

Use of a low-temperature plasma reactor to reduce volatile organic compounds

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Summary

Volatile organic compounds (VOCs), which include a wide range of toxic compounds, pose a significant threat to the environment and public health. Reducing their emissions is therefore an important task for the industries in which they are produced. Given the need to process waste generated by non-destructive technologies, destructive technologies are coming to the fore, especially those that are not low-energy. These include catalytic decomposition and low-temperature plasma (non-thermal plasma – NTP) technology. However, reactors used for the reduction of VOCs must be resistant to corrosion caused by the passage of gaseous substances, they should be made of affordable materials, and they should not negatively affect the efficiency of VOC decomposition.

One solution is to use micro-arc oxidation (MAO) technology to treat the surface of reactor components. The purpose of the experiments carried out on a laboratory NTP reactor with a replaceable cathode was to compare the efficiency of acetone and toluene reduction using a reactor cathode made of structural carbon steel, aluminium, and aluminium with a MAO surface treatment. During the process, the efficiency of the reduction of selected VOCs and process parameters, including the energy intensity of the process, were monitored.

The initial results of the experiments showed that the use of an aluminium cathode with a modified surface has an efficiency comparable to that of the other cathode materials used and, in the case of toluene reduction, it also exhibits lower energy consumption. The use of MAO technology to modify the surface of the NTP reactor cathode appears to be a suitable alternative to the use of more expensive and more corrosion-resistant construction materials (e.g. Ti and Ta).

Keywords: volatile organic compounds, non-thermal plasma, micro-arc oxidation

Introduction

With the gradual reduction of particulate matter, SO_x, and NO_x pollution, the reduction of volatile organic compound (VOCs) emissions from various industries is becoming a focus of attention among experts. Due to their higher saturated vapor pressure, volatile organic compounds have a boiling point between 50 and 260 °C. Volatile organic compounds include several types of organic compounds, mainly aromatic hydrocarbons, alcohols, aldehydes, esters, and organic acids. They are produced by biological processes in nature¹, but the main anthropogenic sources of VOCs are industrial processes, primarily in the petrochemical and chemical industries, coke production, paint manufacturing, and transportation etc.². Most VOCs react easily with NO_x to form ozone^{3,4}. VOCs as precursors of secondary organic aerosol can create significant component of fine particular matter⁵. Secondary organic aerosol

plays a role in the atmospheric particle load, influencing both air quality and climate dynamics⁶. In addition to the negative impact on the environment, many VOCs are carcinogenic, affecting the central nervous system, causing respiratory diseases etc.⁷⁻⁹. For these reasons, it is necessary to reduce their concentration in the air.

The best way to reduce VOCs emissions is to prevent them from being generated in the first place. There are two basic methods to reduce VOCs emissions: (a) non-destructive or (b) destructive techniques. Non-destructive techniques include adsorption^{10,11}, absorption¹², membrane separation¹³, condensation¹⁴. Effective for removing high-concentration VOCs, with activated carbon and zeolites being commonly used.

These technologies are generally inexpensive, widely used, but on the other hand they have a number of limitations. For example, for the adsorption the saturated adsorbent needs to be regenerated. However, the high costs and challenges in adsorbent regeneration limit its practical use^{15,16}. Landfilling used adsorbent is not a solution either. Membrane separation its eminent cost as well as maintenance requirements limit its widespread application¹⁷. The condensation method is particularly suitable and effective for removing evaporating solvents².

Destructive technologies convert organic substances in waste gas into non-harmful substances (CO₂ and water) through chemical reactions. These technologies include biopurification^{18,19}, thermal oxidation²⁰ and advanced oxidation technologies (such as ozonation¹², deep oxidation²¹, photodegradation and catalytic degradation², microwave-assisted catalysis technology²²). Thermal oxidation converts VOCs into CO₂ and H₂O at high temperatures (>1000 °C) and is effective for treating high VOCs concentrations. However, its high energy consumption and the undesirable byproducts formation limit its use^{23,24}. Photocatalytic oxidation operates at lower temperatures, making it a more energy-efficient option and producing fewer harmful by-products. It is particularly suitable for treating diluted VOCs streams (<1% VOCs). Its large-scale use is hampered by the long oxidation time^{23,24}. Alternatively, non-thermal plasma (NTP) and NTP combined with catalysis is widely deemed to have the following merits: (1) Its energy efficiency is higher than that of thermal oxidation. (2) It operates at atmospheric pressure and room temperature. (3) It can be easily integrated with various packing materials. (4) It can be quickly switched on/off²⁵⁻²⁷.

