



Investigating CRDI Engine Performance with ZSM-5 Coated Catalytic Converters for Exhaust Emission Reduction

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Cite this study:

Sethuraman, Karthikeyan, Sarangapani Palani et.al.,(2024)..Investigating CRDI Engine Performance with ZSM-5 Coated Catalytic Converters for Exhaust Emission Reduction. Turkish Journal of Engineering, 9 (3), 471-478.

<https://doi.org/10.31127/tuje.1542632>

Keywords

Zeolite, Flyash,
CRDI Engine, Emission
Catalyst

Abstract

Nowadays, light duty diesel engines and lean burn petrol engines are getting much attention due to improved fuel economy. The present conventional catalytic converter controls effectively the levels of carbon monoxide and hydrocarbon, but it displays poor conversion in harmful oxides of nitrogen emission under lean exhaust condition. But the automobile pollution control regulatory bodies tighten the emission level every year. Zeolite-based catalysts have received a lot of focus recently because of their strong activity and comparatively broad temperature window. In the present work, zeolite-like material and ZSM-5 like material are synthesized by alkali fusion followed by hydrothermal treatment from coal flyash. Cupric Chloride (CuCl₂) and Ferric Chloride (FeCl₃) are used as transition metals. These metals are incorporated separately into the zeolites by conventional liquid phase ion-exchange method. Cu-ZSM5 (In house). Fe-ZSM5 (Inhouse), Cu-ZSM5 (commercial). FeZSM5 (commercial). Emission tests are conducted on CRDI engine. Initially the engine is run without catalytic converter in five different load conditions In all the cases, the concentration of CO, HC, O₂, CO₂ and NO, are measured by AVL Di-gas analyzer. It is observed that commercial catalytic converters at 16 kW load condition, the NO, conversion efficiency is 70%, 65% and 35%; the CO conversion efficiency is 95%, 80% and 82% and HC conversion efficiency is 93%, 79% & 80% respectively. For diesel engine, Cu-ZSMS (IH) like material is more effective in low temperature application.

Research/Review Article

Received:03.09.2024

Revised:05.10.2024

Accepted:07.10.2024

Published:01.07.2025



1. Introduction

With these twin objectives of better fuel efficiency and reduced harmful emissions, the automotive industry has been in continuous evolution. Diesel engines have conventionally been recognized for their superior fuel economy and durability, hence a preferred choice for light-duty vehicles down to commercial applications. However, during the past decades, environmental concern was caused since these vehicles have emitted appreciable amounts of NO_x, CO, HC, and PM. Certainly, this resulted in increasingly stringent emission regulations around the world and urged researchers and manufacturers into developing advanced technologies for emission control [1].

Catalytic converters have been the focus of most emission control strategies because they can be used to effect a chemical transformation in toxic pollutants to relatively benign compounds[2]. Conventional catalytic converters also use noble metals like platinum (Pt), palladium (Pd), and rhodium (Rh) for the oxidation of CO and HC and for the reduction of NO_x to nitrogen and oxygen[3]. These traditional catalytic converters, however, operate under stoichiometric combustion conditions. Under leanburn conditions-a characteristic feature of diesel engines-these converters are much less efficient[4]. Lean-burn engines burn fuel with excess air, which results in higher combustion temperatures and, therefore, in increased formation of NO_x[5]. A serious problem is that NO_x emissions make up the most

significant contributors to smog, acid rain, and respiratory problems in humans[6].

These reasons have stimulated recent work to investigate the use of various alternative materials that can efficiently perform at high temperatures as well as under lean-burn conditions, which characterize diesel engines[7]. Of the various ones investigated, zeolite-based catalysts have been found very promising. Zeolites represent a group of microporous aluminosilicate minerals, characterized by high surface area and good thermal stability with cation exchange capabilities, fitting for a wide range of catalytic applications[8]. The special structure of zeolites, which is represented by a three-dimensional microporous framework, offers only selected adsorption and reaction of certain molecules, which may become very useful and favorable in NO_x emissions reduction [9].

