

# Non-thermal plasma based decomposition of volatile organic compounds in industrial exhaust gases

M. Schmidt<sup>1</sup>  · I. Jõgi<sup>2</sup> · M. Hołub<sup>3</sup> · R. Brandenburg<sup>1</sup>

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**Abstract** The decomposition of various volatile organic compounds (VOCs), namely butyl acetate, styrene, and methanol, by means of non-thermal plasma technology was investigated in different industrial exhaust gases. Although a great deal of effort on the investigation of these VOCs can be found in literature, data regarding the application in real exhaust gases are missing and provided in this contribution. Measurements were performed in an oil shale processing plant in Estonia and in a yacht hall production site in Poland. Up to 71 % of butyl acetate at a specific input energy (SIE) of about 220 J/L and more than 74 % of styrene and methanol at SIE > 300 J/L were decomposed. The energy efficiency of the decomposition processes was analyzed as well as the products formed by the plasma process. Energy constants of  $k_E < 9.85$  L/kJ for butyl acetate and  $k_E < 2.75$  L/kJ for styrene and methanol were obtained. Most of the VOCs were partly oxidized to carbon monoxide and, to lesser extent, totally oxidized to carbon dioxide.

**Keywords** Non-thermal plasma · Dielectric barrier discharge · Exhaust gas · VOC · Decomposition · Pollution control · Industrial scale

## Introduction

Many industrial processes cause emissions of gaseous pollutants endangering public health and the environment. These are e.g., oxides of nitrogen ( $\text{NO}_x$ ), oxides of sulfur ( $\text{SO}_x$ ), and volatile organic compounds (VOCs). Established technologies for gas cleaning are, e.g., catalysis and thermal oxidation, dry and wet scrubbing, and biofiltration (Wang et al. 2004). These methods are efficient with respect to the level of removal of pollutants and energy consumption. Catalytic processes and thermal oxidation require high temperatures ( $\sim 1000$  °C). Beside the large-scale installations of most of the technologies, they suffer from poor applicability to processes varying fast in concentration of pollutants and gas flow volumes. A prospective means to overcome at least the latter is the use of non-thermal plasma (NTP) technology. The term NTP refers to an ionized gas containing, additional to the neutral bulk gas molecules and atoms, also ions, electrons, and excited species. The species are not in thermal equilibrium. A common way to generate an NTP is to apply an electric field to a neutral gas. If the breakdown field strength is exceeded, a gas discharge is forming the plasma. The electric field can be applied by using two electrodes, one connected to ground and the other one to a high voltage supply. Dielectric barrier discharges (DBDs, sometimes also referred to as silent discharges) are a prevalent type of NTP generated at atmospheric pressure and are used in exhaust treatment, for ozone generation,

✉ M. Schmidt  
M\_Schmidt@inp-greifswald.de

<sup>1</sup> Leibniz-Institute for Plasma Science and Technology,  
Felix-Hausdorff-Str. 2, 17489 Greifswald, Germany

<sup>2</sup> Institute of Physics, University of Tartu, Ravila 14c,  
50411 Tartu, Estonia

<sup>3</sup> West Pomeranian University of Technology, al. Piastów 17,  
70-310 Szczecin, Poland



surface treatment, and many other applications (Wagner et al. 2003; Kogelschatz 2003; Brandenburg et al. 2011). In DBDs, at least one of the electrodes is covered with a dielectric material (e.g., glass or alumina). Typical configurations are a metal wire in a glass tube wrapped with a metal foil or metal plates separated by an alumina plate.

The use of NTP allows the initiation of various chemical processes without a significant increase in the gas temperature. The chain of reactions starts among others with ionization, excitation, dissociation, or dissociative ionization of bulk gas molecules (nitrogen, oxygen, and water, if present), by direct collisions of these molecules with free electrons. Besides direct electronic processes, there are, for example, indirect processes like Penning ionization and many others. Thus, various intermediate and reactive species are formed. The most important species utilized for the removal of gaseous pollutants in water containing air are radicals (e.g., O, OH, HO<sub>2</sub>) and ozone (O<sub>3</sub>) with different oxidation potentials. A detailed discussion of plasma chemical processes can be found in (Fridman 2008) and, especially for pollutants decomposition, in (Kim 2004).

