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Examination of Metal Doped Zeolite as Catalyst to Reduce NO_X Emission from Lean Burn Gasoline Engines

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Abstract*— Lean burn gasoline direct injection (GDI) engines are the most preferred gasoline engines because of their low fuel consumption and high thermal efficiency. However, these engines produce exhaust gases that are particularly rich in oxygen and therefore the present three-way catalytic converter (TWC) is not suitable for converting the generated NO_X emission into Nitrogen gases. In this present work, a new method of reducing Nitrogen Oxides emission in a gasoline engine is attempted by using an ordinary oxidation catalyst together with a deNO_X(zeolite-based) catalyst. In this work, Na-form of ZSM-5 zeolite was used as a catalyst and cupric chloride ($CuCl_2$) and ferric chloride ($FeCl_3$) where used as transition metals. Cu-ZSM5 and Fe-ZSM5 catalyst were prepared separately in our laboratory. Na+ ion exchange method is used to prepare the catalyst. After that Cu-ZSM% and Fe- ZSM5 catalyst were washcoated separately onto the blank monoliths. Oxidation monoliths (for oxidation of CO and HC into CO_2 and H_2O) were purchased directly from market. One oxidation monolith and one zeolite coated monolith were placed in a stainless steel container and canned with inlet and outlet cones (forming catalytic convertor). Experiments were conducted on a 2 cylinder Multi Point Port Fuel Injection engine along with a dynamometer. Exhaust emissions such as NO_X, CO, HC, O_2, CO_2 were measured with AVL Di-gas-444 Analyzer. Exhaust gas temperature is measured with the use of a thermocouple. Firstly load tests (4, 7, 10, 13, and 16KW) were conducted on the engine without catalytic convertor was fixed close to the outlet pipe and the test were conducted again with same loading condition as mentioned above. Then by the same above procedure is followed to conduct test with Cu-ZSM5 and Fe-ZSM5 catalytic convertors. From the results it is observed that both Cu and Fe zeolite catalyst minimize emissions than the commercial catalytic converter.

***Index Terms*: catalytic converter, NO_X reduction, oxidation catalyst, ZSM5 zeolite.**

I. INTRODUCTION

In recent years GDI and MPFI engines are most preferable gasoline engines for automobile manufacturers, because of their lean burn mode of operation. The negative side of the lean burn system is, it emit more NOX emission compared to conventional system. And also lean burm engines having slow burning rate and rich in oxygen exhaust. SO more efficient catalytic convertor which can induce complex chemical reactions inside the monolith is necessary to reduce toxic emissions Researchers suggest that selective catalytic reduction of NO_X with a suitable catalyst is the only way to reduce NO_X emission under lean exhaust conditions. The primary function of the catalyst is to increase the reaction rate even at lower temperatures and other kinetic barriers. However, the catalyst does not impact on the extent of chemical equilibrium of a reaction. Many researchers proved from their experiment that zeolite-X and zeolite ZSM5 are the most suitable catalyst to reduce NO_X emission in lean burn petrol engines. Vesna Tomasic and Franjo Jovic[1] & [2] compared the advantages of ceramic and metallic monoliths and explained about the incorporation of active metals into the monoliths walls. The author also discussed heat and mass transfer phenomena in a monolith reactor channel with a mathematical model. Rajakrishnamoorthy , et al. [3] prepared mono metal doped ZSM5 (Cu-ZSM5) catalysts and bimetal doped catalyst (Ce.Cu-ZSM5) to reduce the SI engine emissions in the exhaust in a constant speed engine for varying loads. The author reported that 55.8% of NO_X conversion efficiency and 57.4% of CO conversion efficiency were achieved using this catalytic convertor. The same team [4,5] analyzed Copper-X, Nickel-X and Vanadium-X as a catalyst in SI engine exhaust and observed 61.2% with Cu-X, 59.7% with Ni-X and 56% with V-X catalysts of increased NO_X conversion efficiencies. Elavarasan.G et al. [6] have successfully synthesized the ZSM5 zeolite material from the waste fly ash, the use of zeolite like substance as a catalyst will decrease the cost effectiveness of the catalytic convertor. Elavarasan.G et al. have also explained about the various emission standards of our country and the need to reduce the NO_X emission in the automobiles, thus the zeolite based catalyst substance will be a promising solution to meet the required emission standards [7]. In our earlier work [8], Lignite fly ash was used to synthesize zeolite –X like material and Copper, Nickel and Iron are used as transition metal. Three catalyst were prepared by keeping X-zeolite as base metal namely Cu-X, Ni-X and Fe-X using ion exchange method. The test were performed on the catalyst in a 3-cylinder petrol engine having 800cc and the results were recorded as 60% in NO_X reduction, 85% in CO reduction and 80% in HC reduction. In our other work [9] Copper-X and Iron-x zeolite are prepared and washcoated into a monolith and tested in twin cylinder nano engine. The result obtained is the NO_X conversion efficiency is around 57% at 300 deg C, CO and HC conversion efficiencies are 85% and 87% respectively at 16kw load condition. Ghosh BB et al[10], prepared Copper-ZSM-5 zeolite and experimented in 800cc maruti engine, and recorded that maximum conversion efficiency achieved is 85% of NO_X at 490deg and 71% CO at 495deg and 72% HC at 500 deg Celsius.

