THE ANALYSIS OF COMBUSTION GAS BY METHOD OF FTIR

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Research article

Abstract:	This paper deals with the analysis of wood combustion gases arising under given conditions by methods based on infrared spectrometry. The method according the standard DIN 53436 was used for preparation of wood combustion gas in laboratory scale. As a measurement devices, both the nondispersive infrared analyser S710 (Maihak) and the standard infrared spectrometer with Fourier transform NICOLET IS10 were used for continuous analysis. Mutual comparison of the two way of measurement is based on the analyses results.
Key words:	FTIR analysis, thermal degradation of wood, combustion gas, DIN 53436.

Introduction

The content of chemical compounds in pyrolysis and combustion products is an issue that must be solved with respect to its health and environmental impacts in cases of fire or combustion for residential heating. Reactions that take place during thermal degradation are dependent on the composition of flammable substances, oxygen concentration, temperature and many other conditions. Therefore, their products are very diverse in terms of both quality and quantity. Toxic or harmful substances with negative impact on the environment can be found among these products.

Gas products of thermal degradation do not arise only when the fires or control burning take place, but they accompany the process of spontaneous combustion.

Coal in mines, stockpiles and coal waste are well known sources of gaseous emissions, if the self-heating takes place. The accumulated material of cellulosic nature, such as wood chips, can be a source of gaseous combustion products, too. Carbon monoxide and carbon dioxide are released in larger quantities and other compounds, e.g. acrolein, in smaller quantities.

This work is focused on the wood as an available, frequently used material, which smoke gases, whether originated by fire or by control combustion, are often found in the environment. Wood and other cellulose-based materials are the basic raw material for biofuel production. Although wood is considered a source of „clean energy", the carbon dioxide emitted by combustion is not negligible. Release of other gaseous substances such as polyaromatic hydrocarbons is controlled by the quality of combustion, and there is some correlation with the concentration of carbon monoxide.

In addition to carbon dioxide that is released in high concentrations, the wood combustion products contain other components that arise in lower to trace amounts. Toxicity or other adverse effects of substances increase with increasing concentration. Also, the consequences of substances with lower concentrations may be significant with respect to burning of large quantities of wood or fuels based on cellulosic materials, e.g. in cases of forest fires (Tissari et al., 2008).

The wood combustion appliances in use nowadays are capable to reduce emissions as much as possible, if "natural" untreated wood is used as a fuel. A more complicated situation occurs when "waste" wood, e.g. plywood, wood from buildings, furniture, packaging, etc., is burned. Such wood contains chemical compounds originating from paints or preservatives and therefore unexpected substances of organic nature may be found in gaseous products of combustion. Heavy metals from paints either remain in the ashes, or spread into gaseous fumes, according to their volatility.

The complex structure of wood and conditions during combustion lead to the formation of a aried mixture of substances (Commandré et al., 2011). Wood is one of the carbonaceous fuels with high content of volatile matter. During the thermal degradation, its chemical composition is changed and volatile liquids and gases are formed. The process of thermal degradation is generally divided into several phases according to main features of the changes.

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The results of work (Oren et al., 1987) suggest three stages in dry wood open system combustion. These stages are distinguished by changes in concentration of carbon, oxygen and total hydrocarbons. According to other data (Kačíková et al., 2006) the first gaseous products are generated by thermal degradation of wood at temperatures around 150 °C to 200 °C, but only in very small quantities. At temperatures above 200 °C, the depolymerisation of polysaccharides and lignin takes place and a complicated mixture of many substances, e.g. hydrogen, methane, carbon monoxide and dioxide, aldehydes, ketones and other low molecular weight products, is formed. Many of these substances are toxic or harmful.

The pyrolysis processes of biomass under inert atmosphere were studied (Neves et al., 2011). After drying of cellulosic material, two distinct stages were found and their characteristic products of thermal decomposition were analysed. When the biomass is heated, the primary stage of pyrolysis occurs after fuel drying. The volatile mixture comprises permanent gases such as carbon oxides and methane, and the substances condense according to the ambient conditions (some organic substances and water). Organic compounds that have the ability to condense are often in sum referred to as tar.

The primary pyrolysis stage is completed at relatively low temperatures, under 500 °C, yielding a carbon-rich non-volatile solid that is called char or charcoal. Complex reactions of heterogeneous and homogeneous character can take place upon further heat exposure at higher temperature.

