



How activated carbon improves the performance of non-thermal plasma removing methyl ethyl ketone from a gas stream

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ARTICLE INFO

Keywords:

Non-thermal plasma
Methyl ethyl ketone
Activated carbon
Humidity
Ozone

ABSTRACT

The combination of non-thermal plasma (NTP), operated at room temperature and at atmospheric pressure in air and in combination with activated carbon filters offers a more efficient removal of VOCs from gas streams than each individual method alone. Efficiencies, synergies and mechanisms of this combination were investigated by means of comprehensive quantitative Fourier transform infrared spectroscopy analysis. Therefore, dry and wet synthetic air containing about 90 ppm of methyl ethyl ketone (MEK) were treated with non-thermal plasma (NTP) and an intentionally undersized activated carbon (AC) filter, separately and in combination. As a result, removal of about 50 % was achieved for NTP or AC alone but a removal close to 95 % was found for the combination. Ozone, generated by the NTP, was reduced by 55 % with the AC-filter. For the operation of the NTP with humid air, a decomposition of the pollutant on AC was observed even after the plasma was switched off.

1. Introduction

Ongoing research in the field of exhaust gas cleaning is driven by the persistent need of clean and healthy air and environmental protection. Air pollution from industrial processes and solvent use belongs to the most important sources of environmentally harmful emissions (EEA, 2019). Volatile organic compounds (VOCs) are a significant part of these man-made emissions. Many VOCs are harmful to human health (USEPA, 2019). In addition, their remediation is of interest for their contribution to the formation of ozone and of secondary organic aerosols, as part of airborne particulate matter. Technologies to remove VOCs from gas streams that are already in use are, for example, filtration, scrubbing, condensation, thermal and catalytic oxidation, and adsorption on activated carbon (Wang et al., 2004). While all these methods are focused on removal, non-thermal plasma (NTP) technology, which is also already commercially available (Bäro, 2020), provides a means for the actual decomposition of compounds. In this approach highly reactive and oxidizing species, such as hydroxyl radicals, atomic oxygen and ozone, generated from air by electrical discharges play a major role (Kim, 2004) because these reactive species initiate or accelerate chemical reactions needed to decompose VOCs.

A common method for the generation of a non-thermal plasma is the dielectric barrier discharge (DBD). Generally, an oscillating high voltage is applied between two electrodes, instigating the formation of a plasma.

However, at least one of the electrodes is insulated from the other by a dielectric. Accordingly, only a capacitive current flow is possible and catastrophic short circuits are prohibited. Electrons can gain much higher kinetic energies than the remaining heavier ions due to their much lower mass, which can be described by different characteristic temperatures for both species. The plasma is considered non-thermal for the lack of an equilibrium of the temperatures. Comprehensive summaries and examples for generation of NTP and its use in air-pollution control were described by Kim (2004) and Brandenburg et al. (2014).

The technology is generally combined with activated carbon filters for the retention of ozone and residual pollutants. So far little attention has been paid to possible degradation mechanisms on the activated carbon, which could be promoted by reactive plasma species provided to and retained by the filter, or on the associated significance of the humidity of the processed air. However, this has been suggested by Schiavon et al. (2017) as well as by Paur (1992) as a method to increase the efficiencies of air pollution control systems or at least improve and extend the performance of activated carbon filters (Almarcha et al., 2014). Therefore, we conducted a dedicated investigation on a combination of NTP technology and active carbon adsorption as a tool for removal of VOCs from gas streams. In particular, we studied the removal of methyl ethyl ketone (MEK, C₄H₈O) as a meaningful model with the potential to draw conclusions also on other VOCs. Moreover, MEK is one of the most important industrial used solvents and its vapour is found in

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<https://doi.org/10.1016/j.clet.2021.100234>

Received 30 November 2020; Received in revised form 2 July 2021; Accepted 30 July 2021

Available online 2 August 2021

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exhaust gases from several processes. It is slightly affecting human health (USEPA, 2003) but decidedly unwanted because of its smell.

