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Studies on the nicotine degradation in swimming pool water

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ABSTRACT

The presented research assumes the implementation of experimental studies on the degradation, removal, and transformation of nicotine during processes that occur in swimming pool systems, including chlorination, UV radiation, and ozonation. Nicotine decomposition by-products during these processes have been also identified. The research has documented that nicotine in contact with sodium hypochlorite undergoes degradation that efficiency depends on the contact time, chlorine dose, and type of water matrix. The decrease of nicotine concentration over the time of UV radiation was observed. It achieved about 30% removal rate after 60 min. The effect of the type of aqueous matrix was not as significant in the case of UV irradiation and ozonation as in the chlorination process.

Keywords: Swimming pool; Micropollutants; Nicotine; Chlorination; Ozonation; UV radiation

1. Introduction

The continuous development of analytical tools makes it possible to separate various compounds from environmental samples and to selectively analyze them with increased efficiency, even at trace concentrations. These organic micropollutants, classified as the contaminants of emerging concern (CECs) group, are therefore increasingly the subject of research by scientists from around the world. CECs include several types of chemicals, for example, persistent organic pollutants (POPs), pharmaceuticals and personal care products (PPCPs), and endocrine-disrupting chemicals (EDCs). The growing awareness that they can pose a real threat to the environment and future generations makes these compounds a leading problem in environmental engineering. Research on this topic are very important because the risk that CECs pose to human health and the environment is not yet fully understood. The variety of chemicals labeled as CECs leads to a variety of concerns.

Numerous studies have shown the presence of CECs in effluents from sewage treatment plants, surface waters, tap water, and even bottled water [1–5]. Swimming pool water is of particular interest in this area due to the specific nature of this aquatic environment. Constant water recirculation and its chlorination is the basis of the modern technology of swimming pool systems (Fig. 1). Currently used methods of pool water treatment are not effective in removing most organic microcontaminants. CECs in swimming pool water can be therefore accumulated. They may be also a source of the disinfection by-products (DBPs) formation. The most common procedure of disinfection that efficiently eliminates microorganisms from swimming pool water is chlorination. However, the reaction of active chlorine with dissolved organic matter produces numerous organochlorine compounds. As methods supporting disinfection in swimming pool technology UV radiation and ozonation are used (Fig. 2).

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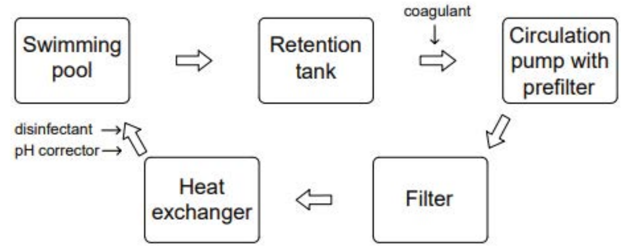


Fig. 1. Scheme of the basic swimming pool water treatment system.

The assessment of CECs occurrence in the pool water is not obligatory according to the applicable regulations [6–8]. Thus, information on the occurrence of these compounds in the swimming pools is quite limited. However, the presence of CECs in swimming pools should be of particular concern, as swimmers are exposed to different routes of exposure to trace chemical contaminants, including accidental ingestion, inhalation, and dermal absorption. There are a lot of researches focusing on the occurrences of DBPs [9–13]. However, some authors have concluded that further researches are needed to evaluate potential health risk not only from DBPs but also from other chemicals occurring in swimming pools [12,13].

