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Emission control of SO_2 and NO_x by irradiation methods

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Abstract

Microwave discharges at 2.45 GHz frequency and accelerated electron beams operated at atmospheric pressure in synthetic gas mixtures containing N_2, O_2, CO_2, SO_2, and NO_x are investigated experimentally for various gas mixture constituents and operating conditions, with respect to their ability to purify exhaust gases. An original experimental unit easily adaptable for both separate and simultaneous irradiation with microwaves and electron beams was set up. The simultaneous treatment with accelerated electron beams and microwaves was found to increase the removal efficiency of NO_x and SO_2 and also helped to reduce the total required dose rate with ∼30%. Concomitant removal of NO_x (∼80%) and SO_2 (>95%) by precipitation with ammonia was achieved.

Keywords: Electron beams; Microwave irradiation; Atmospheric pressure; Gas purification; SO_2; NO_x

1. Introduction

Authorised emission levels of a great number of atmospheric pollutants are being lowered for an increasing number of both large and small industrial companies. Thus the development of new pollution control devices and processes is necessary so that the efficiency, output, flow, rate and costs of processing are not adversely affected.

These needs are particularly acute for gases such as VOCs, NO_x (mostly from fixed sources), as well as gases used by the micro-electronic industry, and for odours and dusts (submicronic particles).

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As one of the post-combustion cleaning techniques to remove NO_x and SO_2 from exhaust gases, electron beams have demonstrated their technical and economical feasibility compared to commercially available techniques [1–8]. The original interest in these techniques is related to the following features: (1) simultaneous removal of NO_x and SO_2 with a single dry process; (2) integration with the developed electrostatic precipitator (ESP) to remove NO_x, SO_2 and fly ash simultaneously; and (3) valuable end by-products, such as ammoniumnitrates NH_4NO_3 and ammoniumsulphates $(NH_4)_2SO_4$. To achieve simultaneous NO_x and SO_2 removal, minimum energy consumption usually depends on the NO_x removal rate and its initial concentration. Various kinds of additives, such as NH_3, H_2O_2, hydrogen carbon (HC), N_2H_4, natural gas and hydrated lime, have been used to improve the energy efficiency and to control final by-products. Although treatment with accelerated electron beams is considered one of the most effective methods for simultaneous abatement of SO_2 and NO_x, the electrical energy consumption for effective purification of flue gases is very high; it requires 2–4% of the capacity of the power station. Thus, reduction of electrical energy consumption for the cleaning of flue gases is important and could be solved by the application of synergetic methods such as combined accelerated electron beam (EB) and microwave (MW) induced non-thermal plasmas irradiation.

In non-thermal plasmas operated at atmospheric pressure most of the electrical energy is consumed to produce free radicals, which have a much greater reactivity than atoms and molecules in the ground state. Non-thermal plasmas at atmospheric pressure can be sustained through various methods. Examples are pulsed corona discharges, barrier discharges, and electron beam induced plasmas. Applications of these plasmas abatement processes are discussed extensively by Penetrante and Schulteis [9], Hackman and Akiyama [10] and Urashim and Chang [11]; plasmas techniques are proposed to clean flue gases of coal-fired electrical power plants, to remove NO_x from the exhaust of Otto or diesel engines, or to improve the emission values occurring during painting and surface cleaning. Depending on the conditions and requirements in question, a variety of discharges have been studied with respect to their ability to reduce toxic pollutants like NO_x and SO_2 or hydrocarbons. Physically, non-thermal equilibrium in atmospheric pressure discharges is a relatively marginal situation. In practice, there exist three generic concepts to generate such discharges. To date, according to Rea [12] and Veldhuizen [13], environmental applications have mainly involved corona and dielectric barrier discharge (DBD) systems. Electronic densities in these types of plasmas (10^9 to 10^{11}) are not sufficient to achieve appropriate conversion rates of the toxic gases. Microwave excited plasmas belong to a fundamentally different category. Non-equilibrium results from the fact that at a sufficiently high frequency, electrons respond solely to the applied electromagnetic field. At atmospheric pressure, microwave plasmas exhibit homogeneous densities of 10^{12} to 10^{15} cm^{-3} and therefore appear much more attractive for the abatement of toxic gases. Microwave plasmas can be excited inside resonant cavities, within waveguide microwave circuits or by means of surface wave field applicators, according to Moisan and Pelletier [14]. The use of microwave energy in addition to electron beam energy, or even microwave energy only, to sustain the energy of the free electrons at optimum levels, depends on several parameters such as electric field amplitude, field distribution, energy distribution, and reaction vessel (applicator) geometry, LePrince and Marec [15].