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The same author in his other papers [11&12] developed CU-ion exchanged ZSM5, Cu-Pt-ZSM5 and Cu-Rh-ZSM5 catalysts and fitted them in a 3 staged converter to compare the efficiencies of CO, HC and NO_X in same 800cc engine. The results achieved are the NOx reduction of CO 90% at 375deg, NO_X 90% at 375 deg and HC 61% at 380deg Celsius. Joseph R Theis [13,14] reported both durability assessment and performance of SCR for reducing NOx emissions from SI engines. Elavarasan.G et al. [15] have reported that implementing the advanced technology in the automobile in terms of reducing emission will increase the overall cost of the automobile, but this zeolite based catalyst will reduce the automobile cost.

Juan M Zomaro et al. [16], Meille V [17] discussed several techniques, to incorporate zeolite material onto the ceramic honeycomb monoliths. L. Lisi et al, [18] have investigated washcoating of Cu-ZSM5 catalysts into cordierite monolith by a dipcoating technique using silica as a binder, with several zeolite/binder ratios in aqueous suspension determining the parameters to obtain the best adhesion and mechanical resistance of the washcoat layer. The author used ultrasound treatment to investigate the adhesion strength and tubular quartz reactor for catalytic activity test. In this work, we used ZSM5 zeolite as a catalyst support.

II. PREPERATION OF METAL DOPED ZEOLITE

A. *Preperation of catalyst*

ZSM5 zeolite sample (purchased from zeolyst International, USA) is used as base material and cupric chloride, ferric chloride (purchased from Fisher scientific) are used as a transition metal. Two types of catalysts (Cu-ZSM5 and Fe-ZSM5) are prepared in our laboratory following the procedure explained below.

B. *Na^+ ion exchange method*

Among various methods adopted to prepare the catalyst, liquid phase Na^+ ion exchange method proves to be effective because of its simplicity. It is a modified process (L Lisi 2009 et el[18], Yang 2008 et al[19] and Metkar P.S 2011 et al[20] . A sample of 100 grams of ZSM5-zeolite powder and 0.5M of $CuCl_2$ of 100ml is mixed with1000 ml of 0.5M $CuCl_2$ solution to form a solution. This mixture is placed in a round bottom flask and stirred continuously for one day at normal room temperature. During this period ion exchange of Na and Cu takes place. After ion exchange the mixture is poured into the vacuum filtered apparatus and filtered and washed with de ionized water, to remove free ions. The slurry is then placed in the oven and heating gradually at 500 °C for a period of six hours. A similar procedure is applied to prepare Fe-ZSM5 zeolite catalyst.

III. CHARACTERIZATION OF ZSM5 ZEOLITES AND METAL DOPED ZEOLITES

A. *X-Ray Fluorescents (XRF)*

The XRF method is applied to determine the chemical composition of ZSM5 and metal-doped zeolites. The Philips Spectrometer PW1404 is used. Table I presents the result of chemical composition of ZSM5, Cu-ZSM5 and Fe-ZSM5. It is noticed from Table I that there is a marked decrease in the % of Na_2O in the zeolite material and also around 6% increase of Cu and Fe species in Cu-ZSM5 zeolite and Fe-ZSM5 zeolite respectively.

