



## NO<sub>x</sub> removal efficiency and N<sub>2</sub> selectivity during selective catalytic reduction processes over Al<sub>2</sub>O<sub>3</sub> supported highly cross-linked polyethylene catalysts



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### ABSTRACT

The performance of a highly cross-linked polyethylene catalyst supported on alumina for low temperature selective catalytic reduction of NO<sub>x</sub> by unburned hydrocarbons (HCs) existing in an exhaust gas was examined at different engine conditions with the addition of exhaust-gas recirculation. The HXPE catalyst was shown to exhibit good NO<sub>x</sub> reduction activity at low temperatures (100–250 °C) where the only reductant was the unburned HC, which was already present in the exhaust flow. The maximum NO<sub>x</sub> reduction of approximately 52% was achieved at a temperature of 150 °C. HXPE demonstrated very good selectivity toward N<sub>2</sub> in the majority of tested conditions (~80%).

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### 1. Introduction

The growth of compression ignition engine usage in the transportation sector has been significantly affected by environmental pressures. Clearly the general trend is toward higher efficiency engines, which improve fuel economy and lower greenhouse gas emissions [1]. While diesel engines are often popular choices based on their durability, cost effectiveness, and high torque, the primary advantages of diesel engines over spark ignition engines are their improved fuel economy, resulting lower

CO<sub>2</sub> emissions (approximately 20–30%) [2]. However, diesel engines generally emit high nitrogen oxides (NO<sub>x</sub>) emissions and are associated with much higher emissions of particulate matter than SI engines [3]. Moreover, the suppression of NO<sub>x</sub> formation by internal means (e.g., exhaust gas recirculation EGR, and retarding the injection timing) has the tendency to cause an increase in particulates, known as the NO<sub>x</sub>-particulates trade-off [4]. Despite the evolution of cleaner fuels and combustion technologies [5] the exhaust from modern diesel engines still require after-treatment to meet the most stringent emission legislation, with the control of NO<sub>x</sub> posing a particular challenge [6]. Control of NO<sub>x</sub> emissions from diesel engines is fundamentally difficult because of the high local peak temperatures and lean average air/fuel ratio in the combustion chamber. However, significant work has been undertaken regarding the methods for NO<sub>x</sub> emissions reduction, which can be classified into conventional and non-conventional methods [7–10]. Selective catalytic reduction of NO<sub>x</sub> with hydrocarbons (HC-SCR) has received much attention as one of the most promising and straightforward methods for reducing NO<sub>x</sub> emissions under conditions of excess

**Abbreviations:** BSFC, brake specific fuel consumption (kg/kWh); BSN, Bosch smoke number; EGR, exhaust-gas recirculation; HC, hydrocarbons; HXPE, highly cross-linked polyethylene; N<sub>2</sub> conversion, the amount of NO<sub>x</sub> that has been converted to N<sub>2</sub>; N<sub>2</sub>O conversion, the amount of NO<sub>x</sub> that has been converted to N<sub>2</sub>O; PM, particulate matter; SCR, selective catalytic reduction; T<sub>r</sub>, reactor inlet temperature.

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oxygen [11]. Extensive research has been undertaken using different types of hydrocarbons as reductants [12,13]. However, although a number of catalysts have been tested in the HC-SCR, to date no catalyst has been considered suitable for the diesel de-NO<sub>x</sub> conditions. In these conditions, the catalysts are required to be hydrothermally stable and active at relatively low temperatures in the presence of SO<sub>x</sub> and water vapor. Although many base oxides/metals (e.g. Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, ZrO<sub>2</sub>, MgO with these oxides promoted by, Pt, Co, Ni, Cu, Fe, Sn, Ga, In, Ag compounds) are active catalysts for HC-SCR of NO<sub>x</sub> [14,15], the NO<sub>x</sub> reduction activity occurs at a narrow temperature window 200–300 °C [16], which is also associated with the formation of N<sub>2</sub>O [17]. Moreover, zeolite-based catalysts, which are the majority of the de-NO<sub>x</sub> catalysts reported in literature, are unlikely to be suitable as an automotive catalyst due to their instability under hydrothermal conditions [18].