The treatment of VOCs in general by NTP-methods alone has been described in detail previously. Yamamoto e.g. investigated the treatment of trichloroethylene and alkyl acetates with different plasma sources (Yamamoto and Futamura, 1998). Schiorlin et al. removed toluene from air with different corona discharges (Schiorlin et al., 2009). Besides catalyst assisted methods for VOC-treatment, Vandenbroucke et al. reviewed various NTP-methods (Vandenbroucke et al., 2011) for that purpose. Matsumoto et al. summarized various electrical discharges like electron beam, pulsed discharges and dielectric barrier discharges for air pollution control (Matsumoto et al., 2012). Xiao et al. published a review of NTP-reactors for VOC-treatment (Xiao et al., 2014) but the article focusses on NTP-systems with catalysts. Karatum and Deshusses studied the removal of various VOCs including MEK in an NTP-reactor (Karatum and Deshusses, 2016) and Holzer et al. investigated the use of non-thermal plasma for cleaning kitchen exhaust air (Holzer et al., 2018). Chang and Chang reported an 80 % removal of MEK with an inlet concentration of 125 ppm and with a humidity of 1.6 % at 100 °C for a specific input energy, SIE, of 720 J/L (Chang and Chang, 1997). Karatum et al. describe a removal of 50 % for about 100 ppm of MEK from a dry gas stream at room temperature with an energy consumption of 320 J/L (Karatum and Deshusses, 2016). Our own previous experiments showed that the MEK-removal efficiency of an NTP-process can be improved by implementing an electric wind, which increases the mixture of plasma-generated reactive species with the bulk gas (Schmidt et al., 2018). Investing 670 J/L of electrical energy, we achieved a 95 % removal for about 100 ppm of MEK under dry conditions at room temperature. A decrease of the rather high energy needed for removal of MEK by NTP is another important motivation for the study presented here.

Activated carbon filters are a standard element for removal of VOCs from air streams as published in a handbook by Wang et al. (2004), studied for MEK and benzene by Huang et al. (2003) and reviewed for VOC-adsorption by Pui et al. (2018). A combination with NTP methods is evident and, hence, a topic of different investigations. Chen et al. showed that VOC-saturated activated carbon can successfully be regenerated by means of NTP (Chen et al., 2013). The oxidation of MEK with ozone on a surface of activated carbon due to the Langmuir-Hinshelwood mechanism was discussed by Wu and You (2007). They observed a significant increase in breakthrough time of activated carbon filters in the presence of ozone. Thus, the combination of NTP and AC can be taken as reasonable regarding improving the efficiency of the NTP-process as well as the performance of the AC. Therefore, we performed experiments with this technique in our process in order to investigate its effects on the removal of MEK with NTP.

In the following, the experimental setup and procedure is described. Then, the MEK-removal under dry and wet conditions is discussed. Subsequently, the influence of the serial connected activated carbon filter is investigated. Finally, the obtained results are summarized.

2. Experimental setup and procedure

The experimental setup used in all measurements is depicted in Fig. 1. NTP was generated by dielectric barrier discharges (DBD) (Wagner et al., 2003). A DBD was chosen because of its good applicability and scalability. It was realized with a stack-type plasma reactor (Müller and Zahn, 2009) combined with an activated carbon filter (AC, 1 g ± 0.05 g of granular activated carbon AFA 4 sized 4 mm). The reactor consisted of a housing with four electrodes (stainless steel grids sized 140 × 82 mm²), separated by dielectric plates and spacers made of phlogopite (thickness 0.5 mm). The electrodes were alternately connected to high voltage and ground. High voltage was provided by a programmable amplifier (Chroma 61604) and a custom-made high voltage transformer (Bremer Transformatoren GmbH, ratio ≈ 1:100). Electrical data were recorded with a digital oscilloscope (Tektronix DPO 4104B) via a high voltage probe (Tektronix P6015A) and a 10:1 voltage divider (Tektronix P6139B) to measure the voltage drop across a measuring capacitor (C_{Meas} = 501 nF). By multiplying this voltage drop with the capacitance of the capacitor the charge transferred into the plasma was calculated. Plotting the charge over the applied high voltage

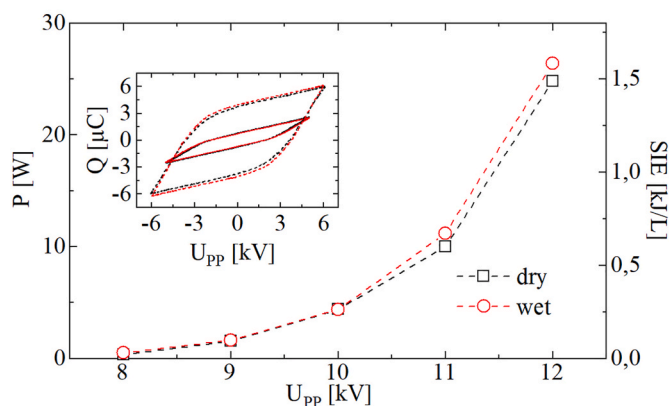


Fig. 2. Power dissipated into the plasma with respect to applied voltage; inset: charge-voltage-plot for 10 kV_{pp} and 12 kV_{pp} under dry and wet (16 % relative humidity) conditions.