Among the most popular and widely studied zeolites for application in automotive is ZSM-5-a trade name (Zeolite Socony Mobil-5). ZSM-5 is noted for its high silica-to-alumina ratio, allowing a large number of strong acid sites for catalytic activity[10]. The addition of transition metals like copper (Cu) and iron (Fe) in the framework of ZSM-5 greatly enhanced its catalytic performance, especially in the selective catalytic reduction of NO_x using hydrocarbons as reductants. For example, Cu-ZSM-5 and Fe-ZSM-5 catalysts have been characterized by strong redox properties and high thermal stability, making them ideal candidates for the reduction of NO_x emissions under leanburn conditions[11].

Presently, this study focuses on the performance of a CRDI engine with ZSM-5 coated catalytic converters for the reduction of exhaust emissions. CRDI technology finds widespread application in modern diesel engines due to the fact that it can control the quantity and exact timing of fuel injected into the combustion chamber with high precision, hence optimizing combustion efficiency and giving reduced fuel consumption[12]. However, the high pressures and temperatures of CRDI engines tend to increase NO_x formation further. More efficient catalytic converters have hence been developed[13]. In the present study, the preparation of ZSM-5-like materials was obtained by alkali fusion of coal fly ash followed by hydrothermal treatment. Coal fly ash is a waste material produced during coal combustion, here used as a raw material that is low in cost and easily available. Indeed, in several studies, coal fly ash has been proved to be a source material for zeolite synthesis[14]. The synthesized ZSM-5 was then doped with copper and iron by the conventional liquid-phase ion-exchange method to enrich its catalytic performance[15]. The synthesized and commercially available ZSM-5 catalysts were then tested on a CRDI engine for their effectiveness in exhaust emission reduction under various load conditions.

Initial tests were conducted on a CRDI engine without a catalytic converter and then with the ZSM-5 coated converters[16]. The emission test measured the concentration of CO, HC, O₂, CO₂, and NO_x in the exhaust gases by using an AVL Di-gas analyzer for a range of

different engine loads. The results indeed showed that the in-house synthesized catalyst Cu-ZSM-5 outperformed its commercial competitors, especially at relatively lower temperatures[17]. For instance, NO_x conversion efficiency of about 70% at a 16 kW load was recorded for the Cu-ZSM-5 (in-house) catalyst, significantly higher than that observed in commercial catalysts under similar conditions[16].

Moreover, it was remarked that with a Cu-ZSM-5 catalyst, CO and HC had conversion efficiencies as high as 95% and this indeed means that not only is NO_x emission reduced effectively by the Cu-ZSM-5 catalyst but also, simultaneously, the overall performance of the engine in controlling emissions is improved[17]. The in-house and commercial Fe-ZSM-5 catalysts also produced very encouraging results, though with relatively lower conversion efficiencies compared to those of Cu-ZSM-5 catalysts. Thus, the potential of such transition metal-doped ZSM-5 catalysts for solving the NO_x reduction problem in diesel engines, especially under lean-burn conditions, is not limited[18]. In addition, the synthesis of the ZSM-5 using coal fly ash as a precursor represents another green and economical way to obtain the catalyst. Fly ash is widely available in industries as waste and hence valorization for synthesizing zeolites; this not only gives value addition to the by-product but also makes it ecologically viable by reducing consumption of virgin raw materials. Besides, high-performance catalysts prepared from low-cost materials are expected to greatly decrease the total cost of emission control technologies, rendering them more feasible for widespread application in the automotive industry[19-21].

Meeting the future emission standards and reducing environmental impact from diesel engines will depend on developing advanced catalytic converters capable of functioning well under a wide range of conditions as regulations further develop. The present study further extends this knowledge on the application of zeolite-based catalysts in the control of automotive emission and points out the Cu-ZSM-5 and Fe-ZSM-5 catalysts as promising candidates for CRDI engines harmful emissions reduction[22-24].

The study of ZSM-5 coated catalytic converters for CRDI engines' exhaust emission reduction specifies the potential of these catalysts in improving air quality by reducing NO_x, CO, and HC emissions[25-29]. The ultimate performance of Cu-ZSM-5 catalysts, especially from coal fly ash synthesis, is directing further research and innovation. In this context, while the car industry is trying to maintain a proper balance between its needs for fuel efficiency and ecological concern, cost-efficient and effective catalytic converters will be highly instrumental in realizing these goals.