The carbon dioxide is considered as the main toxicant of combustion gases from burning wooden materials (Wang et al., 2004). At work (Wang et al., 2004), carbon oxides, nitrogen oxides, sulphur dioxide and oxygen were analysed components. The JM automatic analyser (RBR-ECOM Co., Germany) with electrochemical sensors was used for continuous analysis of these gases.

Composition of wood smokes resulting from residential heating was referred to in the work (Hedberg et al., 2002). Concentrations of benzene, toluene and xylene, polyaromatic hydrocarbons, aldehydes and ketones were monitored and analysed by several methods.

The focus of many research papers is to determine the toxic gaseous components, or soot as particulate matter (Bølling et al., 2009; Tissary et al., 2008). In other works, the particulate matter, carbon monoxide and polyaromatic hydrocarbons are monitored.

The polyaromatic hydrocarbons and other substances resulting from the low-temperature pyrolysis of wood have been measured by gas chromatography with mass detector (Ré-Poppi et al., 2002). Totally 74 compounds were identified. The most abundant compounds were methoxyphenol, phenantren, fluorene, anthracene, acenaphthylene and levoglucosan. Almost one hundred compounds arising under the same conditions were detected by GC-MS (Liodakis et al., 2002). The products of cellulose pyrolysis include many oxygenated products, such as methanol, glycolaldehyde, levoglucosan and oligomers and also methane.

The two standards, ISO 13344 and ISO TS 13571, assess the toxic potency of major component of combustion gases, i.e. carbon dioxide, narcotic gases (carbon monoxide and hydrogen cyanide) and irritant gases (hydrogen chloride, hydrogen bromide, hydrogen fluoride, nitrogen oxides, sulphur dioxide, formaldehyde and acrolein).

Analysis of combustion products that contain such a wide range of chemical compounds, is rather complicated, even using modern technology, and requires time-consuming procedures for each component.

A broad discussion of methods used for the analysis and comparison of toxicity is listed in the work (Hull et al., 2007). The review features standard methods of preparation of combustion products and stages of fire modelled by these methods.

The standard ASTM E-800 provides comparisons between several analysing techniques, which are most often used to assess the composition of gases present or generated during a fire (ASTM 2007).

The analysis of combustion products can be accelerated and simplified in two ways. At the first one, the limited number of substances to be analyzed, e.g. polyaromatic hydrocarbons, is selected. According to this choice, the method of analysis and measuring instruments are selected. By the second way, the number of identified components is almost not limited, and therefore the chosen method must be able to identify relatively wide range of chemical substances occurring in combustion gases. The gas chromatography and spectroscopic methods are suitable for this second way.

Analysis of gas combustion by infrared spectroscopy with Fourier transform is often used in combination with thermal analysis. The combustion products are generated under controlled conditions of temperature growth and atmospheric composition. The kinetics of pyrolysis and combustion of wood under different oxygen concentrations was investigated by this method by (Fang et al., 2006).

The methods of analyzing gaseous combustion products are subject of international European project SAFIR - Smoke gas analysis of Fourier transform infrared spectroscopy (Hakkarainen et

al., 2000). The method of sampling and filtering of combustion products before analysis, the lines of the gas sample into the analyzer, and method of gas preparing in a laboratory scale and their relation to different stages of the fire were studied in the project. Cone calorimeter was used for the preparation of gas combustion products in laboratory scale. The FTIR method (infrared spectroscopy with Fourier transform) was evaluated as a method that allows compensation of certain difficulties in the analysis of combustion or pyrolysis products.

Finnish standard NT FIRE 047 of 1993 describes a methodology of analysis of the hot exhaust gases by FTIR method. In this standard, the emphasis is placed on the use of heated sampling line and its length. The flow rate of the combustion gases shall be high enough and the volume of the sampling line and IR cell shall be small enough for the content of the IR cell to be renewed every 10 s. The value of the flow rate of 7 - 8 liters $\cdot$ min^{-1} is considered as a usual one (NT FIRE 1993).

The standard ISO 19702 also states the analysis methodology of combustion gas (ISO, 2006).

This work deals with the comparison of two methods of analysis of gaseous combustion products of thermal degradation, which are used in the laboratory of the FBI.

