ATMOSPHERIC PRESSURE MW PLASMA FOR WASTE CARBON TREATMENT

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Introduction

The disposal of waste tyres represents a great problem throughout the world, as it is causing many environmental and economic problems in most countries. Pyrolysis seems to be a good way of waste tyre disposal, because it does not only reduce the volume of the waste significantly, but also allows recovering of valuable materials¹. Additional treatment is sometimes needed to improve their qualities^{2–3}. In our case, the MW plasma benefication of the pyrolytic carbon is applied to get rid of the volatile components causing its bad smell, and to make it reusable for other processes, e.g. it could be used as a colouring agent for plastic materials or as an adsorbent.

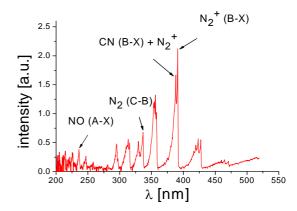
We use Litmas Red MW torch (2.45 GHz, 3 kW), which is able to generate plasma in a state close to Local Thermodynamic Equilibrium (LTE) in temperature range of 1000–5000 K at atmospheric pressure in various gases. In our previous works^{4,5} we investigated the basic characteristics of atmospheric MW nitrogen plasma torch. Here we present the first tests of its application for thermal treatment of waste carbon.

Experimental setup

Atmospheric pressure MW plasma is generated by a Litmas Red plasma torch (2.45 GHz, 3 kW) in N_2 . Experimental setup and the basic torch characteristics are described in more detail in ref.⁴.

Contrary to the typical MW torch systems, in our case the gas is inserted downstream and tangentially through the two holes of the nozzle into the cylindrical plasma chamber, made of Al_2O_3 . This is causing the swirl flow in the cylinder and generated swirling plasma is consequently blown out upstream through the central orifice of the nozzle. Blown-out plasma is then analyzed by optical emission spectroscopy. We identified emission of N_2 molecules, N_2^+ ions and NO and CN radicals in the emission spectra (Fig. 1). CN comes from a minor carbon-containing impurity from the feeding gas, which reacts with N_2 and forms CN radical.

Generated MW plasma was used for waste carbon treatment. In the first approach, the carbon sample was placed on a stainless steel plate in the plasma plume about 1 cm above the nozzle (Fig. 2).



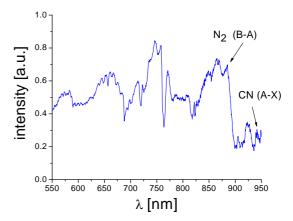


Fig. 1. UV (up) and visible (down) emission spectra of generated nitrogen plasma $\,$

Approximate temperature of the plate, measured by a pyrometer, was 1500 K. The system was closed and exhausted because of possibly dangerous gases being emitted from polluted carbon samples. The carbon samples were heated for 5 or 10 minutes in nitrogen plasma of 13 l min⁻¹ flow rate and 1.4 kW generator power. After plasma being switched off, the sample was cooled at a low N₂ flow 2 l min⁻¹ to avoid oxidation. The diagnostics of carbon samples was performed directly by a scanning electron microscope (SEM) Tescan TS5136MM equipped with wavelength dispersive X-ray (WDX) INCA Wave analyzer, and on KBr pellets analyzed by Fourier-transform infrared (FTIR) spectrometer Perkin Elmer Spectrum BX. KBr powder to the sample ratio was 1:100.

In the second approach (Fig. 3), which is still being developed, the waste carbon samples are continuously inserted directly into the plasma chamber, where the temperature is considerably higher compared to the first approach. The plasma temperature, determined as the rotational temperature by comparing experimental and simulated (LIFBASE⁶) CN spectra, was 4500 ± 250 K and 1.78 kW magnetron power and 4000 ± 250 K at 1.46 kW, both at $151 \, \text{min}^{-1}$ gas flow rate⁴.

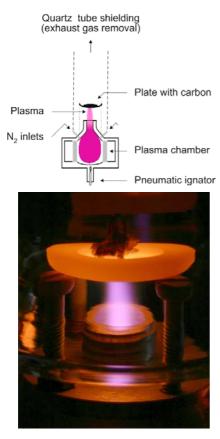


Fig. 2. Treatment of carbon samples on the supporting stainless steel plate

In this arrangement, the treated carbon is also in a direct contact with radicals and active species generated in the plasma. The whole system, including the plasma plume, is turned upside down to enable the collection of the treated carbon powder below the plasma chamber. The carbon collection chamber is water-cooled. It also enables on-line emission spectroscopic analysis, which is performed by portable spectroscope Ocean Optics SD 2000 through the quartz window.

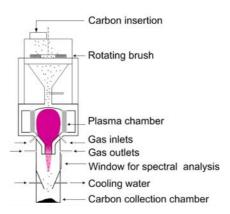


Fig. 3. Treatment of carbon samples passing through the plasma chamber $\,$

Results and discussion

After plasma heating of the carbon samples, we first observed that the sample did not smell anymore and its mass decreased (by 27 % for 5 min and 48 % for 10 min heating). We assume that some volatile substances, most likely aliphatic and aromatic hydrocarbons and their –OH and ether derivates were released from the carbon, which was confirmed by FTIR spectra (Fig. 4). An apparent reduction of CH₂, C-O-C, -OH, and aromatic C-H functional groups was observed. On the other hand, new compounds were created. We assume them to be metal oxides of trace elements found by WDX analysis. Detailed interpretation of the measured FTIR spectra requires further investigation.

