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# Red mud and foundry sand industry wastes for reducing NO<sub>x</sub> in plasma activated diesel exhaust

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**Abstract.** Solid waste in association with plasma was explored as an alternate to commercial catalyst/adsorbents. We have demonstrated reuse of foundry sand and red mud wastes for adsorption of gaseous pollutant from plasma treated diesel exhaust. A novel dielectric barrier discharge reactor with dual metal film is designed to explore the oxidation potential of surface discharge plasma effect onto the exhaust. The metal film was subjected to high voltage pulse/AC energization to assess the effect of plasma in oxidising NO to NO<sub>2</sub>. A separate reactor filled with industry wastes was cascaded with plasma reactor to test its efficacy in NO<sub>2</sub> adsorption. It was observed that some amount of NO was also reduced owing to some visible light-enabled photocatalytic activity. About 85 % NO<sub>x</sub> (oxides of nitrogen) reduction was observed with red mud waste compared to half of that with foundry sand.

**Keywords** — Diesel exhaust, non-thermal plasma, red mud, foundry sand, solid wastes, electrical discharge.

## 1. Introduction

The basis for the present work originates from two issues that is affecting our country india currently: accumulation of industry wastes and abatement of gaseous pollutants emanating from fossil fuels burning. The prominent industry wastes are fly ash, red mud, foundry sand, iron ore tailings etc. [1]. The most caustic amongst these is red mud because of which it has less scope for recycling. Similarly, the waste foundry sand also has limited usage in India. The accumulation of red mud in India is about 3 MTA (million tons per annum) and that of foundry sand is 2 MTA. Any proposition in reusing red mud/foundry sand is a welcoming step. Talking about gaseous pollutants, India is a major consumer of crude oil in the world and about one third of the imported oil constitutes the diesel [2]. 50 % of the NO<sub>x</sub> and hydrocarbons (HC) present in the atmosphere comes from the burning of this diesel in transportation and industry sectors. As such there is no efficient mechanism to completely reduce these NO<sub>x</sub> and HC pollutants in India. The existing catalyst-based converters and adsorbent based techniques are becoming expensive owing to the short life, dependency on noble metals, more vulnerability to acidic coating, bulk usage of adsorbents etc. In this regard the application of non-thermal plasma or electric discharge plasma for pollution control aided by additional technique is slowly gaining popularity in the past few years [3-7].

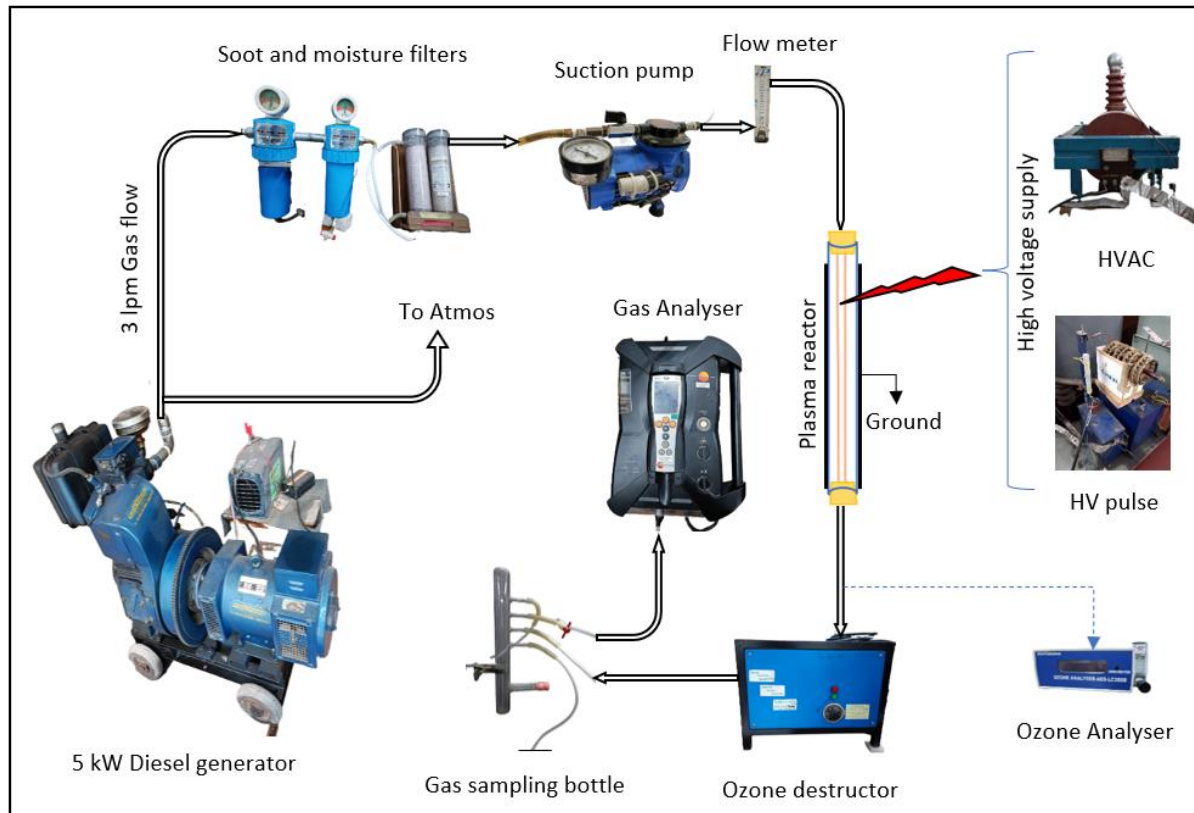
The success in capturing dust particles in electrostatic precipitators by high voltage discharge motivated the researchers to explore the chemistry aspect of discharge in abating the gaseous pollutants in exhaust.



