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Cordierite-like mixed oxide monolith for ammonia oxidation process

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ABSTRACT

The paper deals with modeling of a two-stage catalytic system for ammonia oxidation in an UKL-7 plant, preparation and characterization of cordierite-like honeycomb monoliths (~230 cpsi) modified with (Mn, Co, Fe, V, Bi)O_x oxides as the secondary catalysts. The NO yield for as prepared monoliths was shown to depend not only on the testing temperature, nature and content of the active oxides but also on the cordierite crystallinity degree and on the inlet NH_3 concentration. The best data on NO yield (>80%) at 880–900 °C were obtained with the iron and cobalt based modified monoliths used as the secondary catalysts in the two-stage catalytic system. The data obtained by modeling indicated the possibility to use 8 (instead of 12) gauzes and one layer of as prepared cordierite-like catalysts in the UKL-7 plant to provide the NO yield typical of 12 gauzes.

1. Introduction

The process of ammonia oxidation in nitric acid production ($4NH_3 + 5O_2 \rightarrow 4NO + 6H_2O$) is realized on Pt gauzes at high temperatures at atmospheric, medium and high pressure. During the process, Pt loss is observed. At that, the last gauzes in the catalyst layer polish ammonia conversion while still lose a sound portion of noble metals. Therefore, an idea to replace them by less expensive oxide catalyst is evident. To decrease Pt losses a two stage catalytic system (1 gauze + layer of pelletized oxide catalyst) for atmospheric pressure processes was developed by GIAP in 1960's [1]. In high pressure processes, honeycomb catalysts seem preferable due to more uniform gas flow, low pressure drop and low dust formation. In 1990's a honeycomb catalyst to be used in the high pressure plant UKL-7 was developed at the Boreskov Institute of Catalysis [2]. The BIC-42-1 catalyst is a 70 mm × 70 mm × 50 mm monolith with square channels (5 mm × 5 mm) and wall thickness 1.8–2.2 mm. Substitution the monolith layer for three of 12 gauzes (9 gauzes and one layer) resulted in 15–20% reduction of Pt losses at near the same NO yield and in a longer gauze life time [3].

To decrease the Pt loading and losses needs the monolith with a higher channel density to ensure the high selectivity to NO. Again, very important properties of the catalyst are mechanical strength and thermal shock resistance.

Against oxide catalysts, cordierite-like materials are characterized by a low thermal expansion coefficient and, as a result, by the catalyst stability. Good rheological properties of the raw materials make it possible to prepare monoliths with high channel density. Modification of the material with oxides which are most active to ammonia oxidation (Mn, Co, Fe, V, Bi)O_x looks very promising for preparation of the honeycomb monoliths for ammonia oxidation process.

This paper deals with modeling of a two-stage catalytic system with a smaller number of gauzes, preparation and characterization of modified cordierite-like monolith with high channel density and high NO selectivity to obtain high NO yield in ammonia oxidation process.

2. Experimental

2.1. Mathematical modeling

Two-phase mathematical models were applied for simulation of the two-stage catalytic systems consisting of **n** Pt gauzes and **m** layers of honeycomb catalyst. Approximation of mass transfer limitation of the reaction both on Pt gauzes and on a honeycomb catalyst was assumed and well known correlations for mass transfer coefficients used.

Conversion of NH_3 on one woven Pt gauze may be calculated by formula (1) [4]:

$$X_g(1) = 1 - \exp\left[-2S_g d_g \frac{\beta_*}{U_{sup}}\right], \quad (1)$$

where β – effective mass transfer coefficient, m/sec; U_{sup} – superficial velocity, m/sec; $S_g = \pi/h$– gauze catalyst specific area, m^2/m^3; h – gauze network step, m; d_g – diameter of catalytic gauzes, m.

