

## ***NO Abatement using Microwave Micro Plasma Generated with Granular Activated Carbon***

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**Abstract-** Abatement of NO using microwave micro-plasma is presented in this paper. The micro-plasma is generated using granular activated carbon (GAC) particles of size (2-3mm) in loosely fluidized bed in a microwave cavity operated at 2.45GHz. A single mode microwave cavity reactor (SMMCR) was constructed and microwave was injected through another slotted single mode waveguide in a sandwiched manner. COMSOL Multiphysics software was used to investigate the microwave electric field and the power density within the SMMCR. Gas mixture of air and 500 ppm NO (in N<sub>2</sub>) at the flow rate of 2 l/min was passed through a quartz tube centered within the SMMCR while the supplied microwave power was very low 10-80 W and corresponding NO reduction was greater than 98%. The mass of GAC used for generating the plasma was 5g. The efficiency of NO reduction is found to be 24.84 g(NO<sub>2</sub>)/kWh. When air is mixed with NO (in N<sub>2</sub>), the efficiency of NO<sub>x</sub> reduction achieved vary greatly with respect to the supplied microwave energy and behavior has become complex and is not predictable, which needs further investigation. A gas analyzer (testo 350) was used to measure the gas (NO, NO<sub>2</sub>, CO and O<sub>2</sub>) concentration and temperature.

**Key words:** Non-Thermal plasma, NO<sub>x</sub> abatement, Micro-plasma, Microwave cavity, Granular activated carbon

### I. INTRODUCTION

Marine diesel engine flue contains toxic gases such as NO<sub>x</sub> (NO+NO<sub>2</sub>) and SO<sub>x</sub> (SO<sub>2</sub>) which are harmful to environment and health [1-4]. Marine industry is responsible for more than 15% of the world toxic gas emission and its impact can not only affect the coastal area but also extends to large portion of in-land [3]. Treating these gases before releasing into atmosphere is not only vital but also regulated by International Maritime Organization (IMO) [5-7]. The current state-of-the-art of NO<sub>x</sub> and SO<sub>x</sub> removal in marine exhaust gas requires two separate units which are space ineffective and cost ineffective [5]. Even though their removal efficiency of SO<sub>x</sub> is very high (>90%), NO<sub>x</sub> is relatively low (~70%), which needs to be improved to comply with the requirements set by IMO. Furthermore, current NO<sub>x</sub> removal systems require urea injection and produce ammonia, which requires large storage space and also carry the risk of ammonia leakage. Another successful NO<sub>x</sub> abatement technology is use of activated carbon [8-13]. Activated carbon has been used to adsorb NO<sub>x</sub> and then desorbed using microwave where NO<sub>x</sub> reacts with C and produces CO or CO<sub>2</sub> depending on the temperature of GAC [9]. The main drawback in this process is the disposal of the adsorbed NO<sub>x</sub> after its lifetime or it should be further treated elsewhere. In particular, the amount of GAC stored in ships and regenerated with microwave on board can be very expensive and impractical at this moment in time due to foot print and power consumption of microwave based regeneration system.

Non-Thermal Plasma (NTP) has been successfully employed in many land-based power plants to control NO<sub>x</sub> and SO<sub>x</sub> from high flow rate exhaust emission, such as power plants [14, 15]. These installations use electron beam and microwave to create non-thermal plasma and works really well in such applications, where space and power consumption are not significant issues. When this technology is to be implemented on a ship, the space and power consumption is a major concern. These systems also require the injection of ammonia to convert NO<sub>x</sub> into N<sub>2</sub> and by-product would be urea. In the land based emission

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control systems, the produced urea can be used as sellable fertilizer. However in ships, it is not practical to use ammonia as storage and ammonia leak can cause many challenges.

One of the works reported by Wamadeva et al have shown that excessive O<sub>2</sub> in the marine exhaust can lead to production of NO<sub>x</sub> instead of reduction [16]. One of the alternative methods proposed by Kuwahara et al. for the abatement of NO<sub>x</sub> using NTP to treat NO in an oxygen free system [17]. They used two stage process; firstly to adsorb NO and then de-adsorb NO<sub>x</sub> by purging with N<sub>2</sub> and apply heat to have NO in N<sub>2</sub> which is then treated by NTP, where NO<sub>x</sub> is converted into N<sub>2</sub> and O<sub>2</sub> according to the following chemical equation;



It is also understood that plasma production using microwave is a promising as the system does not require electrodes [18-20]. Microwave technologies are also becoming heavily industrialized and new emerging compact solid state microwave technologies are becoming more popular due its compact size and high power efficiency [21]. However, it has been shown by many researchers that high volume microwave non-thermal plasma is still in research stage [22,23]. This is because the production of electrons-ions from gas requires very high electric field ( $> 3 \times 10^6$  V/m) and producing such high electric field using microwave set-up without any extra electrodes can only be done in either by having very high powered microwave or very small sized wave guides (~few cm depth). Former one is not definitely a feasible solution as high powered microwave means big space, complicated structure and very low power efficiency. The later one is possible and have been demonstrated by many researchers [23] but the volume of the plasmas is well beyond the required for marine exhaust emission control applications.