NTP is a process involving low-temperature plasma formation, in which the energy of electrons reaches 1 to 20 eV. At lower temperatures (close to room temperature), heavy particles such as ions, excited atoms and molecules, and free radicals are formed. Substances in the plasma state usually have the following physicochemical properties: 1) high temperature and high kinetic energy; 2) conductivity similar to that of metals; 3) luminescent properties; and 4) chemical activity. The design of the NTP reactor, known as in-plasma (IPC), includes a catalyst inside that can increase the production of active substances during the discharge process and thus improve the efficiency of the VOCs reduction process. A simpler NTP reactor does not contain any filling, and a controlled plasma discharge and ozone are generated inside.

The equipment in which NTP is implemented is constructed from metallic materials and must be corrosion resistant. It is often made of steel, titanium, or aluminum. However, even these materials have a limited-service life in the aggressive environment of waste gases. Therefore, it seems appropriate to apply a surface treatment that would improve the corrosion resistance of the materials. The solution to this problem may be the use of micro arc oxidation (MAO) technology. The essence of MAO technology lies in the formation of a very thin, porous oxide layer with variable density as a result of the creation of arc discharges in a liquid electrolyte. The resulting oxide layer is robust and its properties (e.g., thickness, porosity, corrosion resistance, etc.) can be adapted to specific application requirements thanks to the adjustable parameters of the technology. This layer then significantly improves the basic properties of the metal or alloy surface by increasing the material's corrosion resistance.^{28,29} In addition, the MAO method is relatively inexpensive and environmentally friendly³⁰.

The essence of the contribution is to verify the effectiveness of reducing selected volatile organic compounds in a laboratory reactor with a cathode made of the perforated aluminum sheet, on which a layer of oxide was created using MAO technology, and to compare the results with the effectiveness of using the perforated aluminum or the carbon steel cathode.

Experiments

Materials, methods, and equipment used

The design of the NTP laboratory test reactor allows for the replacement of cathodes in the form of perforated metal sheets. The laboratory NTP reactor produces ozone as an oxidizing agent, which, together with plasma discharge, decomposes VOCs. In addition to the electrode material, the entire process is influenced by other parameters such as the electrical voltage used, power consumption (generally source parameters), temperature, humidity, and gas flow rate. To verify the influence of the NTP reactor cathode material, or its surface treatment, on the efficiency of VOCs decomposition, cathodes in the form of perforated sheets made of classic carbon steel, pure aluminium, and aluminium with MAO surface treatment were used.

The preparation of ceramic coatings on the surface of perforated sheets was carried out using the MAO method on a semi-industrial unit with a 65 kW alternating current source (DEHOR-elspec. Litvínov s.r.o., Czech Republic). This equipment allows the pulse parameters to be set independently using appropriate electronic amplifiers, which provides great flexibility for preparing a specific coating microstructure. The entire process takes place in a liquid electrolyte environment, the composition of which also influences the chemical composition and properties of the resulting surface. Of the selectable parameters of the technology (voltage, current, frequency, and duty cycle), the pulse duty cycle is the dominant factor for regulating the characteristics of surface discharge. The entire surface treatment process involves several steps:

- No. 1 Degreasing process (1M NaOH; 45 °C)
- No. 2, 3 Rinsing (distilled water; conductivity < 10 μ S/cm)
- No. 4 Pickling (HNO_3 + HF)
- No. 5, 6 Rinsing (distilled water; conductivity < 10 μ S/cm)
- No. 7 MAO process (electrolyte; pH \geq 12)
- No. 8, 9 Rinsing (distilled water; conductivity < 10 μ S/cm).