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Effective mass transfer coefficient β_* can be calculated based on the analogy between heat- and mass transfer processes in accordance with correlation for dimensionless mass transfer coefficient (the Sherwood number) Sh_* for ensembled cylinders (2) [4]:

$$Sh_* = 0.3 + 0.664 \cdot Re_*^{0.5} \cdot Sc^{0.33}, \tag{2}$$

where $Re_* = U_\varepsilon\, d_g/\nu\,(\pi/2)$ – modified Reynolds number; $U_\varepsilon = U_{sup}/\varepsilon$ – interstitial velocity, m/sec; $\varepsilon_g = 1 - \pi/(4h/d_g)$ – gauze pad porosity; ν – dynamic viscosity coefficient, m^2/sec; Sc – Schmidt number.

The conversion of NH_3 in one monolith channel may be calculated by formula (3):

$$X_h(1) = 1 - \exp\left[-\frac{S_h}{U_{sup}}\int_0^L \beta(l)dl\right], \tag{3}$$

where β – local mass transfer coefficient, m/sec; S_h – monolith catalyst specific area, m^2/m^3; U_{sup} – superficial velocity, m/sec; L – channel length, m; l – axial coordinate, m.

Local mass transfer coefficient $\beta(l)$ was defined in accordance with expression of correlation for dimensionless local mass transfer coefficient (the Sherwood number) Sh (4) [5]:

$$Sh(l^*) = Sh_\infty + 6.874(10^3 l^*)^{-0.486}\exp(-57.2 l^*), \tag{4}$$

where l^* – dimensionless axial coordinate.

A total of NH_3 conversion over a multilayer uniform catalyst (monolith or gauzes) may be calculated by expressions (5) and (6):

$$X_g(n) = 1 - (1 - X_g(1))^n; \tag{5}$$

$$X_h(m) = 1 - (1 - X_h(1))^m. \tag{6}$$

The following experimental conditions were used for the mathematical modeling: an UKL-7 plant at pressure 7 atm, temperature 890 °C, linear velocity 5.26 m/s (operation conditions), woven gauzes with diameter $d_g = 0.092$ mm, $h = 10/32$ mm. There were regular triangle channels (asymptotic value Sh_∞ is equal to 2.470) in the monoliths of 50 mm height. We supposed that NO yield on 1 gauze (S_g) was equal to 0.92. The NO yield on the honeycomb monolith (S_h) varied in the range of 0.7–0.9. The data on NH_3 conversion and NO yield were calculated for different compositions of the two-stage catalytic system.

2.2. Catalyst preparation

A series of frame structured modified cordierite-like $2(Mg,Me)O\cdot 2Al_2O_3\cdot 5SiO_2$ honeycomb catalysts were prepared from natural components (talc, kaolin, silica) and Fe_2O_3, Co_3O_4, Mn_2O_3, V_2O_5 and Bi_2O_3 oxides. The mixture of raw materials taken in a stoichiometric ratio was treated in a disintegrator DEZI-15. Then powders were placed in a Z-mixer and, after water adding (24–26% humidity) and mixing during 30–40 min, a uniform plastic pastes were formed. Monoliths were prepared by extruding the pastes as hexahedral prism with a triangle channels side of 2.5 mm and wall thickness of 0.4 mm. The monoliths were first dried at room temperature for 24 h, than at 350 °C for 4 h and at 1000–1200 °C for 4–8 h. There were 230 channels per square inch in the calcined monoliths (Fig. 1).

2.3. Catalyst characterization

The structural and textural features of frame structured cordierite-like catalysts were studied by XRD, SEM, X-ray microanalysis, thermal analysis and adsorption measurements.