The aim of this study was to demonstrate the ability of simultaneous irradiation with electron beam and microwaves to abate NO_x and SO_2 and to explore the mechanisms

involved in the process in order to optimise it from the point of view of both the removal efficiency and the input energy at a temperature below 70 °C.

2. Experimental method

2.1. Experimental system

A schematic diagram of the experimental system is shown in Fig. 1. Sulphur dioxide (SO_2, >99%), nitrous oxide (NO, >99%) and carbon dioxide (CO_2, >99%) were purchased from L'Air Liquide, Romania. Individual flowmeters were used to achieve the desired concentration of SO_2, NO_x and CO_2 in air. A specially made glass chamber was used to mix all gases with air. The flow was passed through a pre-heating chamber thermostatically controlled at 70 °C, then through a second mixing reactor where a solution of ammonia was added in stoichiometric amount calculated as follows:

$$C_{NH_3} = 2C_{SO_2\,\text{initial}} + C_{NO_x\,\text{initial}} \quad (1)$$

where C_{initial} is the initial molar concentration of SO_2 and NO_x, respectively.

The conditioned gas was treated with microwave and/or electron beams in the irradiation reactor, filtered, neutralised with a solution of sodium hydrogen carbonate, and evacuated into the acid exhaust.

The gas effluent was monitored using an on-line quadruple mass spectrometer (HAL 200 Gas Analyser, Hidden Analytical, UK).

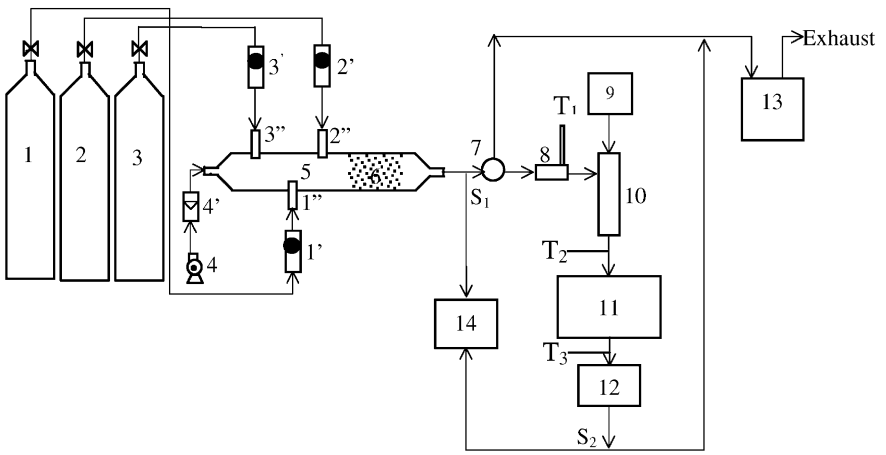

Fig. 1. Schematic diagram of the experimental system, 1: nitrous oxides; 1′: flowmeter; 1″: gas inlet; 2: carbon dioxide; 2′: flowmeter; 2″: gas inlet; 3: sulphur dioxide; 3′: flowmeter; 3″: gas inlet; 4: air; 4′: rotameter; 5: gas mixing chamber; 6: glass spheres; 7: three-way valve; 8: pre-heater, 70 °C; 9: ammonia solution reservoir; 10: liquid–gas mixing chamber; 11: irradiation reactor; 12: filter; 13: neutralization bubbler; 14: mass spectrometer; T_1, T_2, T_3: thermometers; S_1, S_2: sampling ports.

Typical composition of the synthetic gas mixture was: air, water (12–18%), CO_2 (to 5000 ppm), SO_2 (to 2000 ppm), and NO_x (to 1000 ppm). In all experiments, the simulated flue gas was circulated continuously at a flow rate of 10 l/min.

