Experimental Study of Urea SCR Catalyst for Diesel Engine NO_x Abatement

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This paper comparatively describes the studies conducted and the experiments performed on CuZSM5-urea-SCR systems, with a view to lowering NO_x emissions of heavy-duty commercial diesel engines. The innovative outcome of this research is the finding of a relevant indicator for NO_x conversion, depending on the operation modes of the vehicles; this indicator, denominated weighted exhaust gas temperature (WET), predicts NO_x conversion in the SCR catalyst. A copper ion-exchanged ZSM5 zeolite was prepared, using an aqueous solution of $Cu(CH_3COO)_2$ which was deposed on a metallic support of the urea-SCR system. The SCR treatment applied to the exhaust gas emitted by 4-litre heavy-duty diesel engines experimentally revealed a NO_x reduction of 70%, at constant engine speeds and increasing loads, and of and 58.3%, in European Stationary Cycle. Following thorough analysis of the previous research on controlled feed gas reactors and diesel engines, with focus on the differences between NO_x conversions in terms of exhaust gas temperature, space velocity, water content, oxygen content, fuel sulphur content, an indicator of NO_x conversion was proposed, based on the average temperature of the emission test cycle. A practical result of the research work consisted in the compliance of NO_x emission from the tested diesel engine with the standard Euro IV.

Key words: Diesel engine emissions, NO_x conversion, Selective Catalytic Reduction

Diesel engines offer greater advantages than gasoline engines, such as higher efficiency, higher fuel economy and lower CO_2 emissions; however, as regards engine emissions, the abatement technique applied to the gasoline engine (three-way catalyst) is not effective in the case of the diesel engine, as it is difficult to remove the nitrogen oxides (NO_x) from the oxidizing diesel exhaust gas.

Nitrogen oxides is considered a contradictory pollutant: on the one hand, in diesel-engines combustion process, higher values of the NO_x emission generally indicate higher temperatures in the combustion chamber, which leads to increased thermodynamic efficiency and fuel economy; on the other hand, the global environmental problems related to NO_x emissions are utterly challenging for humankind: generation of photo-chemical smog and acid rain, ozone layer depletion and global warming. Furthermore, the basic constituents of NO_x (NO and NO_2) are considered to be toxic; hence a justified concern on air quality arose, which resulted in enforcement of ever more drastic regulations upon these emissions, leading to the introduction in Europe of catalytic reduction upon stationary sources and mobile sources [1-3]. The use of renewable fuels, such as biodiesel, for the operation of diesel engines, is reported in the literature to increase the NO_x emissions [4, 5]. Therefore, the need for an efficient NO_x abatement will be even stronger in the years to come.

Among the methods aiming at the removal of NO_x [6, 7], the classic selective catalytic reduction (SCR) is more efficient than the non-selective catalytic reduction produced by carbon monoxide and unburned hydrocarbons remained in the exhaust gas; the use of urea as reducing agent is preferable to ammonia, the former being nontoxic, dissolvable in water and capable of being stored in a tank. The urea solution is injected in the exhaust gas and ammonia reduces NO_x to N_2. The SCR processes include several steps: at temperatures over 170°C, the aqueous solution of urea evaporates and decomposes in ammonia (1); then, the adsorbed ammonia reacts catalytically with NO and NO_2 in a predominant standard reaction (2) and in a fast reaction (3), both resulting in nitrogen and water.

$$H_2N\text{-}CO\text{-}NH_2 + H_2O = 2\,NH_3 + CO_2 \qquad (1)$$

$$4\,NO + 4\,NH_3 + O_2 = 4\,N_2 + 6\,H_2O \qquad (2)$$

$$2\,NH_3 + NO + NO_2 = 2\,N_2 + 3\,H_2O \qquad (3)$$

The reduction is produced by a catalyst, which can be either a noble metal (Pt), or a metal oxide (vanadium, wolfram, titan [8]), or a metal ion (Cu, Fe, Co)-exchanged zeolite [6, 9]. Among the metal ion exchange zeolites, the copper ion, CuZSM5, proves the best NO-removal properties, within a broad temperature range (180-500°C) [10].