It was then realized that plasma chemistry results in more oxidizing reactions than reductions [8]. However, to a certain extent the oxidised by-products in the plasma appeared to be less hazardous to humans than to nature. This led to the redesigning of plasma reactors with the intension of enhancing the energy in the charged species favoring reduction reactions instead of oxidation ones [9,10] but not without serious limitations with respect to the gas flow. On one hand, pure catalysts/adsorbents-based gas treatment techniques were becoming less efficient and more expensive, the plasma-alone was unable to reduce the gaseous pollutants to the desired level, on the other hand [11-20]. At this juncture, the plasma supported catalysts/adsorbents showed improved results in the gas treatment at laboratory environment [21,22]. This improved result has come from the synergetic effect of plasma and the catalysts/adsorbents [23-33]. The additional expenditure on plasma, though escalates the cost of gas treatment, the benefits due to high pollutant removal efficiency outweigh the cost factor. There can be substantial savings if those catalyst/adsorbents are replaced by freely available industrial wastes that have traces of mineral oxides [34,35]. *The present work is an attempt to explore adsorption and possible catalytic properties associated with solid wastes that were borrowed respectively from aluminum extraction industry (red mud) and metal-casting industry (foundry sand).*

Preliminary experiments in gas treatment showed good results when red mud and foundry sand were used in hybrid plasma catalysis mode [36]. In the current laboratory research, foundry sand and red mud, which were procured from local industry, were grinded and prepared in the form of pellets with considerable degree of porosity to study its adsorption properties. The plasma reactor design is a unique one having just a couple of metal films embedded along the inner surface of the reactor. The studies at room temperature were conducted with unipolar repetitive pulses and power frequency AC to understand energization effect on plasma chemistry. Controlled flow rate of the exhaust was maintained inside the laboratory and results were analyzed.

## 2. Experimental setup



**Figure 1.** Representation of the experimental setup.

A representative picture of the experimental setup is shown in figure 1. Basically, the setup can be divided into three parts: source of pollutants, treatment zone and measuring aspects. Throughout the experiments necessary precautions were taken to regulate the gas flow rate, gas condition, ozone levels, consistency in pulse frequency, alignment of electrodes in plasma reactor etc. The three parts of the setup will now be briefly described.

### *2.1 Source of pollutants*

Current work is about capturing one of the hazardous air pollutants i.e., NO<sub>2</sub>. The major man-made source for NO<sub>2</sub> is diesel engines, be it automobile or stationary. In the present work a 5-kW diesel generator is used as the source of pollutants, and this replicates a condition of exhaust coming out of a typical three-wheeler vehicle in Indian scenario. The generator is a single cylinder, natural cooled, 1500 rpm, class B1 make (Prakash Marketing Pvt. Ltd., India). The exhaust flowrate is about 1000 lpm at a temperature of 350 ~ 400 °C. However, it is just sufficient to maintain a controlled flowrate in the laboratory to understand the chemical reactions in the gaseous phase under the influence of discharge plasma. Therefore, provision was made to divert the excess flow to the atmosphere and allow the rest for experimental research. Stainless steel piping was used to handle the high temperature exhaust and also to prevent corrosion of the material due to the acidic components of the exhaust. Periodic cleaning of the pipelines prevents accumulation of the soot and oil muck and enables consistency in the composition of the exhaust. The raw exhaust entering into the lab is laden with oil vapour, soot particle, moisture in addition to the gaseous pollutants which need to be filtered out.

### *2.2 Treatment Zone*

In this section, two types of exhaust treatment will be described: passive and active treatment. The passive treatment involves filtration of the exhaust and active treatment involves inducing chemical reactions in exhaust under plasma curtain.