2.2. Plasma reactor

Laboratory experimental methods and procedures in microwave and accelerated electron beam treatments have been continuously evolving, Martin et al. [16]. Early experiments to demonstrate the viability of the technique and to identify and test some of the basic ideas governing the use of microwave power in gaseous systems containing SO_2 were illustrated using commercial microwave ovens modified to contain a quartz reactor and connected to a vacuum line for injection of gaseous reagents. At that time, it was found that the interaction of the microwave energy with the reagents used in experiments (SO_2, H_2O and/or NH_3) was so efficient that it was necessary to reduce the energy in the reactor in order to control the temperature and minimise the formation of NO_x. Therefore, a cylindrical microwave cavity (CMC) with controlled variable power 0–850 W was built, Martin et al. [17]. Fig. 2a shows the CMC, a brass tube 90 mm i.d. and 1000 mm long containing a concentric quartz tube. Various diameters, 20–50 mm, of the quartz tube were tested. The CMC was provided with properly sized end microwave chokes and a very efficient water-cooling system. The cavity was excited with a rectangular waveguide propagating the microwave electric field parallel to its axis. The microwave injection system consisted of a controlled microwave generator (2.45 GHz and 850 W maximum output power) and a waveguide launcher. Destruction efficiencies at ~80% for SO_2 and 30% for NO_x were obtained in the experimental conditions described above. However, the stability of the system decreased while the SO_2 and NO_x were converted to ammoniumsulphates and nitrates. The salts formed in the chemical reaction built solid deposits on the reactor walls which affected the intensity of the discharge and required the quartz tube to be washed with water. Fig. 2b shows the quartz tube with a periodical deposit of white powdery material on the inner surface.

Promising results obtained led us to develop a more suitable test unit (irradiation reactor), designed to facilitate the use of both separate and simultaneous electron beam and microwave energies to produce free radicals.

The source of electron beams was an industrial linear accelerator, ALID-7, built in the Electron Accelerator Laboratory of the Institute of Atomic Physics, Bucharest, described in detail elsewhere, Martin et al. [18]. The calibration was made by several chemical systems such as ceric and chlorobenzene dosimeters, described by Dvornik [19]. The values of the maximum beam power (P_B) and the optimum values for the peak beam current (I_B) and electron energy (E_B) to produce maximum output beam power are as follows: $P_B = 670$ W ($f_r = 250$ Hz and $t_B = 3.75$ μs), $I_B = 130$ mA, $E_B = 5.5$ MeV. The microwave injection system consists of a microwave power controlled generator with 2.45 GHz magnetron of 1200 W maximum power output, a rectangular waveguide launcher fitted to a WR430 waveguide, a dual directional coupler for forward and reflected power monitoring, a three stub tuner for impedance matching, and a slotted waveguide (inclined slots cut in the broad wall of a WR430 waveguide) used as a radiating antenna.

In order to minimise the dielectric losses in the internal walls, the rectangular multimode reaction cavity of 240 mm × 240 mm × 450 mm (inner dimensions) was built of aluminium.

(a)

(b)

Fig. 2. (a) Cylindrical microwave cavity (CMC); (b) solid salts distribution inside the quartz tube.

The scanned electron beams are introduced perpendicular to the upper-end plate through a 100 μm thick titanium window. The method of coupling the microwave power to the cavity via the slotted waveguide provided good microwave energy transfer and uniformity over a large area. The gas mixture entrance and exit are branched through two sieve metallic end plates. A photograph and a schematic drawing of the experimental unit is shown in Fig. 3a and b.