The surface of the perforated aluminium sheet was treated using a constant voltage of 450 V for 25 minutes. In the MAO process, the aluminium sheet was connected as the anode and a 2 mm thick stainless steel sheet (1.4301) as the cathode. The experiments were carried out in an alkaline electrolyte of 6 g/l NaOH, 12 g/l Na_2SiO_3 at a source frequency of 96 Hz. Higher electrolyte concentrations were also tested, which, due to their high ionic conductivity, exhibited intense discharge with high heat generation. For these reasons, the process parameters were selected with regard to the lower concentration, or rather the ionic conductivity of the electrolyte (28 mS/cm).

The laboratory equipment is a set consisting of a VOCs source, from which air is extracted by a fan through a plasma reactor and discharged into the atmosphere. The inlet pipe is equipped with an air temperature and humidity sensor, a flow sensor, and the VOCs sensor for determining the concentration of VOCs substances. The outlet pipe is equipped with temperature and humidity measurement, ozone measurement, and a VOCs sensor. The plasma reactor itself is constructed as a "grid" of cathodes arranged perpendicular to the air flow, made of metal rods in glass tubes, and as a counterpoint, one or two symmetrical cathodes made of perforated sheet metal are inserted into one of the grooves in the wall of the reactor frame. The reactor is connected to a high-voltage source, which is connected to an oscilloscope and a wattmeter; the cathode sheets are grounded. The voltage of 17.5 kV was set for all experiments. The Figure 1 shows laboratory reactor and aluminium lattice with MAO treatment and its detail.

Images of surfaces and cross-sections of samples were measured using a JEOL JSM-7610F Plus scanning electron microscope (JEOL, Japan) in secondary electron (SE) and backscattered electron (BSE) modes. The chemical composition of the MAO coatings was determined using an energy-dispersive X-ray spectrometer (EDS, ULTIM MAX 65 mm², Oxford Instruments, England) connected to the SEM.

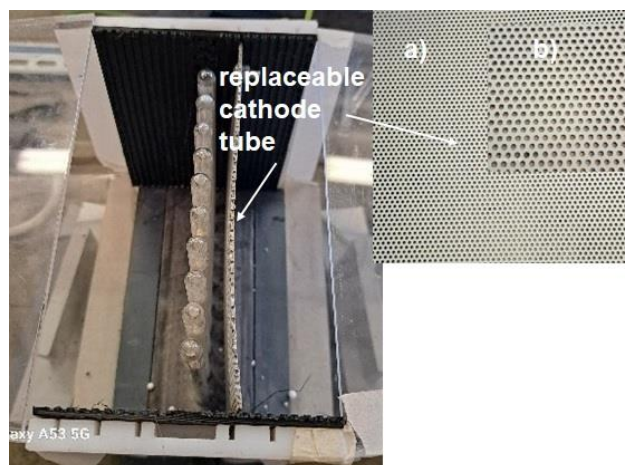


Figure 1: Reactor assembly with one cathode and aluminium perforated sheet with MAO treatment (a) and its detail (b).

The phase structure of the samples was measured by powder X-ray diffraction (XRPD) using a Rigaku Ultima IV diffractometer (Rigaku Corporation, Japan). X-ray diffraction patterns were obtained under conditions using $\text{CuK}\alpha$ radiation at an accelerating voltage of 40 kV, a current of 40 mA, a scanning speed/duration of $1^\circ/\text{min}$, a step width of 0.02° , and a scanning range of $10 - 100^\circ$. The phase composition was evaluated using the ICDD PDF-2 2022 database.

Description of the experiment on the reduction of selected VOCs

The aim of the experiment was to compare the effect of the cathode construction material used on the reduction efficiency. For this reason, the same optional parameters were maintained during the experiments. Acetone (boiling point is 56.29°C) and toluene (boiling point is 110.626°C) were selected for primary VOCs reduction experiments in the NTP laboratory reactor. The VOCs source was filled with acetone or toluene, and the temperature was adjusted to cause the substance to evaporate, which was then drawn in by a stream of air sucked in by a fan located at the end of the device. The stream passes through a plasma reactor, which excites a plasma glow discharge in the gap between the cathodes. During the experiment, a constant flow of air ($31 \pm 2 \text{ m}^3/\text{hour}$) containing $1030 \pm 150 \text{ mg/m}^3$ of acetone or $660 \pm 15 \text{ mg/m}^3$ of toluene entered the plasma reactor. The air then leaves the fan into the atmosphere. By measuring the temperature, the concentration of VOCs at the inlet and outlet, and the amount of O_3 at the outlet at a constant air flow, it is possible to evaluate the efficiency as the ratio of the difference between the inlet and outlet values relative to the inlet value in %. VOCs measurements are performed using a PhoCheck Tiger detector, Ion Science Phocheck Tiger. From the measurements of the source parameters of the plasma reactor, the power consumption of the source can be calculated and converted to the amount of reduced VOCs substances. At the same time, the oscilloscope allows the recording of so-called Lissajous figures, which characterize the behaviour of the plasma source.