The decomposition of VOCs with NTPs is widely studied for laboratory conditions with and without the use of catalysts (e.g., Huang et al. 2011; Vandenbroucke et al. 2011; Aerts et al. 2013). One of the most intensively studied VOCs is toluene (C<sub>7</sub>H<sub>8</sub>) consisting of a benzene ring with a methyl group (Van Durme et al. 2007; Vandenbroucke et al. 2012). It was found that the decomposition process starts with the hydrogen abstraction from the methyl group by electron impact or reaction with hydroxyl radical. The following reaction proceeds to the formation of ring-containing compounds (e.g., benzaldehyde or benzoic acid) or ring-opening products (e.g., formic acid). There are also studies about the removal of other VOCs available in literature (see, e.g., Kim 2004; Fridman et al. 2005).

However, most of the studies in the reviews and contributions mentioned above are performed in the laboratory and thus under well-defined conditions. These studies deal with a general understanding of the mechanisms of plasma chemistry and discharge development which is very valuable and desired for the development of new processes. On the other site, data regarding the application in real exhaust gases are only few, and most of them are concerning SO<sub>x</sub> and NO<sub>x</sub> treatment (Kim 2004; Hammer 2014). One example of the treatment of VOCs, namely methanol, acetone, dimethyl sulfide, and  $\alpha$ -pinene in the exhaust of pulp mills and wood product plants is described in (Fridman et al. 2005). Mizuno is presenting the removal of acetaldehyde (ethanal, C<sub>2</sub>H<sub>4</sub>O) in an indoor air cleaning process (Mizuno 2007).

This work focuses on the removal of butyl acetate, styrene, and methanol in the exhausts of a shale oil production plant in Estonia and a yacht hull production site in Poland, respectively. Butyl acetate (C<sub>6</sub>H<sub>12</sub>O<sub>2</sub>) is widely used as a solvent in the production of coatings, finishes, inks, and cosmetics (MCgroup 2014a, butyl acetate) with a global production of 528,000 tons in 1998 (WHO 2005). Vapor and liquid of butyl acetate are inflammable and form explosive mixtures. Butyl acetate induces drowsiness; the workplace exposure limit is 62 ppm (BGRCI 2014a, BA, all legislative limitations given are valid for Germany). Styrene (C<sub>8</sub>H<sub>8</sub>) is used in the production of rubber, plastics, adhesives, and inks with a global production of more than 26.4 million tons in 2012 (MCgroup 2014c, styrene). Vapor and liquid of styrene are inflammable and health damaging when inhaled and lethal if swallowed. The workplace exposure limit is 20 ppm (BGRCI 2014c, styrol). Methanol (CH<sub>4</sub>O) is used as a feedstock in chemical industry and fuel for combustion engines. The global production of methanol reached almost 63 million tons in 2012 (MCgroup 2014b, methanol). Vapor and liquid of methanol are easy inflammable. Methanol is toxic when swallowed, inhaled or at skin contact. The workplace exposure limit is 200 ppm (BGRCI 2014b, methanol).

To the authors' present knowledge, there are only few data available for the abatement of butyl acetate, styrene, and methanol, which were found to be the most important pollutants in the field tests, by means of NTP (Demidiouk et al. 2003; Zhang et al. 2009). Thus, the plasma reactor, its application, and the removal efficiencies for butyl acetate, styrene, and methanol as well as the formation of reaction products and by-products are presented in this contribution.

## Materials and methods

The experiments took place in an oil shale processing plant in Estonia and in a company producing hulls for boats and yachts in Poland. The experiments were performed in the bypass of the emission duct during the industrial process. This process was not interfered by the experiments. In order to study the general plasma chemistry and to evaluate the plasma effect, sensitive laboratory equipment was applied to industrial environments under rough conditions (dusty air, heat, moisture, and vibrations of installations and devices). Although the conditions of the process gases to be treated were varying (fluctuation of concentrations of the pollutants, moisture, and gas flow), it was possible to obtain sufficient and reliable results after careful selection of the data to be used.