2. Materials and Methodology

From the review of literature, it is observed that catalyst, catalytic converter, selective catalyst reduction technique for NO_x reduction and Urea solution as reducing agent have been studied by number of

researchers. Keka Ojha et al. have synthesized X-zeolite from coal fly-ash and compared the physio-chemical properties with commercial 13-X zeolite. M. Chareonpanich et al. synthesized ZSM-5 zeolite with different SiO₂/Al₂O₃ mole ratio (20-100) from Fly-ash of lignite.

Though number of researches have been made on the potential of catalyst material for reduction of NO_x, [30-32] no literature is available related to fly-ash based catalyst, which is used to reduce oxides of nitrogen emission under lean exhaust conditions.

The primary objective of this study is to investigate the effectiveness of zeolite-based catalytic converters, specifically those containing ZSM-5 material, in reducing exhaust emissions from a CRDI engine under various load conditions. The study aims to evaluate the catalytic performance of Cu-ZSM5 and Fe-ZSM5 materials (both in-house synthesized and commercial versions) by measuring the conversion efficiencies of CO, HC, and NO_x at different engine loads. Additionally, the study seeks to compare the performance of the commercial and in-house synthesized catalysts and determine the suitability of these materials for low-temperature diesel engine applications.

2.1 Materials

The samples of fly ash are collected from three thermal power plants in India namely, (NLC), Mettur Thermal Power plant (MTP) and Tuticorin Thermal Power plant (TTP). Table 1 shows the physio-chemical properties of the fly ash samples as determined by XRF.

Table 1. Samples of fly ash's chemical composition (weight %)

Composition	NLC Fly-ash	TTP Fly-ash	MTP Fly-ash
SiO ₂	48.50	49.50	53.52
Al ₂ O ₃	22.50	30.52	25.61
Fe ₂ O ₃	4.31	4.95	4.21
CaO	15.20	2.52	2.05
MgO	2.40	1.80	2.15
SO ₃	0.79	1.70	2.20
Na ₂ O	0.90	0.50	0.95
K ₂ O	1.50	1.82	1.52
P ₂ O ₅	0.55	1.53	2.01
TiO ₂	1.02	2.50	2.90
BaO	1.22	0.88	0.90
LOi	1.11	1.78	1.98

Coal fly ash: Specify the source of coal fly ash, its composition (e.g., silica, alumina content), and any pretreatment methods before alkali fusion.

Alkali fusion process: Detail the conditions (e.g., temperature, time, and ratio of fly ash to alkali) under which the alkali fusion was carried out. Include details about the type of alkali used (e.g., NaOH or KOH) and its concentration.

Hydrothermal treatment: Provide the specifics of the hydrothermal treatment, including temperature, duration, and pH adjustments (if any).

Transition metals: Mention the source and purity of CuCl₂ and FeCl₃, and explain how these materials were introduced into the zeolites. Describe the conventional liquid phase ion-exchange method in detail, such as the temperature, time, and concentration of solutions used.

Zeolite samples: Indicate if any characterization techniques (XRD, BET surface area, SEM) were used to confirm the synthesis of zeolite materials.

CRDI Engine: Provide the model and technical specifications of the CRDI engine used for testing.

It is observed from the above table that, the calcium oxide content of NLC fly ash is high compared to the other flyashes. Hence high concentration of HCl solution is required to reduce calcium oxide during acid treatment process. Additionally, it can be noticed that the MTP fly ash has a somewhat higher SiO₂ percentage than fly ash from other thermal power plants. Hence MTP fly ash is chosen for this research work to attain higher NO_x conversion.

Zeolites are microporous, crystalline aluminosilicates with a high surface area (300-700 m²/g) and a three-dimensional framework of SiO₄ and AlO₄ tetrahedra. Their well-defined pores enable selective adsorption and catalytic activity, making them effective molecular sieves. Zeolites possess ion-exchange capacity, allowing for the incorporation of metals like Cu²⁺ and Fe³⁺, enhancing their catalytic performance. They are thermally and chemically stable, with both Brønsted and Lewis acid sites that drive catalytic reactions. Their hydrophilicity or hydrophobicity depends on the Si/Al ratio, making them useful in diverse applications like emission control, petrochemical processing, and water treatment.

