DETERMINATION OF OZONE CONCENTRATION AND ITS EFFECT ON DEGRADATION OF MATERIALS

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Research article

Abstract:	The ongoing Covid-19 pandemics showed the need for the effective decontamination of environment and surfaces. One of the options could be the use of gas ozone. Ozone has been well known for its disinfection properties, which were in the past used to decontaminate water. In this article, we present a development of an easy-made and ready-to-use portable detector of ozone, which could be used to easily detect ozone in the environment. In addition, we studied degradation impact of ozone on different materials, which normally occur in households or at working places. Ozone is well detectable and does not cause any damage to materials at concentrations needed for decontamination of the environment.
Keywords:	Detector, FTIR Spectroscopy, Material Degradation, Ozone, Disinfection.

Introduction

In 2020, we met with the worldwide COVID-19 pandemic for the first time. The issue of disinfection/ decontamination of surroundings began to be solved at the same time. In the following periods, disinfection was carried out in large quantities, whether it was the space of hospitals, offices or even bus stops, playgrounds and other open and closed spaces. At this time, the market was saturated with various manufacturers of disinfectants with different, even magical properties. Disinfection procedures of space decontamination were clearly set by Fire and Rescue Service of the Czech Republic. Still, new simpler procedures were still being sought, which could be used as additional disinfection after thorough cleaning of, for example, offices.

One possibility was the use of ozone in the gas phase. This method is used for example to clean car air conditioners. Ozone is also used for the disinfection of water and for the production of ozone water as a disinfectant. First, we were thinking about the possibilities to use ozone for the disinfection of surface or air. In the course of testing this, many questions and unknown answers emerged, which lead us to start a larger project called "Possibilities of using ozone for air and surface decontamination not only for section of Integrated Rescue System of the Czech Republic"

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was created. This project deals with solving and answering all questions, which appeared during the first ozone tests as means of space decontamination. First, it was necessary to learn how to produce and measure the required amount of ozone not only in the laboratory, but also in real conditions for subsequent use for disinfection. The first goal was to create a test methodology for ozone determination and to create a portable simple detector for the use in real conditions.

After we were able to measure the concentration of ozone produced in a given space, we started dealing with two fields of study. One of them is a reaction of ozone with materials occurring commonly in rooms. This issue is very important, because the study of the effect of disinfection on the surroundings cannot be done without the study of the effect of the disinfectant on the surrounding materials. It is very unwanted to have an excellent disinfectant that destroys everything it comes into contact with. The second field dealt with in the project is an influence of ozone on microorganisms. This issue and the results of the influence on microorganisms will be described in a separate article. Based on the first tests, we already know that the process of killing microorganisms using ozone will require time of hours. For this reason, the effect of ozone on the degradation of materials in the specified conditions was studied.

Materials and methods

Materials and ozonation

Common materials ordinarily used in interiors were chosen for the study of material degradation, in particular 12 mm thick white melamine faced chipboard, pine lath of 10x15 mm, 2 mm thick acrylic (PMMA) glass, 5 mm thick nylon carpet, 2.5 mm thick expanded polyvinyl chloride floor covering, 0.4 mm thick cotton T-shirt (100% cotton). All the materials were cut into small pieces of maximum 10 mm of each dimension for ozonation. These pieces were equipped with a hole, which enabled fixing of the sample by a fishing line in the sealed box (Fig. 1).

The apparatus for ozonation of materials was assembled from the source gasses, the ozone generator, the sealed box with a small fan, the FTIR spectrometer and the air pump. The parts of the apparatus were connected by silicone and teflon tubes (to prevent decomposition of ozone). First, we tested which source would be the most suitable for the production of ozone. In particular, we evaluated air from the surroundings, the mixture of O₂ and N₂ from pressure bottles and clean O₂

from pressure bottle. For the required concentrations of O_3 , the mixture of O_2 and N_2 was proven to be the most suitable source.



