

# POSTER PRESENTATIONS

Analytical and Environmental Chemistry

(AEC)





## **Qualitative and quantitative analysis of free amino acids in plant extracts**

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**Keywords:**

Plant extracts,  
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method,  
thin layer chromatography (TLC)  
method.

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**Abstract:** Newly interests for free amino acids have been peaked at its positive effects on immune system responsiveness to infection, blood -pressure reduction, relaxation, neuroprotection and modulation of chemotherapy. The aim of th is study was to determine the free amino acids in garlic, onion, purple onion, onion shallot, leek, bears garlic, fennel bulb, fennel leaf, rucola, corn salad, asparagus, black tea, green tea and artichoke commercially available from bosnian marketplaces by the simple, fast and economical methods. The qualitative analysis of free amino acids was performed by thin layer chromatography (TLC) method. The quantitative analysis of free amino acids was performed by spectrophotometric method (UV/VIS). 1.0 ml of each plant infusion was added to 0.5 ml of phosphate buffer solution (pH 8.04) and 0.5 ml of 2% ninhydrin solution containing 0.8 mg/ml of  $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ . The mixtures were placed on a boiling water bath until the color was changed and then samples were quickly cooled with cold water, and adjusted to 25 ml with distilled water. The absorbance of these blue -purple products was measured after 10 min. The results showed that the highest content of free amino acids was found in garlic (*Allium sativum* - bulb) 0.251 mg/g and the lowest content was found in fennel leaf (*Foeniculum vulgare*) 0.057 mg/g.

### **Sažetak**

Amino kiseline su u posljednje vrijeme u centru pozornosti zbog njihovog pozitivnog uticaja na imuni sistem i njegovu reakciju na infekcije, redukciju krvnog pritiska, relaksaciju, neuroprotekciju i modulaciju hemoterapije. Cilj ovog istraživanja je bio odrediti slobodne amino kiseline i njihov sadržaj u bijelom luku, crvenom luku, ljubičastom luku, luku kozijaku, prasi, srijemošu, glavici i listu komorača, rukoli, matovilcu, šparogama, crnom i zelenom čaju i artičoki komercijalno dostupim na pijacama BiH, jednostavnim, brzim i ekonomičnim metodama, Kvalitativna analiza slobodnih aminokiselina je izvedena metodom tankoslojne hromatografije (TLC). Kvantitativna analiza slobodnih amino kiselina je izvedena spektrofotometrijskom metodom (UV/VIS). Na 1.0 ml supernatanta svake biljke je dodano 0.5 ml rastvora fosfatnog pufera (pH 8.04) i 0.5 ml 2 % rastvora ninhidrina koji sadrži 0.8 mg/ml  $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ . Smjesa se potom zagrijavala na vodenom kupatilu do promjene boje, a zatim brzo ohlađena i razblažena do 25 ml destilovanom vodom. Absorbansa plavo-ljubičastih rastvora je mjerena nakon 10 min. Rezultati su pokazali da je najveći sadržaj slobodnih amino kiselina naden u bijelom luku 0.251 mg/g, a najmanji sadržaj je u listu komorača 0.057 mg/g.



## **Total sulphur content in different plant samples determined by High Performance Ion Chromatography (HPIC) method**

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### **Keywords:**

Sulphur,  
plant material,  
high performance ion  
chromatography (HPIC)

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**Abstract:** Sulphur as an integral part of the protein is an element that is essential for life and human health. Some plants are particularly rich source of organo-sulphur compounds, and these compounds have beneficial effects against several diseases. The aim of this study was to determine the total sulphur content in 17 different plant species which are used as common food in Bosnia and Herzegovina. The total sulphur content in plant material was determined by high performance ion chromatography (HPIC). Plant samples were prepared by dissolving and oxidation with a mixture of nitric and perchloric acid. The HPIC method was performed with a Shimadzu Ion Chromatograph equipped with conductivity detector CDD-10A. Diluted samples were analyzed on Shodex IC SI-90G exchange column, using carbonate buffer solution as mobile phase ( $\text{NaHCO}_3/\text{Na}_2\text{CO}_3$ ). The total sulphur content in the analyzed plant materials varies from 7.87 mg/g in dry black tea (*Camellia sinensis*) to 0.13 mg/g in bear's garlic (*Allium ursinum*). Black tea could serve as beneficial source of total sulphur content.

### **Sažetak**

Sumpor kao sastavni dio proteina je element koji je neophodan za život i zdravlje ljudi. Neke biljke su posebno bogat izvor organo-sumpornih spojeva, a ovi spojevi imaju pozitivne efekte protiv nekih bolesti. Cilj ovog istraživanja bio je utvrditi ukupni sadržaj sumpora biljnih vrsta koje se koriste kao uobičajena hrana u Bosni i Hercegovini. Ukupni sadržaj sumpora u biljnom materijalu određen je jonskom hromatografijom visokih performansi (HPIC). Biljni uzorci su pripremljeni rastvaranjem i oksidacijom smjesom nitratne i perhlorne kiseline. HPIC metoda je izvedena na Shimadzu jonskom hromatografu opremljenim sa konduktometrijskim detektorom CDD-10A. Razblaženi uzorci analizirani su na Shodex IC SI-90G izmjenjivačkoj koloni, koristeći karbonatni puffer kao mobilnu fazu ( $\text{NaHCO}_3/\text{Na}_2\text{CO}_3$ ). Ukupni sadržaj sumpora u analiziranom biljnom materijalu varira od 7.87 mg/g, u suhom crnom čaju (*Camellia sinensis*) do 0.13 mg/g u srijemošu (*Allium ursinum*). Crni čaj može služiti kao koristan izvor ukupnog sadržaja sumpora.



## **Determination of caffeine in green and black teas**

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**Keywords:**

caffeine,  
green tea,  
black tea,  
spectrophotometric (UV/VIS)  
method,  
thin layer chromatography (TLC)  
method

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**Abstract:** Methyl derivatives of xanthine are a group of alkaloids commonly used for their effects as mild stimulants on various organ systems such as cardiovascular and central nervous system (CNS), respiratory system and skeletal muscles. The naturally occurring methylxanthines are caffeine, theophylline and theobromine. The aim of this study was to determine caffeine in the green and black teas commercially available from Bosnian, Croatian, Slovenian, Austrian, Turkish, Indian and Chinese markets by simple, fast and economical methods. The simultaneous quantitative and qualitative determination was based on spectrophotometric (UV/Vis) method and thin layer chromatography (TLC) method. Caffeine content in the green teas was in the range from 33.9 to 110.73 (mg/g dry sample), and in black teas was in the range from 10.32 to 63 (mg/g dry sample). The highest caffeine content was detected in the green tea from the Slovenian market, and in the black tea from Croatian market.

### **Sažetak**

Derivati metil ksantina su grupa alkaloida koji se često koriste kao blagi stimulansi na različite sisteme organa, kao što su kardiovaskularni i centralni nervni sistem, respiratorni sistem i sistem skeletnih mišića. Prirodni metil ksantini su kofein, teofilin i teobromin. Cilj ovog rada je određivanje kofeina u zelenim i crnim čajevima komercijalno dostupnim sa tržišta Bosne, Hrvatske, Slovenije, Austrije, Turske, Indije i Kine, jednostavnim, brzim i ekonomičnim metodama. Simultano kvantitativno i kvalitativno određivanje bazirano je na spektrofotometrijskoj metodi (UV/Vis) i hromatografiji na tankom sloju (TLC). Sadržaj kofeina u zelenim čajevima je bio u rasponu od 33.9 do 110.73, a u crnim čajevima od 10.32 do 63.00 (mg/g suhog uzorka). Najveći sadržaj kofeina određen je u zelenom čaju sa tržišta Slovenije i u crnom čaju sa tržišta Hrvatske.





