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BENZOFENONI I KOFEIN U VODENOJ SREDINI
BENZOPHENONES AND CAFFEINE IN AQUATIC ENVIRONMENT

IZVOD

Benzofenoni i kofein se koriste u proizvodnji kozmetičkih preparata. Pojedini benzofenoni su UV filteri, koji se primenjuju u proizvodnji krema za sunčanje, šampona, losiona, fiksatora u parfemima, dok se kofein, pored upotrebe u kozmetici, koristi najviše u proizvodnji prehrambenih proizvoda, kao i lekova. Njihov sadržaj u vodi, kako otpadnoj, tako i u vodi za piće, nije regulisan propisima. Prema pojedinim autorima mogu se smatrati antropogenim markerima. U ovom radu su sumirani rezultati nekoliko objavljenih studija na temu ponašanja benzofenona i kofeina, od mesta emisije (tretmana otpadnih voda), do mesta tretmana rečne vode koja može služiti kao izvor vode za piće.

Ključne reči: benzofenoni, kofein, tretman otpadnih voda, tretman vode za piće

ABSTRACT

Benzophenones and caffeine are widely used substances. Neither emission limit values, nor environmental quality standards for aquatic environment exist. Several authors suggest that these micropollutants can be considered as anthropogenic markers. This paper summarizes the findings of several previously published studies concerning behaviour of benzophenones and caffeine - from their point of emission (e.g. wastewater treatment process) to the point of surface water intake for drinking water treatment.

Key words: benzophenones, caffeine, wastewater treatment, drinking water treatment

1. UVOD

Benzofenon i njegovi derivati se najčešće primenjuju u sredstvima za čišćenje i održavanje lične higijene, zatim kao aditivi u plastici, fiksatori u parfemima, pojačivači mirisa i ukusa, kao i u proizvodnji pesticida i raznih lekova. Usled sposobnosti da apsorbuju UV zračenje, odnosno da ga spreče da ošteti miris ili boju proizvoda, benzofenoni imaju primenu i u proizvodnji sunčanih naočara, krema za sunčanje, sapuna, losiona i šampona (Pestotnik i sar. 2014). Poznato je dvanaest supstituisanih derivata benzofenona koji se koriste u proizvodnji komercijalnih preparata (Park i sar. 2013), od kojih je za neke Uredbom Evropske Unije br. 1223/2009 regulisan dozvoljen sadržaj u kozmetičkim proizvodima.

Kofein je alkaloid koji se najviše primenjuje u proizvodnji prehrambenih proizvoda (80%), lekova (16%) kao stimulans i diuretik, i u proizvodnji kozmetičkih preparata (OECD, 2014).

Generalno, benzofenoni i kofein dospevaju u vodenu sredinu usled upotrebe proizvoda široke namene koji ih sadrže, nepotpunim uklanjanjem tokom prečišćavanja otpadnih voda, putem ispusta netretiranih otpadnih

1. INTRODUCTION

Benzophenone and its derivatives are commonly used in personal care and pharmaceutical products. Since they are UV filters, they often can be found as ingredients in sunscreens, shampoos, lotions and perfumes (Pestotnik et al. 2014). Twelve substituted derivatives of benzophenones are known as common cosmetic ingredients (Park et al. 2013). European Union Act number 1223/2009 regulates the content of some of them in cosmetics. In addition, they are used as plastics additives and in pesticide manufacturing.

Caffeine is an alkaloid commonly used in food and beverage production (80%), pharmaceutical production (16%) as a stimulant and diuretic, and also in the production of cosmetics (OECD, 2014).

In general, a widely spread consumption of the above mentioned products ensures the simplest route for benzophenones and caffeine to reach water bodies, as a result of their incomplete removal during the wastewater treatment process, and poor waste and water treatment management. Transport through

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voda, kao i lošim upravljanjem otpadom. Transportom kroz porozne slojeve zemljišta ili sedimenta postoji mogućnost prodiranja u podzemne vode, koje mogu biti korištene kao izvorišta vode za piće.

2. PRISUSTVO BENZOFENONA I KOFEINA U VODENOJ SREDINI

U doktorskoj disertaciji Bogunović (2019a) je predstavljen detaljan literaturni pregled prisustva dva odabrana benzofenona i kofeina u različitim vodenim matriksima širom sveta. U tabeli 1 je dat sažet prikaz koncentracionih opsega u različitim vrstama vode, kao i broj država za koje su podaci citirani.

a porous soil and sediment enables them to reach the groundwater, which often serves as a source of drinking water.