The sample's XRD data for SiO₂, Al₂O₃, CaO, and MgO are used to compare the sample's results with those from the JCPDS is shown in figure 1. Most of the 2 values are expected to match the JCPDS patterns for SiO₂ (JCPDS card no. 89-1668) and Al₂O₃ (JCPDS card no. 88-0107). Only a few samples' peaks fit the JCPDS patterns for CaO (JCPDS card no. 82-1690) and MgO (JCPDS card no. 89-7746). It displays the data for comparison. According to the XRD data, SiO₂ and Al₂O₃ are the two oxides that are most common in the raw fly ash employed in this study.

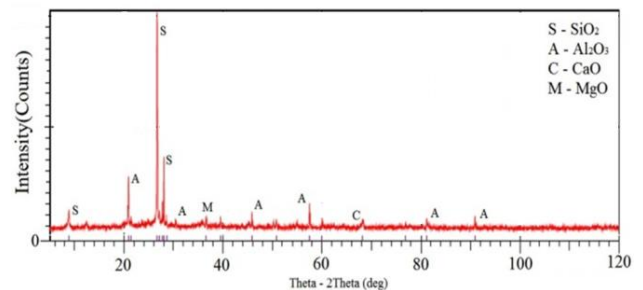


Figure.1 MTP fly ash- XRD Pattern

2.2 Synthesis of ZSM5 like material

A flyash sample is put through a sieve (180 micron) for screening in order to remove coarse particles. Hydrochloric acid is then used to treat the fly ash in order to remove some unwanted components. (e.g., CaO, Al₂O₃, SO₃, etc.). The acid solution is prepared with 20% of concentrated HCl and 80% of de-ionized water. Then the fly-ash sample is added with acid solution in the ratio 20 ml/gram. At room temperature, the mixture is continually stirred for two hours. After reaching a neutral PH, the solution is filtered and repeatedly rinsed with deionized water before being dried for an entire night at 120°C in a Muffle furnace. In a weight ratio of 1:0.5:1, The powdered sodium silicate pentahydrate (Na₂SiO₂5H₂O) and solid powdered NaOH are mixed with the HCl-treated fly ash sample. This combination is ground, then put into an aluminum tray and fired in a furnace for a day at 550 °C. The fused powder is once more crushed and added to a glass flask along with 10g/100ml of distilled water. The slurry is matured for one day at room temperature while being stirred. The mixture is then dried for six hours at 90 °C and atmospheric pressure. The surplus sodium hydroxide and soluble pollutants are then removed by vacuum filtration after all the precipitated particles have been rinsed twice with distilled water. The solid powder (material that resembles X-zeolite) is heated to 80 °C and dried in the air for 12 hours.

3. PREPARATION OF METAL DOPED ZEOLITE

3.1 Preparation of catalysts (for diesel engine)

The ZSM-5-like material used in this study was synthesized using coal fly ash, an abundant industrial waste material, as the primary source of silica and alumina. This process began with an alkali fusion treatment, wherein the fly ash was treated with sodium hydroxide, followed by hydrothermal synthesis to promote the formation of a zeolite framework. This method is environmentally sustainable as it utilizes waste materials, reducing the need for virgin raw materials in catalyst synthesis.

In addition to the synthesized ZSM-5, commercial zeolite samples such as Na-form ZSM-5, Beta, Mordenite, and Ferrierite were also employed as base materials for catalytic studies. These commercial zeolites were purchased from Zeolyst International, a renowned supplier of high-quality zeolites. Figure 2 provides a photographic view of these commercial zeolite samples, highlighting their uniformity in particle size and structure.

For the catalytic enhancement, Cupric Chloride (CuCl₂) and Ferric Chloride (FeCl₃) were used as transition metals, which were incorporated into the zeolite framework via ion-exchange processes. These metals play a crucial role in enhancing the catalytic activity, particularly for the reduction of NO_x and CO emissions under lean burn conditions in diesel engines, making the materials highly effective for emission control applications.