Materials and methods

The analysis was performed in two ways using the same material, i.e. wood, and under the same conditions of preparation of gaseous combustion products for analysis. The strips of dried spruce wood were used as a test samples. Their dimensions were (200 x 15 x 2) mm, according to the requirements of the standard used for the preparation of gaseous fumes. The weight of samples was about 4 grams. Combustion gases of spruce wood were prepared according to the standard DIN 53436. A circular furnace which moves along the quartz tube with the sample, had a constant temperature of 800 °C and the velocity rate of 1 cm.min^{-1}. The ambient air was used as an oxidizing medium. It was drawn into the tube with the sample by the pump, which was placed behind a tube and filters and in front of the analyser. The flow rate of air passing through the apparatus was between 0,66 to 0,70 l.min^{-1}. This very low flow rate was chosen according to the parameters of S710 analyser, which does not allow a higher flow rate of media. The resulting soot particles were trapped by a filter of glass wool; silica gel was used to remove moisture.

The method according to DIN 53436 (DIN, 1981) is intended to evaluate the gas toxicity on the basis of animal experiments, but the numerical

evaluation of toxicity is also applicable. In this work, the method was used as a standard method for the preparation of gas fumes only; therefore the evaluation of toxicity was not made. The gases for analysis by both methods, comparing each other, were prepared by this method.

Proper function of both devices, i. e. the analyser and the spectrometer, was verified before measuring by the use of calibration gases containing carbon monoxide and carbon dioxide. With regard to the possibility of toxic gases formation, the outlet of both devices was directed outside the lab space.

According to the first method, the gases which should be analyzed were determined firstly. Carbon dioxide, which accompanies combustion by considerable concentrations, and carbon monoxide, which is characterized by high toxicity, were chosen. This pair of gases constitutes a high risk at fire, with regard to the synergistic effect that is manifested in their effects on living organisms. Oxygen was another observed component, because oxygen depletion during a fire is assessed as a very important factor with regard to threat to health and lives.

The modular analyzer Sick/Maihak, type S710 with the Multor analyser module, which uses non-dispersive infrared absorption, was chosen to measure the concentrations of both carbon dioxide and carbon monoxide. This analyser was also equipped with the Oxor-E module for determination of oxygen using an electrochemical cell. Results of measured concentrations versus time were recorded using the RD 8800 recorder with A/D converter. The concentrations of all 3 analyzed gases were recorded at time intervals of one second. The data were recorded on a memory card and then transferred to a PC and processed in Excel spreadsheets. A detailed description of the apparatus is shown in work (Loupanec, 2010), in which these analyses were performed. The way of removing soot and moisture from flue gases was also evaluated in this work. Based on observed data, the combination of glass wool and silica gel were evaluated as the best combination.

According to the second method, the FTIR spectrometer Nicolet IS10 was used, which works in the mid-infrared range of 4000 cm^{-1} to 400 cm^{-1} and with a spectral resolution up to 1 cm^{-1}. Gas cell with optical path length of 10 m was heated to a temperature of 150 °C. The delay between the change in concentration in the tube and the detector response was about 5 seconds for a given arrangement. Neither sampling lines nor filters were heated. Two bottles gas washing, with sintered disk, Drechsler, were used as a filter carrier. The glass wool was contained in the first bottle, the silica gel with indicator in the second

one, to capture the soot and moisture generated during combustion. The device allows continuous measurement of spectra in time. Three average spectrums are processed during one minute.

After the introduction of calibration methods, the concentration of each gas showing the characteristic bands in mid-infrared range can be evaluated.

Results

The Sick/Maihak type 710 analyzer is designed to measure three components, namely carbon dioxide, carbon monoxide and oxygen. The concentration of measured components can be monitored during measurement, either directly on the display of analyser or on the connected recorder. The device is also equipped by alarm setting at a certain concentration value of monitored gases. The clear graph allowing a view of the results can be created after the transfer of data to Excel. Example of such processing is shown in Fig. 1. This is a result of the analysis of gas components of spruce wood thermal degradation at oven temperature of 800 ° C and in an atmosphere with inlet concentrations of oxygen 21 %. Soot was captured on a filter of fibreglass and moisture was absorbed on silica gel.

