International Journal of Environmental Science and Technology
https://doi.org/10.1007/s13762-019-02266-x

ORIGINAL PAPER

Selective catalytic reduction of NO_x emissions by hydrocarbons over Ag–Pt/Al_2O_3 catalyst in diesel engine

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Received: 27 October 2018 / Revised: 27 January 2019 / Accepted: 6 February 2019
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Abstract

Selective catalytic reduction is an application used to control NO_x pollutants in diesel engines. Aqueous urea solution, commercially called AdBlue and obtained by mixing pure water and NH_3, is the most commonly used reductant while the V_2O_5–WO_3/TiO_2 structure has a widespread use as catalyst in SCR technology. However, the SCR systems included AdBlue and V_2O_5–WO_3/TiO_2 structure have low NO_x conversion efficiency at low exhaust gas temperatures. The use of hydrocarbons as reductant and catalysts containing silver improves the conversion efficiency of selective catalytic reduction systems at low exhaust temperatures. In this work, selective catalytic reduction of NO_x emissions from diesel engines in the presence of hydrocarbons has been studied. While ethanol and biodiesel mixtures were used as hydrocarbons, the Ag–Pt/Al_2O_3 structure was preferred as the catalyst. Scanning electron microscope and X-ray fluorescence analyses of the catalyst produced by the impregnation method were carried out. In order to determine the NO_x conversion efficiency of ethanol–biodiesel mixtures in the selective catalytic reduction system, tests were carried out at different engine loads and different exhaust gas temperatures under actual operating conditions. As a result of the tests carried out, it was concluded that the reductant, which consists of 15% biodiesel and 85% ethanol, has the highest conversion performance.

Keywords Biodiesel · Diesel vehicles · Emission control technology · Ethanol · NO_x conversion · Pollutant emission

Introduction

NO_x emission is one of the most important problems that hamper the use of diesel engines. Both environmental factors and standard values determined by international organizations necessitate high reduction rates in NO_x emissions of diesel engines (Nakomcic-Smaragdakis et al. 2014). Numerous research and technological developments have been made to achieve desirable conversion rates in NO_x emissions (Shararİar et al. 2018; Ramos et al. 2018; Lee and Min 2014; Chen et al. 2018).

Selective catalytic reduction (SCR) of NO_x emission is a widely used technology nowadays. NO_x emissions from diesel engines can be eliminated at high rates with a reductant and catalyst in SCR system. Ammonia (NH_3) is the most preferred reductant in the present case due to its high conversion efficiency (Hamidzadeh et al. 2018). NH_3 is obtained from the aqueous urea solution called AdBlue to prevent NH_3 from burning at high temperatures. The aqueous urea solution consists of 67% pure water (H_2O) and 33% urea solution ($(NH_2)_2CO$) (Praveena and Leenus Jesu Martin 2018).

The V_2O_5–WO_3/TiO_2 structure is the most commonly used catalyst type in NH_3-SCR systems in which the AdBlue solution is used as a reductant (Liu et al. 2016). In conventional NH_3-SCR systems where the AdBlue reductant is used with V_2O_5–WO_3/TiO_2 catalyst, NO_x emissions can be eliminated at high rates, especially at high exhaust gas temperatures (350–450 °C). However, at low exhaust gas temperatures below 250 °C, conversion efficiency remains low and NH_3 accumulates on the exhaust line and catalyst surfaces. This occurrence, called NH_3 slip, has a very negative effect

Editorial responsibility: R Saravanan.

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Published online: 12 February 2019

on SCR conversion efficiency and can cause damage to the catalyst (Sharariar and Lim 2018).

The use of hydrocarbons (HCs) as reductant improves the low temperature performance of the SCR system (Arve et al. 2005). In these systems, referred to as HC-SCR, the use of diesel fuel or unburned HC as reductant in the exhaust simplifies the system and reduces cost (Ayodhya and Narayanappa 2018). These advantages make HC-SCR systems particularly attractive for light-duty vehicles.