Polyethylene in its various forms is essentially a long chain carbon-based polymer. The chains are not linked directly to each other, but the basic structure is held together by entanglement of the long chains and, in the more crystalline areas, by weak intermolecular forces. Application of heat to this polymer allows the molecules to move relatively easily with respect to each other. The material is thus easily processed and its basic structure gives a tough, flexible material with excellent tensile and impact properties. However, a major drawback of polyethylene is that at higher temperatures it starts to become softer and more elastic due to the polymer chains separating and moving promoting less resistance to tensile and creep forces. The creation of direct links or bonds between the carbon backbones of individual polyethylene chains forms the cross-linked polyethylene (XPE) structure. The result of this linkage is to restrict movement of the polyethylene chains relative to each other, so that when heat or other forms of energy are applied, the basic network structure cannot deform and the excellent physical properties that polyethylene possesses at room temperature are retained at higher temperatures.

Despite the large number of publications on the reduction of NO<sub>x</sub> under lean exhaust gas conditions using SCR, there are only a few papers which focus on the reduction of NO<sub>x</sub> under real engine exhaust conditions using a polymer based material as a catalyst. The aim of the present work is to explore the performance of HXPE as SCR catalyst in reducing nitrogen oxides (NO<sub>x</sub>) by unburned hydrocarbons existing in the actual engine exhaust gas at relatively low temperatures. The combined effect of EGR addition at different engine conditions in parallel with the performance of HXPE polymer catalyst was also investigated.

## 2. Methodology

### 2.1. Test engine

A four cylinder Tempest Engine coupled with dynamometer was used for data collection. The engine was a water-cooled, naturally aspirated, four strokes, and direct injection diesel engine. The main engine specifications are given in Table 1. The exhaust gas was recycled from the engine exhaust to the inlet (external EGR) and the volumetric flow rate of the EGR was calculated according to the reduction in the air volumetric flow rate. An electric dynamometer was used to vary the load the engine.

### 2.2. Engine instrumentation

The engine is a completely self-contained test bed incorporating a swinging field DC dynamometer, which is capable of absorbing 22 kW (30 hp). Services include an oil/water heat exchanger for the main cooling system and a water/oil heat exchanger for the oil cooling system. The engine has manually adjustable ignition timing control with indicator, and one cylinder

**Table 1**

Engine details.

Name of the engine	Tempest
Force arm	0.4 m
Bore and stroke	72.25 mm × 88.2 mm
Connecting rod length	156.0 mm
Swept volume	1447 cm <sup>3</sup>
Compression ratio	22:1
Orifice area	0.00181 m <sup>2</sup>
Injection timing	15° CA, BTDC <sup>a</sup>
Maximum power	22 kW at 3000 rpm

<sup>a</sup> CA: crank angle; BTDC: before top dead center.

is provided with a transducer tapping point (10 mm thread). The orifice air meter uses a cylindrical reservoir or damping chamber to minimize the effect of pulsations, to produce a steadier flow, through the orifice inserted in one of its end walls. The other end wall of the chamber consists of a resilient synthetic rubber diaphragm secured at its edges between rings of marine plywood sealed to the cylinder.

The engine speed was adjusted by revolution speedometer, and then a suitable load (weights) to balance the engine and minimize the vibration of the engine was adjusted. To measure the exhaust gas emissions, the sensor of the gas analyzer was positioned into the outlet of the exhaust. The values of voltage and current were taken by armature, also a multi-point thermometer was used to measure the values of inlet water, outlet water and exhaust temperatures. Fuel measurements were performed by simple gravity pipette type gauge, with two bulbs calibrated for 50 and 100 cm<sup>3</sup> respectively. Flow rate of diesel was measured by a stopwatch (time required to consume 50 mL of fuel). The test rig included other standard engine test rig instrumentation such as to allow monitoring of flows (liquid and gaseous fuels, intake air and EGR), temperatures (oil, air, inlet manifold and exhaust) and pressures (gauges mounted at relevant points). Normal engine test bed safety features were also included. Atmospheric conditions (humidity, temperature, pressure) were monitored during the tests.