The time of analysis was 30 minutes and furnace moved with the speed of one centimeter per minute. Because the sample had a length of 20 cm only, the furnace was outside the sample at the beginning of test but the radiant heat of the oven has already affected the sample. The material carbonised slowly at first, and then an ignition occurred. The sample burned in a bright flame, generating smoke and soot. A thermal ignition of the sample occurred before the furnace reached the edge of the sample. The burning was unsettled; repeatedly re-ignition and extinction take place. Despite the fact that the wood has dried out, a considerable amount of moisture arose, which condensed at the outlet of the tube. Intermittent burning resulted in fluctuations in concentration of carbon dioxide and of oxygen concentration (see Fig. 1).
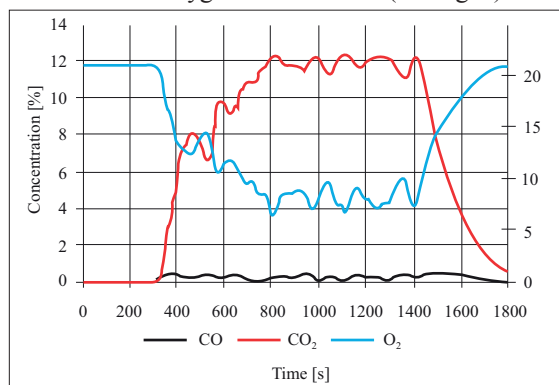

Fig. 1 Gaseous compounds concentration in combustion gas of spruce wood

Concentrations of all compounds began to change approximately in the fifth minute after the test started. The maximum value of carbon dioxide concentration, i.e. 12,5 % vol., was achieved in approximately the thirteenth minute and this concentration fluctuated around a maximum up to the twenty-third minute, then the rapid decline came.

This maximum value of carbon dioxide concentration lies on the border of the measuring range and hence this reading is not unambiguous.

The actual concentration may be higher but its value cannot be found on the basis of measured data (Loupanec, 2010). A similar situation, i.e. exceeding the measuring range, occurred during the measurement of carbon monoxide concentration.

The FTIR spectrometer, Nicolet iS10, cannot be used to measure oxygen, as well as for other diatomic symmetrical molecules. Other components of thermal degradation gas are measurable by this device, if their absorption is in the mid-infrared range. During the determination, the concentration of substances can be observed immediately using the software Series connected with calibration methods. The substance in question should be known in advance, as well as when the dispersion analyzer is used.

Furthermore, the increase in concentration of all combustion gases can be assessed immediately according to the Gram-Schmidt curve, which is a record of the overall detector response versus time (Pásztor et al., 2010). This record is shown in Fig. 2, where the curve is derived from the analysis of combustion gas of spruce wood.
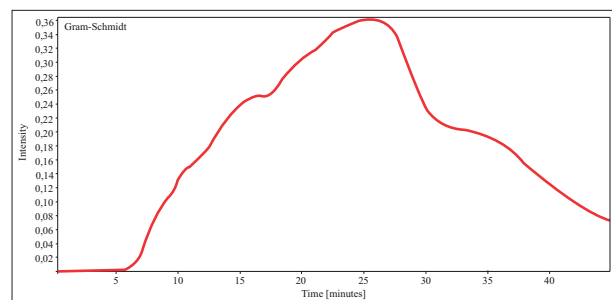

Fig. 2 Gram-Schmidt visualization of the overall response of the detector

The graphic record shows that the first changes occurred in the fifth minute from the start and the maximum change was reached in the twenty-sixth minute. The delay at the beginning of setting is the same as in the evaluation of the Sick/Maihak, type 710, analyzer but the concentrations growth in time is different. Restrictions of measuring range of Sick/Maihak, type 710, analyzer is one of the reasons for this difference. In addition, other components of gas mixtures were analyzed by the FTIR spectrometer.