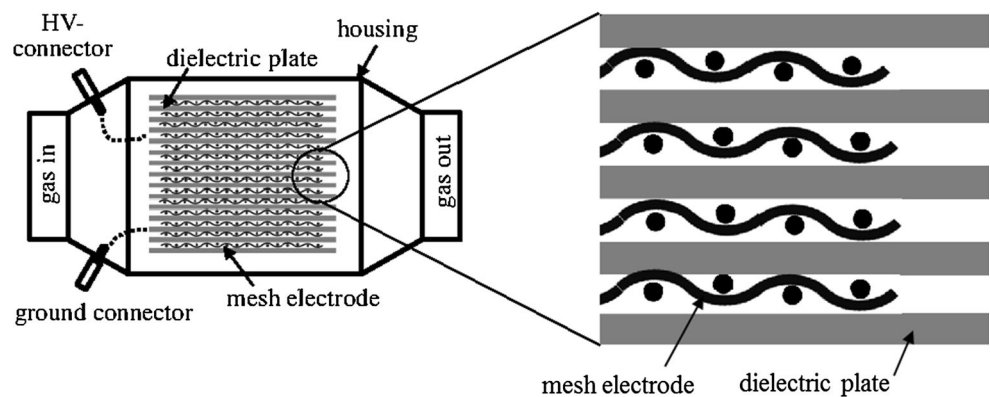
In general, the experimental setup was almost identical for both experiments in Estonia and Poland. It consisted of the plasma source, a power supply to energize the plasma, and a set of diagnostics to analyze the gas composition and the electrical parameters of the plasma source. The gas sample was extracted through the bypass and directly treated by the plasma in all experiments. A more detailed description follows together with the differences of the experimental apparatus.

The treatment of the gas samples was performed with a plasma source (Fig. 1) designed in general at INP Greifswald (Müller and Zahn 2007). The reactor used for the described experiments consisted of 50 mesh electrodes made of stainless steel which were separated by each other by dielectric plates made of phlogopite (magnesium mica). The electrodes were alternating connected to high voltage

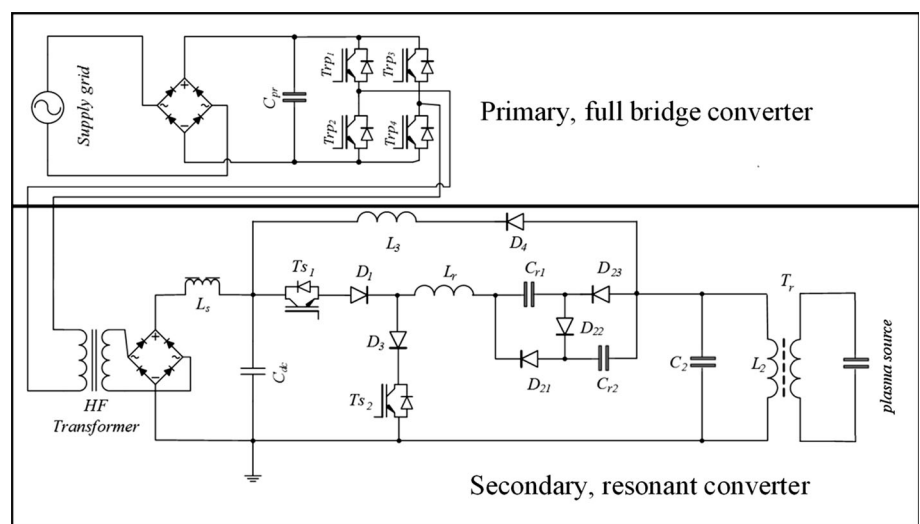
and ground. Thus, the plasma was formed by a stacked volume DBD reactor configuration. This discharge configuration was mounted and sealed in a stainless steel housing also containing the electrical connectors and gas in- and outlet connectors (100 mm diameter).

The plasma source was energized with sinusoidal high voltage with two different power supplies. For the experiments in Estonia, a programmable high-voltage (HV) power supply (Chroma 61604) with a matched high-voltage transformer was used. The amplitude of the high voltage was measured with a digital oscilloscope (Tektronix DPO 4104) via a high-voltage probe (Tektronix P6015A). The current was measured by reading the voltage drop over a shunt ( $R = 50 \Omega$ ). By multiplying the averaged signals and dividing with the resistance of the shunt, the power input into the plasma was calculated

**Fig. 1** Scheme of the plasma reactor



**Fig. 2** Scheme of the resonant power supply



(Weltmann et al. 2010). For the experiments in Poland, the reactor was energized with a high-efficiency, resonant power supply schematically shown in Fig. 2. The system consists of two stages. The DC link voltage value is defined by the first full-bridge stage. The second resonant stage allows highly efficient output voltage control using the zero-current switching technique with the  $T_{s1} - T_{s2}$  transistor timing (Jakubowski et al. 2011, 2013). In case of this set of experiments, the plasma power was calculated from voltage and current waveforms using a digital oscilloscope (LeCroy WaveRunner 6100A) via high-voltage probe (LeCroy PPE 20 kV) and current monitor (LeCroy CT031, Hall-effect sensor). In all experiments, the power input into the plasma was up to 150 W at frequencies around 1 kHz and peak-to-peak voltages well below 10 kV<sub>pp</sub>.