Although extensive studies on CuZSM5-SCR systems have been conducted, the present paper describes the preparation of the catalyst and the achievement of experiments upon an urea-SCR system, which aims at lowering NO_x emissions of a heavy-duty commercial diesel-engine; the aforementioned purpose is underlain by the analysis of the influence exercised by different parameters reported in the literature. The main innovation of this research is the finding of a relevant indicator for the NO_x conversion according to the operation modes of the vehicle; this indicator, called *weighed exhaust gas temperature* (WET) predicts the SCR catalyst NO_x conversion to the driving cycles of the diesel engine.

Experimental part

The experiment included the measurement of the urea SCR activity within the exhaust gas from diesel engines, in real operation modes and not in reactors. The steps of the experiment were: catalyst preparation, construction of the SCR system and NO_x engine testing.

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Catalyst preparation

Based on the literature data, a copper ion-exchanged ZSM5 zeolite was prepared and deposed on a metallic support made of alloyed stainless steel, especially designed for catalysts. The ferritic structure of the steel (Fe-Cr-Al) is very resistant to high temperature corrosion; note that chrome (20 %) and aluminium (5%) increase the sintering resistance of catalytic active layers [11]. The metallic support was preferred to the ceramic one as the former displays higher thermal conductivity, shorter heating time, thinner walls, lower pressure drops, higher resistance to thermal and mechanical shocks.

The catalyst was deposed on two cylindrical monoliths of D=160 mm, H=120 mm, whose volume is 2.4 L for each of them; the cells are triangular, of 2 mm side, 28 cells/cm^2 and 0.05 mm wall thickness, leading to a total active surface of 67.5 m^2.

In order to prepare the catalyst, the cylindrical metallic supports were coated with an active phase consisting of zeolite (69%) and silica (31%). The zeolite ZSM5, delivered by Vega Petrol Processing Plant, has the following characteristics: ratio SiO_2/ Al_2O_3 of 48.97% and ZSM5 crystalline content of 84.28%. The copper ions were introduced in ZSM5 by means of a procedure consisting of ion exchange within an aqueous solution of $Cu(CH_3COO)_2$ 0.06 M, according to the method reported by Iwamoto et al [12]. After this procedure, the catalysts with an excessive load of copper ions have been found to display a higher conversion of the nitrogen oxide to nitrogen, especially at low temperatures. The ion exchange was performed after the coating of the support, at room temperature, for 5 hours. The copper content was afterwards measured by chemical analysis and determined to be 3.63 wt.%. The copper load was controlled while varying the number of ion-exchanged steps. The layer of active phase was made by immersion in suspension, drying (110 °C, 12 h) and calcination (500°C, 5 h), operations which were repeated until the required catalyst mass was obtained (145 g/L active phase or 4.61 g copper /L).

The active layer was analysed according to the following parameters: specific surface, pore volume, radius pore distribution, which are decisive for the catalyst efficiency. The BET (Brunauer-Emmett-Teller) surface area was measured with the static volumetric method using a sorption analyser, based on the amount of N_2 adsorbed at liquid N_2 temperature. The BET surface area and micropore volume were calculated by the plotting method.

SCR configuration

The urea-SCR system was designed based on the parameters of a 3.92 L diesel engine, considering the exhaust engine flow rate measurement with its maximum value of 420 m^3/h [13].

The volume of the catalyst was intended to be 4.8L, *i.e.*1.2 times the engine displacement. By molar calculations, the mass of the aqueous urea solution was established to be 40%, in deionized water, required to be injected to neutralise NO_x. The ratio NO_2/NO_x was approximately 0.1, close to the literature value of 11%, which is in accordance with [14]; the ratio NH_3/NO_x was maintained approximately 1, in stoichiometric conditions, considering that the selectivity of the reducing reactions is 100%. Based on proportionality, the flow rate of the urea solution was determined for every operation mode of the engine, according to the measured engine exhaust flowrate and NO_x concentration. In order to use the SCR on automotive applications, the metering system must be capable of injecting a variable quantity of urea solution, in function of the NO_x concentration from the engine emission map.