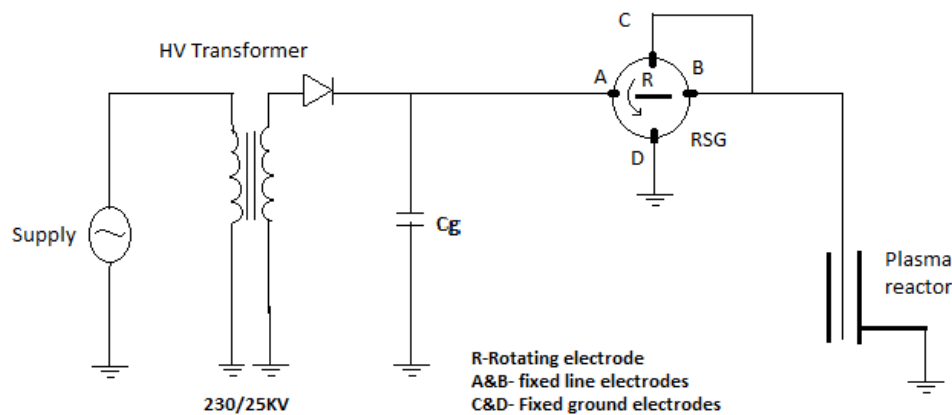
The filtration of the exhaust takes place in two steps. The solid particles (soot) down to the size of 1 micron are removed using stainless steel mesh filters (M/s Ultra filter India Pvt. Ltd.). coarse filtration (5 micron) followed by fine (1 micron) constitute the filter assembly. Thereupon, anhydrous calcium sulphate (M/s Cole Parmer) ensures adsorption of moisture traces, if any, in the exhaust. A suction pump connected in line with the filtration unit maintains a continuous flow which can further be controlled by a combination of three-way cock and a flow meter. Proper care was ensured not to allow excess pressure building up in the laboratory gas lining by using a pressure release valve connected to one of the terminals of the three-way cock. In the present study a steady flow of 3 lpm was maintained at atmospheric pressure and room temperature. This completes the passive treatment zone.

The active treatment zone involves description of the energization unit, plasma reactor unit and the industry waste-based pellets that are used for adsorption of NO<sub>2</sub>, which is the main theme of this paper. The energization unit includes two high voltage sources namely unipolar repetitive pulse and a power frequency AC.

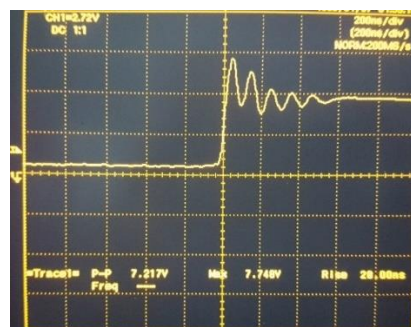
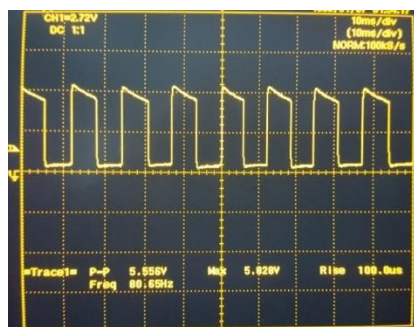
The unipolar pulses were produced by repetitively discharging a DC filter capacitor on to the plasma reactor load, which is basically capacitive in nature due to presence of the dielectric barrier. The DC filter capacitor (0.5 $\mu$ F, 50 kV) is part of a transformer-rectifier unit (25 kV). The DC voltage is fed to a spark gap switch consisting of four fixed and one centrally rotating electrode. The switch in turn transfers the charge from the capacitor to the plasma reactor and then back to ground repetitively, thus, producing fast rising unipolar pulses. In the current work, the pulses were of 80 Hz and 28 ns. The pulse frequency can be varied by adjusting the speed of the motor coupled to the central rotating electrode of the switch. Provision exists in the pulse power supply to vary the voltage from 0 to 25 kV. The rotary spark gap is housed in a shielded cage to prevent electromagnetic interference to the neighbouring digital equipment. Figure 2(a) shows the principle of high voltage pulse generation and fig 2(b) shows the pulse train and rise time of the pulse.

An epoxy filled transformer (1- $\emptyset$ , 50 Hz, 50 kV, Mayog electricals, India) is used for energizing the plasma reactor with power frequency high voltages. During the course of experiment the voltage was varied between 0 to 25 kV (peak) so as to match the levels that were used in unipolar pulse case. The AC energization was intended to check the effectiveness of the newly developed metal film-based

plasma reactor in gas cleaning. The presence of the dielectric barrier facilitates usage of either unipolar pulses or bipolar AC and hence, comparative study was taken up as the reactor design was a unique one being used for plasma-based gas cleaning for the first time.