### 2.3. Exhaust gas and aftertreatment (SCR reactor) products monitoring

A KANE-AUTO SW3 gas analyzer with an external water trap was used to measure exhaust emissions and the after-treatment reactor products. Gas analysis included measurement of carbon dioxide, carbon monoxide (non dispersive infrared), unburned hydrocarbons – hexane equivalent – (flame ionization detector), oxygen (electrochemical method), and NO<sub>x</sub> (chemiluminescence) emissions. Smoke opacity was measured using a Bosch smoke meter, giving smoke emissions in terms of Bosch smoke numbers (BSN). An Ellutia 200 series gas chromatograph fitted with a thermal conductivity detector was used for measurement of the hydrogen (H<sub>2</sub>), and nitrous oxide (N<sub>2</sub>O) content of the SCR reactor product and from the engine.

### 2.4. Tested fuel

The fuel used was conventional diesel fuel available locally. The fuel properties are as follows: cetane number 46, density (at 15 °C) 838.4 kg/m<sup>3</sup>, LCV 45,263 kJ/kg, and sulphur content 57 mg/kg.

### 2.5. Catalyst (polymer) preparation

The highly cross-linked polyethylene (HXPE) or SCR catalyst used throughout this study was obtained from World of Plastic

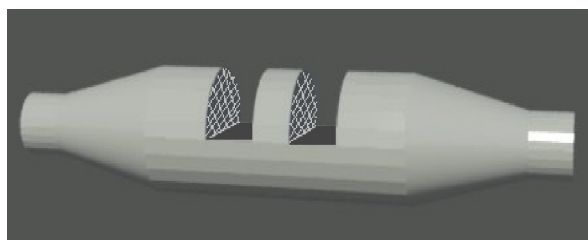


Fig. 1. Selective catalytic reduction (SCR) reactor design.

Factory (Jordan). The HXPE was prepared by irradiation at 120 °C using a 10 MeV electron-beam accelerator. Following irradiation, the samples were melt-annealed above 130 °C for 5 h to substantially reduce the concentration of the residual free radicals. The cross linking of polyethylene (PE) is based on a free radical mechanism. The free radicals generated on thermal decomposition of peroxides can interact with the molten PE free chains thus promoting cross linking of the polymer chains.

## 2.6. SCR reactor design

The SCR reactor was designed as shown in Fig. 1 to have gas hourly space velocity (volumetric flow rate of gas per hour divided by the volume of the catalyst bed) of approximately  $50 \times 10^3 \text{ h}^{-1}$  and  $100 \times 10^3 \text{ h}^{-1}$ , which are typical for internal combustion engines' exhaust gas converters. The catalyst was in the form of granules of approximately 1–3 mm diameter, and it was loaded easily into the SCR reactor. The total length for the entire SCR reactor design was 30 cm with 12 cm diameter. Two SCR beds (5 cm each) were employed in this design, and they were separated by 4 cm. The total catalyst bed employed in the reactor was approximately 160 g in mass and 10 cm long. A stainless sieve was used to support the catalyst. Granular alumina was used as a separator within the SCR bed.

## 2.7. Experimental procedure

The SCR reactor was placed in a tubular uniform distributed heater, which can be adjusted up to 500 °C. The temperature was set by thermocouples in the reactor inlet, middle, and outlet to maintain the temperature distribution throughout the reactor. The polymer catalyst in this study was designed to promote the  $\text{NO}_x$  reduction at low temperatures, and it is proposed that this is an integral part of a complete SCR system. In this study the experimental work has been divided into two parts:

**Part 1:** The performance of HXPE catalyst in reducing nitrogen oxides ( $\text{NO}_x$ ) by (HC-SCR) existing in the actual engine exhaust gas (passive control) was examined by flowing the entire exhaust gas (which ranged according to the engine condition from 640 to 1280 L/min) at three different engine conditions (low, medium, and high loads) shown in Table 2. Throughout this study the low, medium, and high loads refer to 25%, 50%, and 75% full load, respectively.