The urea-SCR system consists of a urea tank, a metering pump system provided with air-urea mixer and atomiser, and a SCR catalyst. The pump may inject different flowrates of urea, within the range 10-4200 cm^3/h, through a micrometric screw. First, the metering urea system was adjusted to spray tiny droplets of urea solution in the exhaust gas. The SCR catalyst was placed as close as possible to the engine exhaust manifold (1m) in order use the high-temperature exhaust gas, which leads to higher NO_x conversion rates.

Engine emission tests

The tested engine 392-L4-DTI has the main characteristics shown in table 1; it was mounted on the facilities of Road Vehicle Institute (INAR, Brasov) on a dynamometric test, instrumented as in figure 1.The engine was supplied with diesel fuel, having 50 ppm sulphur, in line with the European standard EN 590.

Table 1
DIESEL ENGINE SPECIFICATION

Characteristic	Specific value
Engine type	4 stroke, 4 cylinder in line
Bore x Stroke	102 x120 [mm]
Displacement	3.92 L
Net power	93 kW
Rated speed	2800 rpm
Maximum torque	421 Nm
Maximum torque speed	1600–1800 rpm
Minimum specific fuel consumption	206 g / kWh
Intake configuration	Turbocharged /intercooled

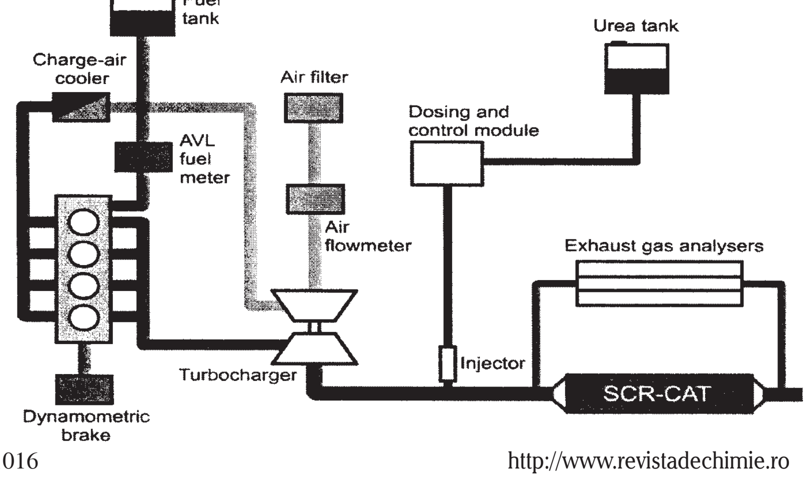

Fig. 1. Engine and SCR system

The measurement system included a pollutant measurement system from the exhaust gas and some specific engine sensors. The emission was analysed with a Beckman/Rosemount analyser made of a heated chemiluminescent analyser for NO_x (model 951A) and a NDIR analyser for the NH_3 slip (model 854).

The speed, load, torque, air and fuel consumption, exhaust-gas flow-rates, exhaust-gas temperatures and pressures, NO_x, upstream and downstream the SCR catalyst, were experimentally determined for different emission test cycles.

Before each test, the SCR was set up and the catalyst was conditioned in a temperature ramp of 5°C/min from the room temperature, up to 170°C, when urea starts to decompose.

Two types of measurements of the NO_x conversion were performed on engine tests: a. at constant speed and increasing load and b. at variable speeds and loads, according to a representative European cycle (ESC) [15]. Preliminary NO_x measurements were made for both tests, in order to calculate the stoichiometric urea to be injected.

Constant-speed emission test

At the constant engine speed of 2800 rpm, the load was modified in 5 steps, from 10%, to 25%, 50%, 75% and up to 100% of rated load, by rising the quantity of injected fuel per cycle.