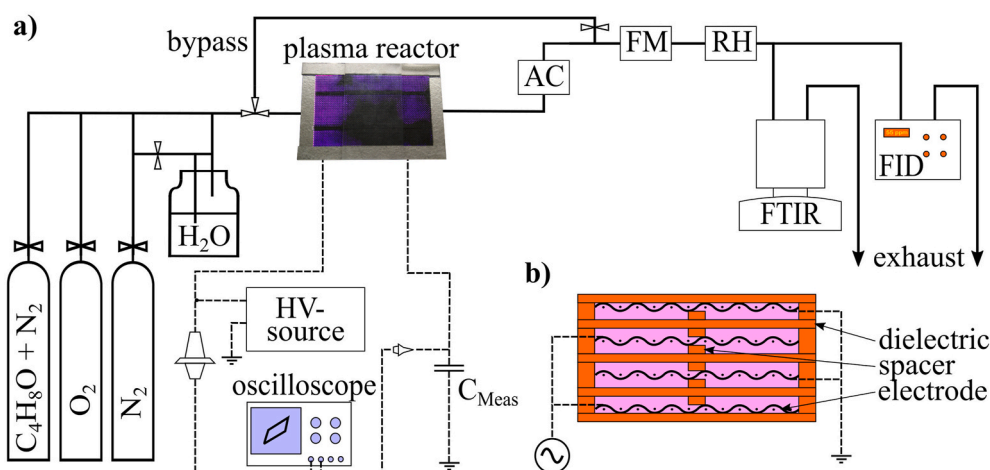


Fig. 1. a) Experimental setup, b) Schematic of the plasma reactor.

gives a Q-V-plot (Manley, 1943), exemplarily shown in the inset of Fig. 2. The area of the parallelogram presents the energy dissipated per high voltage cycle into the plasma. Thus, multiplication with the frequency of the applied voltage results in the respective power dissipated into the plasma. The high voltage was applied at a frequency of 400 Hz and peak-to-peak values of 8 to 12 kV_{pp}. The corresponding power values are presented in Fig. 2.

The process gas was synthetic air with an admixture of about 90 ppm of MEK. The gas mixture was realized with pressurized gases (N₂ 5.0, O₂ 4.8, and 206 ppm ±10 ppm MEK in N₂, Air Liquide) connected via pressure regulators and computer controlled mass flow controllers to the gas supply system. Synthetic air instead of only nitrogen was necessary because oxygen is required to generate the reactive species that are needed to oxidize the targeted VOC methyl ethyl ketone. Moreover, the use of synthetic air as a process gas is of far more practical relevance than the use of pure nitrogen. For the experiments with humidified process gas, which are named wet conditions throughout the text, a part of the nitrogen was passed through a water containing bubbler. With the experimental setup a relative humidity of ≈ 16 % could be realized. Gas flow, humidity, and temperature were monitored with a flow meter (FM, Omega FMA 6610) and a sensor (Rotronic HP 22-A). Although the gas residence time in the active plasma zone, determined by the reactor geometry and the gas flow, influences the removal process significantly (Fridman, 2008), the gas flow was set constant to 60 L/h for all experiments in order to keep the number of operating variables as small as possible. The gas analysis was performed by a Fourier transform infrared spectrometer (FTIR, Bruker Alpha, T_{cell} = 40 °C, cell volume 0.5 L, optical pathlength 5 m, spectral resolution 1 cm⁻¹) and a flame ionization detector (FID, TESTA 2010T). The FID was mainly used to monitor the inlet concentration of MEK, whereas the FTIR-spectrometer served for the detailed observation of the products, e.g. carbon oxides or ozone. Therefore, the recorded infrared spectra were analyzed by using calibration spectra, recorded using certified calibration gases (obtained from Air Liquide). The gas mixture used to record the ozone calibration spectra was generated with ozonized pure oxygen (Air Liquide Alphagaz O₂ 4.8). The respective ozone concentration was measured with a commercial ozone gas analyzer using UV-absorption at 254 nm (Anseros Ozone Analyzer MP). The activated carbon filter element was situated downstream the plasma reactor. For a workable time range of the experimental procedure, the filter element was intentionally undersized, as by Basner et al. (2013). To set and control the process gas parameters without the influence of the plasma source with the activated carbon filter, a bypass was used, guiding the process gas directly from the gas supply system to the gas-diagnostics system. All experiments were performed at room temperature and atmospheric pressure.