2. PRESENCE OF BENZOPHENONES AND CAFFEINE IN WATER

In her PhD thesis, Bogunović (2019a) showcases a detailed literature review related to the presence of two selected benzophenones (benzophenone and benzophenone-3) and caffeine in water worldwide. A brief summary of concentration ranges in different waters has been shown in Table 1, along with the number of countries to which the data corresponds to.

Tabela 1. Prisustvo benzofenona i kofeina u različitim matriksima (Bogunović, 2019a)

Matriksi	Benzofenon (BP)		Benzofenon-3 (BP-3)		Kofein (CF)	
	c (µg/l)	Broj država	c (µg/l)	Broj država	c (µg/l)	Broj država
Sirova otpadna voda postrojenja za tretman otpadnih voda	<0,005-2,43	3	0,01-1195	11	0,1-230	8
Efluent postrojenja za tretman otpadnih voda	<0,003-0,78	3	<0,0028-231	11	nd-34,2	8
Površinska voda	0,03-0,71	6	0,002-44	14	<0,025-50	9
Podzemna voda	<0,01-3,45	1	0,0044-1,12	2	0,15-0,18	1
Voda za piće	0,26	1	nd-0,295	2	0,024-0,036	3

d – nije detektovano

Table 1. Occurrence of benzophenones and caffeine in water (Bogunović, 2019a)

Water matrices	Benzophenone (BP)		Benzophenone-3 (BP-3)		Caffeine (CF)	
	c (µg/L)	Number of countries	c (µg/L)	Number of countries	c (µg/L)	Number of countries
Influent of wastewater treatment plant	<0.005-2.43	3	0.01-1195	11	0.1-230	8
Effluent of wastewater treatment plant	<0.003-0.78	3	<0.0028-231	11	nd-34.2	8
Surface water	0.03-0.71	6	0.002-44	14	<0.025-50	9
Groundwater	<0.01-3.45	1	0.0044-1.12	2	0.15-0.18	1
Tap water	0.26	1	nd-0.295	2	0.024-0.036	3

nd – not detected

Na osnovu prikazanih podataka u tabeli 1, može se uočiti da su najviše koncentracije odabranih mikropolutanata izmerene u sirovoj otpadnoj vodi postrojenja za tretman otpadnih voda. Niže koncentracije su uočene nakon tretmana otpadne vode, odnosno u efluentu postrojenja za tretman otpadne vode, što znači da se supstance uklanjanju, ali nepotpuno. Takođe, benzofenoni i kofein su izmereni i u podzemnoj vodi i u vodi za piće, što znači da prolaze kako kroz prirodne barijere, tako i kroz tehnološke. Utvrđen je uticaj sezonskih varijacija

Based on the data presented in Table 1, it may be concluded that the highest concentrations of the selected micropollutants correspond to the wastewater treatment plant (WWTP) influents (e.g. raw wastewater). Lower concentrations have been observed in the effluent of WWTPs, indicating the incomplete removal of these micropollutants during the wastewater treatment process. The presence of benzophenones and caffeine has been confirmed in groundwater and tap water samples as well - suggesting that the observed micropollutants

na sadržaj odabranih mikropolutanata u otpadnim i površinskim vodama. Za benzofenone su mnogi autori potvrdili da su najviše koncentracije izmerene tokom letnjeg perioda, a kao uzrok toga navode prekomernu upotrebu sredstava za ličnu negu (Loraine i Pettigrove 2006; Pedrouzo i sar. 2009; Tsui i sar. 2014; Česen i sar. 2018). Najviše koncentracije CF u otpadnoj vodi su izmerene tokom zimskog perioda (Papageorgiou i sar. 2016).