The SCR reactions of NO_x emissions using the HCs as reductant are given below.

$$NO + O_2 \rightarrow NO_2$$

$$C_xH_yO_z + NO_2 \rightarrow N_2 + CO_2 + H_2O$$

In HC-SCR systems, the NO gas in the exhaust gas is converted to NO_2 form. NO_2 gas is ingested with HCs, and this reduction results in N_2, CO_2 and H_2O being released.

Unlike V_2O_5–WO_3/TiO_2 catalysts, which exhibit poor performance in low exhaust gas temperatures (< 300 °C) and have toxic effects, ceramic structures containing silver (Ag–Al_2O_3) perform well in SCR systems used HCs as reductant (HC-SCR) (Kylhammar et al. 2007). Apart from silver, many metals such as copper (Cu), platinum (Pt), palladium (Pd), iron (Fe), cobalt (Co), rhodium (Rh) have been tested as catalysts in HC-SCR systems (Mrad et al. 2015). In particular, Pt-based catalysts exhibit high performance in NO_x conversions at low exhaust gas temperatures. Table 1 shows a brief comparison between different catalysts used prevalently in SCR applications.

The conversion efficiency of NO_x emissions in HC-SCR systems varies depending on the ratio of HC to NO_x in the exhaust gas content. In case the amount of HC is 2–4 times higher than that of NO_x, NOx emissions can be converted up to 80% rates. However, the exhaust gas does not contain HC in this amount in diesel engines. Therefore, the amount of HC in the exhaust gas content must be provided by enriching the mixture during combustion or spraying it directly on the exhaust gas (Piumetti et al. 2015). Direct injection of HCs onto the exhaust gas can be controlled by different parameters via an injector and control unit. Increasing the amount of HC in the exhaust gas content by enriching the mixture which intakes in cylinder is an indirect method, and high NO_x conversion efficiency cannot be achieved in this method.

A number of different types of HCs have been studied as reductant in HC-SCR systems (Frobert et al. 2013; Sitshebo et al. 2009; Xi et al. 2017). Oxygenated HCs, such as various alcohols and biodiesel, provide the highest conversion efficiency. Alcohols such as ethanol, methanol, butanol, propanol are widely studied as HC species. Also, diesel fuel can be used as a reductant in HC-SCR systems (Cho et al. 2012).

Biodiesel is an alternative and renewable energy source that contains oxygen in its content. Having similar properties to diesel fuel and the enhancing effect on engine characteristics have made the biodiesel the focus of attention since the past (Knothe and Razon 2017). Biodiesel increasing usage worldwide can be considered as an alternative reductant in HC-SCR systems.

This experimental work focuses on the SCR of NO_x emissions in Ag–Pt/Al_2O_3 catalysts using ethanol and biodiesel mixtures. Its high activity in low exhaust temperatures, advantages in environmental aspects considering the toxic effects of typical catalyst and high performance in reduction of NO_x by HCs have made Ag–Pt/Al_2O_3 structure the preferred catalyst in this study. Catalytic analyses of Ag–Pt/Al_2O_3 were carried out, and the effects of ethanol and biodiesel mixtures on NO_x conversion efficiency

Table 1 Comparison of different SCR catalysts (Praveena and Leenus Jesu Martin 2018; Guan et al. 2014; Wang et al. 2017; Cheng and Bi 2014)

Catalyst	Advantages	Disadvantages
V_2O_5–WO_3/TiO_2	High activity at the medium temperature range (300–400 °C) Superior resistance to sulfur poisoning above 300 °C Widespread use in NH_3-SCR	Low activity at low and high exhaust temperatures High activity of SO_2 oxidation Toxic effects High reactivity temperature
Zeolites (Fe, Cu)	Superior thermal durability Impressive performance at low temperatures High efficiencies at high space velocities Less NH_3 storage capacity at high temperatures	Seriously poisoning by SO_2 High NH_3 storage capacity at low temperatures
Noble metal (Pt, Pd, Ag, etc.) catalyst	Excellent catalytic activity at low temperature Relatively better resistance against SO_2	High cost Narrow operation window Sensitive to SO_2 Low selectivity toward N_2 (typically 30%)
Metal (Mn, Fe, V, Cu, Cr, Co, etc.) oxide catalysts	Outstanding low temperature activity High activity and hydrothermal stability during the reduction of NO_x by HCs	Seriously poisoning by SO_2

were investigated using Ag–Pt/Al_2O_3 catalyst at different engine load and exhaust gas temperatures through a specially designed exhaust system. The study was performed during 2017–2018 at the Department of Automotive Engineering in Cukurova University (Adana, Turkey).