In recent years, some studies have been carried out on the presence of PPCPs, FRs (flame retardants), and parabens in swimming pools [14–19]. Among others, they showed the occurrence of non-steroidal anti-inflammatory substances, stimulants, psychotropic agents, antibiotics, β-blockers, illicit drugs, cosmetics ingredients with the ability to absorb UV radiation, phosphor-organic flame retardants, insect repellents, and parabens used as preservatives in cosmetics, medicines, and food, as well as active substances in antiperspirant and anti-dandruff products. During initial research, the authors of this paper carried out non-target analyzes of swimming pool water samples by the use of gas chromatography coupled with mass detector GC-MS(EI). Based on the interpretation of initial research results using the NIST 17 mass spectral library, 100's of CECs have been identified with more than 70% probability, including pharmaceuticals, phytochemicals, hormones, industrial additives, vitamins and their derivatives, cosmetic ingredients, anabolic steroids, pesticides, stimulants, opioids, food additives, and aroma compounds. The presence of some of these substances in swimming pools has not yet been demonstrated in any literature. Nicotine and its metabolites were among the most commonly identified compounds in the preliminary studies with a concentration in range 1.2 ng/L – 27.4 μg/L. Nicotine is a non-prescription drug to which all members of contemporary society are exposed either through direct or passive smoke inhalation. It is estimated that 5% of nicotine absorbed is unchanged excreted into the urine together with its metabolites: cotinine (10%) and 3′-hydroxy derivative (35%) [20–22] It is also commercially available in some pharmaceutical and botanical products [23–25]. The presence of nicotine was proved in the effluent from sewage treatment plants in Montana, Barcelona, and in 16 plants of northeast Spain [26–28], in eight rivers (four located in the center of Spain and four near Barcelona) [29,30], in surface waters in

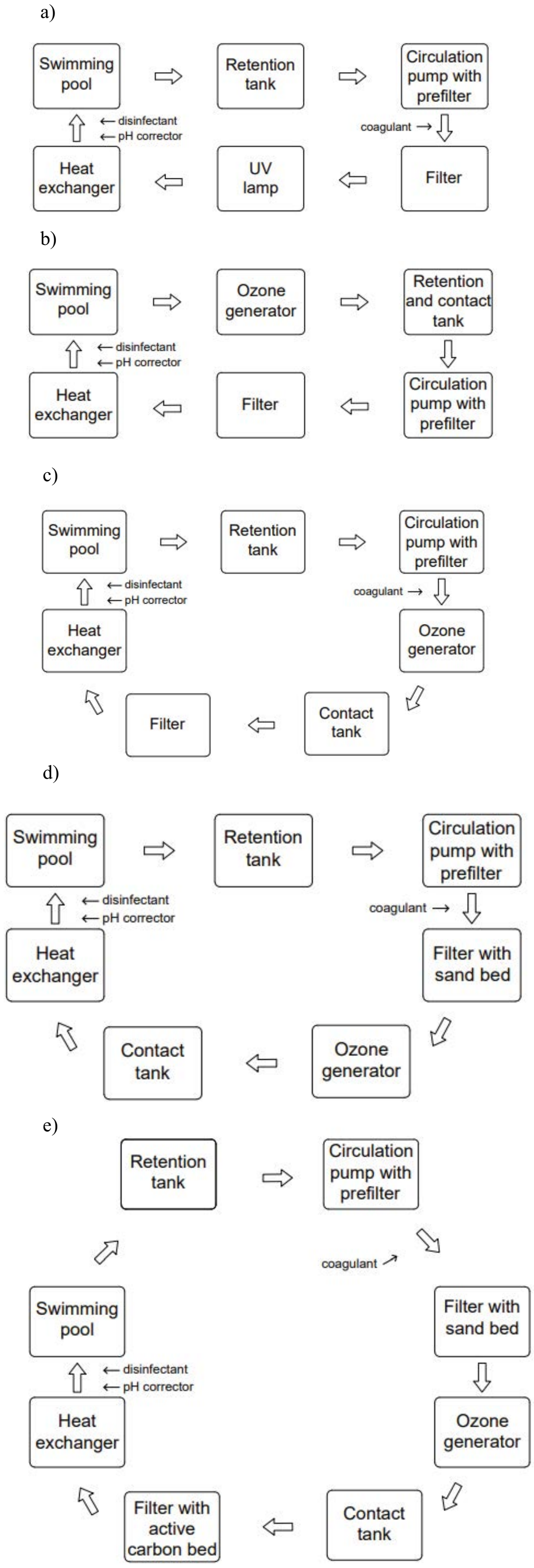


Fig. 2. Schemes of the swimming pool water treatment systems with the disinfection support using (a) UV radiation and (b–e) ozonation.