In the present study, a new method for producing NTP is introduced using only microwave energy for treating NO<sub>x</sub> in oxygen-less system. A single mode microwave cavity (SMMC) was designed in which microwave energy is injected through number of slots on a specially designed waveguide. COMSOL multi-physics software was used to compute the electric field within the microwave cavity. Granular activated carbon (GAC) was used to produce micro-plasma within the single mode waveguide. Due to uneven nature of GAC surface and the fact carbon is good electrical conductor; it will create local regions with high electric field, in which local plasma will be generated. This is referred as micro-plasma. Using this micro-plasma, NO is reduced to N<sub>2</sub> and O<sub>2</sub> where inlet gas consists no oxygen. This method of microwave plasma can potentially be used for large volume NTP that required for marine exhaust emission applications. The efficiency of the NO reduction is calculated as 24.84 g(NO<sub>2</sub>)/kWh which is well above the value reported in the literature.

## II. METHODOLOGY AND EXPERIMENTAL SET-UP

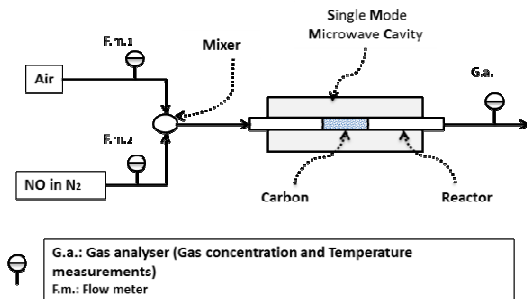


Fig. 1: Gas flow diagram of the NO<sub>x</sub> abatement system

The gas flow diagram of the abatement system is shown in Fig. 1. Two gas cylinders (Air and NO balanced in N<sub>2</sub>) are used as the test gas and their flow rates were controlled by a couple of dedicated separate lab-scale flow meters. Gases from both cylinders are mixed before feeding into the SMMC. In the SMMC a quartz tube (internal diameter of 18 mm) is used to hold granular activated carbon and to guide the gas through for abatement. The quartz tube is transparent to microwave and also able to withstand high temperature. The penetration depth (defined as the distance in which the electric field drops to 1/e) at room temperature for quartz tube for at 2.45GHz frequency is 160 km, whereas for water, it is only 1.4 cm. The melting point of quartz tube is around 1700 °C. The activated carbon used is 1.4 to 2 mm in granule size and has high surface area ( $> 1000$  m<sup>2</sup>/g).

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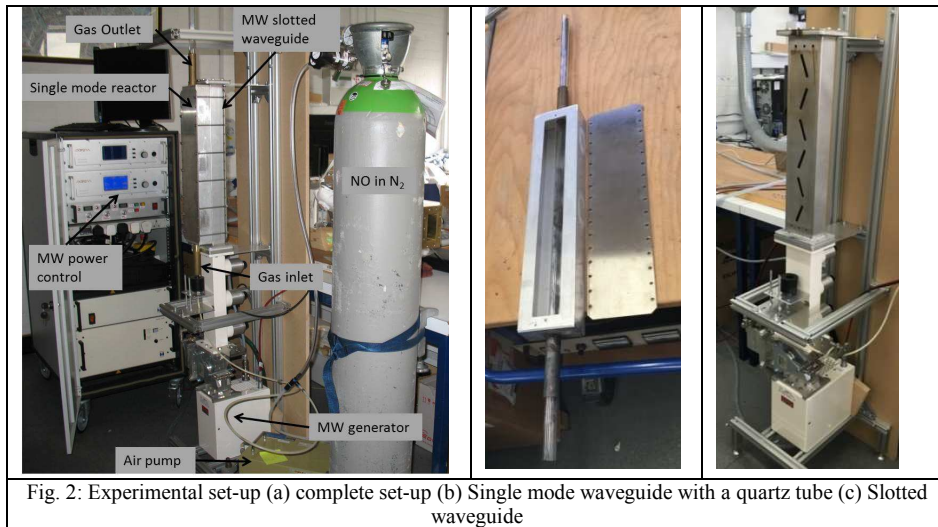


Fig. 2: Experimental set-up (a) complete set-up (b) Single mode waveguide with a quartz tube (c) Slotted waveguide