(a)



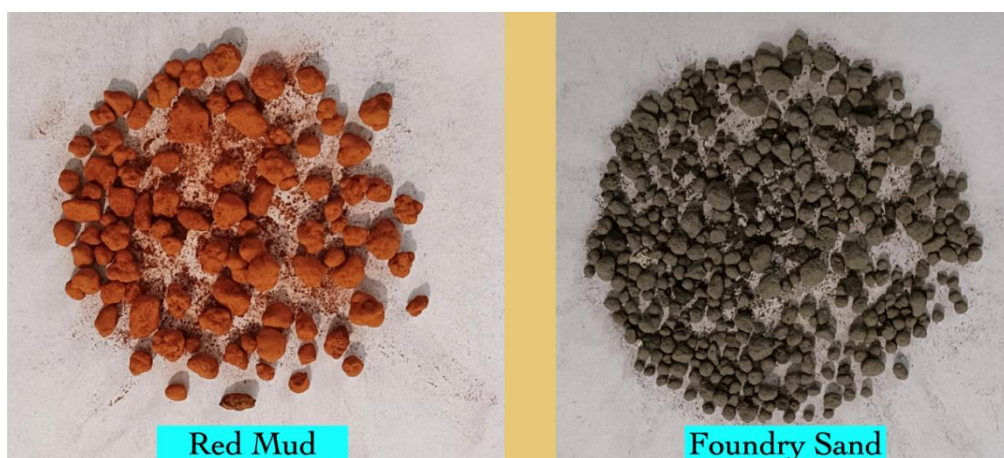
(b)

**Figure 2.** Repetitive pulse energization (a) schematic of circuit (b) pulse train and rise time.

The next part in the active treatment domain is the plasma reactor which consists of a cylindrical quartz glass tube with 35 mm OD, 29 mm ID and an effective length of 29 cm. Two metal films of width 8 mm each, are pasted inside the reactor longitudinally and equidistance from each other. The films occupy about 20 % of the inner area. The films were carefully drawn out of the tube through the silicone stoppers for energization purpose.

The last part in the active treatment domain consists of industrial waste-based pellets filled in a cylindrical reactor for possible use in gas adsorption. Waste red mud from the alumina industry and used foundry sand from metal-casting industry were considered in the current study because their production per annum is quite significant in India (red mud and foundry sand both 3 million tons per annum) and any suggestions that put these wastes for good use is always a welcoming step. The red mud composition mainly includes  $\text{Fe}_2\text{O}_3$  (34 – 40 %),  $\text{Al}_2\text{O}_3$  (17 – 19 %),  $\text{TiO}_2$  (15 – 16 %),  $\text{SiO}_2$  (5 – 6 %) in addition to other metal oxides. On the other hand, the foundry sand mainly contains  $\text{SiO}_2$  (88 %),  $\text{Fe}_2\text{O}_3$  (0.94 %),  $\text{Al}_2\text{O}_3$  (4.7 %),  $\text{TiO}_2$  (0.15 %) and traces of other metal oxides. The raw waste was converted to pellets which are less dense and more porous the procedure involves mixing the raw waste with a binder solution of  $\text{Na}_2\text{SiO}_3$  taken in a proportion of 10 % by volume in 1 liter of water. Utmost care has to be taken during the mixing so that the end product is neither too soft nor too hard but just sufficient enough to spread over a sieve plate to get the desired pellets. These will then be sintered at a predetermined temperature and duration. The pellets so obtained were then tested for pore diameter and surface area. Red mud was having an average pore diameter of 14.53 nm and surface area of 19.96  $\text{m}^2/\text{g}$ . Similar values for foundry sand are 35.56 nm and 1.18  $\text{m}^2/\text{g}$  respectively. Looking into the pore diameter the

pellets fall in the mesoporous range (2 – 50 nm). Figure 3 shows the finished pellets made out of raw industrial wastes.



**Figure 3.** Pellets made from industrial waste (pellet size varies from 3-5 mm).

### 2.3 Measuring aspects

The last part in the experimental setup involves measurement of electrical parameters and the gas composition. A potential divider (ratio 2000:1, 50-kV, EP-50K, PEEC, Japan) is used for voltage profile measurements during the study. The divider comes with the terminating impedance to facilitate measurement of fast rising impulses. The power dissipated in the plasma reactor is assessed by taking the difference of two values of power at the “wall plug” point corresponding to with and without the plasma reactor respectively. It should be noted here that while measuring power under the two circumstances, same voltage magnitudes need to be maintained, be it AC or pulse. While working with pulses, initially, the speed of the motor was kept constant so as to obtain a pulse frequency of 80 Hz throughout the study.