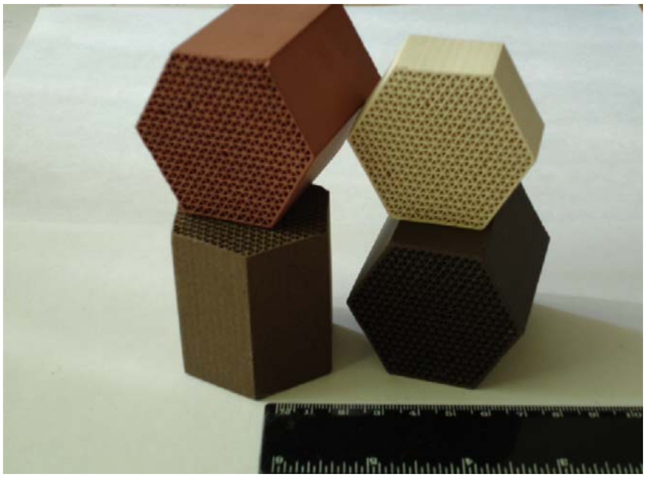

Fig. 1. Frame structured honeycomb monolith catalysts.

The X-ray diffraction patterns were acquired with a URD-63 diffractometer using CuK$_\alpha$-radiation. The 2θ scan region was 5–70°. The cordierite formation was controlled based on intensity of $2\theta = 10.4°$ peak.

The textural features of cordierites were studied by SEM with a JEOL JSM-6460LV machine equipped with X-ray microanalysis.

The pore structure was characterized by the high pressure mercury porosimetry (HPM) using an Auto-Pore 9200 machine, and the specific surface area (S_{sp}) was determined by the routine BET procedure using the Ar thermal desorption data.

The chemical composition was analyzed using atomic absorption spectroscopy (a BAIRD spectrometer).

The thermal stability of the monoliths was characterized as the resistance to thermal cycles as follows. During the thermal cycle a specimen of the monolith was placed to a muffle furnace (preheated up to 700 °C) for 15 min and kept under the ambient conditions for 15 min.

2.4. Catalyst testing

Ammonia oxidation process was carried out in a bench scale quartz tubular reactor with inner diameter of 26 mm at 800–900 °C and atmospheric pressure. Samples of honeycomb catalysts in the form of fragments with diameter of 21–22 mm and length of 50 mm were tested with and without Pt gauzes. Four platinum 80-mesh gauzes were used as a primary catalyst in the two-stage system. The catalyst gauzes were held in the flange between asbestos gaskets. The gauze temperature was measured by attaching a fine Pt/Pt–10%Rh thermocouple. The honeycomb and the gas (above and below the flange) temperatures were measured with chromel–alumel thermocouples.

Before the reaction started, the catalysts were preheated at 700 °C in air for 30 min. Then reaction gazes (5–10% ammonia in air preheated at 450 °C in a quartz mixer) were passed through the catalyst at 7.6 l/min flow rate (standard gas velocity 0.33 m/s). The ignition (reaction start up) was determined by the catalyst temperature increase. Ammonia, NO and NO_2 concentrations were determined using an on-line UV-spectrophotometer by the procedure described elsewhere [6,7]. Experimental conditions were chosen based on our calculations to achieve 100% ammonia conversion at the diffusion-controlled regime on monoliths with prepared geometry to prevent the side reaction between unreacted ammonia and NO after catalyst and a decrease in the NO yield [8].