Fig. 1 Samples of materials prepared for ozonation

The concentration in the box with the samples was adjusted by the change of the O_2/N_2 ratio at the entrance to the ozone generator and by variation of ozone generator output. The concentration was measured by FTIR spectrometer (see below) linked behind the sealed boxed with samples. To ensure the flow of ozone through the box and the spectrometer the air pump was linked behind the spectrometer at the end of the apparatus.

The samples of all of the materials were exposed to different concentrations of ozone (1400 ppm, 3000 ppm) for various intervals (1, 3, 5, 7, 9, 24 h). After removing the samples from the sealed box, they were individually inserted into glass vials and then they were subjected to investigation of degradation (see below).

FTIR Spectrometer

FTIR Spectrometer MATRIX-MG2 from Optik Instruments, s.r.o. (Bruker) with an optical path length of 2 m, a volume of 200 ml and maximum gas cell temperature of 191 °C was used for the measurement of ozone concentration (Fig. 2). The advantage of this spectrometer is

the cooling of the detector using a cryocooler (Stirling cooler) without the need for liquid nitrogen. For measuring ozone concentration, a methodology for 1-500 ppm of ozone has already been prepared by the supplier. Since it was necessary to measure higher concentrations of ozone during the experiments, in cooperation with the supplier of the equipment, the methodology was modified to measure higher concentrations as well. The spectra was continuously measured with the following parameters: spectral range of 4800-750 cm⁻¹, number of scans equal to 10 (for one point of the concentration curve over time), spectral resolution of 0.5 cm⁻¹.



Fig. 2 FTIR spectrometer MATRIX-MG2

The comprehensive software OPUS GA (OPUS Gas Analysis) automatically evaluated the measured spectra in real-time in order to identify and quantify the gas compounds. The methodology developed for the measurement of ozone concentration at room temperature includes other gasses with regard to possible occurrence in an atmosphere or interference in the quantified area. Following components were monitored during the measurements: O₃, CO₂, H2O, NO and NO2. The current concentration of the measured gasses was possible to monitor during the continuous measurement. The result of the measurement was the dependence of the monitored component concentration on time was. Concentration is given in units of ppm. A spectrum measured at a given time can be assigned to each measurement point of concentration.

Detector calibration

In order to calibrate the designed portable detector, it was first necessary to effectively produce and measure the required ozone concentration. We used PROFIZON-X 7G ozone generator with a maximum output of 7 g O_3 per hour with a gas flow rate of 6 l.min⁻¹ and the possibility to supply air from an internal compressor or oxygen from an external input. This device can work continuously and can be used for the disinfection of large spaces,

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water disinfection, deodorization and the production of concentrated ozone water.

The infrared spectroscopy method was used to determine ozone concentration. A spectrum of ozone has a characteristic peak in the region of 1055 cm⁻¹ that is well suitable for evaluating quantitative data - creating a calibration curve (see Fig. 3).



Fig. 3 IR spectrum of ozone

First, the measuring set was assembled to efficiently produce the required ozone concentrations, which would be then used as individual calibration points for calibrating the portable detector (see Fig. 4). Based on the experiments carried out, N₂ and O₂ in ratio of 1:1 were selected as input gasses. In case of producing lower concentrations of ozone, this ratio was reduced in favor of N₂. The production of ozone directly from pure O2 did not work because the production of ozone was too high, even with the reduction of the generator's power to a minimum. When we used ambient air, NO2 was produced, and when we used air from a pressure cylinder, it was not possible to achieve the production of low concentrations of ozone. The ozone generator was placed in a laboratory fume hood (Fig. 4 - 1).

In the beginning, the measuring set for determining the ozone concentration was composed of pressure cylinders of the input gasses. A constant gas flow of 6 l.min⁻¹ into the ozone generator was ensured using a flowmeter (see Fig. 4 - 2). The output was set on the generator to produce the required amount of ozone (see Fig. 4 - 3). The produced ozone flowed into the gas cell of FTIR Spectrometer using a gas pump connected behind the spectrometer (see Fig. 4 - 6, 9). The flow of ozone through the gas cell was reduced to 2 $1.\text{min}^{-1}$ (see Fig. 4 - 7, 8). So a needle valve was connected behind the ozone generator, which served both to remove excess ozone and verify the measured ozone concentration using detection tubes (see Fig. 4 - 4). For the calibration of the portable detector, a glass flanged

vessel was included in the measuring set (see Fig. 4 - 5). A portable detector was placed in this vessel. Different output of the ozone generator was set to produce different concentration of ozone suitable for portable detector calibration. The produced ozone flowed into the gas cell of the MATRIX MG2 FTIR spectrometer using a gas pump connected downstream of the spectrometer.