In order to examine the HXPE polymer catalyst SCR activity window in reducing  $\text{NO}_x$  emissions, the all of the engine exhaust gas was taken (conditioned to around 100 °C to prevent  $\text{H}_2\text{O}$  condensation) and passed directly to the SCR reactor, which was heated to various temperatures with the use of tubular heater. The conversion of  $\text{NO}_x$  emissions was typically measured after 10 min at constant reactor inlet temperature initially at 75 °C then increasing to 100, 150, 200, 250, and 300 °C.

To avoid the expected accumulation of the SCR polymer particularly at high temperatures, granular alumina was used as a separator within the SCR bed. Most importantly, alumina is one of

Table 2  
Experimental conditions.

Engine condition	Speed (rpm)	Load % full load	EGR	Reaction temperature (°C)	Reactor inlet temperature (°C)
1	1200	25	10%	180	75
			20%		100
			30%		150
					200
					250
				300	
2	1200	50	10%	230	75
			20%		100
			30%		150
					200
					250
				300	
3	1200	75	10%	300	100
			20%		150
			30%		200
					250
					300

the best known supports for HC-SCR catalysts, such as Ag-based [19] and Pt-based catalysts [12].

**Part 2:** The effects of EGR addition (10, 20, and 30% vol.) on the diesel engine exhaust gas composition (mainly HC and  $\text{NO}_x$ ) and hence performance of HXPE polymer catalyst in reducing  $\text{NO}_x$  was investigated at three engine operating conditions as shown in Table 3. The SCR reactor inlet temperatures were set to simulate the exhaust gas temperatures for each condition (i.e.: low load  $T_r \sim 180$  °C, medium load  $T_r \sim 230$  °C, and high load  $T_r \sim 300$  °C).

## 3. Results and discussion

In this work, the reaction between the exhaust gases over the HXPE catalyst was allowed to proceed for 1 h to reach steady state. Subsequently, the reactor was switched to air flow and then heated up to the second reaction temperature. When the temperature was reached the reaction was carried out following the same procedure described above. From the concentration of the gases at steady state, both the conversion and the selectivity were calculated according to the following formulae:

$$\text{NO}_x \text{ conversion} = \frac{[\text{NO}_x]_{\text{inlet}} - [\text{NO}_x]_{\text{outlet}}}{[\text{NO}_x]_{\text{inlet}}} \times 100\% \quad (1)$$

$$\text{Selectivity} = \frac{[\text{N}_2]}{[\text{N}_2] + [\text{N}_2\text{O}]} \times 100\% \quad (2)$$

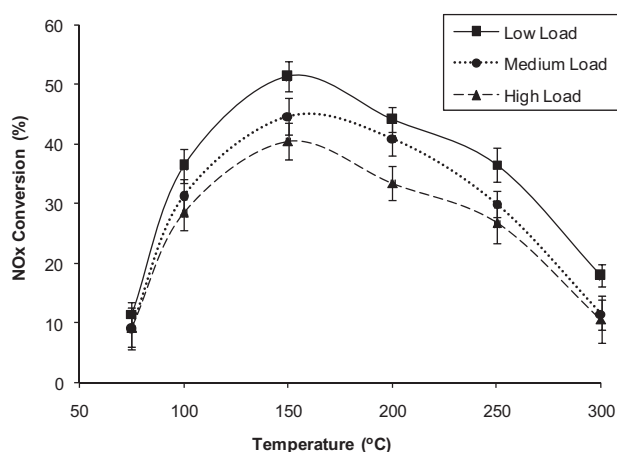
$[\text{NO}_x]_{\text{inlet}}$ ,  $\text{NO}_x$  concentration at the inlet to the SCR polymer catalyst;  $[\text{NO}_x]_{\text{outlet}}$ ,  $\text{NO}_x$  concentration at the exit from the SCR

Table 3  
Composition of the exhaust gas for three different loads @ 1200 rpm.