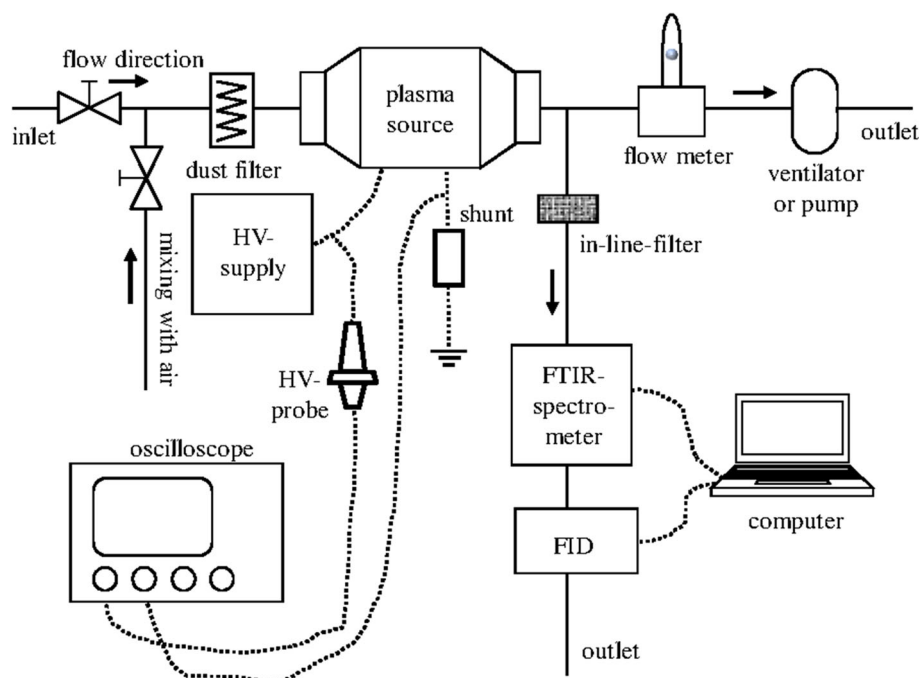
The gas samples to be treated were sucked by a ventilator (Estonia) or a pump (Poland) into the experimental apparatus which is schematically shown in Fig. 3. In the experiments in Estonia, the gas flow was 5 and 2.5 m<sup>3</sup>/h for SIE = 110 and 220 J/L, respectively. In Poland, around 0.3 m<sup>3</sup>/h was realized. With the volume of the plasma source (960 cm<sup>3</sup>), residence times for the gas in the plasma of 0.27–5 s are calculated. Here it was taken into account that around 60 % of the cross section area is covered with

dielectrics and electrodes. The inlet part of the system consists of a pipe connected to the industrial process and a second pipe for air to be mixed with the process gas. Before entering the plasma reactor, the gas passed a dust filter to protect the experimental system from particles. After the plasma source, the gas passed a flow meter, passed the ventilator/pump, and left the system through the outlet. Directly after the plasma source, samples of the gas were led into the gas diagnostics devices, namely an FTIR spectrometer (Fourier transformation infrared spectrometer, Bruker Alpha, 1 cm<sup>-1</sup> spectral resolution, 500 mL volume of the gas cell,  $T_{\text{cell}} = 40$  °C) and an FID (flame ionization detector, Testa 2010T). In Estonia, only the FTIR spectrometer was used. To protect the devices from damage by dust particles, an in-line filter (pore size 2 µm, wall thickness 1 mm, area ca. 120 mm<sup>2</sup>) was used. An influence of this filter on the gas measurement, e.g., by adsorption appearing as a smoothening of the changes in concentration, was not detected.

From the measured power input into the plasma and gas flow through the reactor, the specific input energy (SIE) was calculated as given in Eq. (1)

$$\text{SIE} \left[ \frac{\text{J}}{\text{L}} \right] = \frac{\text{power input [W]} \cdot 3600}{\text{gas flow} \left[ \frac{\text{L}}{\text{h}} \right]} \quad (1)$$

Fig. 3 Experimental setup



The SIE acts as a scaling parameter to evaluate the energy efficacy of the plasma-based conversion of pollutants (Kim 2004).