The experimental set-up is shown in Fig. 2, which shows the single mode cavity with a quartz tube and a slotted waveguide. Microwave energy is supplied from a magnetron operating at 2.5 GHz via a 3-stub tuner into the slotted waveguide, which, in turn, transmit the microwave energy into the SMMC, where NTP is generated. The slotted waveguide and SMMC are identical in terms of its dimensions (110mm (height), 55mm (width) and 500mm (Length) and made from 2mm thick stainless steel. Slotted waveguide has slots on one of its sides and SMMC has a rectangular opening as can be seen in Fig. 2 (b). The SMMC is sandwiched such a way MW can be transmitted into the SMMC from the slotted waveguide through the rectangular opening in the SMMC. The slots are inclined at  $19.9^\circ$  to the horizontal axis as shown in the Fig. 3. There are 6 slots and they are separated by half of the wavelength of the waveguide. Wavelength can be calculated using the following equation.

$$\lambda_g = \frac{\lambda_0}{\sqrt{1 - \left(\frac{\lambda_0}{2a}\right)^2}}$$

(3)

$\lambda_0$  = wavelength of microwave (122 mm)

$a$  = length of the rectangular cross section (96 mm)

For the waveguide used in the experimental set-up,  $\lambda_g$  is calculated to be 158 mm

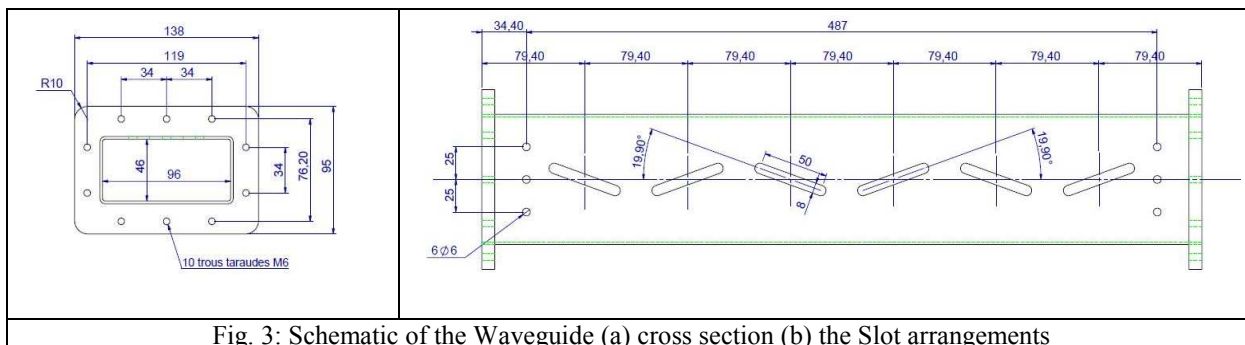


Fig. 3: Schematic of the Waveguide (a) cross section (b) the Slot arrangements

Activated carbon is placed within the quartz tube. The mixture of NO + N<sub>2</sub> + Air from the gas bottles is supplied to the quartz tube in which the gas is treated with the micro plasma generated by the high electric field ( $> 3 \times 10^6$  V/m) around the vicinity of activated carbon particles. Reflected microwave power is diverted into a water load, which continuously circulate from the lab tab. Testo 350, a portable gas analyzer based on chemiluminescent sensors, was used to measure the gas concentration (NO, NO<sub>2</sub>, CO, O<sub>2</sub>) and temperature of the gas was measured at the outlet of the SMMC.

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### III. FEM MODELLING

Understanding the electric field pattern within the SMMC is essential to understand the plasma density within the cavity. Higher the electric field means higher the plasma production and hence higher the electron density. Electrons in gas molecules gain energy and if they gained sufficient energy then they break away from their parent atoms. The broken away electrons gain further energy and move quicker in the electric field and can collide with gas molecules and disassociate more electrons from their parent molecules. These chain reactions will form plasma, which consists of ions, radicals, and excited molecules, which are more reactive than ordinary gas molecules, hence can allow more specialized reactions, which would otherwise not happen.