	25% full load	50% full load	75% full load
$\text{O}_2$ (%)	14.9	12.1	10.7
CO (%)	0.05	0.06	0.07
$\text{CO}_2$ (%)	4.1	5.9	7.1
$\text{H}_2\text{O}^{\text{a}}$ (%)	3.2	4.5	6.0
$\text{HC}^{\text{b}}$ (ppm)	~790	~750	~700
$\text{NO}_x$ (ppm)	300	550	840
HC: $\text{NO}_x$	2.63	1.36	0.83
Exhaust flow (L/min)	~640	~960	~1280
$T_r$ (°C)	180	230	300

<sup>a</sup> Calculated.

<sup>b</sup> Hexane equivalent.



**Fig. 2.** NO<sub>x</sub> conversion as a function of temperature under different engine load conditions.

polymer catalyst; [N<sub>2</sub>]: the amount of nitrogen resulting from NO<sub>x</sub> conversion; [N<sub>2</sub>O]: the amount of nitrous oxide resulting from NO<sub>x</sub> conversion.

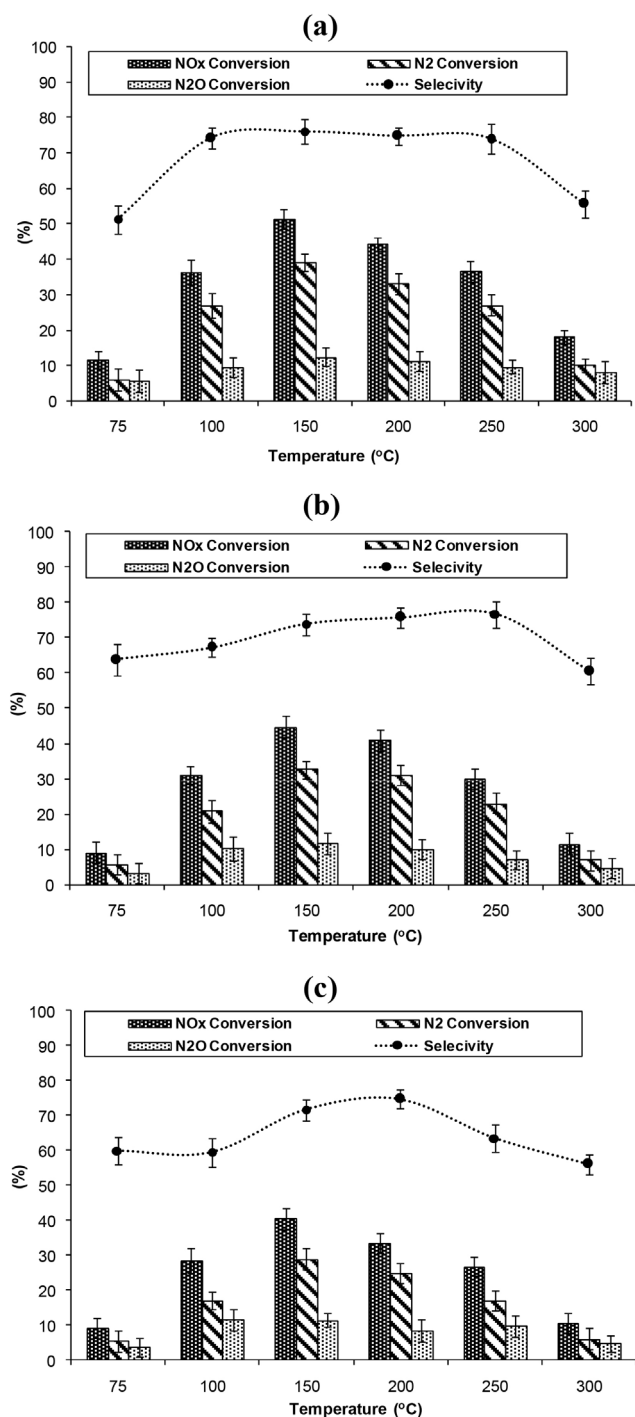
### 3.1. Highly cross-linked polyethylene (HXPE) performance and temperature activity window