Canada, Switzerland, and Croatia [31–33], in groundwaters of Spain near Barcelona and in Montana [26,34]. Its common occurrence in the water environment points out that nicotine survives a conventional treatment process and persists in potable-water supplies [35].

Therefore, the presented research assumes the implementation of experimental studies on the degradation, transformation, and removal of nicotine during processes that occur in swimming pool systems, including chlorination, UV radiation, and ozonation. Nicotine decomposition by-products have been also identified.

2. Material and methods

2.1. Materials and reagents

The analytical standard of nicotine of purity grade >99% was supplied by Merck (Darmstad, Germany). Properties of the studied organic micropollutant are summarized in Table 1. Organic solvents: methanol and acetonitrile of purity grade >99.8% and >99.5%, respectively, by Avantor Performance Materials Poland S.A. (Gliwice, Poland) were also used. Disposable Supelclean™ ENVI™-18 tubes packed with an octadecyl bed of 1.0 g by Supelco were applied to solid-phase extraction (SPE). 0.1 mol/L of HCl (purity grade >99.8%) and 0.1 mol/L of NaOH (purity grade >99.6%) were used to adjust pH during the experiments. Sodium hypochlorite was used as a 15% aqueous solution purchased from Chemoform (Pruszcz Gdański, Poland).

2.2. Analytical procedure for the determination of nicotine

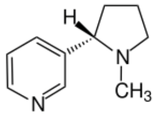
Due to the lack of reference methods for the determination of micropollutants in the swimming pool water environment, authors have developed their own procedure, based on the method presented in paper [36].

The isolation of micropollutants from the water matrix was carried out by SPE in tubes filled with non-polar adsorbent C18. The bed was firstly conditioned in sequence with 5 mL of methanol and 5 mL of acetonitrile. Then it was washed with 5 mL of deionized water. Afterwards, 20 mL of water sample was applied. Extraction was carried at a consistent and reduced flow rate of ~1–2 drops/s. After extraction, the bed was dried for 5 min under vacuum. The extract was eluted in the first step with 1.5 mL of ACN and after with 1.5 mL of MeOH.

The extracts were analyzed using a gas chromatograph coupled to mass spectrometry (GC/MS) with electronic ionization, model 7890B by Agilent Technologies (Santa Clara, United States). The separation of the target compound was carried out by a SLB™ – 5 ms capillary GC Column Sigma-Aldrich (Poznań, Poland) with an internal diameter of 0.25 mm, a length of 30 m, and a layer thickness of 0.25 μm. The oven temperature program was as follows: 80°C (6 min), 5°C/min–260°C, and 20°C/min–300°C. The temperature of the transfer line was 250°C. The support phase was helium with a flow of 1.1 mL/min. Sample injections of 1 μL were performed automatically by the use of the autosampler model no. G4513A by Agilent Technologies (Santa Clara, United States). The mass detector operated in the Full Scan Mode in the range of 50–700 m/s. Quantification was done by interpretation of the mass spectra obtained by chromatographic analysis using the NIST v17 mass spectral library and comparing the response of the mass detector with the standards response. To identify the compounds, also the retention times were compared with reference standards. Chromatogram obtained for a blank sample of the nicotine analytical standard is presented in Fig. 3.