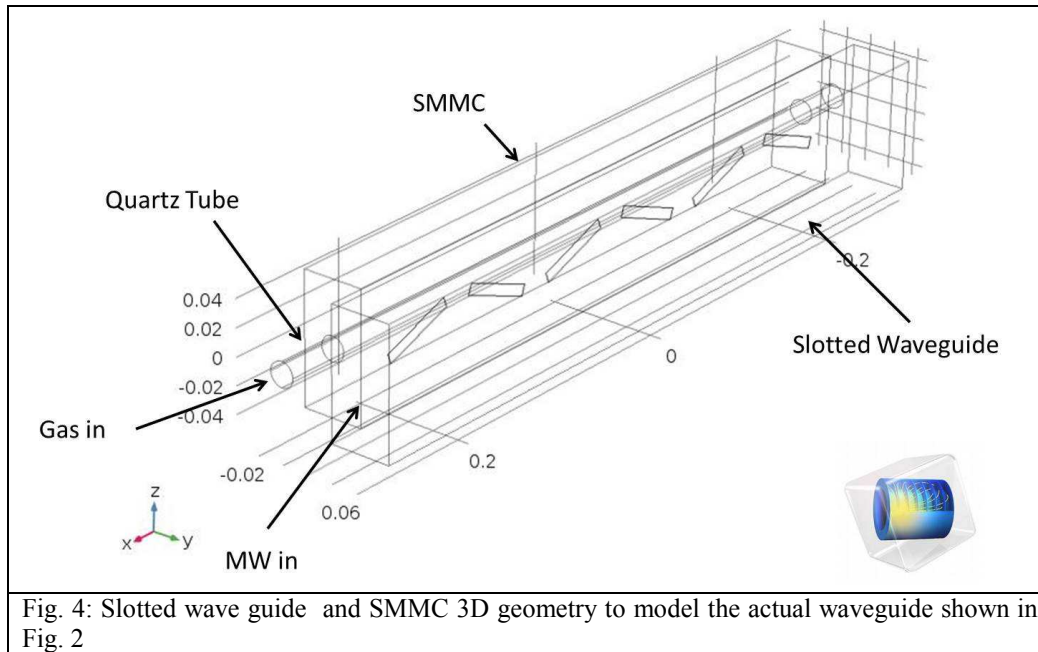


Fig. 4: Slotted wave guide and SMMC 3D geometry to model the actual waveguide shown in Fig. 2

COMSOL multi-physics was used to study the electric field within the SMMC. The geometry of the SMMC developed in COMSOL multi-physics environment is shown in Fig. 4. This geometry was constructed to replicate the actual arrangement of SMMC and slotted wave guide as shown in Fig 2 (b) and (c) and for the dimensions shown in Fig. 3. The microwave generator and stub tuners are not included in the modelling work as they do not influence the electric field standing wave pattern in the slotted waveguide and SMMC. The model is configured in such a way that the microwave energy (MW) is injected through one of the rectangular side as shown in Fig. 4.

The following equation is solved to calculate the electric field in the slotted wave guide and SMMC.

$$\nabla \times \mu_r^{-1}(\nabla \times \mathbf{E}) - K_0^2 \left( \epsilon_r - \frac{j\sigma}{\omega \epsilon_0} \right) \mathbf{E} = \mathbf{0} \quad (4)$$

Where  $\mu_r$  - permeability of the medium,  $\epsilon_0$  - permittivity of the medium,  $\mathbf{E}$  - electric field vector,  $\sigma$  - density of the medium,  $K_0$  - wave number. In our case the medium is air. Walls of the wave guide were assumed to be perfect conductors and hence the following boundary condition was applied:

$$\mathbf{n} \times \mathbf{E} = \mathbf{0} \quad (5)$$

where  $\mathbf{n}$  - normal vector to the walls.

Calculated electric field (absolute) is shown in Fig. 5. These results were obtained when the injected microwave power into the slotted waveguide was set to 600W. It is clear from these figures that highest intensity electric field is seen near the first slot (bottom one) and it decays exponentially towards the top of the SMMC. The period of the electric field maxima-minima is a function of SMMC dimensions and the wavelength of the microwave (equation 3). The maximum electric field in this set-up was somewhere around 20000V/m which is 2 order less than the required field to produce the plasma. It is shown in the experiment that after GAC is introduced in the SMMC, NTP was produced and plasma reactions took place.



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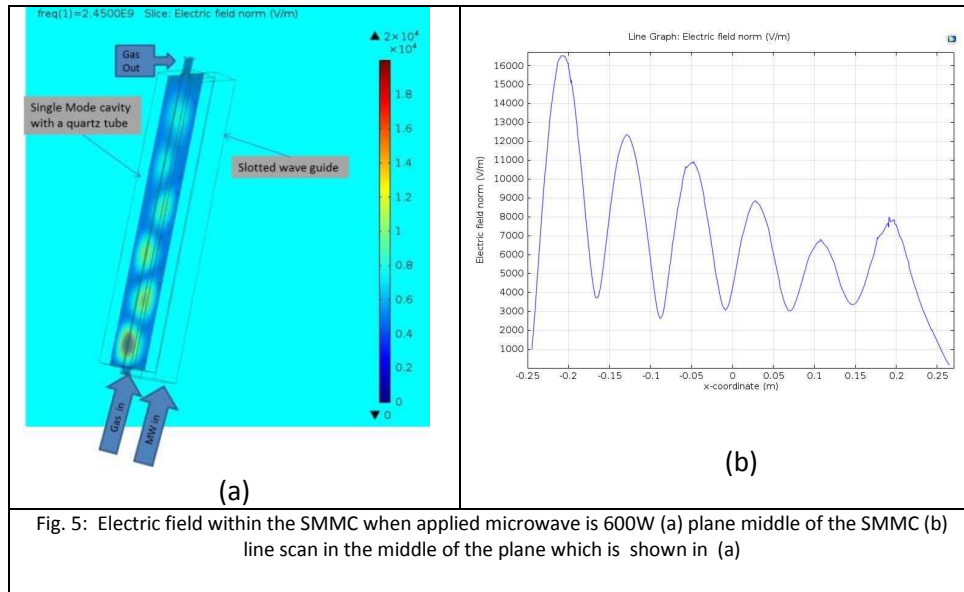