Variable speed and load emission test

The automotive use of an engine requires the broad variation of its speed and load. For every operation mode of the engine identified by a set of speed and load values, it was measured the gaseous emissions, which were averaged with the weight of every operation mode. The measurement of the speed, torque and exhaust-gas flow-rate enabled the calculation of the specific powers for each operation modes, which are reported to pollutant flow rate, revealing the emission reported to produced energy unit (g / kWh).

One of the most representative standard emission tests for heavy-duty commercial vehicles is described in 13 steady-state modes, in European Stationary Cycle ; based on three reference speeds A, B and C and four percent loads (25, 50, 75 and 100%), as well as on the idle mode, the 13-mode test procedure assigns a certain weight . The weight is proportional to the time period in which the engine operated in that particular mode reported to the overall engine life span (fig.2).

For the 392-DTI engine, the speeds of tests shown in figure 2, marked with A, B and C were calculated according to [15] and their respective values were A=1800 rpm, B=2150 rpm and C=2500 rpm.

Results and discussions

Composition aspects

The composition characteristics - specific surface, pore volume, radius pore distribution- are indicators of the activity and stability of the catalysts, and likewise a quality control tool in the process of preparation and manufacture. The measured BET surface areas are similar to the SCR Cu-zeolites described in the literature, having values in the range 270-400 m^2/g. [10, 16]. The characteristics of the BET analysis are shown in table 2.

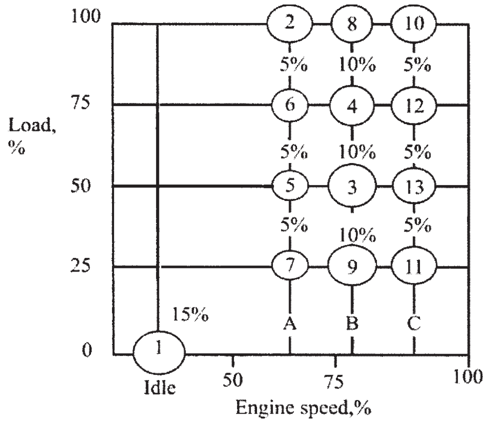

Fig. 2. ESC engine operation modes and weights

Catalyst parameters

The pore-size distribution was dominant in the range of 5-25Å, which led to the characterization of the layer as micropore-preponderent (radius below 20 Å), the diameter of 5 Å specific to the zeolite ZSM5 being also confirmed.

Catalyst activity

In order to check the conversion rate of SCR, a preliminary test was performed. The exhaust gas was circulated through the catalyst without injection supply and the NO_x conversion rate became zero; moreover, the value of the NO_x conversion rate was checked both when urea was being injected and catalyst was being removed; in this case, the value of the NO_x conversion was close to zero, which indicates that SCR is operational only when the exhaust gas mixed with the reduction agent passes through the catalyst.

The NO_x conversion of the SCR system is:

$$NO_x\ conversion = \frac{(c_i - c_o)}{c_i} \times 100 \cdot (\%) \qquad (4)$$

c_i - inlet catalyst NO_x concentration, in ppm,
c_o - outlet catalyst NO_x concentration, in ppm.

Constant-speed emission test

Triplicate tests were carried out and their results were averaged; the relative error of NO_x conversion was below 2%. The average SCR-NO_x conversion of the engine, measured at the constant speed of 2800 rpm, is shown in figure 3, depending on the exhaust gas temperature.

As shown in figure 3, the NO_x efficiency confirms the typical behaviour of the Cu zeolite catalysts, whose values range between 20-70% in the temperature window 180-530°C. The light-off temperature, defined as the temperature at which SCR reaches 50% of the conversion rate, is approximated by interpolation at 270°C.