Table I Chemical Composition Of Zsm5, Cu-Zsm5 And Fe-Zsm5 Zeolite (Mass %)

Composition	ZSM5	Cu-ZSM5	Fe-ZSM5
SiO_2	88.372	87.012	87.092
Al_2O_3	3.256	3.091	3.150
Fe_2O_3	0.114	0.110	5.982
CaO	0.014	0.014	0.011
MgO	1.150	1.050	1.110
SO_3	0.134	0.126	0.123
Na_2O	7.001	1.515	1.095
K_2O	0.00	0.00	0.00
CuO	0.00	5.012	0.00
P_2O_5	0.00	0.00	0.00
TiO_2	0.025	0.22	0.21
BaO	0.00	0.00	0.00
LOi	0.00	0.00	0.00

B. *Scanning Electron Microscopy (SEM)*

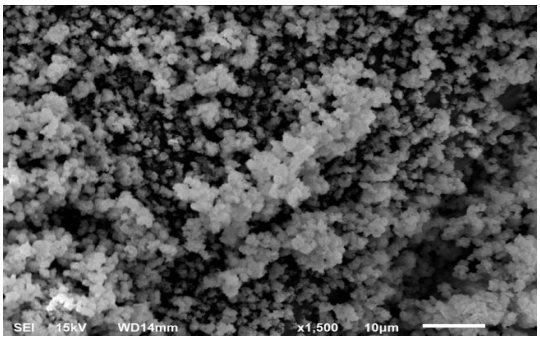

Figure 1.SEM image of ZSM5

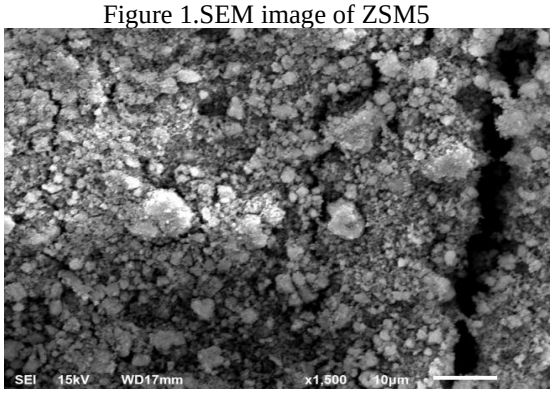

Figure 2.SEM image of Cu-ZSM5

Scanning Electron Microscopy analysis is carried out with a JEOL-JSM 6610LV electron microscope. Micrographs of the samples are recorded with a 10-20 kV accelerating voltage. Figure 1, 2, 3 show the SEM images of ZSM5 zeolite and metal doped zeolites. It is evident from the figure that the microstructure of the metal doped zeolites is completely changed and the particle size is slightly bigger than that of ZSM5 zeolite. This is because, during ion exchange process, the transition metals are distributed or doped on the ZSM5 zeolite particles.

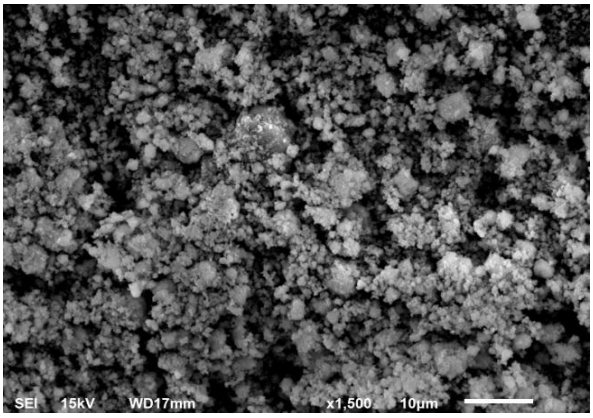

FIGURE.3. SEM image of Fe-ZSM5

C. *X-Ray Diffraction (XRD)*

The crystal structures of the samples are determined by powder X-ray diffraction on a DX-1000 diffractometer using CuK_α radiation (λ=0.15406 nm) and operating at 40 kW and 25 ma. The XRD data are recorded for 2θ values between 0° and 100°. The crystalline phases are identified by comparison with the reference data from the JCDPS card and ICDD.