Materials and methods

The production and analyses of catalyst

Catalyst production was carried out using the infrastructure of the laboratories belonging to Automotive Engineering of Cukurova University in the light of literature information. In the catalyst production, cordierite ($2Al_2O_3$–$5SiO_2$–2MgO) and monolith main carrier structure with 400-cpsi (cell per square inch) squares was used. The carrier structure was obtained commercially. Due to its superior hydrothermal stability, low cost and lower thermal expansion coefficient compared to other constructions, this structure was preferred in this experimental study (Wang et al. 2015).

A silver-based solution was prepared to coat the cordierite structure. To prepare the solution, 15 g of silver nitrate (Merck—$AgNO_3$) and 0.5 g of tetraaminplatinum(II) nitrate (Sigma-Aldrich—Pt $(NH_3)_4$ $(NO_3)_2$) were added to 500 ml of distilled water and stirred in the ultrasonic mixer for about 20 min. Cordierite material was immersed in the resulting solution, and the plugging pores were opened with an air gun.

After immersion, the cordierite material was dried in the furnace at 110° C for 2 h and then was calcined at 500° C for 5 h. Thus, the production of catalyst structures was carried out.

Scanning electron microscope (SEM) and X-ray fluorescence (XRF) analyses of the cordierite structure and catalyst produced by the impregnation method were applied. FEI Quanta 650 SEM was used in the SEM analysis of the samples. Bruker S8 Tiger XRF instrument was used for the chemical analysis of the catalyst as elemental, oxide and special compound.

Preparation of reductants

In this experimental study, ethanol–biodiesel mixtures were used at different rates as reductant. Biodiesel was blended with ethanol at 5%, 10%, 15%, 20% and 25% by volume, and 5 different reductants were prepared in total. Each reductant is named according to its biodiesel and ethanol content (B5E95, B10E90, B15E85, B20E80, B25E75). Due to properties of biodiesel such as higher density, lower evaporation and higher viscosity, ratio of biodiesel in reductant mixtures was lower than ratio of ethyl alcohol. If the higher content of biodiesel is used, mixture formation with exhaust gas will be deteriorated, during the injection of reductant. As a result, this situation reduced reaction efficiency of system. Therefore, maximum biodiesel rate was used as 25% in tests.

The rapeseed oil was used in biodiesel production because it is easily available and has low cost and widespread use in biodiesel production worldwide (Lesnik and Bilus2016). Also the biodiesel derived from rapeseed oil has lower viscosities and pour point (Sajjadi et al. 2016). These specifications made the preference of biodiesel produced from rapeseed oil as a reductant in study.

The flow diagram of biodiesel production is presented in Fig. 1. The transesterification process was conducted in biodiesel production. 3 g sodium hydroxide (NaOH) (brand: Tekkim, purity: 99%) and 200 ml methanol (CH_3OH) (brand: Tekkim, purity: 99.8%) were mixed for resolving of NaOH in CH_3OH. Later they were added in rapeseed oil obtained commercially. The reaction was carried out for 1.5 h at 63 °C in 2-L capacity refluxing glass reactor (ISOLAB GmbH, 280 °C max. heating) with magnetic stirrer and thermometer. After the reaction, solution was allowed for 8 h to rest in a separatory funnel and it was divided into two phases. The upper phase formed methyl esters (biodiesel) while the lower phase formed glycerin. Lastly, the methyl ester was washed and dried at 105 °C. Memmert-UNB 500 Universal oven was used for drying of methyl esters.

The physical properties of biodiesel and ethanol are given in Table 2. Ethanol used in the study has %99 purity. It is shown in table that all properties of ethanol are lower than those of diesel. Flash point of biodiesel was six times higher than that of ethanol. Compared to that of biodiesel, density of ethanol is lower as 10.5%. Kinematic viscosities of biodiesel and ethanol were obtained as 4.21 and 1.05 mm^2/s, respectively.

