to blocking of these ones. The presence of water vapours results in hydrolysing of V_2O_5 - WO_3/TiO_2 catalyst surface and, as a consequence, Lewis active centres lead to regeneration of Brönsted active centres ($V^{4+}=O$), responsible for selective reduction reaction of nitrogen oxides [3]. From here, one may conclude that Lewis active centres are those important for oxidation of volatile organic compounds.

Concordant to the literature [3, 19, 20] and in the basis of obtained experimental results concerning benzene catalytic conversion on SCR-DeNOx (V_2O_5 - WO_3/TiO_2) commercial catalyst, the relevant reaction mechanism is based on two steps:

- a) in the first step, the benzene oxidation on $V^{5+}=O$ occurs, leading to o $V^{4+}=O$ reduced species



- b) in the second step, these $V^{4+}=O$ species are re-oxidized by O_2 from the gaseous stream



a) air + NH_3 + NO_x

b) air + NH_3 + NO_x + H_2O

Figure 4. Conversion of nitrogen oxides on SCR-DeNOx (0.53 % wt. V_2O_5) commercial catalyst in function of gas composition (air, 5 % vol. H_2O ; 60 – 75 ppm C_6H_6 ; 450 ppm NH_3 /600 ppm NO ; 450 ppm NH_3 /400 ppm NO).

CONCLUSIONS

For temperatures ranged between 573 and 623 K, benzene catalytic oxidation in humid air stream on SCR-DeNOx (0.53 % wt. V_2O_5) commercial catalyst occurs with conversions above 80 %. The oxidation reaction is partial, with formation of CO and CO_2 , the selectivity toward carbon dioxide formation being of 60 %. Presence of water in initial gas reduces benzene conversion at temperature of 623 K with almost 15 %, but is favourable to CO_2 formation in detriment to CO.

Benzene conversion decreases in the presence of reactants specific to SCR-DeNOx process. At temperature of 623 K, benzene oxidation in humid air stream (5 %vol. H_2O), parallel to SCR-DeNOx reaction conducted for the molar ratio NH_3/NO_x equal to 0.75, takes place with conversion higher than 60 %. These values are with approximately 10 % lower than benzene conversion obtained in the absence of nitrogen oxides and ammonia.

Results demonstrate that NO_x selective catalytic reduction on SCR-DeNOx (0.53 % wt. V_2O_5) commercial catalyst can occur in parallel to benzene oxidation reaction, the presence of the latter not diminishing the efficiency of nitrogen oxides reduction. Nitrogen oxides conversion reaches the maximum value in function of molar ratio NH_3/NO_x .

Competition between benzene oxidation process and nitrogen oxides selective catalytic reduction may be attributed to activities of the Lewis acid centres of the SCR-DeNO_x catalyst that, in the presence of water vapours, lead to formation of Brönsted acid centres, responsible for nitrogen oxides conversion, but, on the other hand, may participate in benzene oxidation reaction.

At temperatures higher than 573 K, catalytic oxidation of aromatic volatile organic compounds, found in low concentrations in stationary technological gas emissions, using SCR-DeNO_x (V₂O₅-WO₃/TiO₂, with 0.53 % wt. V₂O₅) commercial catalyst constitutes a viable alternative for their treatment with the aim of environmental pollution reduction.

NOTATIONS

$x_{C_6H_6} = (c_{C_6H_6}^0 - c_{C_6H_6}) / c_{C_6H_6}^0$	– conversion of benzene;
$S_{CO_2/CO} = c_{CO_2} / (c_{CO} + c_{CO_2})$	– catalytic selectivity in the oxidation reaction;
$\eta_{C_6H_6} = (c_{CO} + c_{CO_2}) / (6 \cdot c_{C_6H_6}^0)$	– degree of benzene transformation;
$c_{C_6H_6}^0$	– inlet concentration of benzene, ppm;
$c_{C_6H_6}, c_{CO_2}, c_{CO}$	– concentration of gas component <i>i</i> (C ₆ H ₆ , CO ₂ , CO), ppm;
$SV = V_g^0 / V_{cat}$	– space velocity, h ⁻¹ ;
$V_{cat} = L \cdot b \cdot h$	– geometric catalyst volume, m ³ ;
L, b, h	– geometrical dimensions of monolith catalyst, m;
V_g^0	– inlet flow rate, Nm ³ /s.

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