1 - laboratory fume hood; 2 - input gas to ozonation (Q1 = 6 l.min⁻¹); 3 - ozone generator with adjustable output; 4 - conducting of excess O_3 regulated by a needle valve; 5 - glass flanged vessel with a portable detector; 6 -FTIR gas cell; 7 - outlet from the gas cell (Q3 = 2 l.min⁻¹); 8 - needle valve; 9 - gas pump

Fig. 4 The measuring set for calibration of a portable detector

Detectors

As an additional method for real-time ozone concentration measurements we designed and constructed a portable handheld ozone detector that operates in two concentration ranges for low concentrations of 10-1000 ppb and high concentrations of 10-1000 ppm. Two MQ-131 semiconductors were used for ozone detection. These detectors operate on the principle of changing the thermal conductivity of the measuring circuit with respect to the reference circuit. The sensor is heated to a high temperature (150-450 °C) at which oxygen molecules are absorbed onto the surface due to free electrons which changes the resistance of the semiconductor. When an ozone molecule approaches the absorbed oxygen or the sensing oxide layer, a mutual reaction occurs and the resistance of the semiconductor changes, which is directly proportional to the ozone concentration (Starke et al., 2002).

The sensing material of the MQ-131 gas sensor is a semiconductor doped with various metal oxides. WO_3 is the sensitive material for the detection of low ozone concentrations and SnO_2

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for high concentrations. These materials, from which the measuring circuit is assembled, have high conductivity in clean air, but in the presence of gas (ozone) the conductivity of the sensor decreases with increasing gas concentration. By simply converting the conductivity measurement of the circuit, the corresponding output concentration of the gas (ozone) can be obtained. The MQ-131 ozone sensor has such a high sensitivity to ozone, but it is also partially sensitive to strong oxidants such as Cl_2 , NO₂, etc. However, it reacts to organic interfering gasses in the opposite way, by slightly increasing the conductivity of the circuit.

The proposed portable detector was constructed using two MQ-131 ozone sensitive gas sensors, a 128x64 pixel 1.8" LCD graphic display, a DHT22 temperature and humidity sensor, 2x 18650 3.7 V 3500 mAh Li-Ion battery, a Li-Ion battery charging control circuit, a Li-Ion battery status indicator and an Arduino Nano R3 microcontroller with ATmega328P chip. A control program in C++ programming language was developed for the control unit. For the mentioned components, a package made of colored PETG material was designed in Autodesk AutoCAD and printed using a Prusa i3 MK2S MMU1 3D printer (see Fig. 5).



Fig. 5 Designed and constructed portable ozone detector

GC-MS

The effect of ozone on the surface of various materials has been previously investigated. As can be seen from the literature review, ozone influences the materials that are exposed to it. These materials then exhibit a change in the chemical structure of the substances that make up the surface layers of these materials. Most often, ozone causes oxidation of the substances contained in the materials, resulting in the formation of various oxides or peroxides (Destaillats et al., 2006; Shu et al., 2011; Rim et al., 2016; Kruza et al., 2017; Louis, 1967). The most common methods chosen for the analysis of samples of materials exposed to ozone were HPLC-MS or

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GC-MS for this reason GC-MS analysis was chosen for the purpose of our experiments.