Validation parameters of the chromatographic method are listed in Table 1. The detection limit (LOD) and quantification limit (LOQ) were calculated from chromatograms of swimming pool water samples: the LOD was the

Table 1
Properties of the nicotine and parameters of the chromatographic method

Properties of nicotine	Molecular formula	$C_{10}H_{14}N_2$
	Molecular weight (g/mol)	162.23
	CAS number	54–11–5
	NIST number	232,303
	Structural formula	
	LD_{50} (mg/kg)	1.0÷1.5
Parameters of the chromatographic method	Linearity range (μg/L)	0÷100
	Correlation coefficient R^2	0.9875
	Recovery ± SD (%)	84 ± 4.6
	LOD (ng/L)	0.42
	LOQ (ng/L)	1.16
	Retention time ± SD (min)	16.397 ± 0.056
	m/z top peak	84
	m/z second highest	133
	m/z third Highest	162

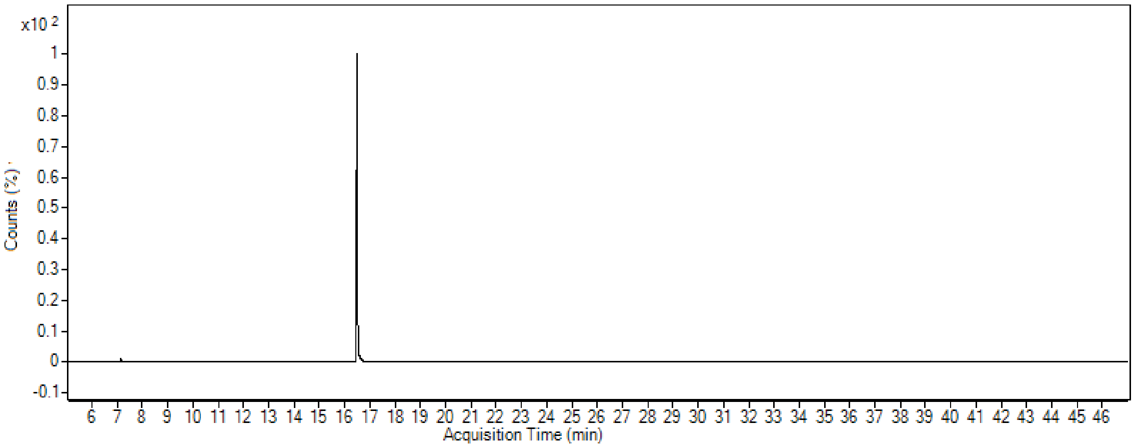


Fig. 3. Chromatogram obtained for a blank sample of the nicotine analytical standard.

concentration with a signal/noise ratio of 3 and the LOQ was the concentration with a signal/noise ratio of 10.

Each sample was analyzed five times. The obtained results are the arithmetical means of repetitions. The marked error bars presented in figures were estimated based on the standard deviation and did not exceed 5%.

2.3. Analysis of water parameters

The following parameters of water matrices were analyzed: pH and conductivity measured by the use of multifunction meter CX-461 by ELMETRON (Zabrze, Poland), total organic carbon (TOC) measured by the use of TOC-L Analyzer, Shimadzu (Kyoto, Japan), the absorbance in wavelength 254 nm with an optical path length of 1 cm (UV_{254}) measured by UV-VIS Cecil 1000 Analytik Jena AG (Jena, Germany), nitrate content by Hach spectrophotometer DR 3900 with RFID technology (Loveland, United States) and water hardness determined by the complexometric method. Free chlorine and combined chlorine during the experiment were measured by Hach Pocket Colorimeter TM II (Loveland, United States).

2.4. Water solutions for experiment

Nicotine analytical standard was dissolved in three various water matrix (deionized water, tap water, or swimming pool water), obtaining the concentration of nicotine equal to 1 µg/L. The pH of the prepared aqueous solutions was adjusted to 7.0 using 0.1 mol/L HCl or 0.1 mol/L of NaOH in order to reproduce the actual environmental conditions of swimming pool water. Tap water and swimming pool water applied as matrices for experiments were dechlorinated by aeration for 5 d prior to each experiment. The parameters of the applied water matrices are summarized in Table 2.

2.5. Chlorination procedure

Prepared water solutions were treated for 1, 5, 15, 30, and 60 min with a 15% solution of sodium hypochlorite at room temperature. Three different doses were used, obtaining the free chlorine concentration between 0.3 ÷ 0.6 mg/L, which meets the requirements of the free chlorine concentration for swimming pool water [7].