Fig. 5: Electric field within the SMMC when applied microwave is 600W (a) plane middle of the SMMC (b) line scan in the middle of the plane which is shown in (a)

#### IV. EXPERIMENTAL RESULTS AND DISCUSSION

Two test gas mixtures were used in this work. The first one is to use NO balanced in  $N_2$  in the absence of  $O_2$ , hence the abatement is a reduction process where NO would be reduced to  $O_2$  and  $N_2$ . In one of the previous works reported by Kuwahara et al. [17], the benefits of the oxygen-less system were explained. In that work, the  $NO_x$  abatement was reported to be 65%. They used adsorption on NO onto Cu impregnated magnesium oxide ( $MnO_x-CuO$ ) and desorbed by purging  $N_2$ , which then treated using non-thermal plasma (NTP). The non-thermal plasma was produced using Electrical discharge (such as DBD), which has its own limitations; such as it required many small units in the scale-up, high voltage power source, contamination of the dielectric material. In the work reported in this paper, microwave is used with activated carbon, so there is no issue of the contamination or need for high voltage sources. Furthermore, scale-up microwave reactor is easy to construct and such reactors have already been an established technology in other industries such as food processing and material processing.

#### NO in $N_2$

Fig. 6 shows experimental results of NTP treatment of NO balanced in  $N_2$  in GAC. The input NO concentration was 500 ppm and gas flow was 2 l/min. Mass of GAC used was 5g. Supplied microwave power (forward power – reflected power) ranged from 10 W to 45 W. Fig. 6(a) shows the outlet NO concentration for various supplied microwave power. The supplied microwave power is shown in Fig. 6(b). As shown in this figure MW power was switched ‘ON’ after 3 mins of start of the experiment (when the outlet NO concentration was constant) and stopped at 39<sup>th</sup> minute for 7 minutes.

Once MW was ‘ON’, NO concentration rapidly went down to a very low level; with almost 100% NO abatement. This clearly shows the effect of microwave on the GAC. As the supplied microwave power varied, the change in NO concentration at the output also showed a similar pattern. When supplied microwave power was around 50W, there was hardly any NO detected at the output of the SMMC. In Fig. 6(a) and (b) during 15<sup>th</sup> to 27<sup>th</sup> minute, the supplied MW power was 45 W and NO concentration was very close to 0 ppm.

Though it was not possible to notice any glow plasma within GAC bed, it can be safely assumed that silent plasma was present while MW was ON. When the supplied microwave power increased to few hundreds of watts, GAC was producing glow plasma, however due to high electron energy in the plasma, more NO was produced rather than the expected reduction. This may be due to recombination of N and O radicals to give NO. It is important to maintain microwave power to a level, where silent plasma is produced to prevent the recombination reaction. Fig. 6(c) and Fig. 6(d) show CO concentration and outlet gas temperature respectively. The reduction reaction of NO ( $2NO \rightarrow N_2+O_2$ ) must be the source for generation of  $O_2$ . This oxygen or oxygen radical on the surface of the carbon further reacts with carbon to produce CO. The production of CO, however, was (averaged value) within the safe limit. This was also confirmed with the built in CO sensor in the lab. The temperature of the gas was gradually increasing from the inlet gas temperature to 60°C within an hour. This was due to microwave heating of carbon, as carbon’s dielectric properties favor microwave heating. Fig. 6 (e) shows the reduction of NO during the experiment and it was always very close 100% when microwave was ‘ON’ and supplied MW power was 45 W. It can also be seen in this figure that NO reduction efficiency simply follows the supplied MW power. A conservative estimate of the efficiency for NO abatement, neglecting the heating effect, was found to be 24.84 g( $NO_2$ )/kWh. This efficiency is much higher than the efficiency

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reported by Okubo et al[24] which was only 16.0 g(NO<sub>2</sub>)/kWh. In the 27<sup>th</sup> minute and 39<sup>th</sup> minute the MW power was reduced and switched off respectively for few minutes and consequently the NO reduction efficiency started to drop quickly until MW power was switched ON and increased to 45 W again.