To determine the effect of exposure of the selected materials to the different ozone concentrations the samples were exposed to 1400 ppm and 3000 ppm of ozone for 1, 3, 5, 7, 9 and 24 hours. These samples were subjected to GC-MS analyses to determine the chemical composition. Samples for GC-MS analyses were treated prior to actual analysis according to US EPA Method SW-846 Test Method 3550C: Ultrasonic Extraction for the extraction of non-volatile and semi-volatile compounds from solid materials (U.S. EPA, 2007). The weighed samples were extracted with hexane, methanol and dichloromethane in an ultrasonic bath for 30 minutes or swabbed from the sample surfaces and then extracted in the ultrasonic bath. The samples were then filtered through a 0.45 μ m PTFE filter and concentrated using a Kuderna-Danish apparatus. The concentrated 1 ml samples were quantitatively transferred into 2 ml vials for the GC-MS analysis.

All samples were analyzed on a Thermo Finnigan Trace GC-MS with an AS2000 autosampler and a Thermo DSQ II mass detector with single quadrupole. The GC-MS analysis conditions for the extracts in methanol/hexane/dichloromethane were as follows: capillary column DB-5ms UI, 60 m x 0.32 mm x 0.25 um, temperature program 50 °C (3 min), 15 °C.min⁻¹, 300 °C (5 min), carrier gas flow rate 0.7 ml.min⁻¹ (helium), splitless, injector temperature 250 °C, ion source temperature 250 °C, transferline temperature 250 °C. Used chemicals: Methanol UHPLC, suitable for mass spectrometry (MS), CAS: 110-54-3, Merck; n-Hexane for gas chromatography ECD and FID, CAS 110-54-3, Merck; Dichloromethane p.a., CAS: 75-09-2, Merck.

Scanning electron microscopy

The analysis of sample morphology with and without ozonification was investigated using scanning electron microscopy (SEM) with a FEG electron gun (FIB-SEM TESCAN LYRA3GMU, Czech Republic) at the acceleration voltage of 5 kV. Prior to the SEM measurement, the sample was placed on a carbon conductive tape and coated by 10 nm thin gold layer in a sputter coater (Quorum Q150R S, United Kingdom). Each sample was analyzed at 6 different magnifications.

Results and discussion

Calibration of detectors

The assembled detector was calibrated using an FTIR spectrometer. The instrument was placed in glass container and exposed to various concentrations of ozone which were simultaneously measured using spectrometer. the FTIR The measurements show that the response of the MQ-131 sensors has a power-law dependence on the detector response. For calibration purposes, the detector response was calculated as the ratio of the resistance of the measuring circuit in clean air R0 to the resistance of the measuring circuit in the presence of ozone for a given concentration Rs as shown in Fig. 6.

Calibration of the sensor for ppm concentrations is shown in Fig. 7.



Fig. 6 The effect of ozone concentration on the change in sensor resistance ratio Rs/R0 for ozone concentrations in the range of 10-1000 ppb at 25 °C and 0 % humidity

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Fig. 7 Effect of ozone concentration on the change in sensor resistance ratio Rs/R0 for concentrations in the range of 10-1000 ppm at 25 °C and 0 % humidity

In addition to the possibility of using the calculation of the detector response depending on the ratio of resistances, the actual sensor voltage measured by the microcontroller can be used for calibration. The detector response above the manufacturer's maximum concentration of 1000 ppm was also tested and the detector response could be observed for concentrations up to 1400 ppm but with lower detector sensitivity as a larger increase in concentration caused a small change in detector voltage, see Fig. 8.

The principle of operation of the MQ-131 sensor shows that the response of this semiconductor detector will depend on the ambient atmospheric conditions, especially temperature and humidity. For this reason, the response of the sensor was found to be within the temperature range of -10 to 50 °C and humidity range of 30 % to 85 %.

The next detector tested was the commercially available GasAlertMicro5 detector with the Cl_2 sensor. The Cl_2 sensor used in this detector responds to ozone in addition to chlorine, but with lower sensitivity. The detection range of this sensor is only 0-50 ppm for Cl_2 and in the case of ozone it responds to concentrations of 0-93 ppm O₃ but with lower sensitivity than our handheld detector, see Fig. 9.