2.6. UV irradiation procedure

The process of UV light irradiation of the prepared water solutions was conducted in a glass reactor with a volume of 700 mL. The mercury medium pressure UV Lamp with a power of 150 W has been used, produced by Helios Quartz (Novazzano, Switzerland). It operated in DURAN 50 sleeve, with radiation flux equal to 24.8 W and 333 mol quanta/h. The lamp was placed in the reactor by the use of a glass jacket, cooled by tap water. The temperature of the water solution during the irradiation process did not exceed 20°C ± 1°C. A magnetic stirrer was used to keep the water solutions in a constant move. Samples for chromatographic

Table 2
Parameters of water matrices

Parameter	Deionized water	Tap water	Swimming pool water
Conductivity (µS/cm)	0.5	65.4	2,754.3
pH (–)	7.0	6.8	7.4
TOC (mg/L)	0.0	1.9	17.3
UV_{254}	0.00	0.008	0.056
Nitrate content (mgNO_3^-/L)	0.0	0.9	19.2
Water hardness (mmol/L)	0.00	1.15	2.40

analysis were taken after 1, 5, 15, 30, and 60 min of UV irradiation.

2.7. Ozonation procedure

Ozone was introduced in the prepared water solutions of volume 1 L by the use of a ceramic diffuser from an O_3 generator ozoner FM500 WRC Multiozon (Sopot, Poland). The concentrations of ozone introduced were equal to 0.6, 1.1, and 1.7 mg/L. They were measured by the use of the photometric method O_3 Spectroquant® by Merck (Darmstad, Germany). The applied ozone doses were about the requirements for swimming pool systems (0.8–1.5 mgO_3/L). The ozonation was stopped after 5 min by the removal of ozone that did not react by the introduction of 24 mmol/L of Na_2SO_3. In order to omit the influence of light on the decomposition of tested compounds all experiments were conducted in a dark chamber.

3. Results and discussion

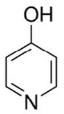
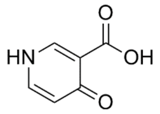
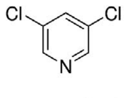
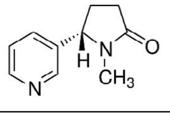
A series of bench-scale tests were conducted to determine the fate of nicotine in swimming pool installations. This study determined the stability of nicotine in this specific system. Aqueous samples of nicotine have been subjected to typical swimming pool water conditions in order to evaluate how it degrades. Fig. 4 presents the influence of different factors on the nicotine concentration in tested aqueous solutions. As shown in Figs. 4a and b, nicotine concentrations decreased significantly both over time and with the increase in chlorine dose, however, depending on the composition of the aqueous matrix. The efficiency of nicotine removal during chlorination was the best in deionized water and the lowest in swimming pool water. The removal efficiency after 60 min of chlorination in deionized water was 45% higher than in swimming pool water. In a deionized water matrix, all the chlorine dose is used to degrade nicotine. Meanwhile, in the pool water, there are many other organic compounds (TOC of blank swimming pool water matrix was equal to 17.3 mg/L) which also reacts with chlorine.

In the deionized water matrix samples (where nicotine was the only compound reacting with chlorine), the combined chlorine content was measured. It enabled to assess indirectly the potential of nicotine for disinfection by-products formation. The obtained values ranged from 0.04 to 0.08 mg/L. Considering the permissible value of combined chlorine in swimming pool water equal to 0.2 mg/L [7], these results indicate the high potential for nicotine to affect this important parameter of swimming pool water quality.

Fig. 4c shows the decrease of nicotine concentration over the time of UV radiation. It achieved about a 30% removal rate after 60 min. The effect of the type of aqueous matrix was not as significant in this case as for chlorination. The decomposition of the nicotine molecule occurred as a result of direct photolysis or during the reaction with reactive species, like hydroxyl radicals generated by the UV irradiation of water. The efficiency of nicotine removal during UV irradiation was much lower than during chlorination in the case of the deionized water matrix. However, for the swimming pool water matrix, the removal rates for UV irradiation and chlorination after 60 min were closer than for deionized water.