The NO<sub>x</sub> conversion over the HXPE catalyst using actual engine exhaust gas at three different engine loads is shown in Fig. 2. It is obvious that the NO<sub>x</sub> conversion is strongly dependent on the reaction temperature and exhaust gas composition, generally the NO<sub>x</sub> and HC concentration in the exhaust gas and more specifically the HC:NO<sub>x</sub> ratio. At low temperatures (<100 °C) the reduction of NO<sub>x</sub> emissions was acceptable (<30%) (especially for a material tested for the first time). The maximum NO<sub>x</sub> conversions of 52%, 45% and 41% were achieved at temperature of 150 °C for the three engine loads of 25%, 50%, and 75%, respectively. At temperatures greater than 150 °C the HXPE catalyst continued to exhibit an optimum performance in NO<sub>x</sub> reduction (~40%), which provides this polymer with a good temperature window (from 100 °C to 250 °C) for NO<sub>x</sub> reduction. The polymer showed good performance at these relatively low temperatures because it still maintained its thermal stability and large surface area despite the glass transition temperature of the polymer being approached (100–250 °C). At higher temperatures (>300 °C) a sharp deterioration in the NO<sub>x</sub> reduction was observed due to the fact that melting temperature of the polymer was reached (~300 °C) and upon melting the microporosity and the specific surface area of the polymer both reduced. Referring to Fig. 2, the reduction in the maximum NO<sub>x</sub> conversion observed with the increase in the engine load, is attributed to the reduction in the HC:NO<sub>x</sub> ratio and the increase in NO<sub>x</sub> concentration (ppm) per catalyst active sites (Table 3). However, increasing the engine load leads to an increase in the amount of water present in the exhaust gas, which may inhibit the SCR reaction.

### 3.2. Selectivity toward N<sub>2</sub>

Selectivity toward nitrogen of any tested catalyst is one of the important factors that must be taken into account, since, if the selectivity toward N<sub>2</sub> is inconsequential, there will be a significant increase in N<sub>2</sub>O (an intermediate product for NO<sub>x</sub> reduction into N<sub>2</sub>). Fig. 3a–c illustrates the total NO<sub>x</sub> conversion, the amount of NO<sub>x</sub> converted to N<sub>2</sub>, the amount of NO<sub>x</sub> converted to N<sub>2</sub>O, and the selectivity toward N<sub>2</sub>.

While there is no clear trend for selectivity with the applied load, the results reveal an adequate selectivity toward N<sub>2</sub> in most conditions. At temperatures from 100 to 250 °C the selectivity was



**Fig. 3.** Overall selectivity toward N<sub>2</sub> and NO<sub>x</sub> conversion efficiencies to N<sub>2</sub>O and N<sub>2</sub> as functions of temperature: (a) 25% engine load; (b) 50% engine load; (c) 75% engine load.

always above 67%, and in some cases (medium load with 200 and 250 °C reactor temperatures) the selectivity reached approximately 80%. These HXPE selectivity data are encouraging compared with well-known base oxides/metals and zeolitic catalysts [20]. The reason for this behavior may be attributed to fact that the material has more resistance to tensile and creep forces upon cross-linking.

### 3.3. Effect of exhaust gas recirculation (EGR)

The performance of the HXPE catalyst in reducing NO<sub>x</sub> emissions under different engine conditions and EGR ratios