## Results and discussion

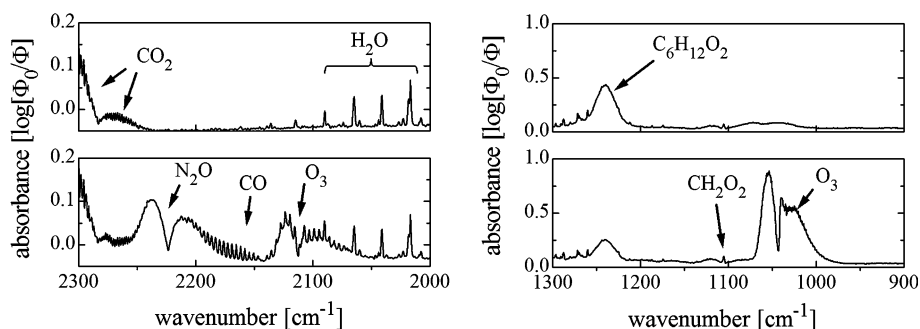
### Oil shale processing plant

During the experiments in the Estonian oil shale processing plant, the inlet conditions were varying strongly. This affected the gas flow, the humidity, and the concentration of the target pollutant butyl acetate. Thus, only such experimental data were used for analysis which provided reliable distinction between changes of the gas composition due to the plasma action or due to changes of the industrial process itself. The criterion therefore is a decrease in the butyl acetate concentration correlated with an increase in the concentration of carbon dioxide and carbon monoxide as well as formic acid ( $\text{CH}_2\text{O}_2$ ) as the main plasma chemical reaction products. Furthermore, the concentration of water ( $\text{H}_2\text{O}$ ) must be

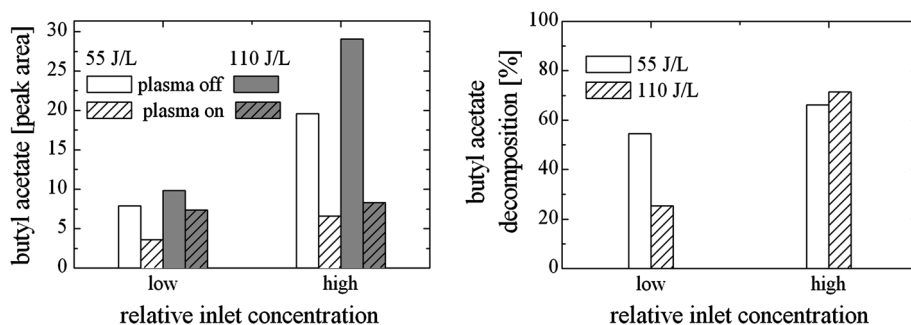
almost stable [variation around 1.8 vol% (40 % RH at 30 °C)], as this is indicating that the inlet conditions were comparable during the plasma treatment. Therefore, data were selected by comparing the infrared absorption spectra recorded before and after switching on the plasma. The infrared absorption spectra given in Fig. 4 are typical examples for the performed measurements for the infrared region 2300–2000 and 1300–900  $\text{cm}^{-1}$ . The upper diagrams display the spectra for gas composition without plasma and the lower lines with plasma on (SIE = 220 J/L). Together with the absorption bands used to fulfill the aforementioned criterion, the markers for plasma operation are shown. Here, the absorption signals of ozone ( $\text{O}_3$ ) and nitrous oxide ( $\text{N}_2\text{O}$ ) are used, since these compounds are typical by-products of NTPs operated in air (Kogelschatz 2003).

Out of the measurements, four representative sets of data were extracted for two different SIE values (110 and 220 J/L). For each SIE, one measurement with low and one with high inlet concentration of butyl acetate were chosen. For all measurements, a reduction in the amount of butyl acetate is found (Fig. 5, left). The white bars

**Fig. 4** Samples of the infrared absorption spectra of the analyzed gas mixture under plasma off condition (top) and with plasma on (SIE = 220 J/L, bottom)



**Fig. 5** Left Concentration of butyl acetate for two different SIE values with respect to the relative inlet concentration. Right decomposition of butyl acetate with respect to the relative inlet concentration



indicate the concentration of butyl acetate without plasma treatment, and the gray bars correlate with the plasma on condition. It is found that the outlet concentration does not exceed 30 ppm, which is well below the legislative limitations, although the inlet concentration varies quite strongly between 30 and 110 ppm. From these data, the decomposition rate was calculated according to Eq. (2)

$$\Delta[\text{VOC}] = \left(1 - \frac{[\text{VOC}]}{[\text{VOC}]_0}\right) \cdot 100 \% \quad (2)$$

where “VOC” stands for the VOC (in this case butyl acetate) and the index “0” indicates its concentration without plasma operation. It is found that at higher inlet concentrations, higher decomposition rates are achieved without a clear difference between the values of the SIE (Fig. 5, right). Up to 71 % of butyl acetate is decomposed at high inlet concentration and at SIE = 220 J/L. From the concentrations with and without plasma and the applied SIE, the energy constant  $k_E$  was calculated according to Eq. (3).