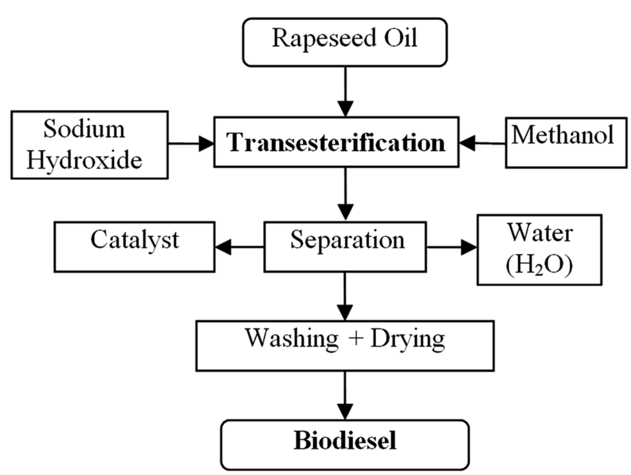

Fig. 1 Flow diagram of biodiesel production

Table 2 Physical and chemical properties of test fuels

Properties	EN590	Biodiesel	Ethanol
Density (kg/m^3)	820–845	885	792
Kinematic viscosity (mm^2/s, 40 °C)	2.0–4.5	4.21	1.05
Lower heating value (MJ/kg)	–	40.11	29.30
Flash point (°C)	Min 55	100	15
Cetane number	Min 51	45	7

Table 3 Technical specifications of the generator motor

Item	Specification
Model	Diesel AKSA A2CRX08
Number of cylinders	2
Cylinder volume (cm^3)	794
Stroke (mm)	79
Compression ratio	23/1
Engine speed (d/d)	3000
Cooling system	Water cooled

The experimental setup

A schematic representation of the experimental setup used to test the NO_x conversion efficiency of reductants in different species is given in Fig. 2. Generally, the test setup consists of a specially designed exhaust system and generator motor (Table 3) which can be operated at different loads. Thanks to the heater located on the exhaust system, the exhaust gas temperature can be adjusted to the desired values. Couple continental NO_x sensors were used downstream and upstream of the catalyst to determine conversion rates for NO_x emissions.

The reductant injection was electronically controlled to provide NO_x conversions in the SCR system. In the injection system, one injector, pump and microprocessor that controlled injection and pump were used.

Thanks to an orifice located on the exhaust system, the exhaust stream is held constant at a space velocity of 20,000 h^{-1}. Space velocity (SV (h^{-1})) is defined as the volumetrically ratio of the hourly flow velocity of the exhaust gas (V_f (m^3/h)) to the catalyst volume (V_c (m^3)) (Resitoglu et al. 2015).

Experiments were performed at 4 different engine loads as 1 kW, 2 kW, 3 kW and 4 kW. In order to determine the performance of the catalyst produced and the reductants used, especially at low exhaust gas temperatures, NO_x conversion ratios in the tests were measured at 10-C intervals in the temperature range of 170–260 °C. Each experiment performed was repeated 3 times, and the average of the values obtained was taken.

Results and discussion

Analysis results of catalyst

The SEM images in 1000/1 and 5000/1 scales of the catalyst and cordierite are given in Fig. 3. When the SEM images are examined, it is seen that the catalyst structure has a porous structure, the coating is homogeneous throughout the entire surface, and the coating materials penetrate the entire material surface including the pores.