Table 2
CATALYST PARAMETERS

Specific surface (m^2/g)	Micro-pore volume (0-300 Å),cm^3/g	Pore volume distribution by radius (%)					
		5-10Å	10-15 Å	15-25 Å	25-50 Å	50-100 Å	100-300 Å
286.09	0.1826	53.98	18.71	14.45	6.27	3.53	3.06

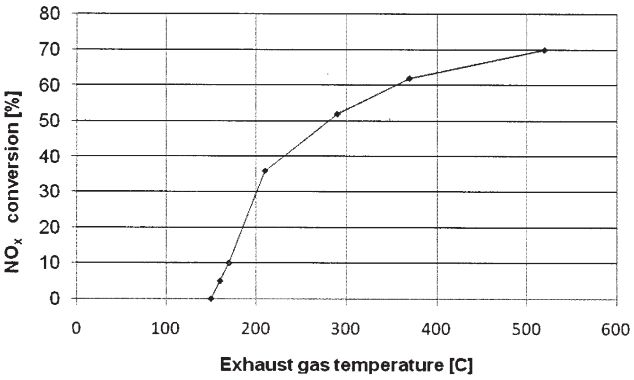

Fig. 3. NO_x conversion versus exhaust gas temperature, at constant speed

Variable-speed emission test

The test was performed three times, in the 13 points of the ESC cycle; the relative error of the NO_x conversion was 2% and the relative error of engine power was 2.5%. The fuel consumption was 3.4% higher for SCR test than without SCR, which indicates that SCR system introduces gas-dynamic losses, which influence engine power and fuel consumption. The maximum NH_3 slip was 15 ppm, measured in the operation mode no.7.

The average results of the ESC emission tests are shown in fig. 4, pointing to the NO_x emissions before and after SCR catalysts, as well as to the number of operation modes.

The overall calculation in terms of emissions, exhaust flow rate and brake power were performed according to [15], resulting the final weighted values of NO_x =5.45 g/kWh (without SCR system); and 2.27 g/kWh (applying SCR); hence a reduction of NO_x on the ESC test of 58.3%. Similar research works with the same type of catalyst and the same ESC cycle indicated values of 53% [17], 62.9% [18] and 67% [19]. In terms of NO_x emissions, the application of the described SCR system has succeeded to turn the pre Euro III engine into an Euro IV-compliant engine, very close to the current Euro V standard, as illustrated in figure 5.

A SCR retrofit is easily feasible in service truck engines, most of them manufactured in Romania, in order to comply with stricter emission standards.

The literature for urea-SCR having CuZSM5 as catalyst describes many research works on NO_x reduction carried out in reactors in conditions of controlled composition of the feed gas. Most of them declare higher conversions, up to 90-95%, in similar Cu-zeolite compositions [10,17] and assess the influence of several factors related to NO_x reduction: exhaust-gas temperature, space velocity, NH_3/NO_x, O_2 concentration, water concentration, NO_2/NO_x ratio, fuel sulphur content.

On the other hand, many research works were conducted on diesel engines, which reported lower NO_x urea-SCR conversions (50%-80%) in different, real-life or standard emission tests [17-21]. As the results of the present tests fall under the second group, there were further discussed several aforementioned factors, whose influences could be separated during experiments:

Exhaust gas temperature has the most significant influence on NO_x; the higher the temperature, the higher the conversion, as described in figure 3. The reactor test at constant feed gas composition generates steady reaction conditions, while in diesel engines the increase of exhaust-gas temperature is produced by the increase of load, based on increased fuel mass which varies continuously the air fuel ratio and exhaust gas composition. When a diesel engine is used on a vehicle, there is a continuous variation of speed and load which may lead, especially in urban driving, to low exhaust temperature (lower than light-off temperature) which points to low NO_x conversions. From this point of view, ESC test is considered to produce relatively high exhaust-gas temperatures; consequently, in real driving conditions where the average temperature ranges between 200-400°C [13], the conversions are even scarcer. As the NO_x threshold limits imposed by environmental regulations become more stringent, some remedies could be the controlled heating of the exhaust gas, the placement of SCR catalyst as close as possible to the engine exhaust manifold or the thermal isolation of the exhaust pipe.