For the experiments, the process gas flowed through the bypass until concentrations were stable. Then, the gas was guided through the experimental apparatus, thus, either through the plasma reactor, or through the plasma reactor with downstream activated carbon filter. After the concentrations reached stable values again, the plasma was switched on and off, each for 5 min. This procedure was repeated until reproducible results were obtained.

3. Results and discussion

3.1. Removal of MEK with NTP only

The performance of the plasma-process was first investigated without the activated carbon filter element but with respect to the humidity of the feed gas. Accordingly, pertinent operating parameters were determined and a baseline established for the subsequent combination with activated carbon. The operating voltage was varied from 8 kV_{pp} (ignition voltage) to 12 kV_{pp} (upper limit for chosen operation mode). Derivations of removal efficiencies are based on the quantification of MEK-concentrations from FTIR-spectra. The corresponding power, dissipated in the discharge, and the associated specific input

energy, SIE, are shown in Fig. 2. Power increased with increasing voltage, mainly due to the progressively larger area of the DBD-electrode that was covered by a plasma until eventually the entire electrode was enclosed (Schmidt et al., 2015). Although, the specific input energy is the more relevant parameter for an evaluation, applied voltage is generally the more directly accessible parameter and system control variable. The specific input energy, SIE, was, hence, calculated according to Equation 1

$$SIE \left[\frac{J}{L} \right] = \frac{P[W] \cdot 3600 \text{ s}}{\Phi \left[\frac{L}{h} \right]} \quad (1)$$

where P denotes the electric power dissipated into the plasma and Φ the gas flow. The specific input energy, SIE, offers a direct comparison for the efficiencies of different plasma sources for a degradation process, whereas efficacies have to be described separately. The energy yield, EY, in comparison, describes the energy needed for the decomposition of a specific compound, such as MEK, according to Equation (2).

$$EY \left[\frac{g}{kWh} \right] = \frac{\Delta c [ppm] \cdot 10^{-6} \cdot \Phi \left[\frac{L}{h} \right] \cdot M \left[\frac{g}{mol} \right]}{V_{mol} \left[\frac{L}{mol} \right] \cdot P [kW]} \quad (2)$$

Here, Δc describes the reduction in concentration of MEK. The parameters M and V_{mol} introduce the molar mass of MEK (72.11 $\frac{g}{mol}$) and the molar volume of an ideal gas (24.5 $\frac{L}{mol}$ at 25 °C) into the calculation, respectively.

The results for the degradation of MEK, provided in either dry or wet air, and depending on different operating voltages are shown in Fig. 3. The data are displayed together with the achieved energy yields. It is known from atmospheric chemistry that photodissociation and oxidation by hydroxyl radicals are of prime importance for the decomposition of VOCs (Atkinson, 2000). In non-thermal plasma applications, accelerated electrons could decompose VOC-molecules by electron impact dissociation but their energy is not sufficient to play a significant role for the investigated system (Chung et al., 2019). Instead, these electrons dissociate bulk gas molecules, such as oxygen, nitrogen, or water, to generate radicals. Thus, for the operation with humid air hydroxyl radicals are provided by the NTP (Rudolph et al., 2003). The much higher rate constant for reactions with hydroxyl radicals, as shown by equation (5), explains the associated better performance for MEK-removal (NIST, 2015). Reactions with ozone and atomic oxygen also play a role for the decomposition of MEK in dry air, according to equations (3) and (4).