In the gas parametric measurement two analysers were used. For the measurement of the ozone a UV based ozone analyser (AES-LC3000, Aurozone, India) with a provision to measure 3000 ppm of ozone was connected at downstream of the reactor. The oxides of nitrogen (NO and NO<sub>2</sub>), O<sub>2</sub>, CO and CO<sub>2</sub> were measured using a multi-component gas analyser (Testo-350 Germany). The analyser has a built-in pump to sample the gas and care was taken to destroy the ozone in the sample gas so that the NO<sub>x</sub> sensors are unaffected. This commercial analyser individually measures NO and NO<sub>2</sub> before adding the same internally to give NO<sub>x</sub>. The entire gas carrying pipeline was either stainless steel or Teflon to avoid reaction with corrosive gases.

### 3. Results and Discussion

Studies were carried out on the diesel exhaust, sampled at 3 lpm, at normal temperature and pressure to check the effectiveness of the newly prepared waste-based pellets in the adsorption of NO<sub>2</sub>. It is well understood that diesel exhaust is rich in NO and when this passes through a shower of electrical discharge, the oxidative environment of plasma enhances the convergences NO to NO<sub>2</sub> depending upon reactor configuration and energization parameters. Thus, it is crucial to design the plasma reactor so as to maximize the NO conversion. The current study focuses on two aspects: NO/NO<sub>2</sub> variations in the newly designed metal film plasma reactor where the discharges are confined at the surface level. The other aspect is understanding the efficacy of red mud and foundry sand-based pellets in the adsorption of NO<sub>2</sub>. Interestingly, in the plasma zone one can expect partial reduction of nitric oxide owing to reaction with hydrocarbon/N radicals and in the adsorption zone, some amount of photocatalysis due to activation of TiO<sub>2</sub> under visible light can be a possibility.

In the current work, filtered diesel exhaust was subjected to a curtain of discharge plasma before passing the same through a bed of pellets made out of industrial waste. The non-thermal plasma comprises of surface discharges (mainly) and volume discharges (partially) owing to the metal film configuration of

the newly designed plasma reactor. In the initial levels of the design, the several metal films were carefully drawn and affixed on the inner walls of the cylindrical glass tube as well as on the rectangular acrylic plates corresponding to pipe type and duct type reactors respectively. Both the reactors were tested for NO conversion & power consumption and pipe type reactor was selected finally. The second level of design involved fixing the optimum number of metal films inside the pipe type reactor. Preliminary tests on energy density were conducted respectively with four, three, two and one metal film pasted inside the reactor. The intention was to have low energy density with high NO conversion so that the total cost incurred on the reactor design comes down when the technology is implemented on a larger scale. It was observed that amongst the combination studied, two metal films were found to be striking a balance between conversion efficiency and power consumption and hence, was selected for rest of the experiments.

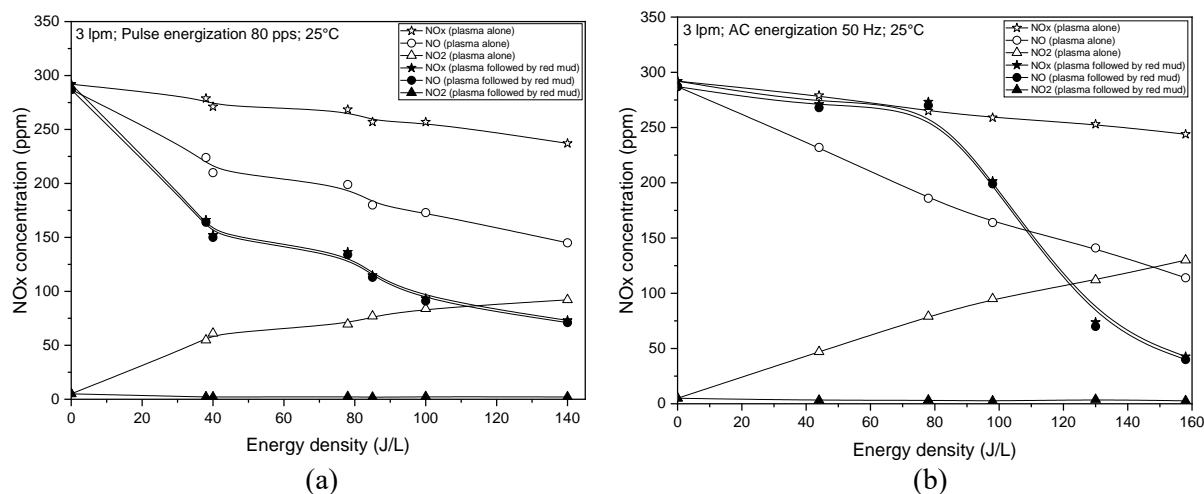
The experiments started with running the diesel engine for sufficiently long time so that the fluctuation in the gas composition reduces to a minimum. Exhaust gas concentration at 0 % load is shown in table 1.