Fig. 8 The effect of ozone concentration on the change in sensor voltage in the range of 0-1400 ppm at 25 °C and 0 % humidity

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Fig. 9 Influence of temperatures from -10 °C to 50 °C and 30 %, 60 % and 85 % humidity on the sensor resistance ratio Rs/R0 $\,$



Fig. 10 Measured response of chlorine sensor 0-50 ppm Cl_2 in GasAlertMicro5 to ozone concentration 0-93 ppm at 25 °C and 0 % humidity

Degradation of material surface

1. Scanning electron microscopy

It is well known that ozone can due to its strong oxidative properties cause the degradation of certain materials. This effect is observed mainly at higher concentrations of ozone or after longer or repeated exposure of the material to ozone. It has been described that ozone causes corrosion of metal surfaces (Yang et al., 1993), damages natural materials and textile fibers (Drahokoupilová, 2018; Siswanto et al., 2020) or degrades plastics and rubbers. In this project, the effect of ozone on the degradation of 6 different materials - plexiglas, linoleum, nylon fiber (carpet), cotton fiber (T-shirt), chipboard and pine wood - was studied. These materials were exposed to two concentrations of ozone (1400 and 3000 ppm) for various times (1, 3, 5, 7, 9 a 24 h). The changes of morphology were subsequently analyzed by scanning electron microscope. As it can be seen in the panels of SEM pictures, ozone at concentration of 1400 ppm does not cause any damage to the tested materials (data not shown), however, when increased to 3000 ppm, certain changes in the morphology of materials were observed (fig. 11-13). While natural materials

are rather unaffected by 3000 ppm ozone, plastic samples undergo degradation.

The shortest time (1 h) of exposure to ozone needed for any changes of the surface morphology was measured by the linoleum samples (fig. 13 B). Already after 5 h of ozonization appeared tiny cracks in the surface of the linoleum. With prolonging time, the damage of the surface deepened. Plexiglas (fig. 12 A) was damaged by ozone at 3000 ppm concentration first after 7 h, when microscopic cracks appeared in the sample, which were visible at the highest magnification used (fig. 12 A, sample area 10 µm²). The most dramatic morphological changes of the plexiglas surface were observed after 24 h exposure to ozone. At this condition, the surface of the sample was covered with microscopic apertures. Similar effect was observed on the sample of synthetic fiber (fig. 12 B), which lost its smooth character after 7 h of exposure to ozone and was rather rough with cracks and holes on its surface. The longer the exposure, the greater the damage of the fiber was observed.



Fig. 11 Scanning electron microscopy images of chipboard (A) and cotton fiber (B) samples exposed to ozone for 0-24 h at concentration of 3000 ppm. Different magnifications are indicated as the depicted area of the sample (5 mm² to 10 μm²)

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Fig. 12 Scanning electron microscopy images of plexiglas (A) and synthetic fiber (B) samples exposed to ozone for 0-24 h at concentration of 3000 ppm. Different magnifications are indicated as the depicted area of the sample (5 mm² to 10 μm²)



Fig. 13 Scanning electron microscopy images of pine wood (A) and linoleum (B) samples exposed to ozone for 0-24 h at concentration of 3000 ppm. Different magnifications are indicated as the depicted area of the sample (5 mm² to 10 μm²)

2. GC-MS

The results of the analyses of swabs and extracts of samples not exposed and exposed to ozone show a significant effect of ozone on the surface of the tested materials, especially for chipboard, wood, linoleum and carpet and partly also for Plexiglas and cotton t-shirt. It is evident from the data that when ozone is applied to the surface of these materials, the substances present in the materials are partially oxidized, resulting in a greater formation of peroxides, oxides, esters and ethers of the primary compounds - see further. The data also show that the longer the ozone is applied to the surface of the materials, the more easily the substances are released from the material, which may be due to the disruption of the integral structure of the surface of these materials.