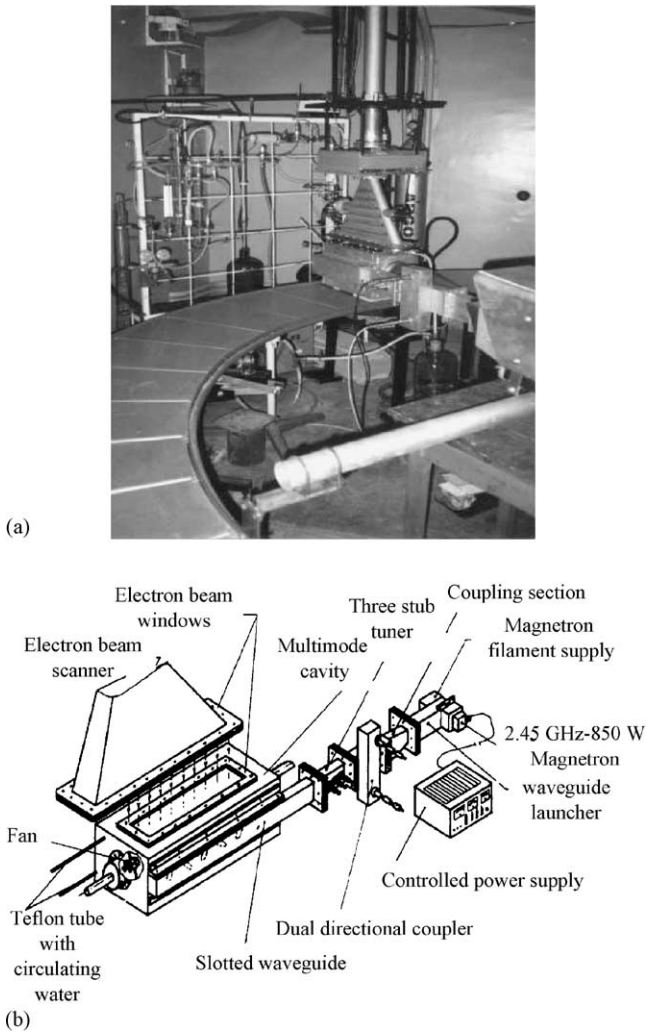

Fig. 3. Irradiation reactor: (a) photograph; (b) schematic drawing.

3. Results and discussion

The primary processes can be represented by:

$$\text{for } NO_x \quad \begin{cases} NO \overset{OH,\,O,\,HO_2}{\rightarrow} HNO_2,\ NO_2 \overset{OH,\,O,\,H_2O}{\rightarrow} HNO_3 \\ HNO_3 + NH_3 \rightarrow NH_4NO_3 \end{cases}$$

$$\text{for } SO_2 \quad \begin{cases} SO_2 \overset{OH,\,O,\,HO_2}{\rightarrow} HSO_3,\ HSO_4,\ SO_3 \overset{OH,\,HO_2,\,H_2O}{\rightarrow} H_2SO_4 \\ H_2SO_4 + 2NH_3 \rightarrow (NH_4)_2\,SO_4 \end{cases} \tag{2}$$

Tests were carried out to estimate the efficiencies of NO_x and SO_2 removal from the flue gases in different irradiation conditions, and to estimate the reduction of the energy consumption required to obtain the same purification effect both with electron beam only and simultaneous electron beam—microwave power.

The NO_x and SO_2 destruction removal efficiencies (DRE_{NO_x} and DRE_{SO_2}) were calculated with the following equations:

$$\mathrm{DRE}_{\mathrm{NO}_x}\,(\%) = \left(1 - \frac{C_{\mathrm{NO}_x\,\mathrm{exit}}}{C_{\mathrm{NO}_x\,\mathrm{initial}}}\right) \times 100 \quad \text{and}$$

$$\mathrm{DRE}_{\mathrm{SO}_2}\,(\%) = \left(1 - \frac{C_{\mathrm{SO}_2\,\mathrm{exit}}}{C_{\mathrm{SO}_2\,\mathrm{initial}}}\right) \times 100 \tag{3}$$

where C_{initial} and C_{exit} are the initial and final molar concentrations of NO_x and SO_2, respectively.

In all experiments, the irradiation was carried out until the gas composition reached a steady state.

3.1. Microwave treatment (MW)

The dependence of DRE on the irradiation time and power was investigated for gas mixtures containing only SO_2 and only NO_x. The results are shown in Figs. 4 and 5.

The results in Fig. 4 indicate that the microwave system can be applied with good destruction efficiency for SO_2, DRE 50–90%. It is noteworthy that a considerable amount of SO_2—up to 40%—was removed without irradiation, by spontaneous reaction of SO_2 with ammonia and water vapour. A microwave discharge appears and remains stable while especially low values of the reflected microwave power rate ($P_r < 10\%$) are realised at forward power (P_f) greater than 800 W. Once the positions of the three screws tuner were adjusted for minimal P_r/P_f, there is hardly any change in the power meter readout.

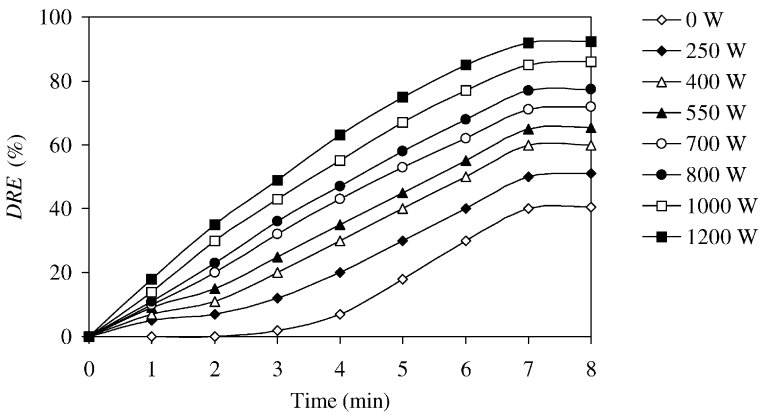

Fig. 4. Removal efficiency of SO_2 vs. microwave irradiation time and power $C_{\mathrm{SO}_2\,\mathrm{initial}} = 2000$ ppm, $C_{\mathrm{NO}_x\,\mathrm{initial}} = 0$, $T = 70\,^{\circ}\mathrm{C}$.