## **Antioxidant status of various mushrooms commercially available from Bosnian market**

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**Keywords:**

Mushrooms,  
total phenol content,  
total anthocyanine content,  
antioxidant activity.

**Abstract:** Mushrooms are well balanced foodstuff that provides definite nutrition and health benefits for human. Mushrooms are known to produce many kind of bioactive compounds, generally linked with mycelial cell wall, that help in enhancing the immune capacity to fight against carcinogens. To consider the importance of polyphenolic compounds and its presence in many varieties of mushrooms, the total antioxidant activity of dry boletus mushrooms, white and brown champignons, oyster mushrooms and shiitake from Bosnian market was determined. Total phenolic content was estimated spectrophotometrically as gallic acid equivalents (GAE /g ) according to the Folin-Ciocalteus method. Total anthocyanine content was analysed by pH differential spectrophotometric method at 525 and 700 nm. The radical scavenging activity (RSA) of mushroom extracts was determined by DPPH assay. The analysis revealed that the total phenolic contents ranged from 4.94 mg GAE/g in oyster mushrooms to 35.56 mg GAE/g in dry boletus mushrooms. The RSA was the highest in brown champignons 88.33 % and the lowest in oyster mushrooms 43.88 %. The mushrooms investigated in the present study could represent easily accessible sources of natural antioxidants.

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### **Sažetak**

Gljive su dobro izbalansirana hrana koja pruža određene prehrambene i zdravstvene pogodnosti za čovjeka. Gljive proizvode mnoge vrste bioaktivnih spojeva, uglavnom povezanih sa micelama ćelijskog zida, koji pomažu u jačanju sposobnosti imunološkog sistema da se bori protiv kancerogenih tvari. Da bi se razmotrile važnost polifenolnih supstanci i njihovo prisustvo u različitim vrstama gljiva, određena je ukupna antioksidativna aktivnost suhog vrganja, bijelih i smeđih šampinjona i shiitaka sa bosanskog tržišta. Sadržaj ukupnih fenola je određen spektrofotometrijski i izražen kao ekvivalent galne kiseline (GAE /g ) metodom po Folin-Ciocalteu. Sadržaj ukupnih antocijanina je analiziran pH-diferencijalnom spektrofotometrijskom metodom na 525 i 700 nm. Antiradikalna aktivnost (RSA) ekstrakata gljiva je određena DPPH metodom. Analiza je pokazala da se sadržaj ukupnih fenola kreće u rasponu od 4.94 mg GAE/g u bukovačama do 35.56 mg GAE/g u uzorku suhog vrganja. RSA je bila najveća za smeđe šampinjone 88.33 %, a najmanja za bukovače 43.88 %. Gljive ispitane u ovoj studij predstavljaju lako pristupačan izvor antioksidanata.



## **Determination of Free and Bound Monoterpenes Content in Aromatic Plants With Simple Distillation-spectrophotometric Method**

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**Keywords:**

free volatile monoterpenes,  
glycosidically bound  
monoterpenes,  
aromatic plants,  
distillation-spectrophotometric  
method

**Abstract:** In this paper the total content of free volatile monoterpenes (FVT) and glycosidically bound monoterpenes (GBT) present in six aromatic plants belonging to Lamiaceae family was determined by using simple and rapid distillation-spectrophotometric method. The investigated wild-growing plants, namely sage, lavender, rosemary, savory and oregano, were collected around Split during spring 2014., while commercial sample of basil was used. The method was originally developed for determination of free and glycosidically bound terpenes in grapes and adapted to analyze these compounds in aromatic plants. The plant materials were hydrodistilled under neutral and acidic conditions to collect two fractions, FVT and GBT. The colorimetric determination of monoterpenes in distillates was performed by adding vanillin-sulphuric acid reagent in order to obtain colour. The intensity of the colour was proportional to the content of monoterpenes. Absorbance was measured at 608 nm. The contents of monoterpenes in the distillates were calculated from the standard curve prepared with linalool standard solutions. According to the obtained results, all investigated plants contained very similar contents of FVT and GBT, except oregano with the lowest contents of these monoterpenes. Only slightly higher contents of FVT and GBT was recorded in basil.

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### **Sažetak**

U radu je brzom i jednostavnom spektrofotometrijskom metodom određen ukupni sadržaj slobodnih monoterpena (FVT) i glikozidno vezanih monoterpena (GBT) šest aromatičnih biljaka porodice Lamiaceae. Istražene su samonikle biljke, kadulja, lavanda, ružmarin, vrijesak i mrvinac, sakupljene u okolici Splita tokom proljeća 2014., dok je bosiljak bio komercijalni. Metoda je izvorno razvijena za određivanje slobodnih i glikozidno vezanih terpena u grožđu i prilagođena za određivanje navedenih spojeva u aromatičnom bilju. Biljni materijal je podvrgnut hidrodestilaciji radi prikupljanja dviju frakcija, FVT i GBT. Kolorimetrijsko mjerenje monoterpena u destilatima provedeno je dodavanjem reagensa vanilin-sumporne kiseline, radi razvijanja boje čiji je intenzitet proporcionalan sadržaju monoterpena, a potom je izmjerena apsorbansa pri 608 nm. Sadržaj monoterpena u destilatima izračunat je iz kalibracionog pravca pripremljenog pomoću standardnih otopina linalola. Iz rezultata je vidljivo da sve istraživane biljke sadrže slične količine FVT i GBT, osim mrvinca koji je imao najmanji sadržaj ovih spojeva, dok je sadržaj navedenih monoterpena bio neznatno veći u bosiljku.



## **Green chemistry by using flow analysis: Determination of iron ions with green tea extracts as reagents**

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### **Keywords:**

sequential injection analysis (SIA),  
green analytical chemistry,  
green tea extract,  
spectrophotometry,  
Fe (II), Fe(III),  
pharmaceuticals

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**Abstract:** The development of flow based technique such as sequential injection analysis (SIA) provides a new opportunity to development of green analytical chemistry methods.

The SIA system with spectrophotometric detector in combination with the extract of green tea as a natural chromogenic reagents were used for determination of Fe(III) and Fe(II). This green analytical method is based on formation of Fe-polyphenol complex that was monitored at 570 nm. In the process of optimization the selected experimental conditions allow determination of iron ions in concentration ranges from  $4.0 \times 10^{-5}$  to  $4.0 \times 10^{-4}$  mol/L with limit of detection of  $5.3 \times 10^{-6}$  mol/L and sampling rate of 122 injections  $\text{h}^{-1}$ . The method was successfully applied to the determination of iron ions in laboratory samples and pharmaceuticals.