$$k_E = \ln\left(\frac{[\text{VOC}]_0}{[\text{VOC}]}\right) \cdot \text{SIE}^{-1} \quad (3)$$

The values are summarized in Table 1.

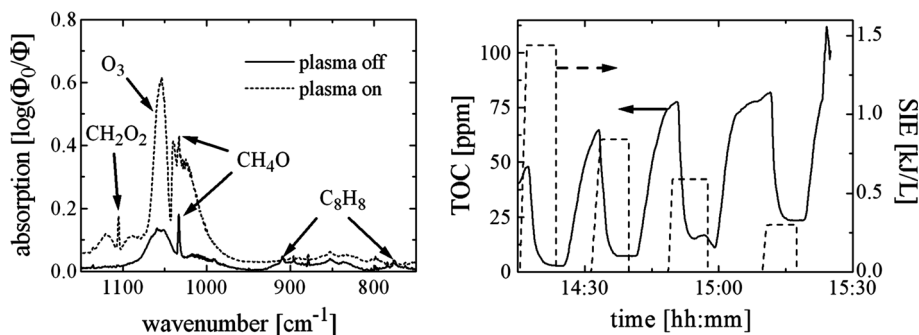
Analyzing the amount of  $\text{CO}_2$  and CO produced, it can be stated that the  $\text{CO}_2$  selectivity is lower than 50 % in all experiments. Most of the butyl acetate is partly oxidized to CO. The other by-products found were ozone in

concentrations of 280–740 ppm and formic acid in concentrations below 3 ppm. Since formic acid is soluble in water and CO together with ozone is removable by a catalyst ( $\text{Pt}/\text{Al}_2\text{O}_3$ , Demidiouk et al. 2003), the combination of NTP technology with catalyst and scrubber could be a promising solution for process gases polluted with butyl acetate. The plasma source used in this work achieves up to 65 % removal of butyl acetate at SIE = 110 J/L. This efficacy is more than twice the best value reported in (Demidiouk et al. 2003) who used a pulsed wire-cylinder corona discharge. One notable difference as compared to the experiments carried out by Demidiouk is the presence of humidity which may strongly affect the removal efficiency. There are no data available, concerning the humidity effect in the plasma removal of butyl acetate. The removal of another ester, namely ethyl acetate ( $\text{C}_4\text{H}_8\text{O}_2$ , ethyl ethanoate), at dry and humid conditions was studied in (Rudolph et al. 2002). In the case of ethyl acetate, the presence of humidity improved the removal efficacy significantly. The positive effect of humidity was explained by the production of OH radicals which have higher rate constants for the reaction with ethyl acetate compared to O radicals [ $\sim 1.5 \times 10^{-12}$  and  $\sim 5 \times 10^{-15} \text{ cm}^3 \text{ molecule}^{-1} \text{ s}^{-1}$ , respectively (Picquet et al. 1998; Caravan et al. 1996)]. In fact, Rudolph et al. suggested that in dry air, the excited states of  $\text{N}_2$  play a more important role than O radicals in the decomposition of ethyl acetate. Butyl acetate has even

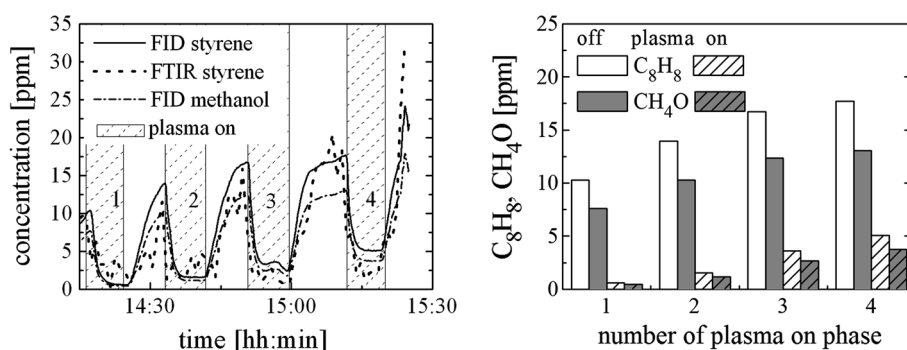
**Table 1** Energy constants  $k_E$  for butyl acetate decomposition