U okviru rada na doktorskoj disertaciji Bogunović (2019a) je, prema saznanjima autora, u Srbiji prvi put uradila merenja ovih mikropolutanata u efluentu postrojenja za tretman otpadnih voda. Rezultati su prikazani na Međunarodnoj konferenciji Hemija i Životna sredina održanoj u Solunu 2019. godine (Bogunović i sar. 2019b). Ispitano je prisustvo BP, BP-3 i CF u efluentu tipičnog konvencionalnog postrojenja za tretman otpadnih voda koje primenjuje primarno i sekundarno prečišćavanje i u rečnoj vodi Dunava. Prisustvo benzofenona i kofeina je potvrđeno u ovim lokalno specifičnim matriksima, sa opsegom koncentracija od ng/l do nekoliko µg/l. Koncentracije BP, BP-3 i CF u efluentu postrojenja za tretman otpadnih voda, kao i u rečnoj vodi Dunava su u skladu sa koncentracijama pronađenim u literaturi (tabela 1). Zaključeno je da se ovi mikropolutanti ne uklanjaju potpuno konvencionalnim tretmanom otpadnih voda. U daljem radu ispitan je potencijal novih tehnologija za moguć unapređeni tercijarni tretman otpadnih voda.

3. PONAŠANJE BENZOFENONA I KOFEINA PROCESIMA PREČIŠĆAVANJA

Tehnologije testirane za uklanjanje benzofenona i kofeina iz efluenta postrojenja za tretman otpadnih voda su: adsorpcija na aktivnom uglju u prahu, koagulacija, kao i hibridni proces adsorpcija na aktivnom uglju u prahu/koagulacija i hibridni membranski proces adsorpcija na aktivnom uglju u prahu/ultrafiltracija (Bogunović, 2019a). Procena efikasnosti uklanjanja mikropolutanata odabranim procesima prikazana je u tabeli 2.

Tabela 2. Efikasnost ispitanih tehnologija za uklanjanje benzofenona i kofeina iz efluenta postrojenja za tretman otpadne vode (Bogunović, 2019a)

Procesi	Efikasnost uklanjanja	
	Benzofenoni	Kofein
Adsorpcija	Visoka	Visoka
Koagulacija	Niska	Niska
Adsorpcija/ koagulacija	Visoka	Visoka
Adsorpcija/ ultrafiltracija	Visoka	Visoka

Visoka efikasnost (>80%); Niska efikasnost (<40%)

successfully pass through natural and technological barriers. The content of the observed micropollutants was found to be closely correlated with seasonal variation. For example, summer periods correspond to the highest benzophenones concentration, most probably due to frequent use of personal care products (Loraine & Pettigrove 2006; Pedrouzo et al. 2009; Tsui et al. 2014; Česen et al. 2018). On the other hand, the highest concentrations of caffeine have been observed in the winter months (Papageorgiou et al. 2016).

By our best knowledge, Bogunović (2019a) was the first one in Serbia who measured the content of these micropollutants in the effluent wastewater treatment plant, as part of her PhD thesis. The resulting findings had also been presented in 2019 at the 17th International Conference on Chemistry and the Environment in Thessaloniki, Greece (Bogunović et al. 2019b). The presence of BP, BP-3 and CF was investigated in the effluent from a typical conventional WWTP that employs primary and secondary treatment, as well as in Danube river water. The presence of benzophenones and caffeine was confirmed in those locally specific matrices, with concentrations ranging between several ng/L to several µg/L. These results are in line with previously reported literature findings (Table 1). It was concluded that BP, BP-3 and CF could not be completely removed via conventional WWTP. The potential and the applicability of advanced technologies in the tertiary wastewater treatment process was further investigated.

3. BEHAVIOUR OF BENZOPHENONES AND CAFFEINE IN WATER TREATMENT PROCESSES

Technologies tested for benzophenones and caffeine removal from WWTP effluent include: powdered activated carbon adsorption, coagulation, hybrid process that employs powdered activated carbon adsorption and coagulation, and a hybrid membrane process that employs powdered activated carbon adsorption with ultrafiltration (Bogunović, 2019a). The estimated micropollutant removal efficiencies of the corresponding processes have been presented in Table 2.

Table 2. Removal efficiency of tested technologies for benzophenones and caffeine from WWTP effluent (Bogunović, 2019a)

Process	Removal efficiency	
	Benzophenones	Caffeine
Adsorption	High	High
Coagulation	Low	Low
Adsorption/ coagulation	High	High
Adsorption/ ultrafiltration	High	High

High efficiency (>80%); Low efficiency (<40%)

Može se zaključiti da su svi procesi efikasni u uklanjanju BP, BP-3 i CF osim koagulacije.