### **Sažetak**

Razvoj protočnih tehnika analize kao što je sekvencijska injekcijska analiza (SIA) daje nove mogućnosti razvoja zelenih analitičkih metoda. SIA sustav sa spektrofotometrijskim detektorom uz primjenu ekstrakta zelenog čaja kao prirodnog reagensa upotrijebljen je za razvoj metode određivanja Fe (III) i Fe (II).

Ova zelena analitička metoda temelji se na nastajanju Fe-polifenol kompleksa čija se apsorbanacija mjeri pri valnoj duljini 570 nm. U postupku optimizacije odabrani su eksperimentalni uvjeti koji omogućuju određivanje Fe iona u koncentracijskom području od  $4,0 \times 10^{-5}$  do  $4,0 \times 10^{-4}$  mol/Ls granicom dokazivanja od  $5,3 \times 10^{-6}$  mol /L i dinamikom mjerenja od 122injektiranja  $\text{h}^{-1}$ . Metoda je uspješno primjenjena za određivanje iona Fe (II) i Fe (III) u laboratorijskim uzorcima i farmaceutskim pripravcima.



## Study of homosalate stability in chlorinated water

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### Keywords:

homosalate,  
free chlorine, by-products.

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**Abstract:** The increasing use of sun-creams containing organic UV-filters has led to increased concentration of these compounds in aquatic environment. Chlorinated water can convert these chemicals into chlorinated products whose toxic effects are of primary concern.

In this paper, stability of the homosalate in chlorinated water was studied. UV/VIS spectroscopy was used to follow the reaction of homosalate in presence of free chlorine. Gas chromatography with mass spectrometry was used to identify the major transformation by-products. Under the experimental conditions used in this work, homosalate reacted with chlorine following zero order reaction. The chemical transformation of the homosalate in chlorinated water led to the formation of chlorinated by-products that was identified as mono and dichloro-products.

### Sažetak

Povećana upotreba krema za zaštitu od sunca koje sadrže organske UV filtere, vode do povećane koncentracije ovih supstanci u vodenom okolišu. Hlorirana voda može prevesti ove supstance u hlorirane produkte koji imaju toksične efekte od primarnog interesa. U ovom radu ispitivana je stabilnost homosalata u hloriranoj vodi. Korištena je UV/VIS spektroskopija da bi se pratila reakcija homosalata u prisustvu slobodnog hlora. Gasna hromatografija sa masenom spektroskopijom je korištena da bi identificirali glavni produkti transformacije. Pod eksperimentalnim uslovima korištenim u radu, homosalat sa hlorom reaguje reakcijom nultog reda. Hemijska transformacija homosalata u hloriranoj vodi, vodi do formiranja hloriranih produkata, koji su identificirani kao mono i dihlor-produkti.



## **Preconcentration of chromium species on Amberlite IR-120 and Amberlite CG-400 ionexchange resins**

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**Keywords:**

specije hroma,  
prekoncentriranje,  
Amberlite IR-120,  
Amberlite CG-400

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**Abstract:** The possibility of separation and preconcentration of chromium species Cr(III) and Cr(VI) from the solutions in which they mutually interfere was examined on ionexchange resins. The following conditions for preconcentration of Cr(VI) on Amberlite CG-400 resin were determined: mass of resin 1.0 g; sample pH 5.5; sample volume of 100 mL (with preconcentration factor of 10), sample flow rate 0.5 mL min<sup>-1</sup>. Cr(VI) was determined by molecular UV/Vis spectrometry with the addition of diphenylcarbazide. The recovery value of Cr(VI) after preconcentration of prepared samples with known concentrations of Cr(III) and Cr(VI) was 63 %. The method for Cr(VI) determination does not give excellent results when it comes to preconcentration, but the results showed that the tested ionexchange resin can be successfully used for separation of chromium species. Cr(III) was quantitatively determined by flame atomic absorption spectrometry (FAAS) after preconcentration on Amberlite IR-120 resin using following conditions: mass of resin 1.5 g; using sample pH 4.5; sample flow rate 0.5 mL min<sup>-1</sup>. The recovery values of Cr(III) after preconcentration of prepared samples with known concentrations for volumes of 250 and 1000 mL (with preconcentration factor of 25 and 100) were 102 % and 99 %, respectively. The method was applied for determination of Cr(III) content in several natural water of Bosnia and Herzegovina (Miljacka, Neretva, Vrbas, stream Faletići), and industrial waste water (metal, leather, industry of coal and cement).

### **Sažetak**

Mogućnost separacije i prekoncentriranja specija Cr(III) i Cr(VI) iz rastvora u kojima međusobno interferiraju je ispitano na ionoizmjenjivačkim smolama. Određeni su sljedeći uslovi za prekoncentriranje Cr(VI) na Amberlite CG-400 smoli: masa smole 1.0 g, pH uzorka 5.5, volumen uzorka 100 mL (uz prekoncentracioni faktor 10), protok 0.5 mL min<sup>-1</sup>. Cr(VI) je određen metodom molekulske UV/Vis spektrometrije uz dodatak difenilkarbazida. Vrijednost *recovery*-ja (analitičkog povrata) za Cr(VI) nakon prekoncentriranja pripremljenih uzoraka sa poznatom koncentracijom Cr(III) i Cr(VI) je bila 63 %. Metoda za određivanje Cr(VI) ne daje odlične rezultate *recovery*-ja kada je u pitanju samo prekoncentriranje, ali su rezultati pokazali da se testirana ionoizmjenjivačka smola može uspješno koristiti za razdvajanje specija hroma. Cr(III) je kvantitativno određen atomskom apsorpcionom spektrometrijom-plamenom tehnikom (FAAS) nakon prekoncentriranja na Amberlite IR-120 smoli pri sljedećim uslovima: masa smole 1.5 g; pH uzorka 4.5; protok 0.5 mL min<sup>-1</sup>. Vrijednosti *recovery*-ja za Cr(III) nakon prekoncentriranja pripremljenih uzoraka poznate koncentracije za volumene od 250 i 1000 mL (uz prekoncentracioni faktor 25 i 100) su bile 102 % i 99 %, redom. Metoda je primijenjena za određivanje sadržaja Cr(III) u nekoliko prirodnih voda Bosne i Hercegovine (Miljacka, Neretva, Vrbas, potok Faletići) i industrijskih otpadnih voda (metalna, kožarska, industrija uglja i industrije cementa).



## **Validation ETA-AAS method for determination of Nickel in Calcium and Magnesium Stearate after Microwave Decomposition**

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### **Keywords:**

AAS (Atomic Absorption Spectrophotometry),  
ETA (Electro thermal atomisation),  
Nickel, microwave acid digestion.

**Abstract:** Heavy metals, including nickel can be found at raw materials for manufacturing pharmaceutical products, but in very low quantity. Calcium Stearate and Magnesium Stearate is used as an excipients for drugs manufacturing. The purpose of this experiment is to develop and validation method which will eliminate very inefficient, long-term classic acid digestion. The samples of Calcium Stearate and Magnesium Stearate were decomposed by microwave acid digestion using nitric acid. Along this mode, it was developed and optimized a method for the determination of nickel traces ETA-AAS direct concentration method. With regard to classic method, in this case addition is not necessary, because the impact of matrix is very effectively compensated using modified (ammonium dihydrogen phosphate) and Zeman's background correction. The procedure is validated and the validation parameters show very good results: linearity for Calcium and Magnesium Stearate  $r^2 > 0,999$ , accuracy 97,1% and 106,1%, reproducibility of system 1,1% and 1,2%, reproducibility of method 3,7% and 2,1%, detection limit 0,35  $\mu\text{g/g}$ , and 0,34  $\mu\text{g/g}$ , quantification limit 1,16  $\mu\text{g/g}$  and 1,13  $\mu\text{g/g}$ .