The spectra measurements were performed using "Omnic Series software", which allows data collection over time. Measurement results are displayed in 3D mode, see Fig. 3.
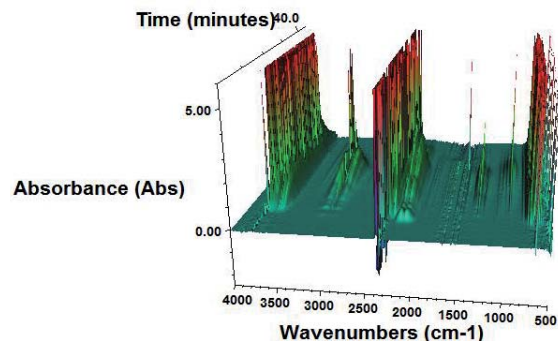

Fig. 3 Dependence of time - wavelength - signal strength at the evaluation of gaseous products of thermal decomposition of spruce wood

From this representation, it is possible to distinguish between majority and minority components and the increase and the decrease in their concentration over time are apparent as well. The individual spectra obtained at certain times can also be viewed from these data. Example of spectra corresponding to the composition of the combustion gas at the time of 24.06 min. from the beginning of the test is shown in Fig. 4.
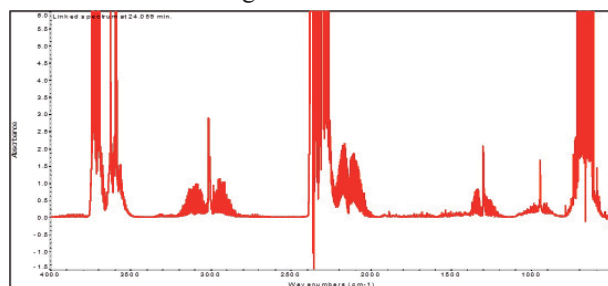

Fig. 4 IR spectrum of combustion gas composition in the twenty-fourth minute of test

Identification of the components contained in combustion gas was performed using a multi-component mixture analysis in the OMNIC Specta Software. First of all, carbon monoxide and dioxide were evaluated. Gaseous carbon monoxide has broad absorption peaks (doublet) at 2130 and 2170 cm^{-1}. Carbon dioxide has a very intense band with a maximum at 350 cm^{-1} (asymmetric stretch vibratory mode), which is in the total absorption, so it cannot be used for quantitative evaluation. Another band of weak intensity (degenerate bending vibratory mode) has its maximum at 666 cm^{-1}.

Simple hydrocarbons and water were also found in gaseous products. The following Tab. 1 summarizes the identified compounds and their detection in time from the start of the experiment.

Tab. 1 Changes in the composition of the gaseous products of thermal degradation of spruce wood

Time [min]	Detekované látky
5,95	carbon dioxide
7,84	carbon dioxide, carbon monoxide, water, methane
12,97	carbon dioxide, carbon monoxide, water, methane
15,95	carbon dioxide, carbon monoxide, water, methane, ethylene
19,46	carbon dioxide, carbon monoxide, water, methane, ethylene, acethylene (tr.)
24,06	carbon dioxide, carbon monoxide, water, methane, ethylene, acethylene
30,00	carbon dioxide, carbon monoxide, water, methane, ethylene, acethylene
35,95	carbon dioxide, carbon monoxide, water, ethylene, acethylene

The carbon monoxide and dioxide and lower hydrocarbons, saturated and unsaturated, were identified in gaseous products of thermal degradation of spruce wood, released under conditions according to DIN 53436. At the end of the test, the methane disappeared and only the unsaturated hydrocarbons were detected.

The concentrations versus time for individual components were determined by means of calibration curves. After converting to the Excel spreadsheet, the chart was processed for particular compounds. An example of such an evaluation is shown in Fig. 5, where the changes of carbon monoxide concentration versus time are recorded.
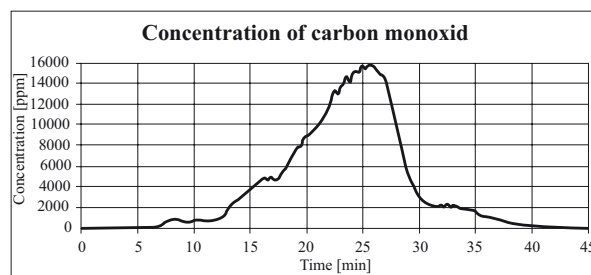

Fig. 5 Changes in the concentration of carbon monoxide in time

The graph shows that the concentration of carbon monoxide has been considerable. The maximum concentration was 15773 ppm, i.e. 1.57 % vol., and it was recorded in the 25th minute after the start. When

evaluating, two different calibration dependences were used, for low concentrations up to 2000 ppm, and for high concentrations up to 99,000 ppm. This shows that measurement results can be evaluated, when a suitable calibration method is available, even if the measurements exceed the expected concentration.