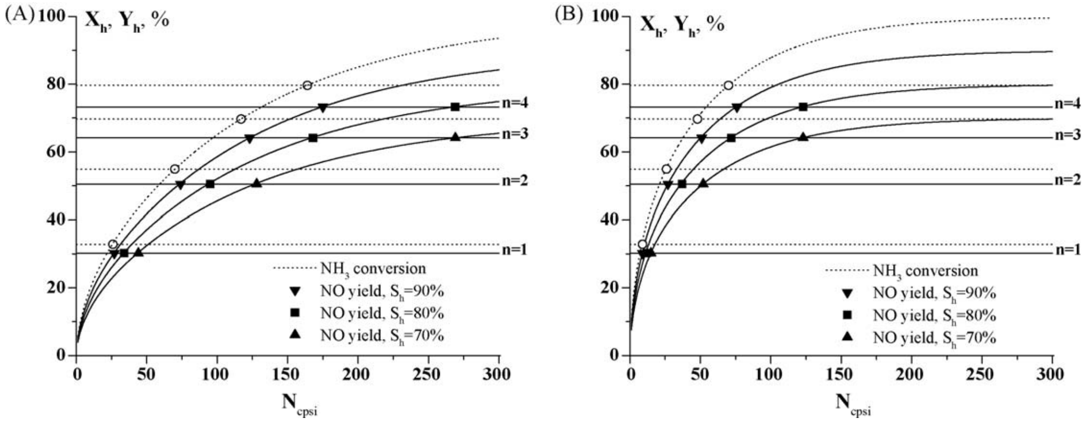

Fig. 2. NH_3 conversion (X_h) and NO yield (Y_h) versus monolith channel density (N_{cpsi}) for monoliths with different selectivity (S_h). (A) One monolith layer; (B) two monolith layers. Horizontal lines demonstrate the NH_3 conversion (dot line) and the NO yield (solid line) on **n** removed gauzes with S_g = 0.92.

3. Results and discussions

3.1. Mathematical modeling

Results of the mathematical modeling are illustrated in Fig. 2 as dependencies of the ammonia conversion and NO yield obtained under the UKL-7 plant conditions over monolith *versus* channel density.

Mathematical modelling demonstrated comparable ammonia conversions as provided by the system of 8 Pt gauzes and one layer of honeycombs (50 mm height and ~130 cpsi) and by 12 woven Pt gauzes. The lower selectivity over honeycomb monoliths may be partly compensated by an increase in the channel density to obtain the same NO yield. The high NO yield (ca. 93% typical of the 12 gauzes system) may be obtained for 8 gauzes and honeycombs with different channel density depending on their selectivity. If the selectivity is about 90%, the monoliths with ~160 cpsi provide usual NO yield. For selectivity 80% the channel density should be increased up to ~250 cpsi. According to the calculations, the NO yield more than 93% may be obtained in the two-stage system consisting of 8 gauzes + 2 layer of oxide catalyst with ~150 cpsi and with selectivity ~80%. Hence, strong requirement for honeycomb catalysts with 150 cpsi is their high selectivity to NO (higher than 90%); for honeycomb catalysts with ~250 cpsi selectivity may be ~80%. According to calculations, it is impossible to replace 4 gauzes by monoliths with 70% selectivity even at $N_{cpsi} \gg 230$ (Table 1).

3.2. The phase composition of frame structured oxides

Figs. 3 and 4 present X-ray data for cordierite-like materials calcined at 1000–1100 °C without modification and with Fe_2O_3 or Co_3O_4 and Bi_2O_3 addition as examples. XRD studies revealed that the cordierite-like frame structure is not fully formed after calcination of treated in the disintegrator raw materials at 1000 °C for 4 h. The admixtures of quartz, mullite and magnesium silicate phases were found. An increase in the calcination time (from 4 to 8 h) or temperature (from 1000 to 1100 °C) leads to better cordierite crystallization, that is seen from the intensity of peak at $2\theta = 10.4°$. The usual ceramic preparation procedure does not lead to cordierite crystallization at 1000 °C. Hence, the preliminary mechanical treatment of the raw materials in the disintegrator considerably reduces the temperature and time of cordierite synthesis during further calcination probably due to not only disintegration (size reduction) effect but also due to the effect of mechanochemical activation as well [9].

The data obtained revealed that modification with 3d-ions (Mn, Co, Fe) promotes cordierite crystallization as well. The single phase cordierite is formed at calcination temperature about 1100 °C, magnesium silicate and individual 3d-oxides being the minor phases.

The highest sintering effect was observed after Bi_2O_3 addition, the amount of cordierite phase was found to increase with an increase in the Bi_2O_3 content. Earlier, Yang also used Bi_2O_3 as a fluxing material to prepare the dense cordierite after 8 h at the calcination temperature 1000 °C [10].