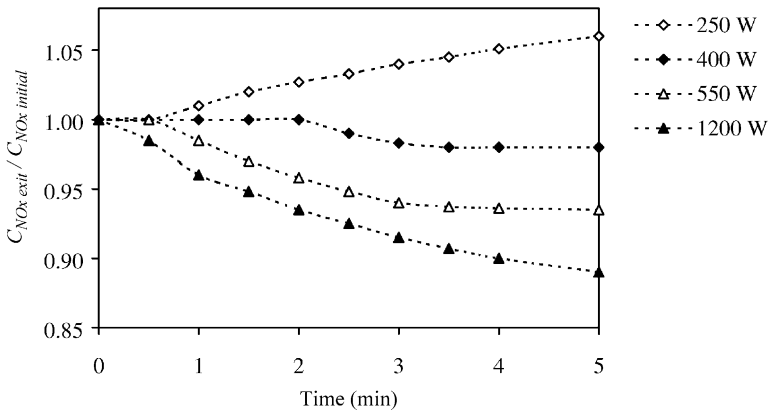

Fig. 5. Removal efficiency of NO_x vs. microwave irradiation time and power $C_{SO_2\ initial} = 0$, $C_{NO_x\ initial} = 730$ ppm, $T = 70\,^{\circ}C$.

Fig. 5 shows that the microwave process is less effective for the removal of NO_x. At low levels of forward microwave power, $P_f < 400$ W, the ratio of NO_x formation from air is greater than its destruction. At power levels in the range of 400–1200 W the destruction of NO_x exceeds recombination. However, DRE remains very low, between 6–10%, indicating that microwave irradiation, in the described conditions, cannot be applied as an efficient method for the destruction of NO_x.

3.2. Irradiation with accelerated electron beam (EB)

The basic principle of removing SO_2 and NO_x from flue gases by electron beams consists of irradiation of the gas mixture with high energetic electrons. In this method, NH_3 is injected into the gas mixture just before the irradiation section and the solid by-products (ammonium salts) are separated by filtration after irradiation.

Figs. 6 and 7 show the effect of electron beams on the removal rate of SO_2 and NO_x.

SO_2 is easily removed even at low total absorbed dose levels; at 1 kGy, DRE was 50% and increased to 90% for 40 kGy.

For SO_2 removal, the initial reaction rates were found to be linear in the studied range of total absorbed doses; for NO_x, the initial reaction rate seemed to reach a plateau, indicating that, especially at high absorbed dose levels, the formation reactions of NO_x (recombination from ions and radicals and N_2 from air oxidation) are in equilibrium with those involving its removal.

3.3. Simultaneous irradiation with electron beam and microwave (EB + MW)

It has been proved that both external accelerated electron beam and microwave can be regarded as means of generating free electrons and active species. In the case of external electron accelerators, the active species are concentrated along the accelerated electrons path. At low and medium dose rates, the chemical reactions consume the majority of the

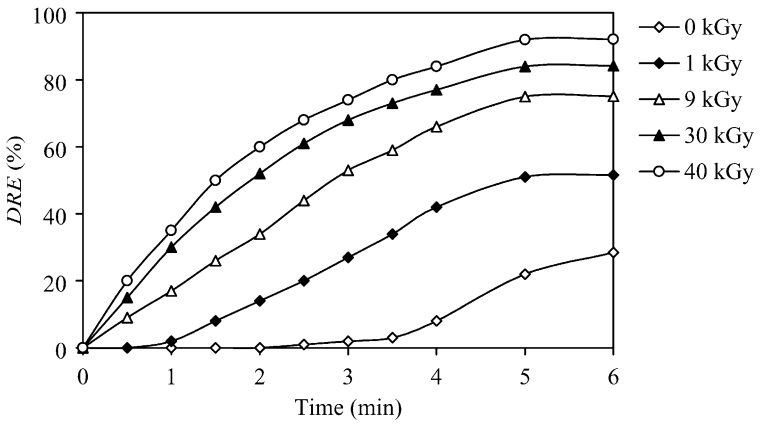

Fig. 6. Removal efficiency of SO_2 vs. electron beams irradiation time and total absorbed dose $C_{SO_2\,\text{initial}} = 2000\,\text{ppm}$, $C_{NO_x\,\text{initial}} = 0$, $T = 70\,^{\circ}\text{C}$.