The results of the XRF analyses carried out in order to understand the percentages of the elements and compounds in the catalyst are presented in Table 4. In the table, the elements and compounds in the high position in the cordierite and the percentages of the Ag and Pt materials used in the coating are given. The elements and compounds below 1% in the cordierite structure are not included in the table. When the results are examined, it is seen that Al_2O_3 and SiO_2 structures in both the cordierite and catalyst have the highest percentages. The reason for this is that they are the main constructs and they are used as the main template. The ceramic structures are coated with a very thin catalyst surface. Therefore, the amounts

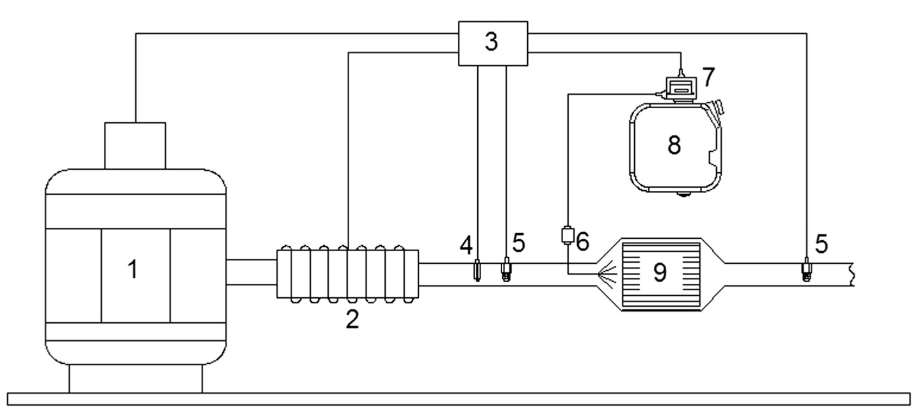

Fig. 2 Experimental setup (1—generator motor, 2—exhaust gas heater, 3—control unit, 4—temperature sensor, 5—NO_x sensor, 6—injector, 7—pump, 8—reduced tank, 9—catalyst)