It should be noted that UV lamps currently used in swimming pool water systems act as flow devices. The contact time in a real installation is therefore tens of seconds to several minutes. Based on the results obtained, it is worth considering the possibility of extending the contact time

Table 3
Identified intermediates produced during the experiment

Name	CAS number	Molecular weight (g/mol)	Structural formula	Probability of identification (%)
4-Hydroxypyridine	626–64–2	95.10		71
5-Chloro-3-hydroxypyridine	74115–12–1	129.54		83
4-hydroxynicotinic acid	609–70–1	139.11		79
Myosmine	532–12–7	146.19		74
3,5-Dichloropyridine	2457–47–8	147.99		87
Cotinine	486–56–6	176.22		72

of UV rays with swimming pool water in swimming pool installations.

An interesting phenomenon was also observed during UV radiation. In the initial phase of the process, nicotine removal was the most effective in deionized water, and least effective in swimming pool water, the same as it was in the case of chlorination. However, after about 15 min there is a rapid increase in efficiency of nicotine removal in the pool water matrix and finally, after 30 min, the nicotine concentration in the swimming pool water matrix is the lowest among all matrices tested. The organic matter present in the pool water could have been a precursor to create free radicals [37], capable of breaking down nicotine. Such radicals could be formed, for example, from nitrates [38] present in swimming pool water (19.2 mg NO_3^-/L in blank swimming pool water matrix).

The effect of matrix type on nicotine removal in the ozonation process was insignificant, close to the error limits. From the range of acceptable doses for swimming pool water, the optimal dose for nicotine removal was determined as equal to 1.1 mg/L. The efficiency of nicotine removal in ozonation was lower than the efficiency of chlorination and