The formation of Bi_2VO_5 phase [JCPDS 47-0734] stable at high temperatures was revealed in the case of cordierite modification with Bi_2O_3 and V_2O_5.

Table 1
Needed monolith channel density (N_{cpsi}) at different monolith S_h for equivalent (on NH_3 conversion and on NO yield) replacement of **n** gauzes in the case of one monolith layer (upper) and two monolith layers (lower, italic).

n	S_h (%)			
	70	80	90	100 (X_h)
1	44	34	27	26
	15	*12*	*9*	*9*
2	128	95	74	70
	52	*37*	*27*	*26*
3	269	168	123	117
	123	*72*	*51*	*48*
4	Unattainable	269	175	164
		123	*76*	*70*

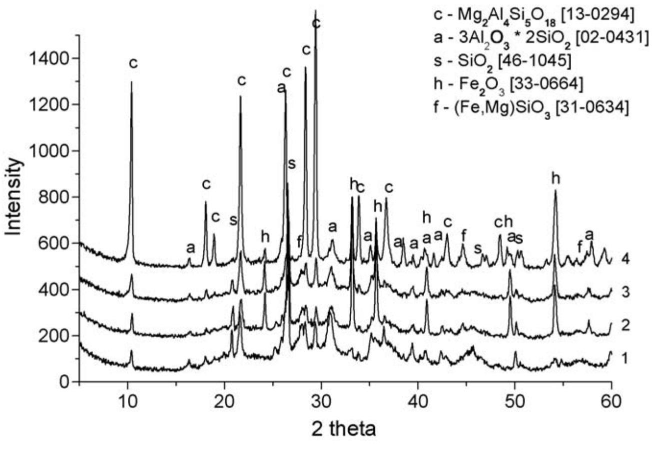

Fig. 3. X-ray data for cordierite-like materials: (1) cordierite calcined at 1100 °C, 4 h; (2) Fe_2O_3 modified cordierite calcined at 1000 °C 4 h; (3) (4) Fe_2O_3 and Bi_2O_3 modified cordierite calcined at 1000 and 1100 °C, 4 h, respectively.

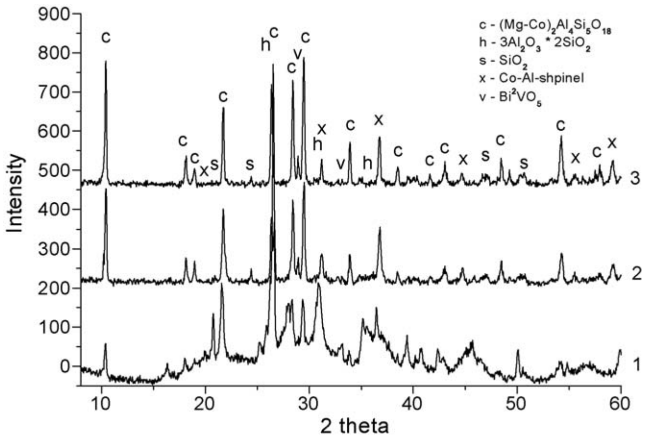

Fig. 4. X-ray data for cordierite-like materials: (1) cordierite calcined at 1000 °C; (2 and 3) Co_3O_4, Bi_2O_3 and V_2O_5 modified cordierite calcined at 1000 °C and 1100 °C, respectively.

3.3. *Texture of frame structured oxides*

BET data of some prepared cordierites calcined at 1100 °C are presented in Table 2. In general, the samples have low specific surface area (0.2–9.5 m^2/g) and it was found that better cordierite formation results in a lower specific surface area. So, elevation of the calcination temperature and modification with 3d-cations (V, Co, Mn, Fe) or addition of Bi_2O_3 that affects the cordierite crystallization leads to a decrease in the specific surface area of the samples. Also monoliths with formed cordierite structure were found to have a good thermal shock resistance and high durability.