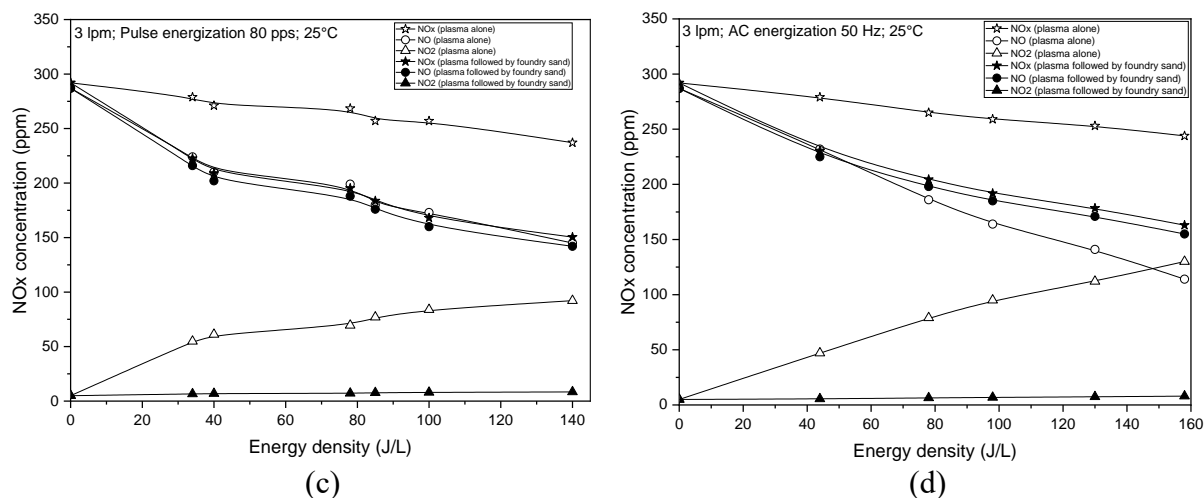
**Table 1.** Diesel exhaust composition under 0 % load

Engine Load (%)	O <sub>2</sub> (%)	CO <sub>2</sub> (%)	CO (ppm)	NO (ppm)	NO <sub>2</sub> (ppm)	NO <sub>x</sub> (ppm)
0	16.32	3.38	1046	287	5	292

The filtered gas was subjected to plasma treatment followed by adsorption treatment, possibly by the industry waste-based pellets. In order to understand synergetic effect of adsorption and plasma, the studies were carried out individually with plasma and then with a combined unit of plasma-adsorption. It was ensured that similar power inputs were applied in these two sets of experiment. At every power input ozone and other exhaust gas components were measured after providing sufficiently long time of 30 minutes for the readings to stabilize. The whole experiment was repeated at least for three times and the data shown in the graph is the average of 3 sets of readings. The deviation from the average value is less than 1 % and hence error bars have not been shown in the graphs. Figure 4 presents the comparative analysis of red mud and foundry sand effectiveness in plasma-adsorption treatment of diesel exhaust. Further, the plasma alone case has also been shown to understand the synergy effect.

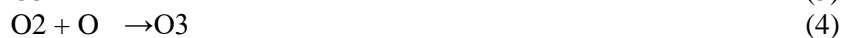
Before the graphs are discussed at length, it will be more appropriate to identify the reaction pathways pertaining to plasma species. Reactions 1 to 35 describe group-wise the species formation, interaction of species with NO<sub>x</sub>, that with hydrocarbons etc. It should be noted here that the ozone that was formed in the plasma got fully utilized in the NO/NO<sub>2</sub> oxidation reactions. This was indirectly verified by the ozone measurement conducted at the downstream of the plasma reactor. Throughout the studies, the ozone concentration was almost negligible at the outlet of plasma reactor.



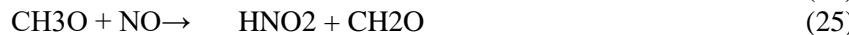
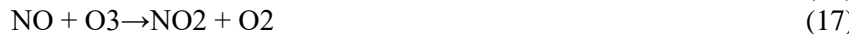
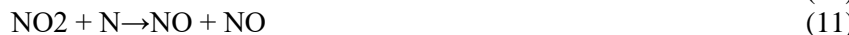


**Figure 4.** Variation of NO/NO<sub>2</sub>/NO<sub>x</sub> in plasma and plasma assisted adsorption cases: (a) pulse energization with red mud (b) AC energization with red mud (c) pulse energization with foundry sand (d) AC energization with foundry sand.