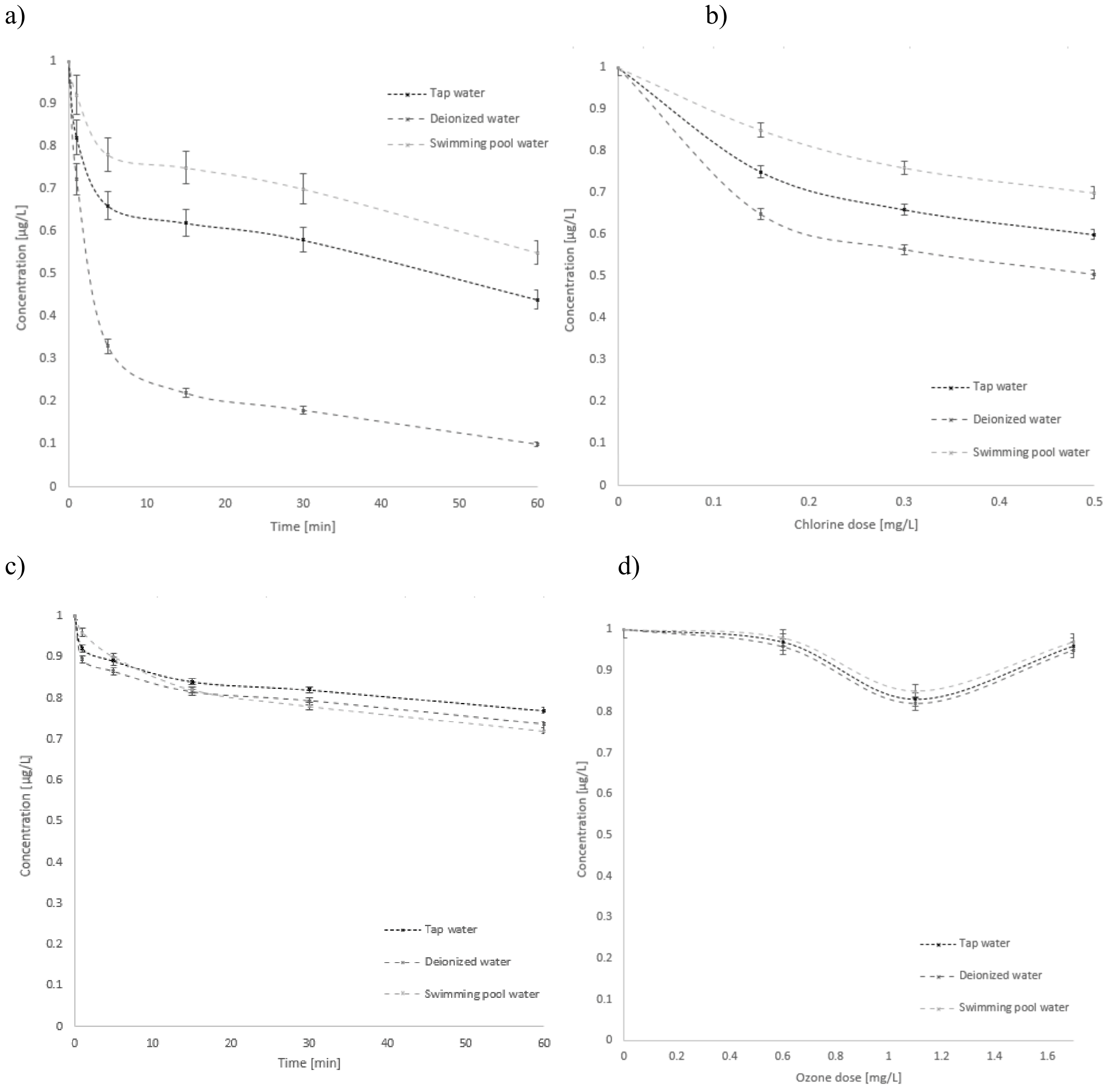


Fig. 4. Effect of (a) time of contact with chlorine, (b) chlorine dose, (c) UV radiation time, and (d) ozone dose on nicotine content in different water matrices.

similar to the efficiency of UV radiation. Because ozone molecules are very unstable in water, it transforms from O_3 to O_2, leading to the formation of reactive radicals OH^- [39]. Due to ozone instability in water and its self-destruction, its efficiency at the very beginning (shortly after its introduction into water) is high but then it weakens.

Qualitative identification of intermediate compounds and byproducts of the applied processes was performed. It can allow the identification of the nicotine reaction mechanism and its degradation pathways in swimming pool systems. In addition, the intermediate compounds may be more toxic than the initial compound [40]. Therefore, it is important to identify the intermediate final compounds. Fig. 5 presents examples of obtained GC/MS chromatograms that were used to identify nicotine removal by-products. The nicotine peak is the one with the highest intensity in the acquisition time RT = 16.397 ± 0.056. Other peaks indicate the formation of by-products of its decomposition as they were not present in Fig. 3. Mass spectra from these peaks allowed the identification of some intermediates that