radicals. At high dose rates, the radical concentration is much higher and radical recombination reactions may no longer be neglected, leading to low SO_2 and NO_x removal efficiencies. With microwave energy applied by properly designed applicators, promising results are expected, as microwaves penetrate large volumes and lead to the formation of a volume of active species, thus avoiding fast radical recombination reactions.

The essential feature of combined microwave and electron beam irradiation is the additional use of microwave energy for increasing the number of free electrons and sustaining their energy at optimum levels. This leads to decreased levels of electron beam total dose by maintaining the same removal efficiency.

Fig. 8 summarises the SO_2 destruction removal efficiency over time for the simultaneous irradiation experiments. Although the process conditions were slightly varied throughout

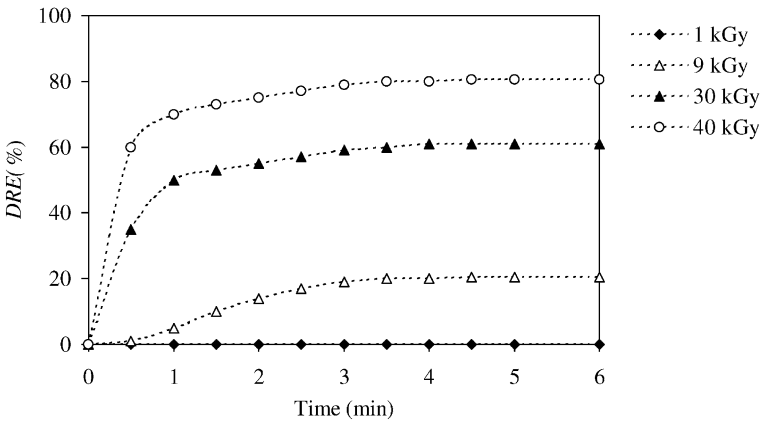

Fig. 7. Removal efficiency of NO_x vs. electron beams irradiation time and total absorbed dose. $C_{SO_2\,\text{initial}} = 0$, $C_{NO_x\,\text{initial}} = 730\,\text{ppm}$, $T = 70\,^{\circ}\text{C}$.

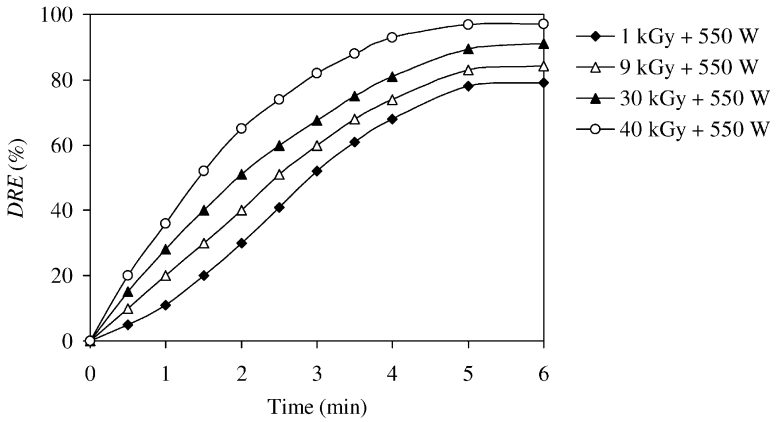

Fig. 8. Removal efficiency of SO_2 vs. electron beam total absorbed dose and microwave power and irradiation time $C_{SO_2\ initial} = 2000$ ppm, $C_{NO_x\ initial} = 0$, $T = 70\,^{\circ}C$.

the many tests performed, the input concentration of SO_2 remained close to 2000 ppm and the temperature maintained at 70 °C. The air dilution flow rate was 10 l/min.

The additional use of MW energy to the EB energy increases the SO_2 removal speed and efficiency. SO_2 removal efficiency for simultaneous EB and MW irradiation of 1 kGy + 550 W has the same value as for EB irradiation at 9 kGy; thus, for the same removal efficiency, the required dose in simultaneous EB + MW is approximately 10 times smaller than used in EB irradiation. The process carried out at 9 kGy + 550 W gives the same DRE as obtained at 30 kGy; the required total absorbed dose level being three times lower. This effect decreases with the increase of the absorbed dose level because, at high dose, the EB irradiation becomes very effective by itself (Table 1).