Reactions pertaining to formation of O/N radicals and ozone (O<sub>3</sub>) molecules in the plasma environment are:



Reactions involving O/N radicals and ozone (O<sub>3</sub>) molecules with oxides of nitrogen in plasma environment are:



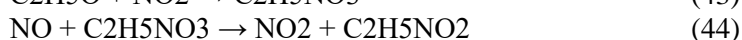
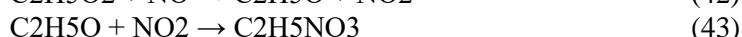
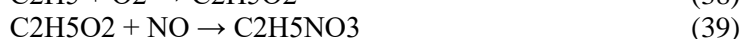




The sampled exhaust, which was filtered to eliminate moisture and soot, is regulated at a flow of 3 lpm throughout the current experiment. The exhaust is made to pass through the plasma reactor and the concentrations of NO/NO<sub>2</sub>/NO<sub>x</sub> are recorded at different power inputs. The specific energy of the applied plasma is then calculated as a ratio of power input to gas flow rate and expressed in J/L. Figures. 4(a) and 4(b) represent the impact of red mud on NO<sub>x</sub> variation for pulse and AC inputs. Looking at the plasma alone case, conversion of NO to NO<sub>2</sub> was about 50 % with either of the voltage inputs and formation of higher oxides of nitrogen (N<sub>2</sub>O<sub>4</sub>, N<sub>2</sub>O<sub>5</sub>) through reactions 19 and 21 was negligible as is evident with the near-constant curve of the NO<sub>x</sub>.

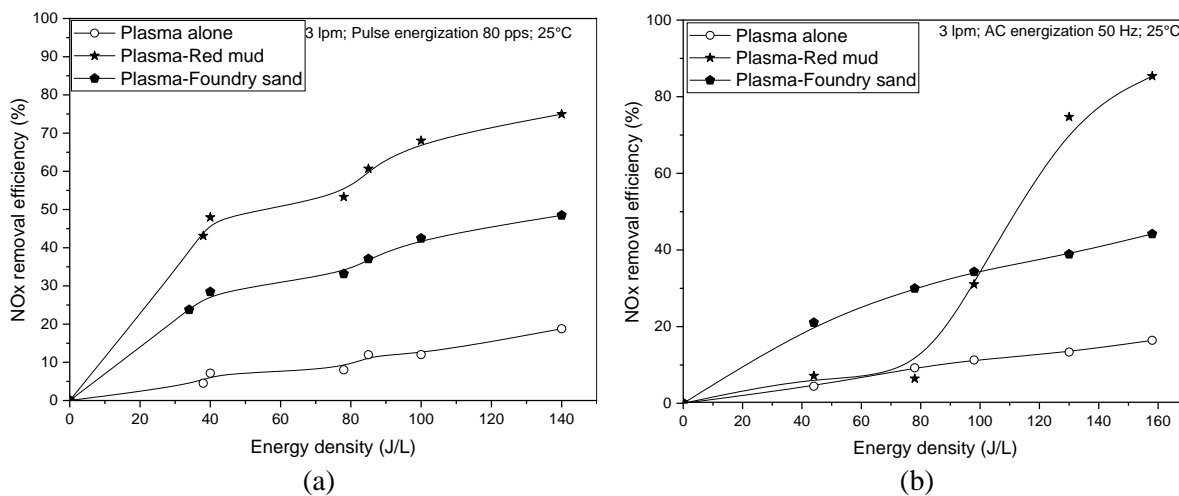
When the plasma treated exhaust flows through the reactor filled with red mud pellets almost all the NO<sub>2</sub> got adsorbed as shown in figures. 4(a, b), where the NO<sub>2</sub> curve almost touches the X axis. The adsorption efficiency of NO<sub>2</sub> was close to 98 % both with pulse and AC energizations. Interestingly the nitric oxide, that is coming out of the plasma reactor, when it enters the cascaded adsorbent reactor has shown a decreasing trend. This is not due to adsorption of NO as the critical point of NO is around -150 °C which is highly unfavourable for the adsorption process. One possible explanation for this could be photocatalysis of ethyl nitrate by visible light activated TiO<sub>2</sub>/Fe<sub>2</sub>O<sub>3</sub> during adsorption process. Ethyl nitrate can be from ethylene/propylene (C<sub>2</sub>H<sub>4</sub>/C<sub>3</sub>H<sub>6</sub>) in diesel exhaust during plasma process through a chain of oxidative processes as shown in reactions 36 to 43. The ethyl nitrate thus formed goes along with the exhaust into the adsorbent reactor filled with red mud. The TiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub> compositions in red mud are respectively 15 % and 35 %. Both being good photo catalysts at visible light itself, they would have contributed for the photocatalysis of nitric oxide with ethyl nitrate producing nitrogen dioxide and ethyl nitrite as shown in equation 44. This has resulted in the conspicuous decrease of NO by almost 75 % at an energy density of 140 J/L in the adsorbent reactor that was filled with red mud. The NO<sub>2</sub> thus formed in this reactor might have got adsorbed in red mud owing to its critical point close to ambient temperature as is evident from the near-zero graph of NO<sub>2</sub> in figures. 4(a) and 4(b).