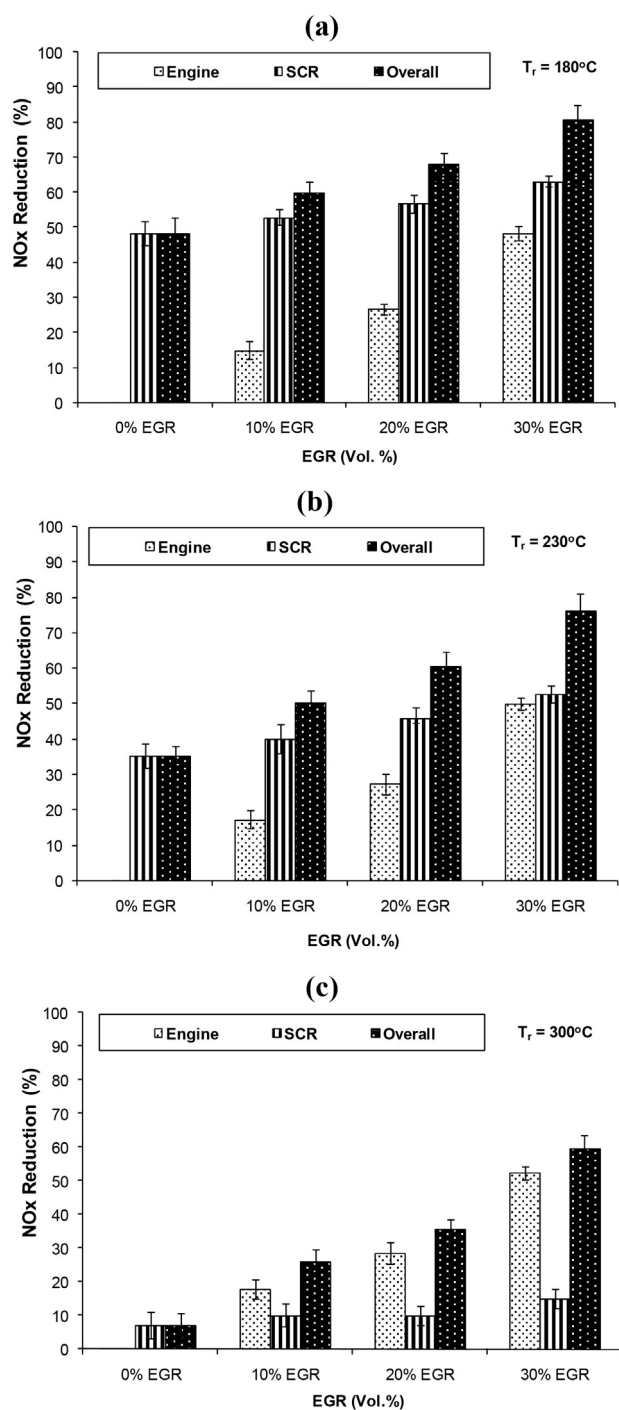


Fig. 4. Overall NO<sub>x</sub> reduction, NO<sub>x</sub> reduction in the SCR reactor, and in the engine due to EGR addition: (a) 25% engine load; (b) 50% engine load; (c) 75% engine load.

(10%, 20%, and 30%) is illustrated in Fig. 4a–c where the reactor inlet temperatures were set to simulate the exhaust gas temperatures for each condition (i.e.: 25% low load  $T_r = 180^\circ\text{C}$ , 50% medium load  $T_r = 230^\circ\text{C}$ , and 75% high load  $T_r = 300^\circ\text{C}$ ).

Although the EGR has the tendency to cause an increase in both engine particulate matter (PM, i.e., smoke) and fuel consumption as shown in Tables 4–6, it also has an effect in reducing NO<sub>x</sub> by direct suppression of NO<sub>x</sub> formation during the combustion process, and by improving the performance of the catalyst in HC-SCR activity. The favorable effects from the engine operation with EGR on the SCR catalyst activity arise from its effect on the exhaust gas composition as shown in Tables 4–6. It is apparent that

Table 4

Exhaust gas composition: low load (25% full load) @ 1200 rpm.

	0% EGR	10% EGR	20% EGR	30% EGR
H <sub>2</sub> O <sup>a</sup> (%)	3.2	3.0	2.7	2.1
HC <sup>b</sup> (ppm)	790	815	840	890
NO <sub>x</sub> (ppm)	300	255	220	155
HC:NO <sub>x</sub>	2.63	3.26	4.00	5.74
BSFC (kg/kWh)	0.62	0.71	0.82	0.90
BSN	1.1	1.2	1.5	2.0

<sup>a</sup> Calculated.