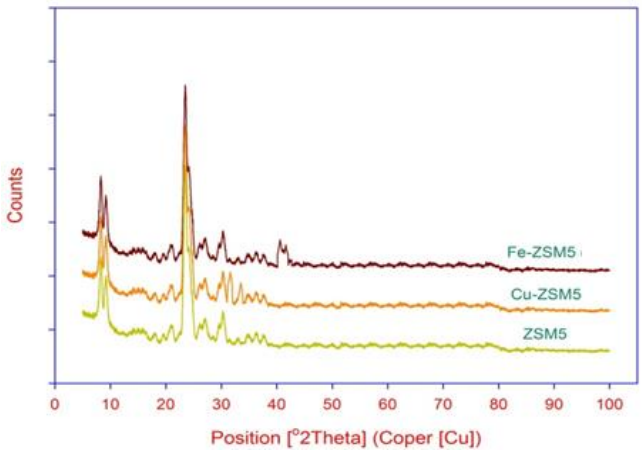

Figure.4 XRD Patterns Of ZSM5, Cu-ZSM5, Fe-ZSM5 zeolites

Figure 4 shows the XRD pattern of ZSM5 zeolite and metal (Cu, Fe) doped on the zeolites. It is observed from the pattern that the major original structure of zeolites (i.e. the major peaks of SiO_2 and Al_2O_3) remains unchanged after the ion exchange process. It is also observed that some low-intensity diffraction lines (peaks) disappear and new diffraction lines are formed, which is identified as metallic (Fe and Cu) species. It is evident from the figure that there is no increase or decrease in the content of the SiO_2 and Al_2O_3 species after the ion exchange process and only Na^+ ions are replaced by metal ions.

D. *Braunauer-Emmett-Teller's (BET) method and particle size analyzer*

Table II shows the results of average particle size distribution and surface area of ZSM, Cu-ZSM5, and Fe-ZSM5. It is noted that when the particle size is increased or becomes larger it resulted in decreased surface area. It is evident from the table that the particle size distribution is increased in Cu and Fe doped zeolites. It is because the transition metals are well distributed on the ZSM5 zeolite particles during ion exchange process.

Table II Result Of Average Psd And Bet

Sample	Average Particle size Distribution (d.nm)	BET (m^2/g)
ZSM5	315.4	425
Cu-ZSM5	336.3	395
Fe-ZSM5	328.2	401

IV. CORDIERITE MONOLITH

Oxidation monoliths and blank monoliths were commercially ordered from Bocent Advanced Ceramics Co Ltd, China. The diameter and the length of the monolith is having the same dimension of 90mm. 400 cpsi is the cell density of both the monoliths and the 0.17mm of wall thickness. Figure 5 shows the photographic view of blank monoliths.

Figure.5. Photographic View Of Blank Monoliths

V. MONOLITHIC WASHCOATING

Among the various methods adopted for deposition of catalyst on the monolith, the conventional dip coating method proves to be effective because of its simplicity and versatility (M.A. Ulla et al., 2003[21]; Valerie Meille, 2006[17]). The Slurry is prepared using 50 weight% of Cu-ZSM5 zeolite, 4 weight % of silica gel and remaining weight % of water. Uncoated monolith is immersed completely into the slurry for a minute and then the excess slurry can be removed with the help of blowing compressed air on the monolith for about five seconds from both the ends. Then the monolith is kept in a box type muffle furnace and dried for two hours at a temperature of 120 °C. The dipping and drying processes are continued until the deposited 16% weight of monolith is wash coat. At last, the substrate is calcined for five hours at 500 °C. The same procedure is applied to wash coat the Fe- ZSM5 zeolite.

VI. FABRICATION OF CATALYTIC CONVERTER

Oxidation monolith (purchased from market) and Cu-ZSM5 zeolite-coated monolith are placed together, housed in a steel casing and then welded with inlet and outlet cones. Proper provisions are also made in order to fix the catalytic converter in the outlet pipe of the engine. In a similar method, Fe-ZSM5 zeolite coated monoliths are also fabricated. Figure.6 shows the photographic view of the in house made catalytic converter and Figure.7 shows the photographic view of the OEM catalytic converter.