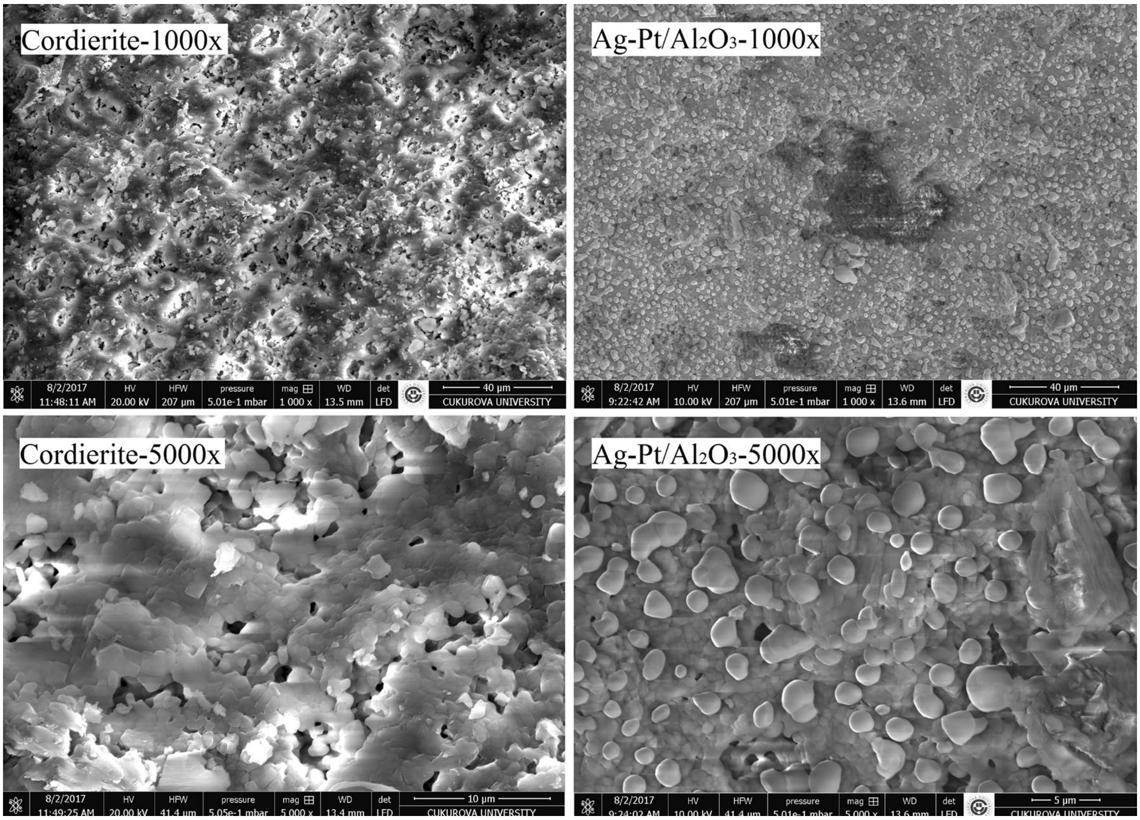

Fig. 3 SEM images of cordierite and Ag–Pt/Al_2O_3 catalyst

Table 4 XRF analysis results

Element/compound	Cordierite	Ag–Pt/Al_2O_3 catalyst
SiO_2	34.10	34.07
Al_2O_3	24.12	24.53
MgO	9.85	10.11
Si	15.20	15.80
Al	10.77	11.67
Mg	4.31	4.88
Ag		9.75
Pt		0.23

of Ag and Pt materials are low compared to these constructions. The amounts of Ag and Pt in the catalyst were 9.75% and 0.23%, respectively. The Pt material has a low percentage as it is added in trace amounts to the coating solution when compared to Ag. The obtained XRF results also support the SEM results and show that the coating materials are well penetrated into the cordierite structure.

NO_x conversion rates

Figure 4 shows the NO_x conversion ratios of the SCR system in which the Ag–Pt/Al_2O_3 structure is used as the catalyst and biodiesel–ethanol is used as reductant. NO_x conversion ratios were measured at four different engine loads, 20,000 h^{-1} SV and 10 different exhaust gas temperatures between 170 and 260 °C with 10-C intervals. The increase in exhaust gas temperature generally has an enhancing effect on the NO_x conversion efficiency. The effect of the exhaust gas temperature on the catalyst performance is quite high. The increase in exhaust gas temperature has an effect of increasing the catalyst efficiency (Kang et al. 2018). However, at temperatures of 600 °C and above, the catalyst can be damaged due to high temperatures (Resitoglu et al. 2015).

The addition of biodiesel to ethanol has improved the NO_x conversion performance. The highest conversion rates were generally obtained with B15E85 blend containing 15% biodiesel content. Considering the given all engine loads and exhaust gas temperatures, the average conversion rates were obtained as 69.15%, 70.21%, 72.86%, 71.45% and 70.91% for the B5E95, B10E90, B15E85, B20E80 and B25E75 reductants, respectively. In terms of NO_x conversion efficiency, the order of the reductants was B15E85 > B20E80 > B25E75 > B10E90