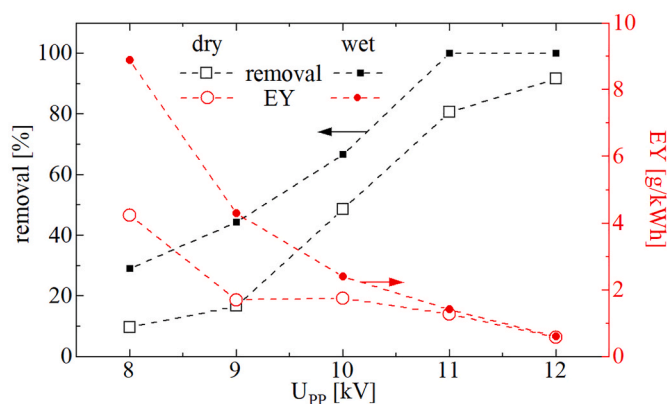
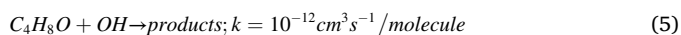
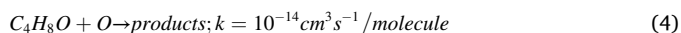
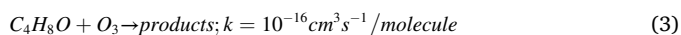


Fig. 3. Removal (black symbols) and energy yield (red symbols) of MEK under dry (empty symbols) and wet (full symbols) conditions. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)



Although the ozone concentrations are lower under wet conditions, the production of hydroxyl radicals apparently more than compensated for the reduced generation of ozone and associated diminished generation of nitrous oxide, which is shown in Fig. 4 and well-known for operation of DBDs with humid air.

Lower ozone concentrations, found under wet conditions, were due to a lower ozone production caused by the competing reactions, like OH- and HO₂-production as published by Kogelschatz (2003) and further investigated by Zhang et al. (2016). Along with the increased removal of MEK and a change in production of ozone and nitrous oxide, an increased oxidation to CO₂ was observed (Fig. 5).

The data clearly show that the oxidation to CO₂ is much more efficient under wet conditions. Calculating the carbon balance, according to Equation (6), suggests that the removed MEK was not completely mineralized to CO₂ and CO.

$$C_{balance} = \frac{4 \cdot MEK_{removed} [ppm]}{CO_2 [ppm] + CO [ppm]} \quad (6)$$

This suggests at least one intermediate oxidation state of MEK. Thus, the increasing concentration of CO₂ at 11 kV_{pp} and 12 kV_{pp}, where MEK already completely vanished from the gas stream, can be attributed to the oxidation of this intermediate compound. For an operation at 11 kV_{pp}, corresponding to a value for SIE of 600 J/L, most of the MEK was decomposed (c.f. Fig. 3) but the concentrations of the byproducts N₂O and O₃, needed for oxidation of MEK but not wanted in the exhaust, were also higher (c.f. Fig. 4). The emission of byproducts can be reduced for an operating voltage of 10 kV_{pp} or less, which is still economically interesting for the associated considerably lower required energy consumption (SIE = 264 J/L, c.f. Fig. 2). The reduced performance of the NTP could be compensated by a combination with an activated carbon filter, which motivated the studies that are described in the subsequent section.

3.2. Removal of MEK with NTP and activated carbon

Consequently, following the suggestion from the results for a most promising combination of NTP with AC with respect to an efficient combination of both methods, the plasma was operated at 10 kV_{pp}, corresponding to an SIE of 264 J/L, and effluents subsequently treated by an intentionally undersized activated carbon filter. Its size was chosen based on the experiences of Basner et al. (2013).

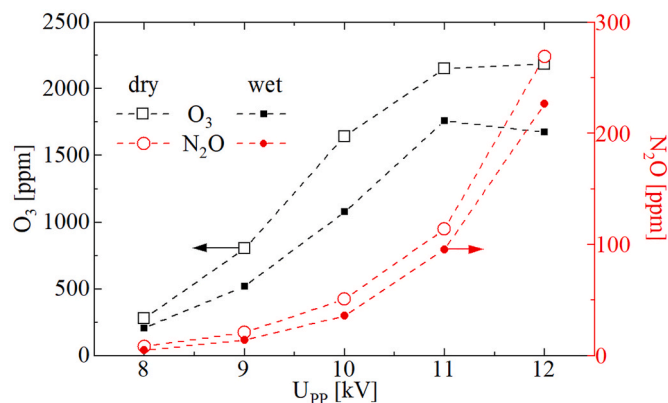


Fig. 4. Concentrations of O₃ and N₂O with respect to the applied voltage under dry (empty symbols) and wet (full symbols) conditions.