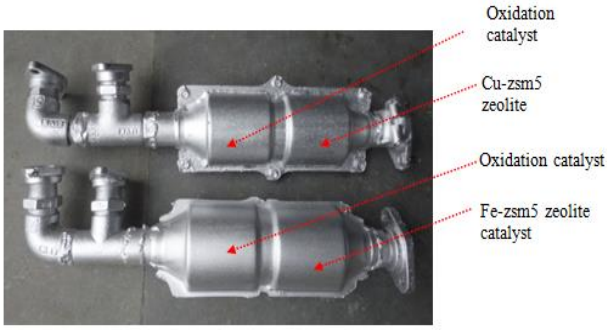

Figure.6. In-House Made Catalytic Converter

Figure.7. OEM Catalytic Converter

A. *Experimental Study*

The tests are conducted on TATA Nano stationery, 2 cylinder, 4 stroke, water cooled gasoline engine. The engine air fuel ratio is altered to run at 17.5:1 condition by company's authorized representative. Figure 8 represents the block diagram of the experimental setup used in this work. The eddy current dynamometer is used for varying the engine load conditions while running. Emission of CO, HC, CO_2, O_2 and NOx are measured using AVL-Di gas analyzer. The Chromel-Alumel thermocouple is used to measure the temperature of exhaust gas. Tests are conducted at different load and temperature conditions. The readings was taken with the for the engine without fitted with catalytic convertor (4, 7, 10, 13 and 16 kW) at a constant speed of 2500 rpm. In each load condition, the concentration of CO_2, O_2, HC, CO and NOx emissions are measured. Then the commercial catalytic converter is placed along with the engine exhaust pipe and the engine is run at the same load conditions. The concentration of various emissions like CO, HC, CO_2, O_2, and NOx were measured in each load condition. For temperature test, the exhaust gas temperature is raised from 100°C to 500°C in step-raise of 50 °C, by suitably raising the load of the engine. In each increment, after reaching the steady-state condition, the NOx emission is measured. Then, the same process is repeated by fiting Cu-ZSM5 zeolite and Fe-ZSM5 zeolite catalytic converters. The specification of the petrol engine is shown in Table.III and the eddy current dynamometer specification used in this study is shown in Table IV.

TABLE III Specification of PETROL ENGINE

Type	MPFI Engine
Number of cylinders	2
Displacement	624 cc
Bore	73.5 mm
Stroke	73.5 mm
Compression Ratio	9.5:1
Fuel	Petrol
Cycle	4-Stroke
Max. Engine output	25.74 kW @ 5250 rpm
Max. Torque	48 Nm @ 3000 rpm
Speed	2500 rpm
Orifice Diameter	20 mm
Cooling System	Water
Loading Device	Eddy current dynamometer

TABLE IV Specification of EDDY CURRENT DYNAMOMETER

Make	Shenck Avery
Serial No	ASE- 010/05
Type	ASE- 50
Year	2005
Max	50 kW

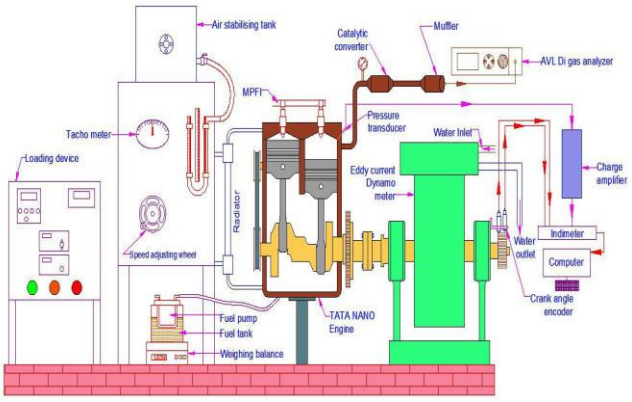

FIGURE.8 Experimental Setup

VII. RESULTS AND DISCUSSIONS

A. *Nitrogen oxides emission with load*

Figure 9 shows that NO_x conversion efficiency with the brake power of the engine. It figure.9 shows that the zeolite-based catalytic converter reduces NO_x emission significantly high when compared to the OEM catalytic converter. The maximum NO_x reduction by OEM catalytic converter is around 35%, On the other hand, the zeolite-based catalytic converter reduces NO_x emission around 55% at 4KW brake power, and slowly increases and reaches 70% at 16KW load condition. The reason is that in OEM catalytic converter, three reactions (reduction of CO, HC and NO_x to CO_2, H_2O and N_2) occur simultaneously and also the active sites for NO_x reduction are available only in the corners of the monolith channels.