<sup>b</sup> Hexane equivalent.

Table 5

Exhaust gas composition: medium load (50% full load) @ 1200 rpm.

	0% EGR	10% EGR	20% EGR	30% EGR
H <sub>2</sub> O <sup>a</sup> (%)	4.5	4.1	3.6	3.0
HC <sup>b</sup> (ppm)	750	770	800	875
NO <sub>x</sub> (ppm)	550	455	400	275
HC:NO <sub>x</sub>	1.36	1.64	2.00	3.02
BSFC (kg/kWh)	0.39	0.47	0.54	0.61
BSN	2.8	3.2	3.8	4.4

<sup>a</sup> Calculated.

<sup>b</sup> Hexane equivalent.

Table 6

Exhaust gas composition. High load (75% full load) @ 1200 rpm.

	0% EGR	10% EGR	20% EGR	30% EGR
H <sub>2</sub> O <sup>a</sup> (%)	6.0	5.5	5.1	4.7
HC <sup>b</sup> (ppm)	700	725	760	820
NO <sub>x</sub> (ppm)	840	690	600	400
HC:NO <sub>x</sub>	0.83	1.05	1.26	2.05
BSFC (kg/kWh)	0.26	0.30	0.35	0.40
BSN	3.7	4.5	5.2	6.3

<sup>a</sup> Calculated.

<sup>b</sup> Hexane equivalent.

there is a significant decrease in NO<sub>x</sub> accompanied with a gradual increase in the HC, which means that more reductant (increased HC:NO<sub>x</sub> ratio) is available for the SCR process. A NO<sub>x</sub> conversion of ~81% was achieved with 30% EGR for the low load engine operating condition, while the NO<sub>x</sub> conversion at the same EGR percent (30%) for the medium engine load was 77%. Furthermore, the increased fuel consumption and PM (smoke) can be controlled by using an optimized injection set-up (e.g. injection-rate, pressure and timing) and diesel particulate filter [16]. For the low and medium engine operating conditions used in this study, both NO<sub>x</sub> reduction techniques (EGR and HC-SCR) were effective in reducing NO<sub>x</sub>. However, at the high engine load condition the use of EGR has significantly increased the PM emissions (Table 6). The HXPE catalyst was not effective in reducing NO<sub>x</sub> (where the NO<sub>x</sub> reduction in the SCR reactor did not exceed 15%, Fig. 4c) due to increased exhaust gas temperature (300 °C). The significant overall NO<sub>x</sub> reduction at high load with 30% EGR arose from the direct effect of EGR on engine emissions.

#### 4. Conclusions

The performance of highly cross-linked polyethylene (HXPE) supported on Al<sub>2</sub>O<sub>3</sub> as a selective catalytic reduction of nitrogen oxides (NO<sub>x</sub>) by unburned HC existing in an exhaust gas was examined at different engine conditions and temperatures. Under real diesel exhaust gas conditions, HXPE catalyst supported on Al<sub>2</sub>O<sub>3</sub> has shown satisfactory performance in the reduction of NO<sub>x</sub>, with unburned hydrocarbons existing in the exhaust gas at low temperatures (i.e. 150–250 °C). The reaction temperature, HC:NO<sub>x</sub>

ratio significantly affected the NO<sub>x</sub> reduction efficiency. There was no clear trends between the selectivity toward N<sub>2</sub> and HC:NO<sub>x</sub> ratio, or the selectivity and the reaction temperature. HXPE has shown very encouraging selectivity toward N<sub>2</sub> in all tested conditions, reaching in some cases 80%. For most of the conditions examined the combination of the EGR and HXPE catalysts significantly improved the overall NO<sub>x</sub> conversion (more than 80%). However, this significant reduction deteriorated at high loads because of high associated temperatures in the reactor.

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