Results and discussion

The X-ray diffraction analyse was proven that the ceramic layer prepared MAO technologies and the layer of corundum was covered the aluminium cathode as shows Figure 2. Figure 3 shows the SEM imagine of the ceramic layer prepared using the MAO technique, including a representation of the thickness of the oxide layer. The ceramic surface is porous, which increases the surface area of the cathode and can positively affect the reduction efficiency. The layer thickness is approximately 660 nm (Figure 3b).

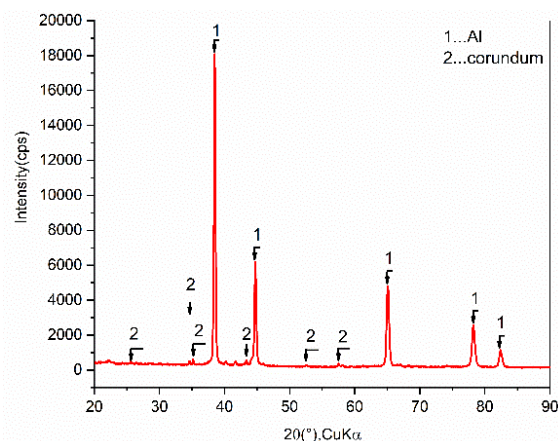


Figure 2: Diffraction pattern of the aluminium cathode surface after MAO treatment.

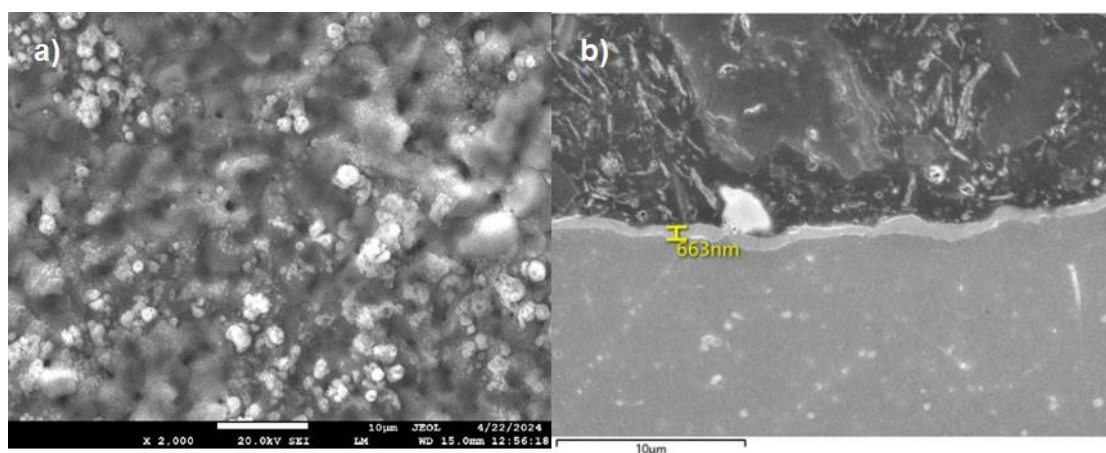


Figure 3: SEM image of aluminium cathode surface (a) and the thickness of the layer (b) created by MAO on the aluminium cathode. Magnification 2000x.