In the samples analyzed, especially of a plastic nature (PMMA acrylic glass, linoleum, carpet), various plasticizers were primarily detected, and in the case of wood and chipboard samples, the primary compounds present were various terpenes (limonene, pinene, carene), various resins, etc. Formaldehyde and urea originating from ureaformaldehyde resins used for gluing were also detected in chipboard. In the case of the cotton T-shirt sample, mainly carboxylic acid esters and the dyes used were analyzed. In addition, the samples that were exposed to ozone for varying periods of time from 1 hour to 24 hours showed the presence of substances that may have been formed mainly by oxidation of chemical compounds contained in the samples. Higher concentrations of various esters and ethers or oxides and peroxides of these substances were detected in the tested samples. The measured spectra also show an increase in peak intensity of the individual detected substances with increasing ozone exposure time.

In the case of samples exposed to ozone concentrations of 1400 and 3000 ppm for 1, 3, 5, 7, 9 and 24 hours, the following substances were detected in samples of plastic nature (plexiglass, linoleum, carpet): Butylated Hydroxytoluene, 3.5.5-Trimethyl-1-hexanol, 4-Hydroperoxy-2nonenal, butyl methyl ester of terephthalic acid, 3-(2-Hydroxyphenyl)propionic acid, 2-ethylhexyl ester of Methylchlorophenoxyacetic acid, etc. In the case of wood and chipboard samples, the following substances were analyzed: a-Pinene oxide, butoxyethyl ester of 2,4,5-Trichlorophenoxyacetic acid, 2,3-diphenyl-6-Quinoxalinecarboxylic acid, dinonyl ester of 1,2-Cyclohexanedicarboxylic acid, etc. For the cotton T-shirt sample, primarily carboxylic acid esters and 3-(2-Hydroxyphenyl) propionic acid were analyzed.

As an example, chromatograms of the extracts of a sample of linoleum unexposed to ozone and exposed to ozone at a concentration of 3000 ppm for 1, 3, 5, 7, 9 and 24 hours were selected. GC-MS chromatograms of samples extracted with dichloromethane are shown in Fig. 14, samples extracted with methanol in Fig. 15 and samples extracted with hexane Fig. 16.



Fig. 14 GC-MS chromatograms of samples of linoleum extracted in dichloromethane unexposed to ozone (0) and exposed to 3000 ppm of ozone for 1, 3, 5, 7, 9 and 24 hours

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Fig. 15 GC-MS chromatograms of samples of linoleum extracted in methanol unexposed to ozone (0) and exposed to 3000 ppm of ozone for 1, 3, 5, 7, 9 and 24 hours



Fig. 16 GC-MS chromatograms of samples of linoleum extracted in hexane unexposed to ozone (0) and exposed to 3000 ppm of ozone for 1, 3, 5, 7, 9 and 24 hours

Conclusion

Ozone is an extremely reactive molecule, which could be potentially used for the decontamination of environments. However, ozone is also a very toxic compound for people and therefore it is important to easily detect its concentration in the environment. Thus, we designed and built a portable detector, which can be used with great advantage as a cheaper alternative to commercially available ozone analyzers. This device could be potentially used to detect ozone in households or working places and determine the safety of such environments.

Since ozone is a strong oxidative molecule, it could potentially degrade materials in the surroundings. Using GC-MS, we confirmed that materials undergo dose- and time-dependent chemical degradation by ozone, which was evidenced by the increased presence of different esters, ethers, oxides and peroxides of common substances present in the studied materials. Plastic materials are more susceptible to ozone, since these oxidation products were more increased after shorter exposure of PMMA glass or linoleum to ozone. This is probably due to complete decomposition of the tested substances already before the material extraction. However, ozone does not cause any

dramatic microscopic changes of the material surface structure, which was observed in SEM.

Given the presented data, ozone could probably be used for the decontamination of environments, because it does not cause any dramatic damage to commonly occurring materials. Given this fact, ozone could probably be used also for the decontamination of public places with high risk of microbial contamination, such as public transport vehicles, waiting rooms, hospital areas or motor vehicles of the Integrated Rescue System. Since ozone could potentially be harmful to people, when occurring at too high concentrations in the air, we also developed a ready-to-use ozone detector, which could be used to determine the safety of ozonized places. Next, it is important to study the effectiveness of ozone in the decontamination of surfaces and air.

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