SEM data on the prepared materials presented in Fig. 5 indicate that cordierites modified with Fe_2O_3, Co_3O_4 and Bi_2O_3 have low open porosity and the external pore diameter does not exceed 1 μm. Well-developed internal pore structure of cordierite is observed for these samples. The formation of well crystallized cordierite phase with well-defined crystals of cordierite was revealed for V_2O_5 modified materials. The cordierites with high open porosity were produced at 1100 °C in this case that is confirmed by mercury porosimetry measurements presented in Table 2. So, addition of 3d-cations affects the degree of cordierite crystallization and the pore structure as well.

X-ray microanalysis revealed no noticeable surface 3d ions enrichment, all active cations were homogeneously distributed between the bulk and the surface to form different alumosilicates.

3.4. *Catalytic activity*

High temperature ammonia oxidation process occurs in the diffusion limited regime where the reaction rate is limited by ammonia diffusion to the catalyst walls [11]. So, under the same reaction conditions, the NH_3 conversion depends mainly on the monolith geometrical parameters, whereas the NO yield depends strongly on the chemical composition of the catalysts.

The non-modified cordierite shows low NH_3 conversion and NO yield in the ammonia oxidation reaction (Table 2). For all modified

Table 2
Phase composition, specific surface area (S), total pore volume (V), mean pore diameter (d), ammonia conversion (X_{NH_3}) and NO yield (Y_{NO}) at 900 °C ($C^0 \sim 5\%$) for some prepared modified cordierite-like (2Mg, MeO·2Al_2O_3·5SiO_2) honeycomb catalysts calcined at 1100 °C.

Modificating oxides	Phase composition	S (m^2/g)	(V) (cm^3/g)	d (nm)	X_{NH_3} (%)	Y_{NO} (%)
Without modification	Cordierite, $MgSiO_3$, α-SiO_2	4.9	0.194	109	69.0	12.2
MnO_2	Cordierite, α-SiO_2, α-Al_2O_3	2.8	0.176	105	100	43.2
MnO_2 + V_2O_5	Cordierite, mullite, Mn_3O_4	0.2	0.283	1120	100	71.4
Fe_2O_3 + Bi_2O_3	Cordierite, α-Fe_2O_3	1.3	0.187	374	100	41.1
Fe_2O_3 + V_2O_5	Cordierite, α-Fe_2O_3, (Fe,Mg)SiO_3	0.7	0.253	1538	100	74.9
Fe_2O_3 + Bi_2O_3 + V_2O_5	Cordierite, α-Fe_2O_3, Bi_2VO_5	0.4	0.294	1930	100	78.2
Co_3O_4 + Bi_2O_3	Cordierite, Co–Al–spinel	–	–	–	100	34.8
Co_3O_4 + V_2O_5	Cordierite, Co–Al–spinel, α-SiO_2	4.1	0.169	216	100	78.0
Co_3O_4 + Bi_2O_3 + V_2O_5	Cordierite, Co–Al–spinel, Bi_2VO_5	0.9	0.251	1930	100	80.6