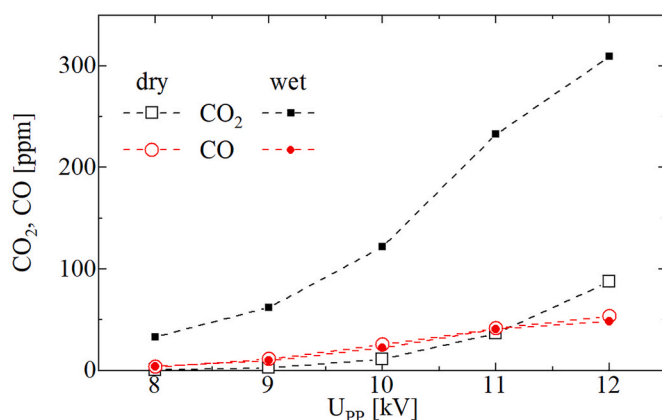


Fig. 5. Concentrations of CO₂ and CO with respect to the applied voltage under dry (empty symbols) and wet (full symbols) conditions.

The results of the combination are presented in Fig. 6. (energy yields, EY, only apply to the removal by NTP). The shaded bars show the removal of MEK for an operation with either dry or humid air and the overlaid white bars describe the respective energy yields. Under dry conditions, the activated carbon alone provided a removal efficiency that is similar to the NTP-results. The individual removal efficacies add up to an almost complete elimination of MEK especially for humid air. Conversely, no significant synergisms were observed. Overall, the investigations established the possibility to significantly reduce operational expenses for the remediation of VOCs in air streams by NTPs for a combination with a rather marginal filtration by activated carbon.

Another advantage of an AC-filter is the removal of process by-products from the gas stream. This applies in particular to the removal of ozone and nitrous oxide from the plasma as shown in Fig. 7. The elimination of ozone is much better (35 % and 48 % for dry and wet air, respectively) than for nitrous oxide (10 % and 11 % for dry and wet air, respectively). This is explained by the adsorption capacity for nitrous oxide, which is the smallest of the investigated species. The molecular weight and especially the polarizability of N₂O are smaller than those of O₃ and MEK (Lide, 1999) and the adsorption capacity of active carbon increases with these respective characteristics (Sorrels, 2018).

Removal efficiencies were generally higher under wet than under dry conditions. This applies in particular also to a filtration by activated carbon only. The adsorption capacity of activated carbon for VOCs was rather independent from water concentration in air until a relative humidity of 50 % was approached (Cal, 1996). At higher values, the activated carbon adsorbed less VOCs. However, our results suggest a dependency on humidity for the removal of MEK already for a change from dry air to a relative humidity of only 16 %. Since the plasma was

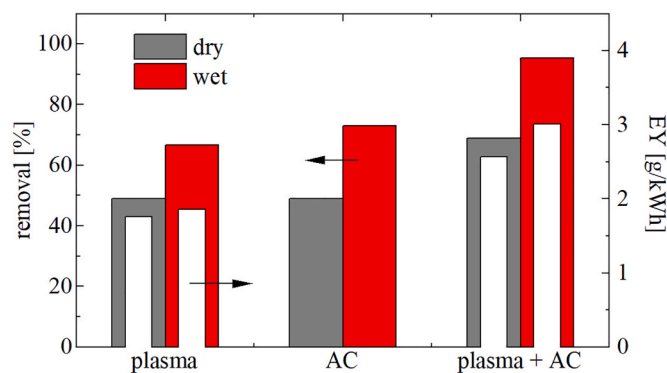


Fig. 6. Removal (coloured bars) and energy yield (white bars) of MEK with plasma alone, activated carbon and plasma with activated carbon under dry and wet conditions.

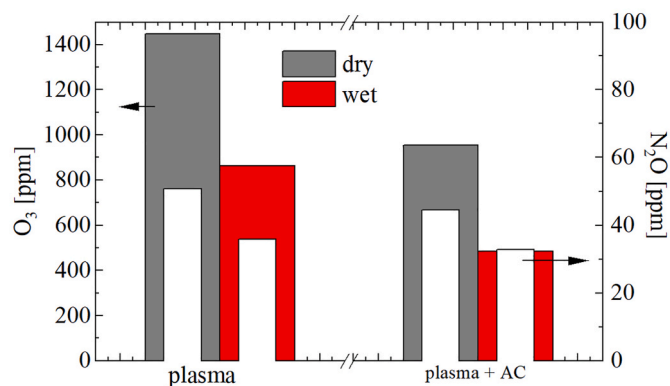


Fig. 7. Concentrations of O_3 (coloured bars, left scale) and N_2O (white bars, right scale) under dry and wet conditions with and without activated carbon.