In case of zeolite-based catalytic converters more active sites are available for NO_x conversion. When the exhaust gas after leaving the oxidation monolith contacts the zeolite-based monolith, the following heterogeneous reactions take place (i) the adsorption of NO on the active metal sites, followed by dissociation into $N_{(ads)}$ and $O_{(ads)}$, (ii) two $N_{(ads)}$ combined to give N_2, (iii) the dissociated $O_{(ads)}$ available on the metal sites react with remaining CO in the exhaust gas and produce CO_2.

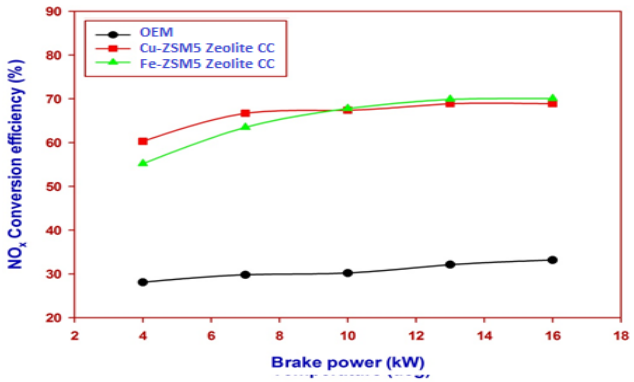

Figure 9 Percentage reduction of NOx Vs load

B. *Nitrogen oxides emission with temperature of exhaust gas*

Figure 10 shows the NO_x reduction efficiency with respect to the temperature of exhaust gas. It is observed from the figure that the conversion efficiency of the zeolite-based catalytic converter is suddenly increases and reaches 60% at about 190 ºC and steadily increases up to 500 ºC, after which there is no significant increase. The light-off temperature of the zeolite catalyst is estimated to be around 180 °C. So when the temperature of the catalyst is below 150 °C, the zeolite is less active. When the temperature exceeds a threshold level of 180 °C, the zeolite becomes more active and hence there is a sudden increase in the conversion efficiency of NO_x. The same behavior is not observed with the commercial converter as the exhaust is rich in oxygen and they could not reduce NO_x significantly.

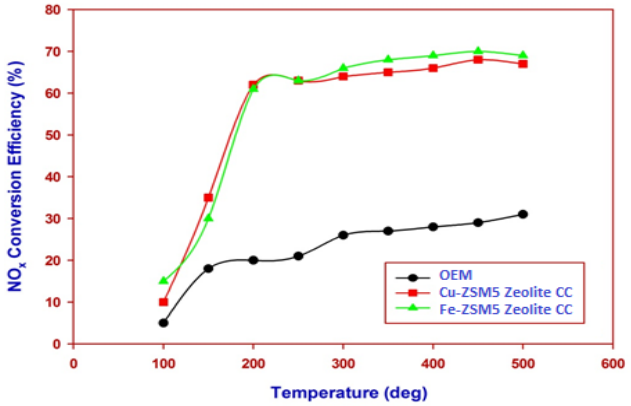

Figure.10 Percentage reduction of NOx Vs temperature

C. *CO Conversion efficiency*

Figure 11 shows that the CO conversion efficiency of OEM catalytic converter and zeolite-based catalytic converter. It is noticed from the graph that, the OEM catalytic converter also reduce significantly the CO emission level under lean burn exhaust condition. It is seen from the figure that around 93% of CO conversion efficiency is detected at 4 kW load condition and reaches around 95% at 16 kW load condition. This is since the remaining unreacted CO, leaving the oxidation monolith, again reacts with adsorbed oxygen (generated by NO_x dissociation) on the surface of the zeolite-based catalyst and gets converted into CO_2.