A control experiment was also carried out without MW but with same amount fresh GAC see the NO<sub>x</sub> reduction with GAC alone. Fig. 6(f) presents the NO abatements with and without MW presence. It is clear from the control experiment that without the supply of microwave, GAC simply adsorbs NO initially and the ratio of NO<sub>out</sub>/NO<sub>in</sub> slowly increases with time, whereas with microwave the ratio remains very low as long as the MW was on throughout the experiment. This is also evident from Fig. 6a and 6b that when MW was switched off between time 40 min and 45 min, the NO content in the outlet gas had gone up and restored to the original level when the MW was switched on.

#### NO in N<sub>2</sub> plus Air

The second set of tests was performed to investigate the effects of O<sub>2</sub> in the test gas. Air from a gas cylinder was mixed with the test gas (NO in N<sub>2</sub>) before admitting to the charcoal bed. Table 1 shows the inlet gas compositions and flow rates used in this experiment. The mixed gas was fed into the SMMC and NO concentration was reduced to 340ppm and O<sub>2</sub> concentration was 5.5%. Total flow rate of the inlet gas was kept at 2.67 l/min

	NO (in N <sub>2</sub> )	Air	Mixture of Air and NO (N <sub>2</sub> )
Flow rate (l/min)	2	0.67	2.67
NO concentration (ppm)	450	0	340
O <sub>2</sub> concentration (%)	0	21	5.5

Table 1: Inlet gas composition and flow rates

Typical results of NO abatement with oxygen are shown in Fig. 7. Microwave power was switched on from 1<sup>st</sup> minute, though the power absorbed in the SMMC was very small and the actual MW power absorbed could not be recorded as shown in Fig 7(b). Tuning the 3-stub tuner in the microwave supply system to supply the maximum MW power was a very challenging task in this experiment. Fig. 7(a) shows the effect of MW power on NO concentration at the outlet of the SMMC reactor. NO concentration started declining from the time the MW power was on and reached a steady value around 50 ppm from time 5 min. (from 340 ppm to 50ppm) onwards. When the MW power was increased at 13<sup>th</sup> minute, the NO concentration started to go up and at the same time CO concentration went up unacceptably very high (up to 6000ppm) for a short period as can be seen in Fig. 7 (c). The outlet gas temperature also went up to 60<sup>o</sup>C during this time as shown in Fig. 7(d), which shows similar trend as in the previous experiment {Fig 6 (d)}.

When the MW power was increased at 13 min., most of the power was used to heat carbon. This heating increased the local temperature of the carbon sharply causing sudden desorption of CO from the adsorbed state. It may also be possible for the oxygen ion/radical generated can react with carbon at these elevated temperature to give CO and CO<sub>2</sub>. The rise in temperature is also responsible for the sudden increase of desorbed NO during this time. This temperature rise also noticed in the outlet gas from 15<sup>th</sup> minute to 17<sup>th</sup> minute was due to the fact that the supplied MW power was higher during these periods.

Fig. 7 (e) shows the percentage (%) NO reduction by the SMMC and it clearly shows excellent reduction of NO except those instances where supplied MW power was higher than required due to carbon heating. Fig. 7(f) shows O<sub>2</sub> concentration and there was no O<sub>2</sub> observed in the outlet gas from 16<sup>th</sup> minute onwards. It was also found that concentration of carbon dioxide, which was measured with the use of testo 535, started appearing in the outlet gas more than 10000 ppm after the MW power was increased. The possible explanation for this behavior was that oxygen started reacting with carbon to produce CO<sub>2</sub> at higher power levels (high temperature). Overall, further investigation needs to be carried out to understand the dynamics of the plasma chemistry and ways to reduce the CO and CO<sub>2</sub> production while O<sub>2</sub> presents in the inlet gas. It is also important to operate the system at lower power levels when oxygen is present in the exhaust so that the heating effect and the production of CO and CO<sub>2</sub> are kept minimum.

## V. CONCLUSIONS

Microwave micro non-thermal plasma was generated using granular activated carbon (GAC) in a single mode waveguide at the microwave frequency of 2.45GHz. Experimental results show that near 100% reduction of NO is achievable when O<sub>2</sub> is not in the inlet gas mix. Microwave power required for treating 2l/min was between 10 and 45 W and this can be further improved through better position of GAC within the SMMC. The efficiency of the NO abatement was calculated to be 24.84 g(NO<sub>2</sub>)/kWh when there is no oxygen present in the inlet gas. Though the experimental results suggest that excellent abatement

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of NO can be achieved in the presence of O<sub>2</sub>, the plasma chemistry become complex and the behavior of NO reduction is unpredictable. This requires further investigation.