A comparison of the average reduction efficiency of three experiments with selected of selected VOCs using an NTP laboratory reactor with different cathodes is shown in Figure 4. The comparison shows that the aluminium cathode with MAO surface treatment does not reduce the decomposition efficiency; in the contrary, in the case of acetone reduction, the presence of corundum on the cathode surface has a slightly positive effect on the reduction efficiency. The reduction efficiency of selected VOCs reached a maximum of 70% which agree with available studies that show efficiency ranges 70 – 85% without the presence of a catalyst, while at higher power and with an optimal catalyst composition, efficiency of up to 90 – 96% can be achieved³¹⁻³³. The effectiveness of NTP in the decomposition of acetone or toluene depends on the process conditions (type of plasma, presence of a catalyst, gas composition, power, speed of flow gas)³². However, the use of catalytic effects in NTP operating reactors increases the economic parameters of the process. The efficiency of the reactor used in this work can be increased by optimizing parameters of the process, which will be the next step before the construction of a semi-industrial equipment.

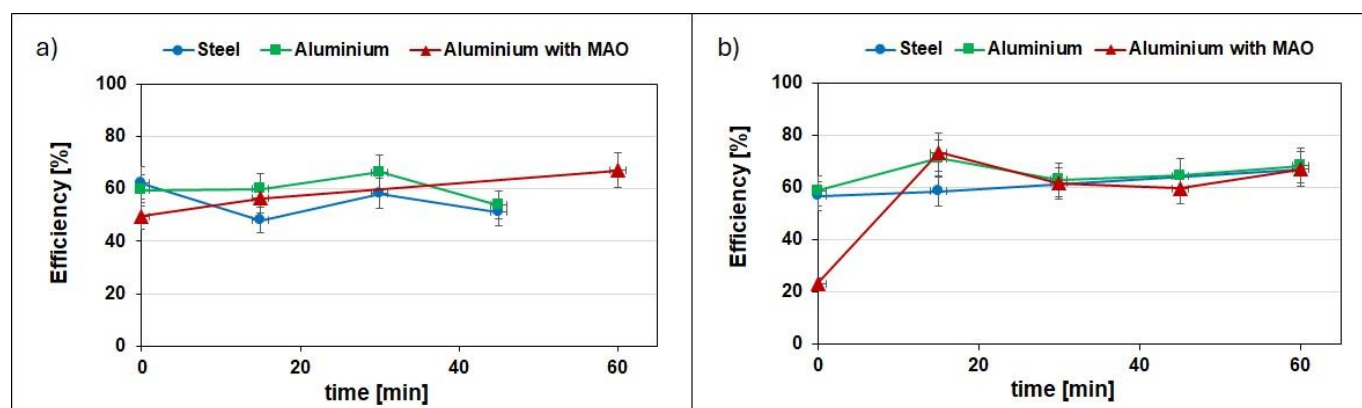


Figure 4: Reduction efficiency of acetone (a) and toluene (b) using laboratory reactor of NTP with different cathode.

During all experiments, the volt-ampere characteristics of the plasma were measured at a constant voltage (17.5 kV), and the plasma power was calculated. The energy consumed to reduce the concentration of a given substance was calculated from the difference in acetone/toluene concentrations in the unit air flow in front of and behind the reactor and the plasma power, and the results are shown in Figure 5. The difference between the energy consumption of acetone and toluene decomposition stems from the nature of the chemical compound. Reducing the acetone concentration in the air stream is most energy-intensive when using a carbon steel cathode, while the process using the aluminium cathode and the MAO-coated aluminium cathode is comparable. Conversely, the reduction in toluene concentration is comparable for all electrodes used, taking into account standard deviations, but error bars calculated as standard deviations showed that the decomposition process using the aluminium or the MAO-modified aluminium cathodes proceeds under more stable electrical conditions. These results showed that the process conducted at a stable voltage affects the plasma performance of individual electrodes and thus the efficiency of the process.