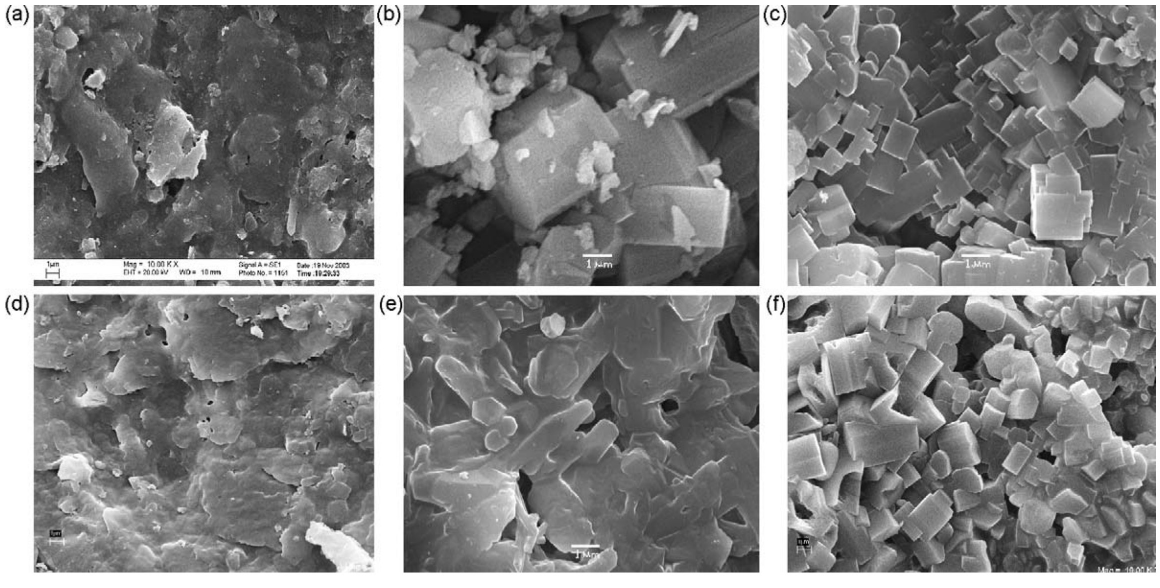

Fig. 5. SEM data for (a) cordierite (1100 °C) and cordierite (1100 °C) modified with (b) Co_3O_4 and V_2O_5, (c) Co_3O_4, Bi_2O_3 and V_2O_5, (d) Fe_2O_3, (e) Fe_2O_3 and Bi_2O_3, (f) Fe_2O_3, Bi_2O_3 and V_2O_5.

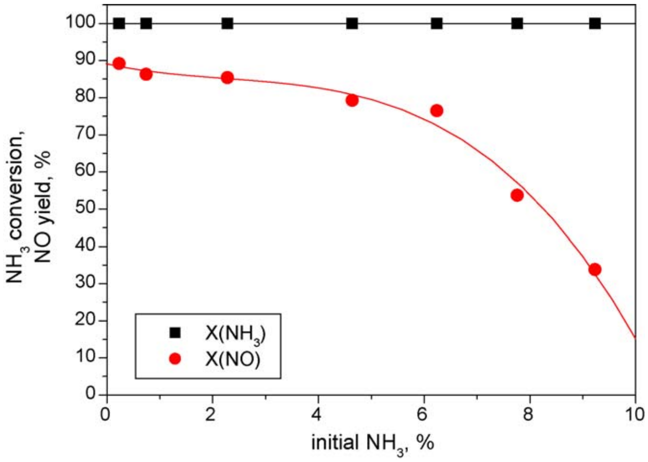

Fig. 6. NH_3 conversion and NO yield at 900 °C versus inlet NH_3 concentration as measured for honeycomb catalyst modified with Fe_2O_3, Bi_2O_3 and V_2O_5.

cordierites, the ammonia conversion at 850–900 °C was found to be 100% that is in a good agreement with our calculations for a lab reactor. The NO yield was shown to depend on the testing temperature, nature and content of active oxides, and crystallinity degree of cordierite. The best data (NO yield ~80%) at 880–900 °C were obtained for iron and cobalt based modified frame-structured monolith calcined at 1100 °C (at 5% NH_3 inlet). Manganese based catalysts show lower, about 70%, NO yield. For comparison, NO yield ~90% at 830–900 °C was detected for BIC-42-1 iron oxide-based monolith prepared with channel density about 180 cpsi [12].