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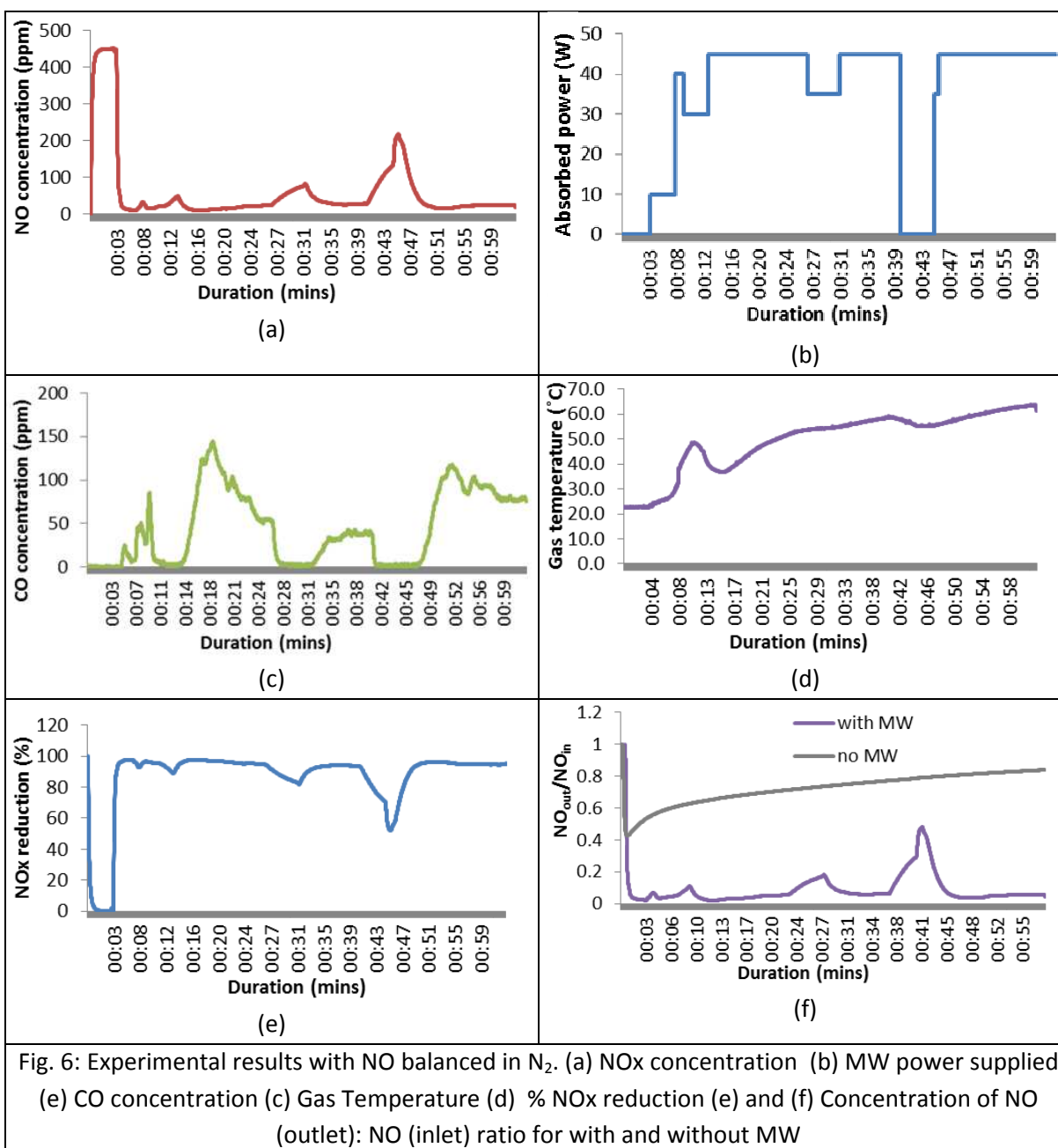


Fig. 6: Experimental results with NO balanced in N<sub>2</sub>. (a) NOx concentration (b) MW power supplied (c) CO concentration (d) Gas Temperature (e) % NOx reduction (e) and (f) Concentration of NO (outlet): NO (inlet) ratio for with and without MW