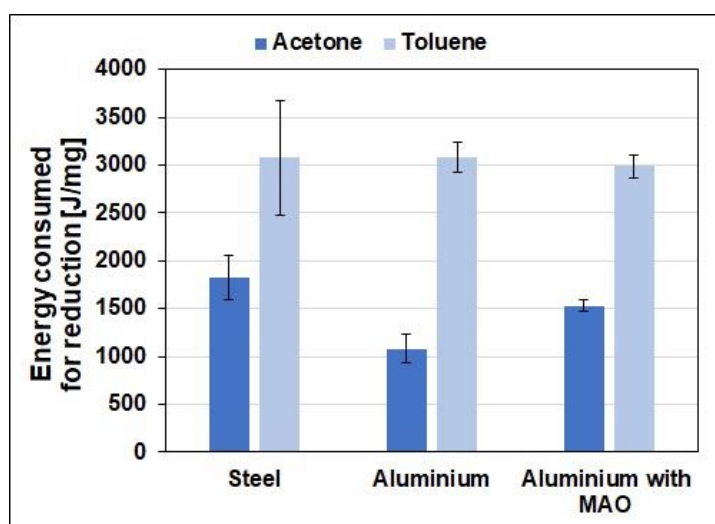


Figure 5: Energy consumption per unit quantity of decomposed acetone and toluene.

After completing the experiments, the surface of the electrodes was examined using an electron scanning microscope; the results are shown in Figure 6. During the decomposition process, the surface of the cathode made of the carbon steel (Figure 6a) was damaged most significantly. No significant changes were observed on the surfaces of the cathodes made of aluminium and aluminium treated with MAO technology. It is clear, that the carbon steel cathode is unsuitable for long-term use. Both cathodes base of aluminium will be tested to corrosion resistance.

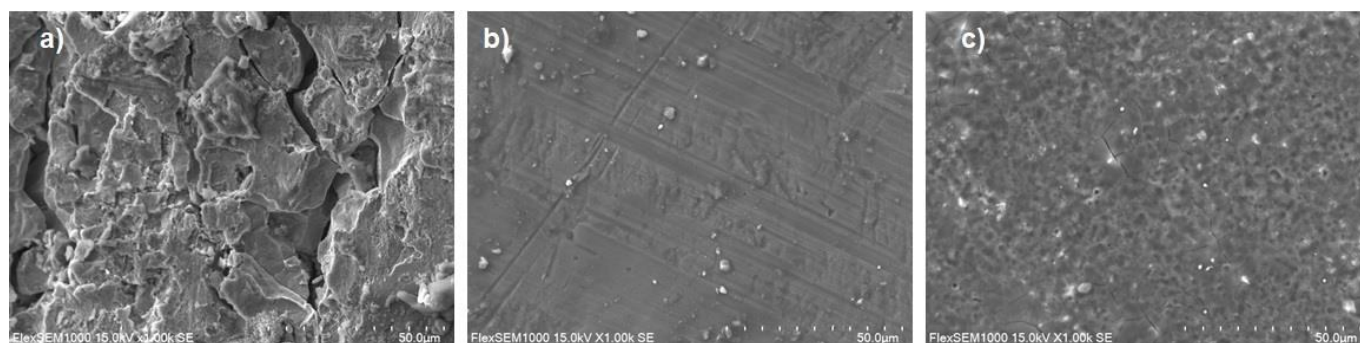


Figure 6: SEM image of carbon steel (a), aluminium (b) and aluminium cathode modified MAO (c) after NTP processes. Magnification 1000x.

Based on the results of corrosion tests on surfaces treated with MAO^{35,36} technology, it can be assumed that aluminium cathode with treated surfaces will be more resistant to corrosion than electrodes without surface modifications.

Conclusion

Reducing the VOCs concentration emitted from industrial operations is an urgent task for improving the environment and quality of life of people living near industrial agglomerations in particular. Despite existing technologies, it is necessary to increase their efficiency and applicability to specific types of VOCs. A new alternative appears to be a combination of ozone oxidation and plasma discharges generated in low-temperature plasma reactors. The efficiency of NTP is influenced by a number of parameters, including the construction material of the reactor and the electrodes themselves, which must also be resistant to the corrosive environment of gaseous substances. Corrosion resistance can be improved by surface treatment of construction materials, e.g., using micro-arc oxidation technology.

The aim of the experiments, carried out under the same operating parameters, was to compare the efficiency of acetone and toluene reduction in a laboratory NTP reactor using cathodes made of structural carbon steel, aluminium, or aluminium treated with MAO technology. During the process, the efficiency of the reduction of selected VOCs and process parameters, including the volt-ampere characteristics of the process, were monitored.