	Low inlet concentration (L/kJ)	High inlet concentration (L/kJ)
SIE = 110 J/L	7.14	9.85
SIE = 220 J/L	1.33	5.69

**Fig. 6** Left Infrared spectra of the untreated gas mixture (straight line) and the gas treated with plasma (SIE = 300 J/L, dashed line). Right total organic carbon content (straight line) and SIE (dashed line) with respect to experiment operation time



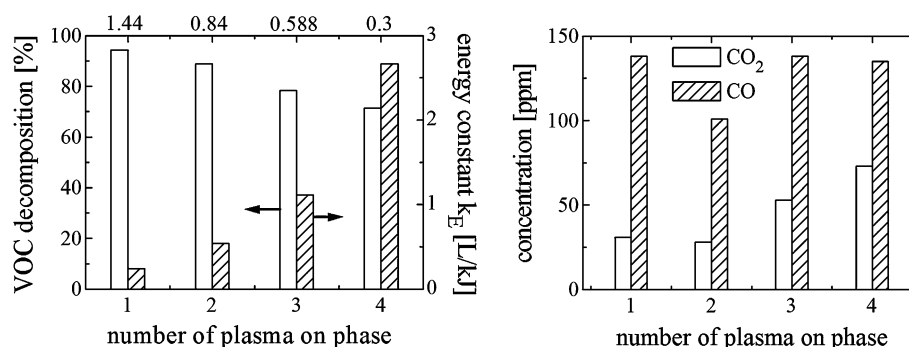




**Fig. 7** Left Concentrations of hydrocarbons measured with FID and FTIR with respect to experiment operation time; the numbers 1–4 indicate the numbers of plasma on phases. Right concentrations of

styrene (white) and methanol (gray) during plasma off phase (hollow) and plasma on phase (patterned) with respect to the number of plasma on phase

**Fig. 8** Left Decomposition of VOCs (hollow) and energy constant  $k_E$  (patterned) with respect to the number of plasma on phase (bottom) and the applied SIE in terms of kJ/L (top). Right concentrations of the reaction products  $\text{CO}_2$  and CO with respect to the number of plasma on phase



a higher rate constant for the reaction with OH [ $\sim 5 \times 10^{-12} \text{ cm}^3 \text{ molecule}^{-1} \text{ s}^{-1}$  (Picquet et al. 1998)] as compared to ethyl acetate. Thus, the presence of humidity may have a strong effect for butyl acetate decomposition.

As a conclusion, the easy-to-apply and easy-to-use system of a stacked DBD reactor driven by a sinusoidal high voltage showed ability to convert butyl acetate in realistic industrial conditions. Legislative guidelines on butyl acetate concentration can be reached by means of plasma treatment under certain conditions. Supporting the NTP-based removal process with a catalytic or scrubbing process could enhance the overall performance.

### Yacht hull production site

In the Polish yacht hull production site, four measurements at different inlet concentrations of the target pollutant styrene were performed, accompanied by a change of the

power energizing the plasma source and, thus, the SIE. This is displayed in the right part of Fig. 6. The dashed line shows the SIE applied to the plasma source as a function of the experiment operation time. The straight line displays the response of the FID instrument, i.e., the total organic carbon compounds. It is clearly to be seen that the total amount of hydrocarbons in the untreated process gas increases slightly with time but decreases significantly when operating the plasma.

In the left part of Fig. 6, samples of typical infrared absorption spectra are displayed. The straight line corresponds to the untreated process gas and the dashed line to the gas treated with plasma at  $\text{SIE} = 300 \text{ J/L}$ . These spectra reveal that along with the target pollutant styrene, another compound is found, namely methanol ( $\text{CH}_4\text{O}$ ). As products of the plasma process ozone are found as well as nitrous oxide (not present in this spectral range) and formic acid ( $\text{CH}_2\text{O}_2$ ). The spectra also show that the peaks related to styrene vanish at plasma on conditions. Moreover, the area of the methanol signal (superimposed with the ozone absorption peak) decreases when



the plasma is operated. The FID signal represents the sum of styrene and methanol, since the FID is not sensitive to the other carbon-containing compounds  $\text{CO}_2$ ,  $\text{CO}$ , and  $\text{CH}_2\text{O}_2$  (Kosch 2005). With the response factor 1 for styrene and 0.74 for methanol (Testa 2014), the corresponding concentrations were calculated. The comparison with the values obtained by calibrated FTIR spectroscopy showed good agreement (left part of Fig. 7). Here, the numbers 1–4 indicate the number of plasma on phases.