Figure 2 Photographic view of zeolite samples

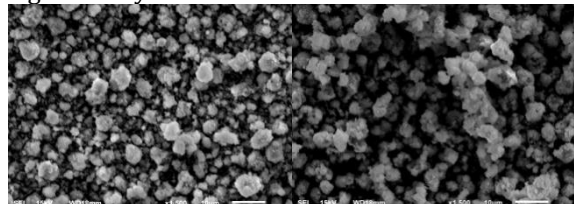
3.2 Catalysts preparation

Metal (Cu, Fe) / zeolite [ZSM5 (in house), ZSM5 (commercial), beta mordenite and ferrierite catalysts are prepared by conventional liquid phase ion exchange method. 100 grams of zeolite powder are combined with 1000 cc of 0.5 M metal solution to create a sample. The mixture is then continuously mixed for 24 hours at room temperature. After ion exchange, the mixture is vacuum filtered and completely de-ionized water washed until no free ions are present. The aforementioned sample is then dried and heated to 550°C for six hours while being calcined in air. The above process is followed to prepare ten different catalysts namely, Cu-ZSM5 (IH), Fe-ZSM5 (IH), Cu-ZSM5 (commercial), Fe-ZSM5 (commercial), Cu-beta, Fe-beta, Cu-mordenite, Fe-mordenite, Cu-ferrierite and Fe-ferrierite. The amount of metal (Fe or Cu) content loaded into the zeolite is around 12%.

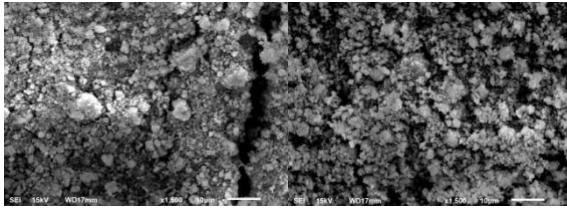
3.3 Scanning Electron Microscopy (SEM)

The electron microscope is JEOL-JSM 6610LV used for scanning electron microscopy examination. To stop charging of the samples, a tiny layer of platinum is applied to them. The samples' micrographs are captured using an accelerating voltage of 10–20 kV.

Fly ash, X-zeolite-like material, 13X-zeolite (commercial), ZSM5-like material (in-house), and ZSM5 zeolite (commercial) are all depicted in SEM images in Figure 3. The X-zeolite-like substance and the ZSM5-like materials don't include any spherical particles, as seen in the picture, indicates a high rate of fly ash conversion into zeolite during hydrothermal treatment. It should be observed that the surface of the fly ash particles is smooth because aluminosilicate glass phase has coated the surface. After hydrothermal treatment, the treated fly ash's surface turns rough, indicating the zeolite phase. Additionally, it can be seen that the crystal structures of X-zeolite (IH) and 13X-zeolite (commercial), as well as between ZSM5 (IH) and ZSM5 (commercial), do not differ significantly.



(a) Cu-ZSM5(IH)Fe-ZSM5 (IH)



(c)Cu-ZSM5 (Commercial) (d)Fe-ZSM5 (commercial)

Figure 3. displays the SEM images of numerous metal-doped zeolites and parent zeolites.

The image clearly demonstrates how drastically different the microstructure of the metal-doped zeolites is. (Fig. 3. a, b, c, d), and the particle size is slightly bigger than that of parent zeolites. This is due to the transition between the Braunauer-Emmett-Teller (BET) method and the size of particles analyzer during the ion exchange process. The average particle size distribution is shown in table 2.

Sample	Average Particle size Distribution (d.nm)	BET(m ² /g)
Fly ash	2519	3.5
X-Zeolite	397.5	332
ZSM5 (IH)	346.7	405
ZSM5 (Commercial)	315.4	425

Table 2 Result of average PSA and BET

4. EXPERIMENTAL STUDY FOR DIESEL ENGINE

The Mahendra Maximo diesel engine used in the experiment has a rated power of 5.2 kW at 1500 rpm. Figure 4 depicts the experimental setup's schematic setup. The eddy current dynamometer utilized in this work is described in appendix 3, and the specifications for diesel engines are provided in appendix C. The engine has a three-hole injector and a piston with a hemispherical bowl. The standard injection pressure for the inline mechanical fuel pump is 220 kg/cm², and the suggested injection timing is 23 bTDC. To lower engine heat, water is sent through the cooling jackets in the cylinder head and engine block. To accomplish the desired results, the engine is driven for 15 to 20 minutes.