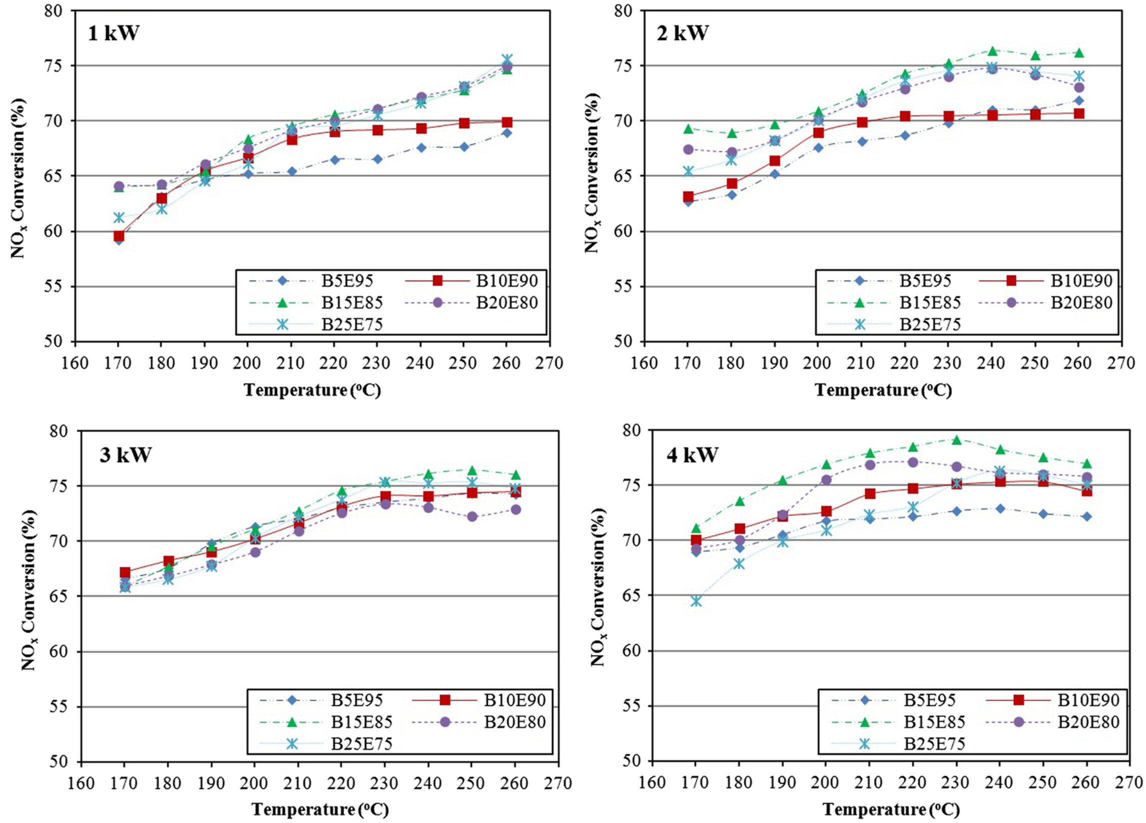

Fig. 4 NO_x conversion rates at 20,000 h^{-1} space velocity, different engine loads and different exhaust gas temperatures

> B5E95. When compared to the B5E95 reductant, the average NO_x conversion rate of the B15E85 reductant was averagely 3.71% higher. This suggests that the addition of biodiesel to ethanol increases the NO_x conversion performance. However, the B20E80 and B25E75 reductants with biodiesel ratios above 15% had a tendency to decrease when the NO_x conversion rates were compared with the B15E85 reductant. In tests, the highest NO_x conversion rate was achieved with the B1585 reductant as 79.17% at 230 °C and engine load of 4 kW.

The increase in engine load caused an increase in NO_x conversion efficiency. The average NO_x conversion ratio of all the reductants at 4 kW engine loads was found 5.76% higher than that at 1 kW engine load. Significant increases in NO_x conversion rates have been observed, especially at low exhaust gas temperatures at a 4 kW engine load. The increase in engine load enriches the mixture in the cylinder, which results in an increase in both the HC content of the exhaust gas content and exhaust temperature to improve the conversion efficiency (Bai et al. 2018; Boriboonsomsin et al. 2018; Liu et al. 2014).

Conclusion

In this experimental work, the SCR of NO_x emissions from diesel engines in Ag–Pt/Al_2O_3 catalysts using ethanol and biodiesel mixtures was carried out. The catalyst production and analysis were first carried out, and then, tests were performed on the specially prepared experimental setup to determine the NOx conversion ratios of different reductants. SEM analysis of the catalyst and cordierite structure showed that the catalyst structures had a porous structure, the coatings were homogeneous throughout the surface, and the additives penetrated the entire surface including the pores. The percentages of the coating materials in the catalyst were obtained with the XRF results. After analyzing the catalyst, it was placed in a specially designed exhaust system and NOx conversion ratios were tested at 20,000 h^{-1} SV, different engine load and exhaust gas temperatures to see the NO_x conversion rates of reductants formed blends of biodiesel and ethanol. As a result of the tests performed, the highest NO_x conversion rate was obtained as 79.17% with B15E85 reductant at 230 °C exhaust gas temperature and 4 kW engine load. Increases in engine load and exhaust gas temperature increase NO_x conversion rates. Considering given all the reductants, the best NO_x conversion performance was obtained with the B15E85 reductant.

Acknowledgements This work was supported by Mersin University Scientific Research Projects Unit (Project Code: 2018-2-AP3-2964).

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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