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### **Sažetak**

Teški metali se mogu naći u sirovinama za proizvodnju lijekova, ali samo u vrlo malim količinama. Kalcijum stearat i magnezijum stearat se koriste kao pomoćna sredstva u proizvodnji lijekova. Cilj ovog ispitivanja je da se razvije i validira metoda koja će isključiti klasičnu razgradnju uzoraka, koja je neefikasna i dugotrajna. Uzorci kalcijum stearata i magnezijum stearata su raščinjani mikrovalnom kiselinom digestijom sa nitratnom kiselinom. Razvijena je i optimizirana ETA-AAS metoda direktne koncentracije za određivanje tragova nikla u ovako pripremljenim uzorcima. U odnosu na klasičnu metodu u ovom slučaju adicija nije neophodna iz razloga što uticaj matriksa moguće efikasno kompenzovati uz korištenje modifikatora (amonij dihidrogen fosfata) i uz Zemanovu pozadinski korekciju. Cijeli postupak je validiran, a validacioni parametri su pokazali veoma dobre rezultate: za linearnost slučaju kalcij stearate i magnezij stearata  $r^2 > 0,999$ , tačnost ide u intervalu 97,1% do 106,1%, preciznost sistema 1,1% odnosno 1,2%, preciznost metode, 3,7%, odnosno 2,1%, limit detekcije 0,35  $\mu\text{g/g}$ , odnosno 0,34  $\mu\text{g/g}$ , limit kvantizacije 1,16  $\mu\text{g/g}$ , odnosno 1,13  $\mu\text{g/g}$ .



## **The use of pulverized *Cucurbita pepo* peel for the preconcentration of Co and Ni ions from aqueous solutions**

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**Keywords:**

SPE,  
AAS,  
heavy metals,  
pulverized pumpkin peel,  
optimal conditions,  
recovery

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**Abstract:** In this study, the use of pulverized pumpkin peel (*Cucurbita pepo*) for the preconcentration of Co and Ni ions, from aqueous solutions in a column system, was described. After preconcentration, these metal ions were analysed by flame atomic absorption spectrometry. In the optimization procedure three important variables were investigated: sample pH, sample flow rate and preconcentration factor. Under optimized conditions the detection limits of the method ( $3.3 \cdot S_{bl}$ ) were 87.50 and 96.65  $\mu\text{g L}^{-1}$  for Co and Ni, respectively. The precision expressed as a relative standard deviation (RSD.s, %) of the method was 3.89 % (Co) and 4.37 % (Ni), calculated from ten measurements. The recoveries of analytes under the optimum conditions were 100.2 % (Co) and 99.1 % (Ni). The optimal pH for investigated metals was in the range of 5.0 to 10.0; optimal sample flow rate was 2  $\text{mL min}^{-1}$  and optimal preconcentration factor was 10. Pulverized pumpkin peel as a biosorbent can be reused for a large number of cycles with insignificant loss of the initial sorption capacity. The results obtained show that *Cucurbita pepo* shell based biosorbent can be used as an effective and low-cost means for removal of toxic metals from natural water and wastewater.

### **Sažetak**

U ovom radu opisano je korištenje sprasene kore bundeve (*Cucurbita pepo*) za prekoncentriranje iona Co i Ni iz vodenih rastvora primjenom kolona. Nakon prekoncentriranja, metali su određivani plamenom atomskom apsorpcionom spektrometrijom. U postupku optimizacije su istražene tri bitne varijable: pH rastvora, brzina protoka rastvora i faktor prekoncentriranja. Pod optimalnim uslovima limiti detekcije metode ( $3.3 \cdot S_{bl}$ ) su bili 87.50 i 96.65  $\mu\text{g L}^{-1}$  za Co i Ni, respektivno. Preciznost metode, izražena kao relativna standardna devijacija (R.S.D., %), za deset mjerenja bila je 3.89% (Co) i 4.37% (Ni). Recovery vrijednosti analita dobivene pri optimalnim uslovima analize su 100.2 % (Co) i 99.1 % (Ni). Optimalna vrijednost pH rastvora određenih metala je u intervalu od 5.0-10.0; optimalni protok rastvora je 2  $\text{mL min}^{-1}$  i optimalni faktor prekoncentriranja je 10. Sprasena kora bundeve, kao biosorbent, se može koristiti za veliki broj ponovljenih ciklusa prekoncentriranja sa zanemarljivim gubitkom početnog kapaciteta sorpcije. Dobiveni rezultati su pokazali da se biosorbent *Cucurbita pepo* može koristiti kao efikasno i jeftino sredstvo za uklanjanje toksičnih metala iz prirodnih i otpadnih voda.



## **Optimization of Biological Surface Adsorption Index Approach for Applications in Surface Characterization of the Nanomaterials**

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### **Keywords:**

nanoparticles,  
characterization,  
interactions,  
biomolecules.

**Abstract:** A complete characterization of the different physiochemical properties of nanomaterials is necessary for the evaluation of their impact on health and the environment. Surface characterization of the nanomaterial is the least developed among the surface properties of nanomaterials.

The biological surface adsorption index approach (BSAI) for characterization of surface adsorption properties of nanomaterials has been recently introduced. This approach offers in principle the possibility to characterize the different interaction forces exerted between a nanomaterials surface and biomolecules.

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In this work we further developed the BSAI approach which, as an outcome, gives a better defined quantification of the adsorption properties on nanomaterials.

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### **Sažetak**

Detaljna karakterizacija fizičkih i hemijskih osobina nanomaterijala je neophodna za evaluaciju njihovog uticaja na zdravlje čovjeka i na okoliš. Među različitim osobinama nanomaterijala, površinska karakterizacija je najslabije razvijena.

Biološki površinski indeks (biological surface adsorption index – BSAI) je metoda nedavno razvijena za karakterizaciju površinskih osobina nanomaterijala. Metoda je bazirana na karakterizaciji različitih interakcija koje se javljaju između nanomaterijala i biomolekula.

U ovom radu smo detaljno razradili i optimizirali BSAI metodu, koja će kao takva, dati bolju i detaljnu kvantifikaciju površinskih osobina nanomaterijala.





## **SPE extraction and TLC Identification of Tetracycline and Fluoroquinolone in Surface Water**

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**Keywords:**

antibiotics,  
TLC,  
identification,  
surface water.

**Abstract:** Simultaneous identification of the antibiotics tetracycline, oxytetracycline, chlortetracycline, ciprofloxacin and enrofloxacin in surface water is reported. The method is based solid-phase extraction (SPE), separation and identification by thin-layer chromatography (TLC). TLC separation was performed on TLC silica gel 60 F254 plate using a mobile phase system water/methanol/dichlormetan (6/35/59) (v/v). The plates are previously impregnated with 10% solution EDTA pH 9,0. The method was optimized with spiked surface water. Antibiotics were extracted on a OASIS HLB 6cc/500 mg cartridges. 10 µl aliquots of the water sample and reference solutions were applied to the plate. After development, the chromatograms were visualized under UV light at  $\lambda = 254$  nm and  $\lambda = 366$  nm.