The application of an indicator is economical and time-saving: the *weighted exhaust gas temperature* (WET), defined as a function of the test emission cycle, can rapidly predict the NO_x removal, comparing WET with the light-off temperature and the conversion curves of the catalyst. For example, in terms of steady-state operation modes, WET, an average temperature of the exhaust gas during the test cycle, can be written as:

$$WET = \sum_{i=1}^{n=13} w_i \cdot t_i \qquad (5)$$

with:

w_i- weight of the steady operation mode i in the steady-state emission test cycle,

t_i- exhaust gas temperature in operation mode i in the emission test cycle.

As regards the tested engine, the weighed exhaust temperatures were calculated for ESC, ECE Regulation 49 [22] and for two original urban and motorway cycles, which were aggregated based on continuously recording the vehicle speeds and loads on Romanian roads, in urban and motorway operation modes, as described in detail in [23] and presented in table 3.

The meaning of the data from table 3 is that urea-SCR conversion depends strongly on the vehicle duty cycle; the higher the WET, the higher the predicted NO_x conversion. Unfortunately, the urban driving, where a higher NO_x conversion is needed, has lower WET, due to lower speeds and loads; consequently, the SCR system is more efficient in motorways cycles and steady state regulation cycles such as ECE R 49 and ESC.

Space velocity (or Gas Hourly Space Velocity-GHSV) shows the time spent by the exhaust gas in the catalyst, being measured as the ratio between the exhaust gas volumetric flow-rate (at standard temperature and pressure - STP) and the catalyst volume. The reduction of the space velocity would increase the NO_x conversion. The typical values of GHSV in SCR diesel engines are 10 000-150 000 h^{-1}. The reduction of GHSV also lowers the light-off temperature.

The current experiments have shown that GHSV varied in the range of 26 000-125 000 h^{-1}. The influence of GHSV on the NO_x conversion at 500°C-constant temperature of the exhaust gas is shown in fig.6. The results are in line with other research works [17, 19].

A practical solution for decreasing GHSV is to design SCR systems with higher volumes of catalysts; for the tested engine, a method of raising GHSV is to increase the catalyst volume by a higher coefficient than the value 1.2, characteristic of the engine displacement. A reasonable value in terms of bulk volume on the vehicle is 1.8.

Sulphur content acts as a poison upon Cu-zeolite catalysts, as it lowers their activity and durability. The literature mentions that in tests performed with reactor

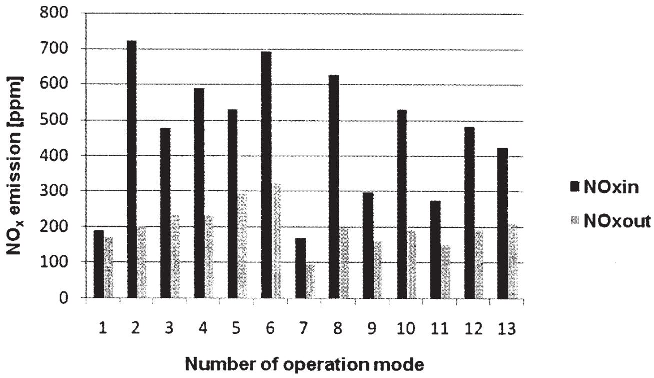

Fig. 4. NO_x reduction in European Stationary Cycle

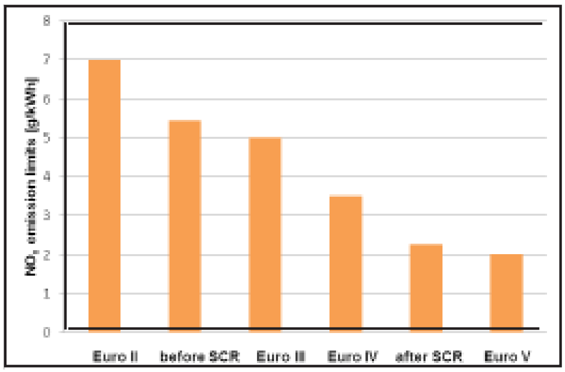

Fig. 5. SCR NO_x mesurements and limits in European legislation

Table 3
WEIGHTED EXHAUST TEMPERATURES FOR TESTED ENGINE IN DIFFERENT OPERATION MODES

Cycle	Weighted exhaust temperature [°C]	Predicted NO_x conversion [%]
ESC	350	58
Reg. 49	356	61
Urban cycle	225	40
Motorwaycycle	455	65