A set of experiments was performed to evaluate the efficiency of the process when SO_2 and NO_x are added together in the gaseous mixture. Fig. 9 shows the destruction efficiency of SO_2 and NO_x related to EB and EB + MW methods. The presence of both SO_2 and NO_x in the gas mixture proved to increase the removal efficiency of the toxic gases in both EB and EB + MW processes, especially when low doses were applied.

Table 1
The reduction of total absorbed dose in EB + MW method vs. EB in destruction of SO_2; $C_{SO_2\ initial} = 2000$ ppm, $C_{NO_x\ initial} = 0$, $T = 70\,^{\circ}C$; $P_f = 550$ W

Total absorbed dose level (kGy)	DRE_{SO2} in EB process (%)	DRE_{SO2} in EB+MW process (%)
1	51	79
9	75	84
30	84	91
40	92	97

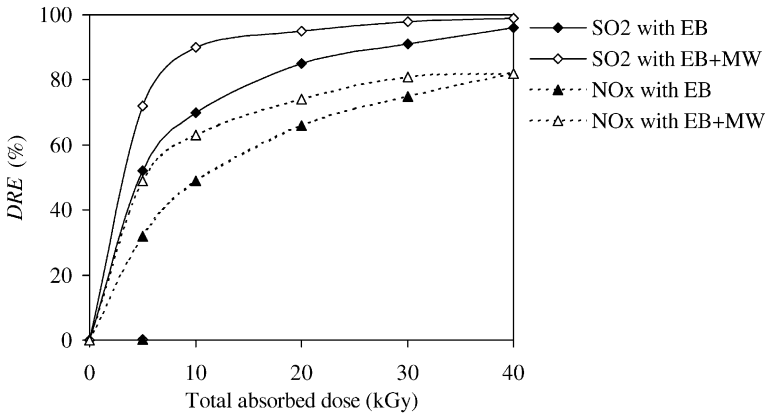

Fig. 9. Removal efficiency of SO_2 and NO_x vs. electron beam total absorbed dose and microwave power ($P_{MW} = 550$ W) $C_{SO_2\,initial} = 1350$ ppm, $C_{NO_x\,initial} = 900$ ppm, $T = 70\,^{\circ}C$.

Table 2
Results of quantitative and qualitative analyses of the by-product separated after irradiation

Compound	Analytical method	Weight (%)
Sulphate (SO_4^{2-})	Barium sulphate	30.50
Nitrate (NO_3^-)	Devarda's alloy and Kjeldahl	41.70
Ammonia (NH_4^+)	Kjeldahl	23.60
Water	Gravimetric	3.10
Total		98.90

3.4. By-product

X-ray diffraction of the solid by-product separated in the filter installed downstream the irradiation reactor showed characteristic peaks for $(NH_4)_2SO_4$, $(NH_4)_2SO_4(2NH_4NO_3)$ and $(NH_4)_2SO_4(3NH_4NO_3)$.

Quantitative and qualitative chemical methods described by Mendham et al. [20] were also used to identify and analyse the solid by-product. The results of these analyses are presented in Table 2. However, in order to demonstrate and clarify how the irradiation method and concentration of reactants influence the reaction, we shall further extend these results with more studies focused on the by-product formation and changes as the reaction proceeds.

4. Conclusions

Removal of SO_2 and NO_x by irradiation with accelerated electron beams and/or microwaves was studied using ammonia and water as reagents. The by-products, solid salts of ammonia, were separated post-irradiation by filtration. Original laboratory equipment that allows for irradiation with accelerated electron beams (EB) only, microwaves (MW)

Table 3
Influence of the irradiation method on the destruction removal efficiency (DRE) of SO_2 and NO_x

Irradiation method	DRE_{SO_2} (%)	DRE_{NO_x} (%)
MW 550 W (2.45 GHz)	65	6.5
EB 40 kGy	89	80
EB + MW 30 kGy + 550 W	91	81

only, and simultaneous treatment with microwave and electron beams (EB + MW) has been developed for this research.