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The analysis of surface water samples taken from three different locations not confirmed the presence of the tested antibiotics. The described method is simple and suitable for the rapid identification of investigated residues of tetracycline and fluoroquinolone antibiotics in surface water with LOD of 0.5 ppm.

### **Sažetak**

Provedena je simultana identifikacija antibiotika tetraciklina, oksitetraciklina, hlortetraciklina, ciprofloksacina i enrofloksacina u površinskoj vodi. Postupak se zasnivao na ekstrakciji na čvrstim fazama (SPE), razdvajanju i identifikaciji primjenom hromatografije na tankom sloju (TLC). Razdvajanje je provedeno na TLC silikagel 60 F254 pločama uz mobilnu fazu voda / metanol / dihlormetan (6/35/59) (v/v). Ploče su prethodno impregnirane sa 10%-tnom otopinom EDTA pH 9,0. Metoda je optimizirana primjenom površinske vode u koju je dodana poznata koncentracija antibiotika. Antibiotici su ekstrahirani primjenom OASIS HLB 6cc/500 mg ketridža. 10 µl uzoraka vode i standardnih otopina aplicirano je na ploču. Nakon razdvajanja, hromatogrami su vizualizirani pod UV lampom na  $\lambda = 254$  nm i  $\lambda = 366$  nm. Analizom uzoraka površinskih voda uzetih sa tri različita lokaliteta nije potvrđeno prisustvo ispitivanih antibiotika. Opisana metoda je jednostavna i prikladna za brzu identifikaciju rezidua ispitivanih antibiotika iz grupe tetraciklina i fluorokinolona u površinskoj vodi sa limitom detekcije 0.5 ppm.



## Synthesis of biobased antioxydant and antiusure additives for diesel fuel

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### Keywords:

biodiesel,  
fatty acids,  
transesterification,  
antioxydant,  
antiusure additives

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**Abstract:** Biodiesel as a biodegradable, sustainable and clean energy has worldwide attracted renewed and growing interest in topical years, chiefly due to development in biodiesel fuel and ecological pressures which include climatic changes. In general, fatty esters are used as bio additive, cosmetic ingredients, polymers, and, more recently, biofuel or bio lubricant. This review describes important features related to be the synthesis, stability to, and activity acid or basic catalysts in bioadditifs production reactions. The maximum bioadditif I yield obtained were about 94%, 83%, in acid and basic middle respectively. Overall, the acid catalyst (H<sub>2</sub>SO<sub>4</sub>) in isoamylic alcohol showed high performance at operating conditions (95°C, 3h, H<sub>2</sub>SO<sub>4</sub> at 1% wt, ratio of oil/isoamylic= 3/1).

Oxidation test was carried out in dispositive (ASTM D 2274). The oxidative degradation of diesel with or without antioxidants was investigated by UV-visible spectroscopy was used to monitor the changes using peroxide value at optimal concentration about 1% of bioadditif.

### Sažetak

Širom svijetu raste interes za bio-dizelom kao bio-razgradivoj, održivoj i čistoj energiji, uglavnom zbog razvoja bio-dizela kao goriva i ekoloških zahtjeva izazvanih klimatskim promjenama. Najčešće se masni esteri koriste kao bio-aditivi, kozmetički sastojci, polimeri i u novije vrijeme kao bio-gorivo ili bio-mazivo. Ovdje će biti istaknut značaj same sinteze, stabilnosti, aktivnosti kiseline ili osnova katalize u reakcijama proizvodnje bio-aditiva. Maksimalno dobiveni prinos bio-aditiva I su oko 94% i 83% u kiseloj i baznoj sredini, redom. Općenito, kiselu katalizator (H<sub>2</sub>SO<sub>4</sub>) u *izoamilnom* alkoholu pokazuje visoki učinak pri radnim uslovima (95 ° C, 3h, 1% wt H<sub>2</sub>SO<sub>4</sub> i odnosu ulje/*izoamilalk.* = 3/1). Oksidacijski test je proveden po uredbi (ASTM D 2274). Ispitivana je oksidacijska degradacija dizela sa ili bez antioksidansa UV-VIS spektroskopijom, koristeći vrijednost peroksida pri optimalnoj koncentraciji bio-aditiva oko 1%, za praćenje promjena.



## **Isolation and Antioxidant activity of betulinic acid and oleanolic acid obtained from *Betula pendula* L., *Betulaceae***

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### **Keywords:**

betulinic acid, oleanolic acid,  
isolation, dry column  
chromatography, antioxidant  
activity

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**Abstract:** *Betulae cortex*, *Betula pendula* Roth., *Betulaceae*, comprise triterpene substances which are confirmed to possess very important pharmacological activities such as anti-inflammatory, anticancer and antiviral. In this study, extraction and isolation of triterpenes, betulinic acid and oleanolic acid from external birch bark, was carried out using method of dry column chromatography. To those substances infrared (IR) spectra were recorded and compared with IR spectra of adequate standards. Further, we tested the antioxidant capacity of betulinic acid and oleanolic acid isolated from investigated plant material using 1-diphenyl-2-picrylhydrazyl radical scavenging method (DPPH).

Working solutions of the tested substances, betulinic acid (0,006 g), oleanolic acid (0,025 g), were dissolved in ethanol (10 ml). For every analyzed substance is calculated calibration relationship diagram between inhibition and concentration of dilution. Following IC<sub>50</sub> values were obtained: betulinic acid 302,49±9,45, oleanolic acid 302,29±15,543. Since the *Betula pendula* is very widespread plant species a positive results of the antioxidant activity could be the basis for its use as dietary supplement and production of special recipe preparation in the prevention of cardiovascular disease, thrombosis, strengthen the immune system, particularly in terms of the stress of everyday life.

### **Sažetak**

*Betulae cortex*, *Betula pendula* Roth., *Betulaceae*, sadrži triterpenske supstance za koje je potvrđeno da ispoljavaju vrlo važna farmakološka djelovanja kao što su protuupalno, antivirusno i antikancerogeno. U ovoj studiji, ekstrakcija i izolacija triterpena, betulinske kiseline i oleinske kiseline iz vanjske kore breze, je provedena metodom suhe kolonske hromatografije. Snimljeni su Infracrveni (IC) spektri i upoređeni s IC spektrom odgovarajućih standarda. Nadalje, testirali smo antioksidativni kapacitet betulinske kiseline i oleinske kiseline izolirane iz ispitivanog biljnog materijala pomoću 1-difenil-2-picrylhidrazil radikal metode vezivanja (DPPH). Radne otopine ispitivanih supstanci, betulinske kiseline (0,006 g), oleinske kiseline (0,025 g), otopljene su u etanolu (10 ml). Za svaki analizirani uzorak napravljen je kalibracioni dijagram, odnosa između inhibicije i koncentracije supstance. Dobivene su sljedeće IC<sub>50</sub> vrijednosti: betulinska kiselina 302,49 ± 9,45, oleinska kiselina 302,29 ± 15.543. Budući da je *Betula pendula* vrlo rasprostranjena biljna vrsta, kao i na osnovu pozitivnog rezultata antioksidativne aktivnosti, otvara se mogućnost njene upotrebe kao dodatka prehrani i proizvodnji specijaliziranih pripravaka za prevenciju kardiovaskularnih bolesti, tromboze, jačanju imunološkog sistema.