Similar trend in NO decrease in the plasma treated exhaust was observed in the reactor filled with another industry waste i.e., foundry sand pellets as shown in figures. 4(c) and 4(d). However, the amount of NO decreased due to photocatalysis was about 50 % at 140 J/L. Owing to presence of lesser amount of TiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub> in foundry sand, perhaps, the amount of NO conversion to NO<sub>2</sub> was also less in foundry sand case compared to that with red mud case.



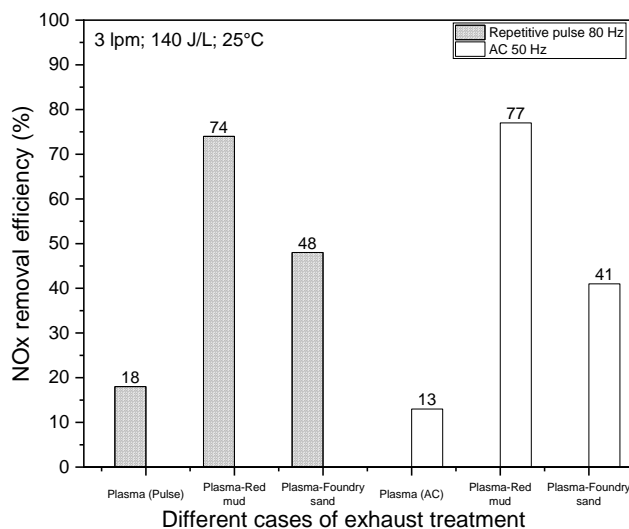
Figures. 5(a) and 5(b) present the comparison of NO<sub>x</sub> removal efficiency for the two industry wastes studied. It can be inferred that irrespective of the type of energization red mud offered more NO<sub>x</sub> reduction efficiency compared to foundry sand. For example, at about 140 J/L the DeNO<sub>x</sub> efficiency

with red mud is about 74 – 77 % (pulse or AC) and the same with foundry sand is about 41 – 48 % (pulse or AC).



**Figure 5.** Comparison of DeNOx efficiency between the two industrial wastes (a) for pulse energization (b) AC energization.

The point to be noted here is that the existing commercial adsorbents can be replaced by the cheaper industry wastes in the post plasma treated exhaust environment. Figure 6 shows for a given energy density the variations in the DeNOx efficiency as one move from plasma alone case to plasma-adsorbent case while treating the diesel exhaust.



**Figure 6.** Comparison of different plasma cases studied for NOx reduction.

**4. Feasibility of the current work**

At the industry level, the technique can be used immediately by the aluminium plants with little investment on the plasma side. The red mud residue can be reused by the plant itself for capturing the NOx which in turn can be resold to the fertilizer industry. The plasma-adsorbent units, with provision for air-suction, can take care of huge inflow of polluted air simultaneously reducing the NOx and particulate matter (up to some extent). Since industrial wastes are abundantly available and power requirement for plasma is almost negligible, larger cross section of plasma-adsorbent can be designed to suit the ever-increasing traffic junctions. The NO emitted by the auto-exhaust gets captured in the foundry sand or red mud pellets after plasma oxidation and occasional desorption of the pellets lead to

recapture of NO<sub>2</sub> in cylinders which can then be transported to end users of NO<sub>2</sub> such as nitric acid plants or fertilizer industries.

## 5. Conclusion

Industry waste-based adsorbents were tested for possible NO<sub>x</sub> reduction in plasma treated exhaust environment. Studies were limited to laboratory scale to understand the principle and mechanism associated with NO<sub>x</sub> reduction in the presence of mineral oxide components of the industry wastes. Major inferences can be listed as:

- Fe<sub>2</sub>O<sub>3</sub>/TiO<sub>2</sub> present in the industry waste can act as photo catalyst in the presence of visible light and aids the NO oxidation through plasma generated ethyl nitrate.
- The newly developed metal film based DBD reactor enhances surface discharges thus facilitating more plasma oxidation-based reactions.
- The NO<sub>x</sub> removal efficiency gets enhanced by a factor of 4 – 6 between plasma-alone and plasma-adsorbent approaches, which is quite significant amongst the plasma studies for pollution control.

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