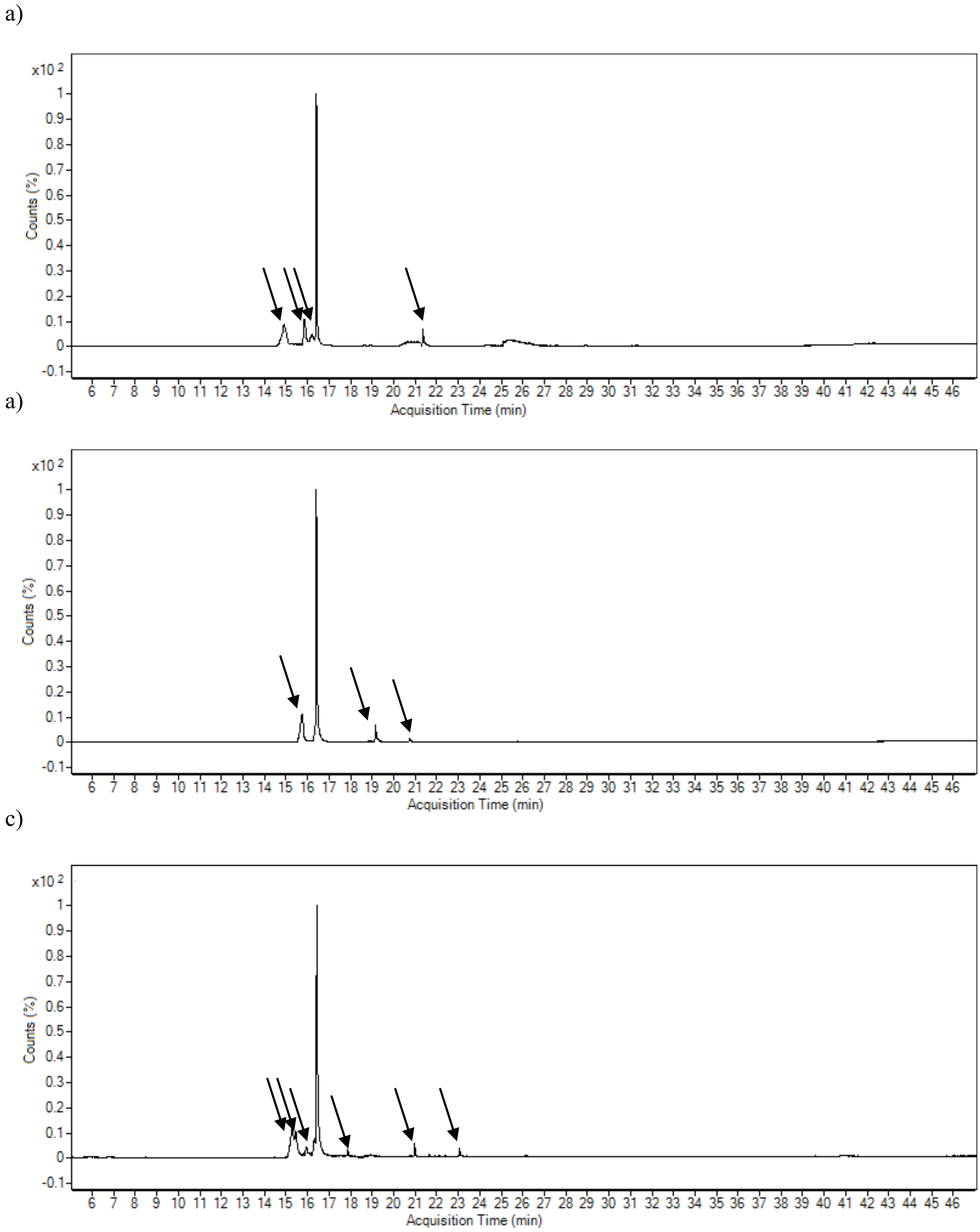


Fig. 5. (a–c) Examples of obtained GC/MS chromatograms presenting nicotine removal by-products.

are summarized in the Table 3 list of intermediates and by-products identified with a probability higher than 70%.

4. Summary and conclusions

- The research has documented that nicotine in contact with sodium hypochlorite undergoes degradation that efficiency depends on the contact time, chlorine dose, and type of water matrix. The effect of the type of aqueous matrix was not as significant in the case of UV irradiation and ozonation as in the chlorination process.
- Chlorination was the most effective among tested processes of nicotine removal.
- The decrease of nicotine concentration over the time of UV radiation was observed. It achieved about a 30% removal rate after 60 min. The possibility of extending the contact time of UV rays with swimming pool water in swimming pool installations is worth considering.
- The optimal dose of ozone (1.1 mg/L) allowed for about 15% removal of nicotine from water.
- Six nicotine decomposition by-products have been identified in the post-processed samples: 4-hydroxypyridine, 5-chloro-3-hydroxypyridine, 4-hydroxynicotinic acid, myosmine, 3,5-dichloropyridine, and cotinine.

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