## **Photochemical investigation of arbutin content in herbal drugs from family *Ericaceae* and *Vacciniaceae***

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### **Keywords:**

arbutin, uva, cranberry,  
blueberry, phytochemistry

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**Abstract:** The phytochemical analysis of arbutin content were performed on the plant species of the family *Ericaceae* and *Vacciniaceae* as follow: Uvae leaf and fruit (*Uvae ursi folium et fructus*, *Arctostaphylos uva ursi*, *Ericaceae*), domestic cranberry leaf and fruit (*Vitis idaea folium et fructus*, *Vaccinium vitis idaea* L. *Vacciniaceae*) and blueberries leaf and fruit (*Myrtilli folium et fructus*, *Vaccinium myrtillus* L., *Vacciniaceae*). Plant material was tested using method of thin layer chromatography (TLC) in the presence of arbutin. TLC conditions: mobile phase was ethylacetate-methanol-water (100:13,5:10), visualization was performed under UV lamp at 254nm and splash spray reagent in Berlin blue. Identification of the components was performed by comparison with standard substances (arbutin, rutin). By colorimetric method was examined the content of arbutin in plant material. Content of arbutin amounted as follow: uvae leaf (6.38%), cranberry leaf (4.32%), leaf blueberry (1.23%) and uvae fruit (1.02%).

### **Sažetak**

Fitohemijiski su ispitivane biljne vrsta iz porodica *Ericaceae* i *Vacciniaceae* na sadržaj arbutina. Fitohemijiskom ispitivanju podvrgnutii su: list i plod uve (*Uvae ursi folium et fructu*, *Arctostaphylos uva ursi*, *Ericaceae*), list i plod domaće brusnice (*Vitis idaea folium et fructus*, *Vaccinium vitis idaea* L., *Vacciniaceae* i list i plod borovnice (*Myrtilli folium et fructus*, *Vaccinium myrtillus* L., *Vacciniaceae*). Biljni material je ispitivan metodom hromatografije na tankom sloju na prisustvo arbutina. Korištena je mobilna faza etilacetat-metanol-voda (100:13,5:10), vizuelizacija vršena pod UV lampom na 254nm i prskanjem sprej reagensom berlinsko plav o. Identifikacija komponenti vršena poređenjem sa standardnim supstancama (arbutin, rutin). Kolorimetrijskom metodom je ispitan je sadržaj arbutina u biljnom materijalu i iznosio je: list uve (6,38%), list brusnice (4,32%), list borovnice (1,23%) i plod uve (1,02%).



## **Determination of Fe and Mn from Aqueous Solutions after Preconcentration on Yttrium (III) Oxide**

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### **Keywords:**

yttrium oxide,  
preconcentration,  
recovery,  
optimum conditions,  
foreign ions,  
water samples

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**Abstract:** In this study, we described the use of a column packed with unmodified yttrium (III) oxide as sorbent for preconcentration of Fe and Mn prior to their analysis by flame atomic absorption spectrometry (FAAS). We determined factors affecting the preconcentration of known low concentration of Fe and Mn such as pH of the sample, sorbent mass, sample flow rate and volume (preconcentration factor) from pure aqueous solutions. The recovery values of prepared samples of known concentration of analytes were: 103.9 % (Fe) and 95.8 % (Mn), under optimum conditions (pH 10, sorbent mass 400 mg, sample flow rate 4 mL/min, preconcentration factor 10). Detection limits of the method were 37.1 µg/L for Fe and 10.3 µg/L for Mn. Influence of foreign ions (Mg, Ca), which are in water present in high concentrations, on recovery values was examined. Mg concentration in range from 25 to 1000 mg/L showed no influence on recovery values. Ca concentrations 25, 50 and 100 mg/L had no significant influence on recovery values, while Ca concentration of 250 mg/L and higher caused decrease in recovery values for approximately 15 %. Proposed method for metals preconcentration was also tested on spring and river water samples.

### **Sažetak**

U ovom radu su korištene kolone punjene sa nemodificiranim itrij (III) oksidom kao sorbentom za prekoncentriranje Fe i Mn neposredno pred njihovo određivanje plamenom atomskom apsorpcionom spektrometrijom (FAAS). Određeni su faktori koji utiču na prekoncentriranje poznate, niske koncentracije Fe i Mn iz čistih vodenih rastvora i to pH uzorka, masa sorbenta, brzina protoka uzorka i volumen korištenog uzorka, odnosno prekoncentracioni faktor. Vrijednosti *recovery*-ja pripremljenih uzoraka metala poznate koncentracije su bile: 103.9 % (Fe) i 95.8 % (Mn), pod optimalnim uvjetima (pH 10, masa sorbenta 400 mg, brzina protoka uzorka 4 mL/min i prekoncentracioni faktor 10). Detekcioni limiti metode su bili 37.1 µg/L za Fe i 10.3 µg/L za Mn. Ispitivan je uticaj stranih jona koji su u visokoj koncentraciji prisutni u vodi (Mg, Ca) na vrijednosti *recovery*-ja. Koncentracija Mg od 25 mg/L do 1000 mg/L nema uticaja na vrijednost *recovery*-ja. Koncentracije Ca, 25, 50 i 100 mg/L nemaju značajan uticaj na vrijednost *recovery*-ja, dok koncentracije Ca od 250 mg/L i više dovode do smanjenja vrijednosti *recovery*-ja približno 15 % za oba metala. Predložena metoda za prekoncentriranje metala je testirana i na uzorcima izvorske i riječne vode.



## **Determination of copper, chromium and cadmium in food-packaging materials by atomic absorption spectrometry – flame technique**

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**Keywords:**

Heavy metals  
Atomic absorption spectrometry,  
Packaging materials.

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**Abstract:** The issue of assessment of health risks of food packaging materials represents an ongoing challenge of producing safe and wholesome packaging materials. Selected heavy metals, Cu, Cr and Cd in five different food-packaging materials were analysed by flame atomic absorption spectrometry. The chosen packaging materials were: paper, carton, cellophane, plastic coated paper and tetra pak. Considering the fact that analysed samples were of different materials and percentage of coloration, significant differences were found in concentrations, especially in the case of copper and cadmium. Results show that the Cu, Cr and Cd concentrations were found in the range of 1.06 - 55.62  $\mu\text{g/g}$ , 4.13 - 7.73  $\mu\text{g/g}$  and 1.54 - 5.57  $\mu\text{g/g}$ , respectively. Heavy metals in packaging material mainly originate from the colors that are used to obtain the final product, rather than the base material. The contents of all analysed metals mainly decrease with decreasing the percentage of coloration surface of the packaging material.