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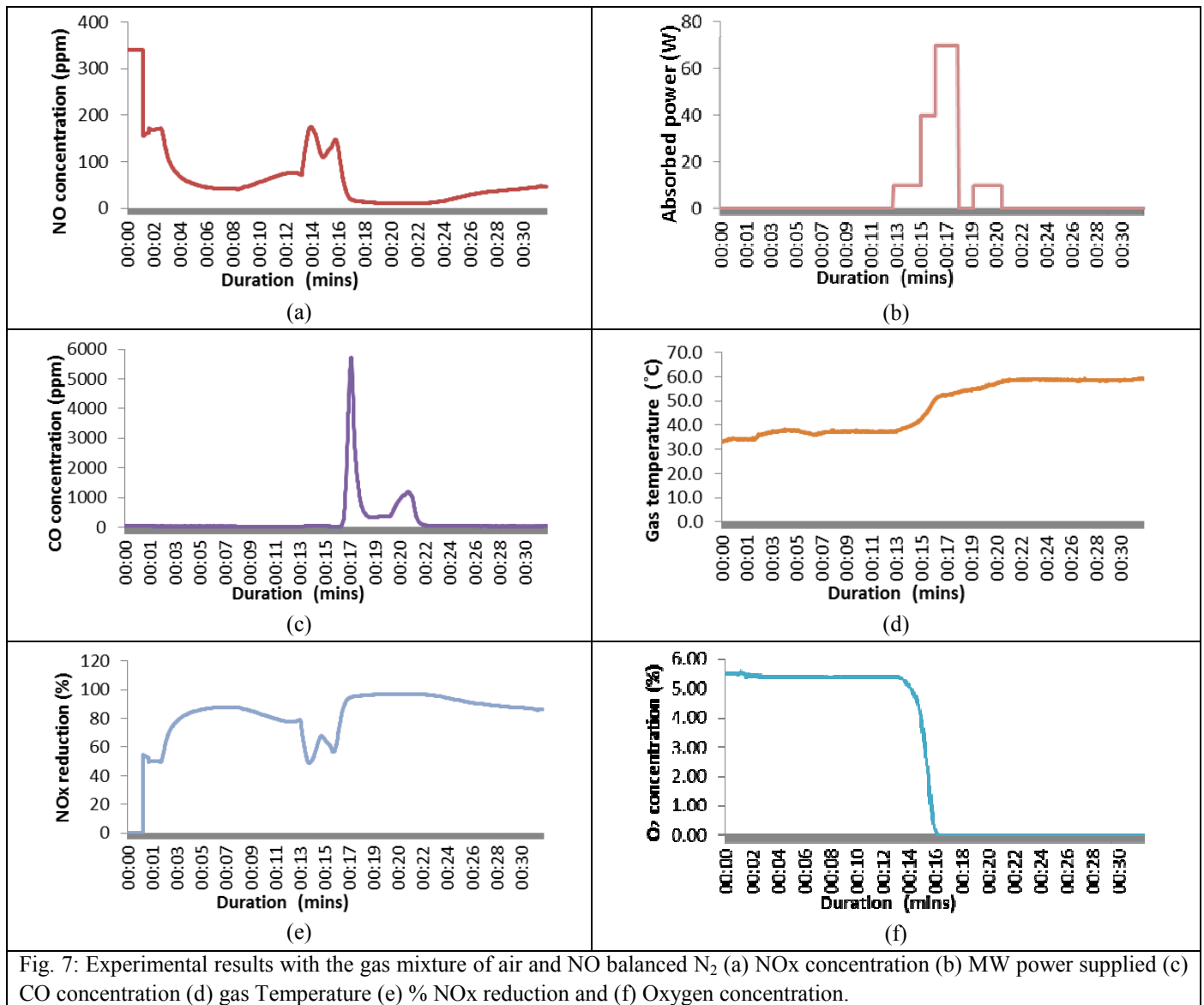


Fig. 7: Experimental results with the gas mixture of air and NO balanced N<sub>2</sub> (a) NOx concentration (b) MW power supplied (c) CO concentration (d) gas Temperature (e) % NOx reduction and (f) Oxygen concentration.



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## **Title: NO Abatement using Microwave Micro Plasma generated with Granular Activated Carbon**

### *Response to the comment of Reviewer 1*

#### Comments to the Author

For the description in the last paragraph in section III:

"It was also found that carbon dioxide started appearing in the outlet gas (not shown) up to 0.25% after the MW power was increased."

you should show the graph of CO<sub>2</sub> concentration as a function of elapsed time to allow readers to understand the disappearing of O<sub>2</sub> shown in Fig. 7 (f).

#### Response by Author

CO<sub>2</sub> was only measured using testo 535 which is only capable to perform spot measurement and hence it was not possible to take a continuous measurements to plot a graph of CO<sub>2</sub>. This one of area suggested in the future work, where we will be able to analyse the CO<sub>2</sub> and co concentration in relation various O<sub>2</sub> level.