actually switched on and off during the experiments to compare results with and without NTP operation, reactive species that had been provided to the AC might still be present in the AC and encourage chemical reactions on the surface of the granules even some minutes after the plasma was turned off. Since the effect was not observed for an operation with dry air, probably hydrogen species, e.g. hydrogen peroxide, or reactions instigated by hydroxyl radicals are involved. The assumption of reactive species lingering in the AC-filter was further supported by the fact that even 5 min after the plasma was turned off, the effluent from the AC-filter still contained more CO_2 than measured under wet conditions without plasma and also than measured under dry conditions with plasma (data not shown). Conversely, the ozone adsorption capacity of activated carbon does not depend on relative humidity, which was demonstrated at least for ppb-levels of ozone concentrations (Lee and Davidson, 1999). Notably, the utilization of active carbon raises several questions on the AC itself. Adsorption efficiencies depend on a broad variety of parameters like the type of carbon (von Kienle et al., 1994) or internal surface area, porosity or pore volume (Pui et al., 2018). Details on the influence remains subject to further investigations.

The activated carbon also reduces ozone better for humid air (55 % compared to 42 % for dry air) because ozone is chemisorbed. Consequently, reactions associated with water, as described by Equations (7)–(9), could enhance the reduction of ozone (Lee and Davidson, 1999).



The radicals that are released by these surface processes may also be the reactants required for the increased MEK removal efficiency that was found for wet conditions when the plasma was turned off. However, despite the evidence for a plasma species enhanced activation of carbon depending on humidity, further studies need to be conducted for a more thorough and unambiguous evaluation of the underlying mechanism.

The removal of the VOC methyl ethyl ketone (MEK) from a gas stream with non-thermal plasma (NTP) alone and in combination with an activated carbon (AC) filter was investigated for dry and humid air. MEK can be removed with NTP with increasing efficiency by increasing the energy input into the plasma. However, a removal efficiency of 95 % was also achieved by including an AC-filter downstream the plasma-module and humidifying the intake air for much lower power input to the plasma. The main objective of the presented research was the degradation of MEK by the plasma itself, especially under economic considerations, such as power input. Therefore, no direct combination of NTP and AC e.g. by placing the AC in the active plasma zone, was investigated. It was found that the subsequent treatment of the exhaust from even a “weak” plasma by active carbon improves the overall

efficiency significantly by adsorbing residual MEK, ozone and other gaseous species. The results also indicate a lasting beneficial effect of humidity on the MEK-removal even after the plasma was switched off. Accordingly, already an intermittent operation of an NTP-reactor can improve the overall efficiency of an activated carbon filter significantly, with respect to the removal of MEK and the concentration of the unwanted by-product ozone, which, although necessary for the oxidation of MEK, is not wanted in the exhaust air.

In order to evaluate the improved performance of the combined system compared to the parts operated separately, the assets and drawbacks are discussed as follows. The main advantages of activated carbon are a high adsorption capacity and a low price. Unfortunately, loaded activated carbon has to be replaced and disposed as hazardous waste with the respective costs. The combination with an NTP offers a way to extend the intervals for an exchange of the AC-unit. The main advantages of non-thermal plasma methods are controllability (by operating parameters) together with good scalability. A caveat of the technology are expenses for electrical energy and the generation of some unwanted by-products. The contribution and advantage of adding an active carbon filter is the possibility that even for relaxed requirements on the plasma-stage, i.e. electrical power and size, MEK-concentrations, as they are relevant in practical applications, could be removed sufficiently – although not entirely. Residual concentrations could be prevented by increasing the power to the plasma or a larger active carbon filter. According to our findings, the plasma process converts a considerable part of the MEK, the active carbon filter adsorbs another certain part of the MEK. The combination of the technologies shows an additive removal of MEK from the gas stream, which correlates with the removal rates of the separate technologies. This shows that the active carbon filter not only removes the reaction products of the plasma process but also the target compound. The presented combination of NTP- and AC-technology merges the advantages of the separate technologies to alleviate their individual disadvantages and, thus, provides a strategy for a cost-efficient, easy-to-use and stable process to treat contaminated gas streams.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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