### **Sažetak**

Procjena zdravstvenih rizika zbog upotrebe ambalažnih materijala za prehrambene proizvode predstavlja stalni izazov u smislu proizvodnje sigurnih i sa aspekta ljudskog zdravlja prihvatljivih ambalažnih materijala. U ovom radu su analizirani teški metali, Cu, Cr i Cd u pet različitih tipova ambalaže, metodom atomske apsorpcione spektrometrije - plamena tehnika. Korišteni su uzorci: papira, kartona, celofana, plastificiranog papira i tetra paka. S obzirom da su analizirani uzorci različitog sastava i obojenosti, uočena je značajna razlika u koncentracijama prije svega bakra i kadmija između različitih uzoraka. Koncentracije Cu, Cr i Cd kretale su se u rasponu 1.06 - 55.62  $\mu\text{g/g}$ , 4.13 - 7.73  $\mu\text{g/g}$  i 1.54 - 5.57  $\mu\text{g/g}$ , respektivno. Teški metali u ambalažnom materijalu uglavnom potiču od boja koje se koriste za dobivanje finalnog proizvoda, a ne od baznog materijala. Sadržaj svih analiziranih metala se uglavnom smanjivao sa smanjenjem postotka obojenosti površine ambalažnog materijala.



## **New kinetic method for determination of N-acetyl-L-cysteine in pharmaceutical formulations**

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**Keywords:**

N-acetyl-L-cysteine,  
Kinetic spectrophotometry,  
Initial rate method,  
Fixed time method,  
Pharmaceutical analysis

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**Abstract:** N-acetyl-L-cysteine (NAC) is a synthetic aminothiols antioxidant that has been in clinical use for more than 40 years, primarily as a mucolytic agent and in the management of paracetamol (acetaminophen) poisoning. There is need for a new, robust, inexpensive, and rapid method of determination of NAC in pharmaceutical formulations, in order to assure proper quality control (QC).

Novel flow-injection spectrophotometric method for the determination of NAC has been developed and validated. The proposed method is based on the reduction of Cu(II)-neocuproine reagent to Cu(I)-neocuproine with the analyte, in a Britton-Robinson buffer solution (pH = 3.0). The non-steady state absorbance of the formed yellow Cu(I)-neocuproine complex is measured at 458 nm. A linear calibration curve is established in a concentration range of  $6 \times 10^{-7}$  to  $4 \times 10^{-5}$  mol L<sup>-1</sup> NAC with regression equation  $y = 3.89 \times 10^3 x - 0.0015$  ( $R^2 = 0.9996$ ) ( $n = 9$ ) and detection limit of  $9.4 \times 10^{-8}$  mol L<sup>-1</sup>. The proposed method is simple, rapid, sensitive and reproducible (RSD 0.9%,  $n = 100$ ). In addition, proposed method is sensitive enough to enable determination of near nanomole amounts of NAC without expensive instruments with the analytical frequency of 120 h<sup>-1</sup>.

**Sažetak**

N-acetil-L-cistein (NAC) je sintetski aminotiolni antioksidans koji je u kliničkoj upotrebi već više od 40 godina, prvenstveno kao mukolitički agens te pri liječenju trovanja paracetamolom. Kako bi se osigurala odgovarajuća kontrola kvalitete, postoji potreba za novim, robustnim, jeftinim i brzim metodama određivanja NAC-a u farmaceutskim pripravcima.

Razvijena je te vrednovana nova protočna injekcijska spektrofotometrijska metoda određivanja N-acetil-L-cisteina. Predložena metoda zasniva se na redukciji Cu(II)-neokuproin reagensa do Cu(I)-neokuproin kompleksa, djelovanjem analita, u acetatno-boratno-fosfatnom puferu (pH = 3.0). Promjenjiva apsorbancija stvaranog Cu(I)-neokuproin kompleksa mjerena je pri 458 nm. Krivulje umjeravanja su linearne u području koncentracija  $6 \times 10^{-7}$  do  $4 \times 10^{-5}$  mol L<sup>-1</sup> s regresijskom jednadžbom  $y = 3.89 \times 10^3 x - 0.0015$  ( $R^2 = 0.9996$ ) ( $n = 9$ ) i granicom dokazivanja od  $9.4 \times 10^{-8}$  mol L<sup>-1</sup>. Razvijena metoda je jednostavna, brza, osjetljiva i ponovljiva (RSD 0.9%,  $n = 100$ ) te je dovoljno osjetljiva za određivanje nanomolarnih količina NAC-a izbjegavajući skupe instrumente uz analitičku učestalost od 120 h<sup>-1</sup>.



## **Content of As, Cd, Pb and Sn in parsley (*Petroselinum crispum*) from different districts in Serbia**

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### **Keywords:**

Parsley,  
content of metals,  
ICP OES

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**Abstract:** Parsley (*Petroselinum crispum*) is one of the most popular vegetables in Serbia and during the past decades its consumption has been increasing. Parsley belongs of the *Apiaceae* family, which also includes celery, coriander and carrot. Accumulation of metals in plants is very important, due to their effects on human health. The aim of this study was to determine the content of arsenic, cadmium, lead and tin in the parsley from different geographic origin. Vegetable samples were collected from nine locations in three districts in Serbia: Toplica, Bor and Rasina district. The concentrations of As, Cd, Pb, and Sn were determined using the ICP OES technique and results show different level of contamination in various district. The highest amount of arsenic, cadmium, lead and tin was found in parsley from Bor district, ( $3.734 \mu\text{g g}^{-1}$ ), ( $0.052 \mu\text{g g}^{-1}$ ), ( $0.804 \mu\text{g g}^{-1}$ ) and ( $1.951 \mu\text{g g}^{-1}$ ), respectively, as it was expected, because the considered locations are representatives of potentially most polluted. The lowest concentrations of As, Cd, Pb and Sn were registered in Rasina district, ( $0.321 \mu\text{g g}^{-1}$ ), ( $0.038 \mu\text{g g}^{-1}$ ), ( $0.399 \mu\text{g g}^{-1}$ ) and ( $0.595 \mu\text{g g}^{-1}$ ), respectively. Amount of selected metals is reflection of the environmental pollution and soil type.

### **Sažetak**

Peršun je dvogodišnja biljka koja se često koristi u kuhinjskom u Srbiji. Pripada porodici *Apiaceae*, gde spadaju celer, korijander i šargarepa. Od velike važnosti za ljudsko zdravlje je akumulacija metala u biljkama, pa je shodno tome cilj ovog rada bio određivanje sadržaja arsena, kadmijuma, olova i kalaja u peršunu različitog geografskog porekla. Biljni material je sakupljan na devet lokaliteta Topličkog, Borskog i Rasinskog okruga. Koncentracija As, Cd, Pb i Sn je određivana ICP OES tehnikom i rezultati pokazuju različiti nivo zagađenja u različitim okruzima. Najveća količina arsena, kadmijuma, olova i kalaja detektovana je u Borskom ( $3.734 \mu\text{g g}^{-1}$ ), ( $0.052 \mu\text{g g}^{-1}$ ), ( $0.804 \mu\text{g g}^{-1}$ ) i ( $1.951 \mu\text{g g}^{-1}$ ), respektivno, a najniža u Rasinskom okrugu, ( $0.321 \mu\text{g g}^{-1}$ ), ( $0.038 \mu\text{g g}^{-1}$ ), ( $0.399 \mu\text{g g}^{-1}$ ) i ( $0.595 \mu\text{g g}^{-1}$ ), respektivno. Različiti sadržaj analiziranih metala ukazuje na različiti tip zemljišta i zagađenja živone sredine.