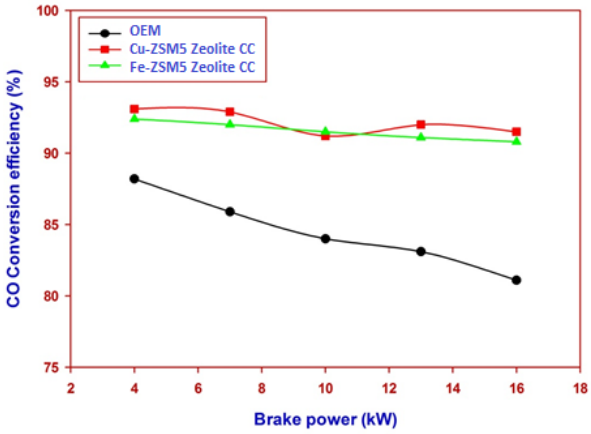

Figure 11 Carbon monoxide against brake power

D. *HC Conversion efficiency*

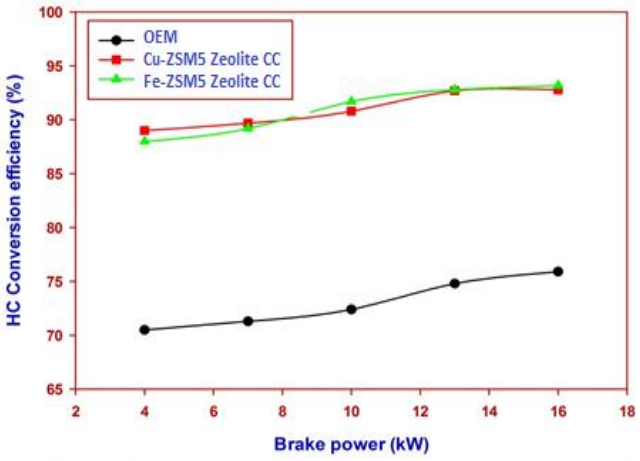

Figure 12 Hydrocarbon Vs brake power

Figure 12 shows the % reduction of HC emission with the load of the engine. It is viewed from the figure that the NO_x conversion efficiency of OEM catalytic converter is 72% at 4 kW load condition and gradually increases and reaches 79% at 16 kW load condition. On the other hand, in the zeolite-based converter system, HC conversion efficiency is around 90% at 4 kW load condition and gradually increases to 93% at 16 kW load condition. It is since the unreacted HC emission leaving the oxidation coated catalyst again reacts with adsorbed oxygen and produces CO_2 and H_2O. Many researchers (Jan Kaspar et.al, 2003, Liu Zhiming et al., 2004) have mentioned that HC acts as a reducing agent and rapidly removes the $O_{(ads)}$ produced by NO_x dissociation reaction and keeps the active sites in reduced stages (Cu^+ or Fe^+), for further NO_x reduction[22].

E. Brake Thermal efficiency

Figure 13 represents the brake thermal efficiency against the brake power of the engine. It is witnessed from the figure that the brake thermal efficiency is slightly reduced using zeolite based monolith catalytic converter compared to OEM catalytic converter. The reason is that small flow resistance of exhaust gases across the monolith walls exists. This may be due to conventional washcoating of zeolite material in the channels of monoliths, will reduce the total exhaust gas flow area.

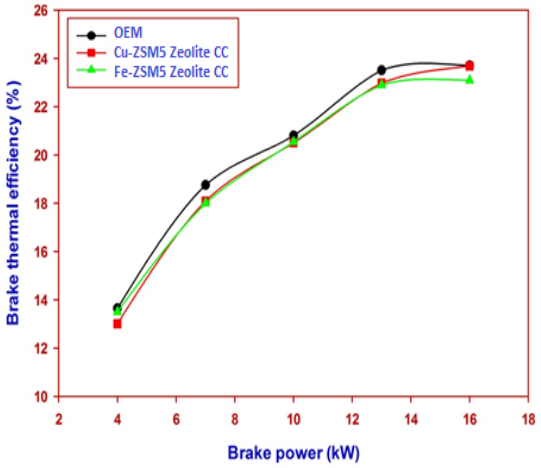

Figure 13.Brake thermal efficiency against brake power

VIII. CONCLUSION

- The in-house made zeolite based catalytic converter is more active then OEM catalytic converter at wide range of exhaust gas temperature.
- NO_x conversion efficiency achieved by Fe-ZSM5 zeolite, Cu-ZSM5 zeolite and OEM catalytic convertor at 16 kW load condition were 70%, 68% and 30% respectively.
- The CO conversion efficiency achieved by zeolite based catalyst is 94% and 92% respectively at 4 kW and 16 kW load conditions.
- The HC conversion efficiency achieved by zeolite based catalyst is 89%, 90% and 92% at 4, 10 and16 kW load conditions respectively.
- Back pressure developed is well within the acceptable limits across the catalytic bed.
- No appreciable deactivation of converter was observed, even after 100 hours of experimental examination of zeolite based catalytic converter.

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