Poznato je da se rečna voda može direktno zahvatati i koristiti za pripremu vode za piće, a može se koristiti obalska filtracija kao prvi stepen obrade. Procesi hemijske degradacije (oksidacije, fotodegradacije), biodegradacije, kao i sorpcije na sedimentu, u zavisnosti od prirodnih uslova, mogu predstavljati efikasne barijere za dalji prolazak mikropolutanata u podzemne vode. Biodegradacija benzofenona i kofeina u smeši sa karbamazepinom u rečnoj vodi Dunava, primenom laboratorijskog test filtra po Sontheimer-u (Sontheimer, 1988) je eksperimentalno potvrđena (Bogunović i sar. 2017). Efikasnost uklanjanja BP, BP-3 i CF pri početnoj koncentraciji od 20 µg/l je 90-99%. Dobijene konstante biodegradacije su za BP 0,5 h^{-1}, BP-3 0,33 h^{-1} i CF 0,29 h^{-1}, za vremena poluraspada od 1,4 h, 2,1 h and 2,4 h, redom. Time je pokazano da rečna voda Dunava ima potencijal biodegradacije ovih mikropolutanata. U saradnji sa timom prof. dr Ivane Teodorović sa Departmana za biologiju i ekologiju, Prirodno-matematičkog fakulteta Univerziteta u Novom Sadu, urađeni su testovi toksičnosti pre i posle pomenute biodegradacije. Efikasnost procesa biodegradacije, u smislu ukupnog smanjenja toksičnosti, procenjena je standardnim testovima toksičnosti: *Daphnia magna* test akutne toksičnosti i test inhibicije luminiscencije sa *Vibrio fischeri*. Oni su pokazali smanjenje ukupne toksičnosti smeše mikropolutanata nakon prolaska kroz laboratorijski test filter. Rezidualna toksičnost može biti posledica nedetektovanih transformacionih proizvoda, ali, i bez obzira na nju, u zavisnosti od stepena emisije ovih supstanci, problem pseudoperzistentnosti može biti prisutan.

Ispitivanje transporta benzofenona i kofeina kroz sediment Dunava, primenom kolonskih testova, pokazalo je da je sediment za benzofenone efikasna barijera, dok CF lako prodire usled izražene mobilnosti (log Kow -0,07), i stoga predstavlja potencijalnu opasnost za zagađenje podzemnih voda.

Poznato je da se u tretmanu komunalne otpadne vode može primenjivati UV dezinfekcija u završnoj obradi. U obradi rečne vode, UV dezinfekcija se može primenjivati u početnoj fazi obrade. Zbog toga je ispitana fotodegradacija benzofenona u uslovima različitih model vodenih matriksa (Ivančev-Tumbas i sar. 2019). Ona je potvrđena, iako je poznata otpornost BP u tom smislu koji se pre svega koristi kao UV filter. Pokazano je da stepen fotorazgradnje zavisi od sadržaja soli i rastvorenog organskog ugljenika u vodi, što može biti značajno za učinak eventualno primenjene UV dezinfekcije vode.

U doktorskoj disertaciji Bogunović (2019a) je testirana primena hibridnih procesa kojima se direktno zahvaćena rečna voda može obraditi, a to su: adsorpcija na aktivnom uglju u prahu/koagulacija,

It can be concluded that all processes, except coagulation, successfully remove BP, BP-3 and CF from WWTP effluent.

It is well known that a direct intake of river water in the production of drinking water is possible. Frequently, riverbank filtration serves as a primary treatment stage. Processes such as chemical degradation (oxidation, photodegradation), biodegradation, sorption onto sediment, depending on the natural conditions, may also serve as effective barriers for the micropollutants transport towards the groundwater. The biodegradation of benzophenones and caffeine in a mixture with carbamazepine, has been experimentally confirmed in laboratory test filter (Sontheimer, 1988), in a Danube river water, by Bogunović et al. (2017). The obtained removal efficiencies for BP, BP-3 and CF (initial concentration of 20 µg/L) were 90-99%. The corresponding biodegradation rate constants are as follows: BP 0,5 h^{-1}, BP-3 0,33 h^{-1} and CF 0,29 h^{-1}, for half-times of 1,4 h, 2,1 h and 2,4 h, respectively. The results confirmed the biodegradation potential of the Danube river water with respect to the examined micropollutants. The efficacy of the biodegradation process, in terms of overall toxicity reduction, was estimated in collaboration with prof. Dr. Ivana Teodorović and her team at the Department of Biology and Ecology, at the Faculty of Sciences in Novi Sad. Two standard toxicity tests were used – *Daphnia magna* (immobilisation) and bioluminescence inhibition with *Vibrio fisheri*. The substantial reduction of the overall mixture toxicity in a laboratory test filter was shown. Residual toxicity was associated with the undetected transformation products. Despite that, pseudo persistence could still be occurring, depending on the degree of the micropollutant emissions.