The initial results of the experiments showed that the use of an aluminium cathode with a modified surface has a comparable efficiency to the other cathode materials used, i.e., approximately 70%. The cathode made of aluminium and surface-treated aluminium using MAO technology did not show significant surface damage after use in the NTP. It can be assumed that under long-term using, MAO technology used to treat the surface of the NTP reactor cathode will be a suitable alternative to the use of more expensive and anticorrosive construction materials (e.g., Ti and Ta). Optimization of the control parameters of the NTP source will lead to an increase in the efficiency of VOCs decomposition, which will enable the production of semi-industrial equipment that could be tested in real industrial production conditions.

Acknowledgement

This work was created as part of the project Application of low-temperature plasma to reduce gaseous emissions, which is co-financed with state support from the Technology Agency of the Czech Republic under the TREND Programme (No.FW10010383) and project Materials and technologies for sustainable development within the Jan Amos Komensky Operational Programme, financed by the European Union and from the state budget of the Czech Republic (No. CZ.02.01.01/00/22_008/0004631).

Data Availability Statement

The data presented in this study are available at Zenodo <https://doi.org/10.5281/zenodo.17249859>.

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Využití reaktoru s nízkoteplotním plazmatem k redukci těkavých organických látek

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Souhrn

Těkavé organické látky (VOC), zahrnující celou řadu toxických sloučenin, jsou výraznou hrozbou nejen pro životní prostředí a lidské zdraví. Jejich emise mohou způsobovat sekundární znečištění ozonové vrstvy a vytvářet fotochemický smog, především v oblastech s regionálními zdroji znečištění. Je proto nezbytné věnovat pozornost snížení jejich emisí do ovzduší. Současné technologie zpracování/odstranění těkavých organických látek lze rozdělit na dva typy: technologie nedestruktivní (zejména adsorpce, kondenzace nebo absorpce) a destruktivní, ve kterých se chemickými reakcemi VOC přemění na CO₂, H₂O a případně další méně škodlivé sloučeniny (např. ozonizace, termická oxidace, katalytický rozklad, nízkoteplotní plazma aj.). S ohledem na nutnost zpracování odpadu vzniklého nedestruktivními technologiemi se do popředí dostávají destruktivní technologie a to zejména ty, které nejsou energeticky náročné. Mezi ně patří katalytický rozklad a využití nízkoteplotního plazmatu (NTP). Aplikace NTP je spojena s konstrukcí reaktoru, který by měl odolávat korozi a přitom měl dostatečnou účinnost rozkladu VOC. Řešením může být volba vhodnějšího materiálu než je konstrukční uhlíková ocel nebo využití korozivzdorné povrchové úpravy povrchu při zachování dostatečné účinnosti procesu. Jednou z technologií úpravy povrchu vybraných konstrukčních materiálů je technologie mikro-obloukové oxidace (MAO).

Cílem experimentů provedených na laboratorním reaktoru NTP s vyměnitelnou katodou bylo porovnat účinnost redukce acetonu a toluenu při použití katody vyrobené z konstrukční uhlíkové oceli, hliníku a hliníku s povrchovou úpravou technologií MAO. Experimenty byly provedeny za konstantního napětí zdroje plazmatu a rychlosti proudění vzduchu se známou koncentrací acetonu nebo toluenu. V průběhu procesu byla sledována účinnost redukce zvolených VOC i procesní parametry, včetně energetické náročnosti procesu.

Prvotní výsledky experimentů ukázaly, že použití hliníkové katody s upraveným povrchem má srovnatelnou účinnost rozkladu vybraných látek (cca 70 %) jako zbývající použité materiály katody a je srovnatelná s literárními údaji. Zvýšení účinnosti procesu lze dosáhnout optimalizací volitelných parametrů, především napětí zdroje plazmatu a rychlosti proudění plynu. Využití technologie MAO k úpravě povrchu katody reaktoru NTP se jeví jako vhodná alternativa k dražším a korozivzdornějším konstrukčním materiálům (např. Ti, Ta).

Klíčová slova: těkavé organické látky, nízkoteplotní plazma, mikro-oblouková oxidace