Discussion

The subject of this study was the composition analysis of combustion gas mixtures prepared according to DIN 53436 (DIN, 1981). Many other methods are used for the preparation of gaseous products of combustion and pyrolysis, for example, the cone calorimeter was used in the project SAFIR (Hakkarainen et al., 2000). The thermal analysis is frequently used for the preparation of gaseous fumes.

The method according to DIN 53436 was chosen because it allows the evaluation of combustion gas toxicity of thermal degradation of solids and liquids. Such evaluation is important when assessing the risk associated with the processes of thermal degradation of materials, for example by heating during self-heating or during unexpected chemical reactions.

Several variants of filtration of released gaseous mixture were tested. In addition to soot, the resulting moisture was removed, especially with regard to non-dispersive analyzer that could be damaged by excessive moisture. Infrared spectrometer can also analyze the humid exhalations, as far as the measuring cell is heated to a temperature higher than about 130 °C.

Therefore, the cell was heated to a temperature of 150 °C during the test. In addition, the bands of water could cover the bands of other compounds in the measured spectrum.

Sampling line between the device according to DIN 53436 and the nondispersive infrared analyzer and/or the standard infrared spectrometer was not heated. Therefore, much of the moisture condensed on the output of a quartz tube intended for preparation of the gaseous sample. At the end of the quartz tube, the probably organic nature substances condensed in various shades of brown. Certain amount of these substances was also captured on the filter of glass wool. This condensation was apparently the reason why only the gaseous organic compounds, particularly lower hydrocarbons, occurred in the spectra. The heated sampling lines are vital for the analysis of these substances.

The relatively low air flow rate was set during the test; about ten times lower.in comparison with similar work (NT FIRE, 1993). The very low flow rate is probably the reason for the evolution of

high concentration of carbon monoxide. Unsteady combustion in a quartz tube according to DIN 53436 is explained by too small a diameter of tube (ASTM International, 2007), although this fact is related to insufficient air supply, too.

Both the non-dispersive analyzer and the infrared spectrometer have the option to transfer the measured data into an Excel spreadsheet for further processing. Results from the infrared spectrometer are directly recorded and processed in the PC. Other device is needed to be connected to non-dispersive analyzer for the data transfer.

When working with the non-dispersive analyzer, the gas species and its approximate maximum concentration must be known in advance. If the maximum concentration was exceeded the actual value cannot be traced. In addition, the values recorded in this situation can be the source of an erroneous evaluation of the concentration.

The infrared spectrometer data can be evaluated, even if they exceed the estimated concentration obtained using the calibration dependence for the higher values of concentration. There are also other possibilities of higher concentrations solution, for example utilization of cells with a shorter optical path, dilution of sample, or the analysis in the near infrared (NIR), depending on available equipment. The infrared spectrometer records all substances, even the unexpected ones, contained in combustion gas. These substances can be identified and quantitatively evaluated, if calibration method is prepared. The device allows continual and discontinual analysis of particular samples.

Conclusion

A comparison of two methods of analysis provides their main advantages and disadvantages.

The analyser of NDIR type is satisfactory for the analysis of pre-defined components of combustion gas arising from thermal degradation. It allows the continuous monitoring of concentrations and the direct observation of their value on the device display. It is equipped with a possibility of alarm setting. When the appropriate data acquisition device is connected, analyser can also store data with a later transfer to PC for further evaluation. Before executing the analysis out, the chemical compound concerned must be known, as well as its maximum concentration range. The measures should be taken to avoid incorrect interpretation of data, if the maximum value of measuring range is reached and/or exceeded.

If the aim of analysis is finding of complex composition of gaseous combustion products,

including substances of very low concentration, it is preferable to use FT-IR spectrometer. The software of the device allows rapid analysis of a mixture of gaseous substances. The evaluation of data during the ongoing analysis is not possible, but the data are automatically recorded for further evaluation.

This method is used not only as a basis for evaluation of toxicity of combustion gas. It is also appropriate for stage assessment of self-heating, because the composition of the exhaust gas depends on temperature and conditions of the material. Using this method, the minor components of gas mixtures arising in chemical and other plants can be identified. Despite of their lower concentration, some of these compounds can threaten the health and lives and contribute to environmental pollution by long-term action.

Acknowledgments

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