The results of the column tests conducted on the transport of benzophenone and caffeine through the Danube river sediment, suggested Danube sediment to be an effective barrier for benzophenones, whereas this was not the case for CF. High mobility of CF (log Kow -0,07) allows for easy penetration through the sediment, thus potentially posing a threat to the groundwater quality.

UV disinfection can be applied as one of the final stages in the wastewater treatment process. When it comes to the drinking water treatment UV disinfection may be applied also in the initial stages. This was the reason why photodegradation of benzophenone in various model water matrices was tested (Ivančev-Tumbas et al. 2019). Despite the reported resistance of BP to UV-irradiation, it was successfully degraded. The results show that natural organic matter, anions and concentration level of BP influence the process. Such outcome could be significant for the assessment of UV disinfection efficiencies in different water matrices.

Bogunović (2019a) PhD thesis also examines hybrid process applications that can be applied in the case of

adsorpcija na aktivnom uglju u prahu/ultrafiltracija, kao i koagulacija/ultrafiltracija. U tabeli 3 je sažeto prikazana procena efikasnosti primenjenih procesa.

Tabela 3. Efikasnost ispitanih tehnologija za uklanjanje benzofenona i kofeina iz rečne vode (Bogunović, 2019a)

Procesi	Efikasnost uklanjanja	
	Benzofenoni	Kofein
Adsorpcija/ koagulacija	Srednja-Visoka za BP-3/ Niska-Visoka za BP*	Niska
Adsorpcija/ ultrafiltracija	Visoka	Srednja
Koagulacija/ ultrafiltracija	Visoka za BP-3/ Srednja za BP	Niska

Visoka efikasnost (>80%); Srednja efikasnost (40%-80%); Niska efikasnost (<40%); *Efikasnost uklanjanja zavisi od primenjenih procesnih kombinacija aktivnog uglja u prahu i koagulanta.

Hibridni proces adsorpcija/koagulacija se pokazao efikasan u uklanjanju hidrofobnih BP i BP-3, dok se za CF nije pokazao efikasan. Tokom hibridnih membranskih procesa (adsorpcija/ultrafiltracija i koagulacija/ultrafiltracija) se simultano odigrava dezinfekcija ultrafiltracijom i uklanjanje mikropolutanata, odnosno organskih materija primenom uglja i koagulanta. Ovi procesi su se pokazali efikasni u uklanjanju hidrofobnih BP i BP-3, dok je za CF efikasnost bila niska do srednja. Pri tome je uočen uticaj matriksa na njegovo uklanjanje, što ukazuje potrebu za dodatnim istraživanjima (Bogunović i sar. 2019b).

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direct surface water intake: powdered activated carbon adsorption/coagulation, powdered activated carbon adsorption/ultrafiltration, as well as coagulation/ ultrafiltration. The results of the study have been shown in Table 3.

Table 3. Removal efficiency of tested technologies for benzophenones and caffeine from river water (Bogunović, 2019a)

Process	Removal efficiency	
	Benzophenones	Caffeine
Adsorption/ coagulation	Medium-High for BP-3/ Low -High for BP*	Low
Adsorption/ ultrafiltration	High	Medium
Coagulation/ ultrafiltration	High for BP-3/Medium for BP	Low

High efficiency (>80%); Medium efficiency (40%-80%); Low efficiency (<40%); *The removal efficiency depends on the process combinations of powdered activated carbon and coagulant used.

The adsorption/coagulation hybrid process has shown to be efficient for the removal of hydrophobic BP and BP-3, which cannot be observed for CF. During hybrid membrane processes (adsorption/ ultrafiltration and coagulation/ultrafiltration), both disinfection by ultrafiltration and removal of micropolutants and/or organic matter using powdered activated carbon and coagulant are simultaneously performed. Such hybrid processes proved to be efficient for the removal of hydrophobic BP and BP-3, whereas the efficiencies were low-to-medium for CF. The effect of matrices has been observed in case of the CF, suggesting a need for further testing (Bogunović et al. 2019b).

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