It was established that the destruction removal efficiency (DRE) of SO_2 in MW processing is greater than 90% at 1.2 kW (2.45 GHz); the irradiation with EB at 40 kGy total absorbed dose removes ∼90% SO_2. The simultaneous treatment EB + MW also provides DRE ∼90% but the required energy introduced into the system by electron beams is 1.4 times lower (Table 3). It was also found that ca. 40% of SO_2 can be removed without irradiation, by spontaneous reaction with ammonia at temperatures below 70 °C.

It was documented that the microwave treatment of gaseous mixtures containing NO_x cannot be applied as an efficient method for the destruction of NO_x. At power levels below 400 W, the reaction rate of recombination is greater than destruction. In the EB and EB+MW processes, NO_x is removed with 80% efficiency.

Further studies of EB + MW on the removal of NO_x and SO_2 are continuing. Although, we have demonstrated that EB + MW can be applied as the energy source for this process, it must be pointed out that the whole system has not been totally optimised. The potential of MW + EB technologies for the destruction of toxic gases has been addressed in this paper. This simultaneous irradiation pathway has been proved to have advantages over the conventional processes. The scope of this paper is to demonstrate the ability of simultaneous irradiation with electron beam and microwave to induce a variety of physical and chemical

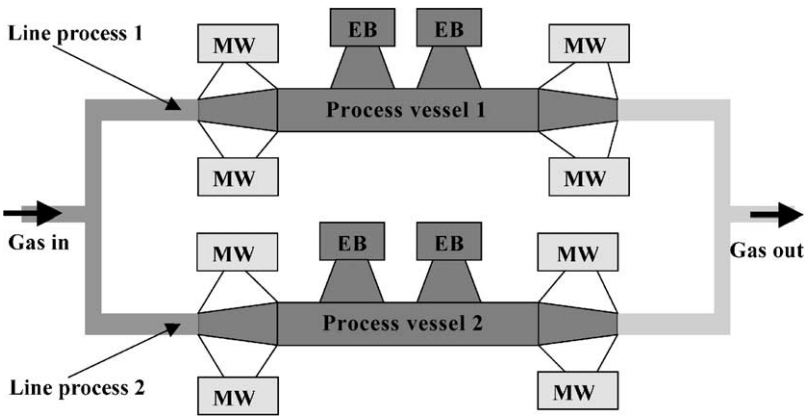

Fig. 10. Thermo-Power Plant CET-West Bucharest. Process for simultaneous SO_2 and NO_x removal by combined electron beam and microwave irradiation.

Table 4
Installation for SO_2 and NO_x removal by combined electron beam and microwave irradiation; proposed for a boiler of 525 t/h at Thermo-Power Plant CET-West Bucharest of 550 MW

Parameter	
The source of flue gas	Oil + gas burning
Flow rate	345,100 Nm^3/h
Conditioned gas composition	$CO_2 = 140.5\ g/Nm^3$; $CO = 0.0035\ g/Nm^3$ $O_2 = 165.2\ g/Nm^3$; $N_2 = 779.51\ g/Nm^3$ $SO_2 = 3.46\ g/Nm^3$; $NO_x = 0.458\ g/Nm^3$ $H_2O = 100.45\ g/Nm^3$; $NH_3 = 3.464\ g/Nm^3$ Dust $= 0.05\ g/Nm^3$
Conditioned gas temperature	70 °C
EB power per 1 Nm^3	4.40 W
MW power per 1 Nm^3	4.00 W
Total EB + MW power	1520 + 1380 kW
Estimated removal efficiency	SO_2 = 95–99%, NO_x = 75–80%
The main items of the process	Number of process lines: 2 Process vessel: $1.1 \times 1.96 \times 0.245\ m^3$ Number of accelerators per line: 2 Number of MW sources per line: 4 EB energy: 0.8–1 MeV EB power per accelerator unit = 400 kW MW power per source unit = 200 kW

phenomena, which can play a significant role in the development of new and existing technologies in toxic gases abatement.

Based on this research and the results obtained by a pilot project built in collaboration with Electrostatica Bucharest, a project for an installation involving simultaneous SO_2 and NO_x removal by irradiation with accelerated electron beams and microwaves has been proposed. The main parameters and the process flow diagram are shown in Fig. 10 and Table 4.

To make EB + MW removal process more economically attractive, further investigation of the mechanism of the chemical effects from microwaves and electron beams, on reactor technology and process technology is needed. The general conclusion of this study is that simultaneous electron beam and microwave irradiation is a viable and promising method for flue gas cleaning in view of the reduction of power consumption and flue gas treatment process cost.

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