The analysis of the obtained concentration values shows an almost constant difference between plasma on and plasma off conditions of around 12 ppm for styrene and 9 ppm for methanol, resulting in 71–94 % decomposition of these VOCs (left part of Fig. 8, hollow bars, left scale).

The energy constant  $k_E$  was calculated according to Eq. (3) with “VOC” being styrene. As displayed by the patterned bars in the left part of Fig. 8 (right scale),  $k_E$  increases in the set of measurements because of decreasing applied power, whereas the inlet concentration increases. This suggests that even higher  $k_E$  values are achievable, for example, by increasing the flow rate and, thus, decreasing the SIE. In (Kosch 2005, 2009) a closed loop corona system was used, and  $k_E$  values of over 20 are reported (as  $\beta$ -value,  $\beta = k_E^{-1}$ ). The right part of Fig. 8 shows the amount of the reaction products  $\text{CO}_2$  and  $\text{CO}$  formed by plasma-based decomposition of styrene and methanol. Similarly to the experiments in Estonia, again a poor  $\text{CO}_2$  selectivity is found, saying that most of the pollutants are partly oxidized to  $\text{CO}$ . For methanol, this is already reported in (Hsiao et al. 1995), also stating that the  $\text{CO}_2$  selectivity improves with temperature increasing up to 300 °C. Besides this, the carbon balance found (difference of number of carbon atoms with and without plasma) is in all measurements positive, suggesting that there is an additional carbon-containing product which could not be identified. It is very likely that part of the VOCs or aerosols formed by plasma activity are adsorbed in the plasma source (e.g., on the surface of the dielectric plates). Adsorbed compounds may be desorbed and oxidized during plasma on phase. To clarify this, additional experiments with simultaneous gas-phase diagnostics before and after the plasma source are necessary. The concentration of formic acid, which is also detected in the infrared spectra, did not exceed 1 ppm and is not further discussed here. The ozone concentration ranged from 250 to 650 ppm. Further by-products as reported in (Zhang et al. 2011) could not be found. The obtained results show a readily attack of plasma species on styrene and other VOCs, but a combination of NTP technology with a catalyst for  $\text{CO}$ -

conversion is needed for efficient reduction in pollutants in the exhausted process gas.

## Conclusion

Non-thermal plasma treatment of industrial exhaust gases in real processes was performed. A stacked DBD NTP source in combination with different power supplies was installed for the direct treatment of exhaust gases in an oil shale processing plant in Estonia and in a yacht hull production site in Poland. Moreover, sensitive laboratory equipment, namely an FTIR spectrometer, an FID instrument, and digital storage oscilloscopes were successfully operated in the on-site experiments, i.e., under rough conditions. The devices worked properly and stable under such rough environmental conditions. It was shown that gaseous pollutants can be decomposed with NTP even under industrial conditions as expected from numerous laboratory studies found in literature. The concentration of the pollutant butyl acetate was reduced by up to 71 % and styrene, and methanol concentrations decreased by up to 94 %. The reaction products were mostly carbon monoxide and carbon dioxide as well as comparably small amounts of formic acid. As expected from the operation of an NTP in air, ozone and nitrous oxide are produced.

The utilization of catalysts together with NTP technology is widely discussed in the literature, and thus, it should be also investigated if a catalyst can be implemented in the investigated situations. In particular, the removal of  $\text{CO}$  by a platinum/alumina-catalyst after plasma treatment appears to be promising. The removal of formic acid can be utilized by scrubbing. The combination of different processes can improve the overall cleaning efficiency and applicability. Potential for improvement of the energy efficiency is given by the geometry and size of the plasma source which can be optimized with respect to the gas flow of the special industrial process. Further work is needed to clarify the reaction pathways of the decomposition of the VOCs treated in complex gas compositions of real exhaust gases.

One should also take in mind that plasma can be operated “on demand,” i.e., in situations when the VOC concentration is fluctuating and exceeding certain thresholds only in limited time windows. Cyclic processes, switching between absorption phase and plasma-assisted desorption/





regeneration phase, as suggested, e.g., in (Mizuno 2007) and (Hsiao et al. 1995, 2004), are promising concepts.

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