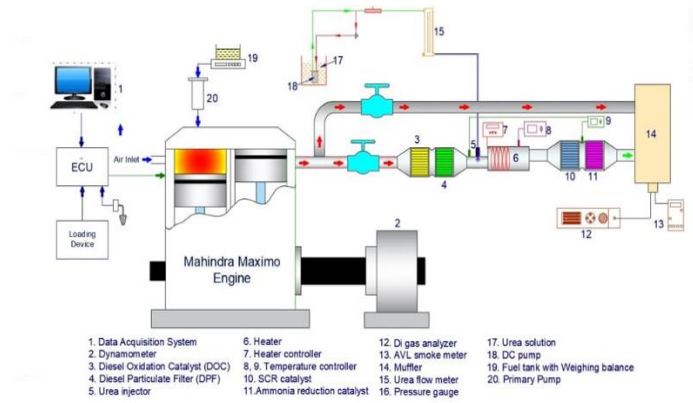


Figure 4 Photographic view of catalytic converter fitted in diesel engine

5. RESULTS AND DISCUSSION

In the present chapter, the results of experiments conducted in petrol engine and discussion about the same are dealt with in the first phase. The second phase deals with that of diesel engine. Results of these experimental investigations are represented in the form of graphs showing percentage reduction of emission. Inferences drawn out from the results are also explained in this chapter.

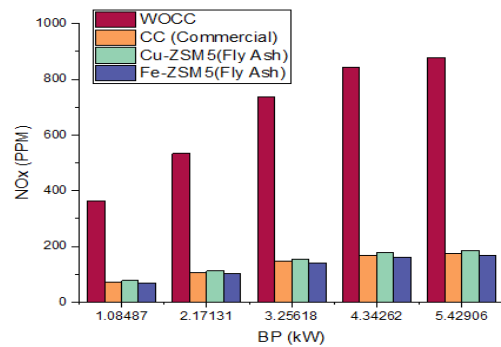


Figure 5: Speed 1300 /IP 300 – NOx Vs Brake power

The figure 5 shows that all of the domestically produced catalytic converters considerably lower NOx emissions when compared to commercial catalytic converters. It is because the commercial catalytic converters are designed to work efficiently under stoichiometric air-fuel mixture condition. But the engine used in the present investigation is a lean burn engine (MPFI), which means exhaust is oxygen rich, which reduces the conversion efficiency of the standard convertor. Hence a better catalytic converter system using zeolite is designed to reduce this harmful emission. Due to the higher affinity of zeolite active sites on NO, they are easily disassociated into its basic form N and O. Hence it can be proved from Figure 5 that the conversion efficiency of modified system is better.

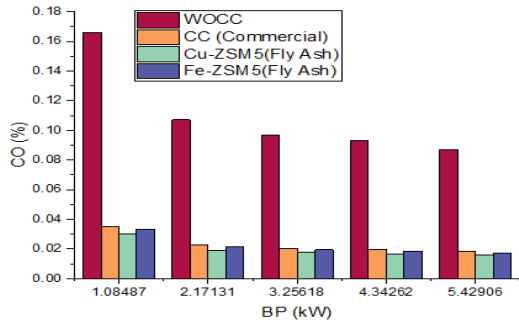


Figure 6. Speed 1300 /IP 300 – CO Vs Brake power

The CO conversion efficiency of a dual layer monolith catalytic converter and a deNOx and CO catalytic converter are shown in Figures 5 and 6, respectively. As can be seen from the graph, the commercial monolith efficiently regulates the amounts of CO output and HC emission during lean exhaust conditions, which is consistent with the findings of numerous other researches. The CO conversion efficiency is only slightly lower than that of a commercial catalytic converter, as can also be seen in Figure 6. The zeolite layer in the dual layer monolith partially blocks the Pt-layer, which is why there aren't enough active CO conversion sites. In contrast, the deNOx catalyst (Figure 6) detects around 93% of CO conversion efficiency at 4 kW load conditions and approximately 95% at 16 kW load conditions. This is because the unreacted CO that is still present after the Pt-coated monolith is removed reacts once more with adsorbed oxygen (produced by NOx dissociation) on the surface of the deNOx catalyst to produce CO₂.