gas having 50 ppm SO_2 (and no water), the NO_x conversion was approximately 20% lower than without SO_2 [18].

For operation in the present tests, conventional diesel fuel was used, in whose composition 50 ppm of sulphur are also included; therefore one of the explanations for the different results between reactor tests and diesel engine tests could be the sulphur content in the fuel. As a remedy, which is also in line with the tendency followed by diesel fuel reformulation, only ultra low sulphur diesel fuel with maximum sulphur content of 10 ppm must be used.

Water content in exhaust gas inhibits SCR reactions and increases the light-off temperature [24]. The literature mentions a 10-20% loss as regards the NO_x conversion in Cu –zeolites caused by their low hydrothermal stability, when feed gas has 10% water [25, 26]. The research work [19] confirms the aforementioned, which constitutes therefore the weak point of this type of catalysts.

For the proposed SCR system, the water content varied between 4% (air in excess λ=2.86, measurement point no.7 in fig.4) up to 12% (λ=1.1, measurement point no.2 in fig.4) and it was calculated based on the combustion equation. The water content in the diesel exhaust gas is not avoidable, any drying system being for the moment difficult to implement.

Oxygen content negatively influences the NO_x conversion only when its concentration is lower than 2%. The higher values do not have noticeable effects on the standard reaction expressed in equation (2) [24, 27]. As in the diesel exhaust, the oxygen content ranges between 1.9 – 18 % in volume, it must be verified for the current tests the condition of having oxygen content higher than 2%, in order to exclude it as the cause of NO_x lower conversion. By applying air-fuel measurements and combustion equation, the oxygen content varied from 2% (for λ=1.1 measurement point no.2 in fig.4) to 14%, (for λ=2.86, measurement point no.7 in fig.4), excluding the influence of this factor.

Conclusions

An experimental SCR system based on the urea-Cu-ZSM5 zeolite, aimed at reducing the NO_x, confirmed the

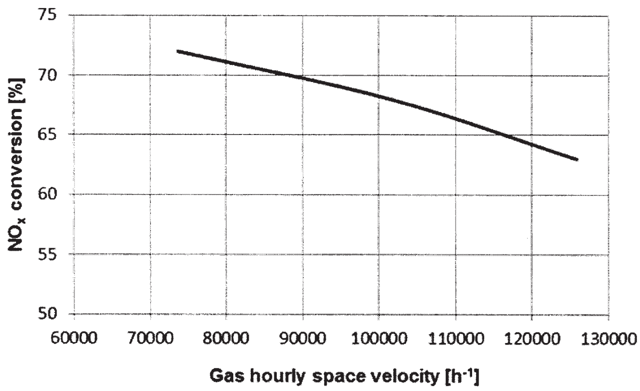

Fig. 6. NO_x variation with space velocity

quality of the prepared catalyst and its activity, during engine tests.

NO_x conversion efficiency proved to be highly dependent on the test cycle temperature; the weighted exhaust-gas temperature (WET) is a reliable predictor for SCR –engine behaviour .

At constant exhaust-gas temperature, the reduction of space velocity raises the NO_x conversion by 10%, due to a longer time spent by the gas in the catalyst; further research work benefit from the reserves of GHSV, which allows an increase of catalyst volume.

A practical result of the research work is the retrofit of 392-L4-DTI engine with SCR – urea catalyst which has a considerable reduction of NO_x emission, meeting the Euro IV heavy-duty engine emission test standard.

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