The inlet NH_3 concentration was shown to affect the NO yield as well. Fig. 6 presents the dependence of NO yield on the initial NH_3 concentration on Fe_2O_3, Bi_2O_3 and V_2O_5 modified cordierite-like monoliths. It was found that ammonia conversion was 100% in all cases; however, the NO yield reduced with an increase in the inlet ammonia concentration that could be a result of the side reaction between ammonia and NO in the monoliths channels or during the sampling.

Hence, an important characteristic of catalysts for ammonia oxidation is their operability in a two-stage catalytic system as far as different reaction conditions could occur: the temperature of the inlet gas on the first and second stage could differ significantly. The gas coming to the second stage (honeycombs) consists mainly of NO, NO_2 and water vapor, nitrous oxide and unoxidized ammonia, as well. So, the cordierite catalyst should oxidize the rest NH_3 and do not interact/decompose/ with NO or NO_2.

Data on NH_3 conversion and NO yield for few modified cordierite-like catalysts consisting of two-stage catalytic system are presented in Table 3. All catalysts provide 100% ammonia conversion, no ammonia slip after two-stage catalytic system, and NO yield growth. The NO yield for modified cordierites was found to be somewhat higher as compared with previous experiments. Probably, the low NH_3 concentration at the honeycomb inlet (0.20–0.37%) results in an increase in the NO yield.

The frame structured cordierite ($2MgO{\cdot}2Al_2O_3{\cdot}5SiO_2$) honeycombs are characterized by a low TEC (2×10^{-6} 1/K) and often used as refractory carrier for high temperature catalysts [6,13]. As prepared monoliths were characterized by good durability (no less than 4000 Newton/monolith) and thermocycle stability (more than 12 thermocycles without destruction). The stability of cordierite modified with Fe_2O_3, Bi_2O_3 and V_2O_5 (1100 °C) was checked in 24 h running test as the second stage. After the test neither changes in the geometry nor were cracks observed in the walls. The sample revealed nearly the same NH_3 conversion and NO yield in comparison with the fresh one (Table 3). Further life test in the high pressure ammonia plant is required to confirm the operability of prepared catalysts. In the case of supported catalyst activity after life tests was shown to decrease [6].

Table 3
Data on ammonia conversion (X_{NH_3}) and NO yield (Y_{NO}) on two-stage catalytic system where the 1st stage is four Pt gauzes and the 2nd stage is modified cordierite-like monolith honeycomb catalyst. $C^0 \sim 10\%$.

Modificating oxides	1st stage		2nd stage		Total
	X_{NH_3}	Y_{NO}	X_{NH_3}	Y_{NO}	Y_{NO}
$Co_3O_4 + V_2O_5$	97.4	91.8	100	75.4	93.8
$Co_3O_4 + Bi_2O_3 + V_2O_5$	95.2	91.4	100	81.2	95.3
$Fe_2O_3 + Bi_2O_3 + V_2O_5$	97.2	92.9	100	87.7	95.3
−//− after 24 h	97.6	92.4	100	86.7	94.5

4. Conclusions

The modification of cordierite with 3d active cations (Mn, Fe, Co, V) allowed obtaining cordierite-like honeycomb monoliths to provide a high NO yield in the process of ammonia oxidation. Catalysts texture and cordierite crystalline degree were found to depend on the type and amount of added 3d-oxides and on the preparation conditions. Prepared monoliths with 230 cpsi calcined at 1100 °C have high mechanical strength and thermal stability. Co/Fe–V-containing cordierites were found to be the most promising to provide nearly 90% NO yield in the two-stage catalytic system.

According to our mathematical modeling for UKL-7 plant conditions, the catalytic system consisting of 8 gauzes and one catalyst layer with 50 mm height, 230 spci and nearly 90% NO yield should provide NO yield even more than 93% (typical of 12 gauzes). Taking into account our experience with BIC-42-1 catalyst, more than 4 gauzes may be removed using the prepared cordierite-like honeycomb monoliths because the NO yield under industrial conditions could be higher than calculated due to a better uniformity of the gas flow, gauzes temperature and other additional factors.

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