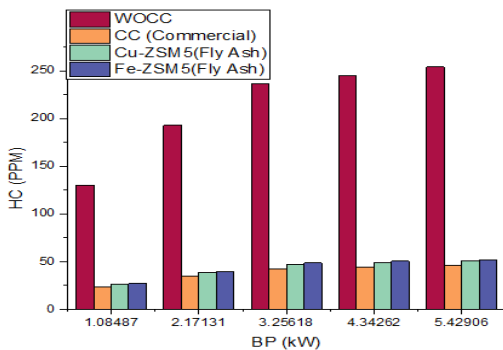


Figure 7. Speed 1300 /IP 300 –HC Vs Brake power

The % reduction in HC emission with engine brakepower is shown in Figures 6 and 7. The figure shows that the NOx conversion efficiency is 72% at 4 kW load conditions, gradually rises to 79% at 16 kW load conditions, and then progressively decreases. In contrast, the HC conversion efficiency in a deNOx converter system is roughly 90% at 4 kW load condition and gradually rises to 93% at 16 kW load condition[31]. This is because the unreacted HC emission that is released from the Pt-coated catalyst includes with the oxygen adsorbed there again to form CO₂ and H₂O. The 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.

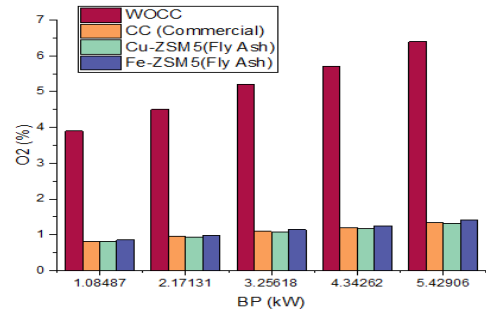


Figure 8. Speed 1300 /IP 300 –O2 Vs Brake power

Using zeolite as a catalyst in a CRDI (Common Rail Direct Injection) engine to reduce oxygen reduction aims to enhance combustion efficiency and lower emissions. Zeolites, with their unique porous structure and catalytic properties, can facilitate the breakdown of fuel molecules, leading to more complete combustion. This improved combustion can reduce the formation of pollutants such as unburned hydrocarbons and nitrogen oxides (NO_x). Additionally, zeolites can help in the adsorption and subsequent reduction of these pollutants, contributing to cleaner exhaust emissions. Therefore, integrating zeolite catalysts in CRDI engines could potentially optimize fuel usage and reduce harmful emissions, aligning with stringent environmental regulations.

6. Conclusion

The primary focal point of the present study is to develop a low cost catalyst for reduction of Measure the output and NOx emission under actual engine exhaust conditions. Discussions in the previous chapters highlighted these concepts, methodology, result and inferences. The principal points that can be highlighted are as follows: Zeolites from Class ‘F’ fly-ash are successfully synthesized.

- Transition metals (Cu and Fe) are incorporated into the zeolites by liquid phase ion- exchange method.
- Metal doped zeolites are washcoated on the uncoated monoliths.
- Catalytic converters are fabricated housing these monoliths separately for petrol engine and diesel engine.
- The in-house made deNO_x catalysts (zeolite synthesized from coal fly-ash) are more active then commercial catalysts at low exhaust gas temperatures.
- The NOx conversion efficiency of Cu ZSM-5 (IH) is 72%, 80%, and 84%, respectively, at 225 °C, 300 °C, and 375°C of exhaust gas temperature.

- The NO_x conversion efficiency of Fe ZSM-5(IH) at 300°C, 375 °C and 400°C of exhaust gas temperature is 72%, 80% and 82% respectively.
- CO conversion efficiency is 80% and 85% respectively at 1.02 and 5.2 kW load conditions and HC conversion efficiency is 80% and 84% at 1.02 and 5.2 kW load conditions which are achieved by DOC.
- Smoke density is reduced by DPf; the reduction rate is 70% at 1.04 kW and 85% at 5.2 kW load conditions.

Finally, it can be concluded from the experimental findings that ZSM-5 zeolite made from coal flyash has a higher NO_x conversion efficiency than ZSM-5 zeolite sold commercially. When temperatures are lower, the Fe-ZSM (IH) catalyst performs worse than the Cu-ZSM5 (IH) catalyst. Consequently, compared to heavy duty diesel engines, which have relatively high exhaust gas temperatures, light duty diesel engines are more suited for Cu-ZSM5 as a catalyst.

Author contributions

Sethuraman Narayanan: Conceptualization, Writing-Reviewing and Editing. **Karthikeyan Duraisamy:** Data curation, Writing-Original draft preparation. **Sarangapani Palani:** Visualization, Methodology and Investigation.

Conflicts of interest

The authors declare no conflicts of interest.

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