WDX analysis (Fig. 5–6) of all samples demonstrated C as a dominant element. Trace amounts of S, Ca and Zn were found in the original carbon sample and decreased after the heating, but new elements, such as Fe, Al, Si, K, Mg were found. We assume that some of these metals were released

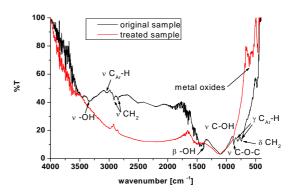
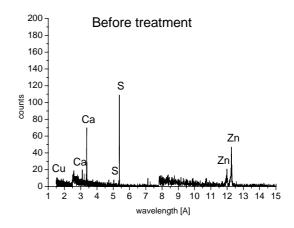


Fig. 4. FTIR spectra of carbon samples



 $Fig.\ 5.\ \textbf{WDX}\ \textbf{analysis}\ \textbf{of}\ \textbf{carbon}\ \textbf{samples}\ \textbf{before}\ \textbf{treatment}$

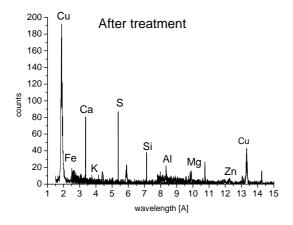


Fig. 6. WDX analysis of carbon samples after treatment

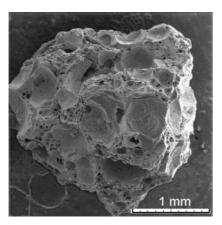


Fig. 7. SEM picture of carbon sample before treatment

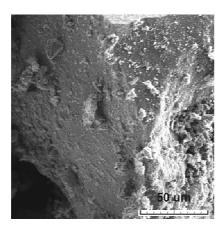


Fig. 8. SEM picture of carbon sample after treatment

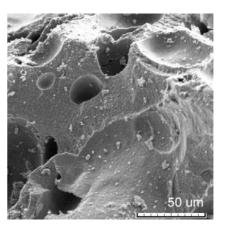


Fig. 9. SEM picture of the cleaving area before treatment

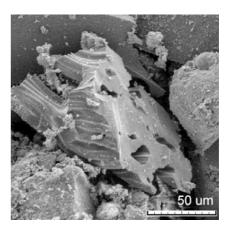


Fig. 10. SEM picture of the cleaving area after treatment

from the stainless steel plate during the heating (Fe, Si). Cu is from the supporting tape used during SEM analysis.

Despite the conductivity of the carbon samples being weak, it was good enough for SEM analysis at low magnifications. Microscopic pictures showed that the carbon has an amorphous structure with pores (Fig. 7–8). Pictures of the treated samples revealed the steps-like shaped cleaving area (Fig. 10), which compared to the smooth shape in the original sample (Fig. 9) shows that material became more compact.

Summary and perspectives

Atmospheric pressure nitrogen microwave plasma was used as a heat source for used tyre waste carbon benefication. The plasma heat treatment removed the smell and caused a mass loss, a composition change and a structure change of the waste carbon. The treated carbon could be potentially used as a colouring agent. We are currently testing the adapted microwave torch system in which the carbon powder is inserted directly into the plasma chamber. This provides higher temperatures compared to when the sample is heated on the

supporting metal plate. In the new setup, the plasma chamber is turned upside down to enable the collection of the treated carbon powder in the cooled collection chamber. Because of the presence of pores in the treated carbon we also plan its further investigations, such as measuring its adsorption capacity. This could be interesting for its potential use as an active carbon.

This work was carried out under the support of the Slovak Grant Agency VEGA 1/0293/08 and 1/3043/06, EOARD FA8655-08-1-3061 and APVV 0267-06 grants. We gratefully thank Sencera, Ltd. for loaning us the MW torch, KFS Stavstroj Delta Ltd. for waste carbon samples and Dr. V. Foltin for technical assistance.

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L. Leštinská^{a*}, V. Martišovitš^a, M. Zahoran^b, and Z. Machala^a (^a Division of Environmental Physics, Department of Astronomy, Earth Physics and Meteorology, ^b Department of Experimental Physics, Comenius University, Mlynská dolina, Bratislava, Slovakia): Atmospheric Pressure MW Plasma for Waste Carbon Treatment

The first tests of benefication of waste carbon from used tyre pyrolysis are presented. The treatment of carbon samples is performed by microwave (MW) plasma generated by Litmas Red MW torch (2.45 GHz, 3 kW) in nitrogen at atmospheric pressure. We employ two ways of waste carbon treatment – heating the samples on the supporting plate and direct treatment inside the plasma chamber. The diagnostics of carbon samples was performed by gravimetry, SEM equipped with WDX element analyzer and FTIR spectroscopy. The plasma heat treatment causes a mass loss, a composition change and a structure change of the waste carbon.