## **Simultaneous Determination of Caffeic and Chlorogenic Acid in Green Coffee Extracts by Gas Chromatography and Mass Spectrometry (GC-MS)**

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**Keywords:**

caffeic acid,  
chlorogenic acid,  
gas chromatography  
Mass spectrometry

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**Abstract:** Rapid and reliable method for simultaneous separation, identification and quantitative determination of selected phenolic acids in commercially available green coffee (*Coffea canephora*) extracts has been developed. Caffeic and chlorogenic acid were determined as trimethylsilyl (TMS) derivatives by GC-MS. GC-MS has the advantage because it allows simultaneous separation and identification of geometric *cis*- and *trans*-isomers of the investigated compounds. The results confirmed that *cis*-chlorogenic acid and *cis*-caffeic acid are not naturally occurring compounds but were produced after sample preparation procedure as a result of isomerization from their *trans*-forms. Stability studies were performed and it was found that »*trans-cis*« isomerization is solvent, temperature, and time-dependent. Anyhow, final silylated derivatives were stable at least 2 days as in this time the peak-area ratios for *cis*- and *trans*- isomers were constant throughout the whole concentration range. Linearity of the method was confirmed in the concentration range from 20 to 200 mg L<sup>-1</sup> with  $r^2$  greater than 0.9942. It was proven that this method is repeatable (precise) (RSD<10%), and accurate. It was established that the commercially-available dry weighed green coffee extract contains in average 16.1±1.1 mg g<sup>-1</sup> of caffeic acid and 197.7±13.6 mg g<sup>-1</sup> of chlorogenic acid.

### **Sažetak**

Razvijena je brza i pouzdana metoda za simultano odvajanje, identifikaciju i kvantitativno određivanje odabranih fenolnih kiselina u ekstraktima komercijalno dostupne zelene kafe (*Coffea canephora*). Kofeinske i hlorogenske kiseline određivane su kao derivati trimetilsilil-a (TMS) pomoću GC-MS-a. GC-MS ima prednost jer omogućava istovremeno razdvajanje i identifikaciju geometrijskih *cis*- i *trans*-izomera ispitivanih spojeva. Rezultati su potvrdili da *cis*-hlorogenska kiselina i *cis*-kofeinska kiselina nisu prirodni spojevi, već su nastali kao rezultat izomerizacije iz njihovog *trans*-oblika u postupku pripreme uzorka. Izvedene su studije stabilnosti i utvrđeno je da je »*trans-cis*« izomerizacija zavisi od otapala, temperature i vremena. U svakom slučaju, konačni sililirani derivati bili su stabilni najmanje dva dana, jer su tokom ovog vremenskog perioda površina pika za *cis*- i *trans*- izomere bila je konstantna kroz čitav koncentracijski opseg. Linearnost metode potvrđena je u rasponu koncentracija 20-200 mg L<sup>-1</sup> sa  $r^2$  većim od 0,9942. Dokazano je da je ova metoda ponovljiva (precizna) (RSD <10%), i tačna. Utvrđeno je da odvagani suhi ekstrakt, komercijalno dostupne zelene kafe, sadrži u prosjeku 16,1±1,1 mg g<sup>-1</sup> kofeinske kiseline i 197,7±13,6 mg g<sup>-1</sup> hlorogenske kiseline.



## **Measurement Uncertainty for the Determination of Monosaccharide Content in Different Olive Leaves Extracts Using Gas Chromatography and Mass Spectrometry**

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### **Keywords:**

olive biomass,  
olive leaves,  
glucose,  
mannitol,  
gas chromatography,  
mass spectrometry.

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**Abstract:** Analytical method for the simultaneous determination of D-glucose and D-mannitol in olive biomass, was developed, optimized and validated. The measurement uncertainty for the determination of glucose and mannitol concentrations was evaluated and all possible sources of measurement uncertainty arising from the analytical procedure implementation were determined. Olive leaves were extracted by subcritical water extraction at different temperatures (80° C, 120° C and 180°C), different pressure levels (30 bar, 80 bar, 100 bar and 150 bar) and different extraction time (5 min, 10 min, 30 min, 60 min, 90 min). Samples were analyzed by gas chromatography and mass spectrometry (GC-MS) and sugars were quantified as oxime- trimethylsilyl derivatives from corresponding calibration curves using phenyl-β-D-glucopyranoside as an internal standard (ISTD). The results confirmed that the highest contents of glucose (2,576 g/L) and mannitol (4,031 g/L) were present in the sample extracted with subcritical water at 80°C for 90 min and the minimum contents of glucose (0,489 g/L) and mannitol (1,321 g/L) were determined in the sample extracted with subcritical water, at 180°C for 90 min.

### **Sažetak**

Analitička metoda za simultano određivanje D-glukoze i D-manitola u biomasi od maslina je razvijena, optimizirana i validirana. Mjerna nesigurnost za određivanje koncentracije glukoze i manitola je evaluirana, određeni su svi mogući izvori mjerne nesigurnosti proizašli iz primjenjenog analitičkog postupka. Ekstrakcija maslinovog lišća je izvedana subkritičnom vodenom ekstrakcijom na različitim temperaturama (80°C, 120°C i 180°C), različitim pritiscima (30 bara, 80 bara, 100 bar i 150 bara) i različitim vremenima ekstrakcije (5 min, 10 min, 30 min, 60 min, 90 min). Uzorci su analizirani gasnom hromatografijom i masenom spektrometrijom (GC-MS) a šećeri su kvantificirani kao derivati oksimtrimetilsilila iz odgovarajućih kalibracionih krivulja pomoću fenil-β-D-glukopiranozida kao internog standard. Rezultati su potvrdili da je najveći sadržaj glukoze (2,576 g/L) i manitola (4,031 g/L) bio prisutan u uzorku ekshiranom subkritičnom vodom na 80°C u toku 90 min a minimalni sadržaj glukoze (0489 g/L) i manitola (1,321 g/L) nađen je u uzorku ekshiranom subkritičnom vodom na 180°C za 90 min.



## **Chemically modified Silica gel with Zirconium (IV) Oxychloride Octahydrate for Solid Phase Extraction and Preconcentration of Cr (III) and Pb (II)**

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**Keywords:**

Silica gel,  
zirconium (IV) oxychloride octahydrate,  
chromium,  
lead,  
SPE,  
FAAS.

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**Abstract:** Silica gel modified with zirconium (IV) oxychloride octahydrate was used for the preconcentration of chromium and lead prior to their determination by flame atomic absorption spectrometry (FAAS). Surface characterization of the silica gel (activated silica gel) before and after chemical modification was determined by Fourier Transform Infrared Spectrometer (FT-IR) in order to ensure the immobilization of zirconium (IV) oxychloride octahydrate onto the silica gel surface. The retention and recovery of the analytes were studied by applying the column technique. The experimental parameters, such as effect of pH of the sample, mass of silica and preconcentration factor (sample volume) were determined. At optimum conditions (pH 9, mass of silica gel 250 mg, preconcentration factor 10), the recoveries of Cr (III) and Pb (II) were 101 % and 98 %, respectively, with relative standard deviations (RSDs) of approximately  $\pm 2$  %. Detection limits of the method ( $3.3s_b$ ) were  $41.8 \mu\text{g L}^{-1}$  and  $69.7 \mu\text{g L}^{-1}$  for Cr (III) and Pb (II), respectively. Good results of recovery show that the used method can be applied to preconcentration of chromium and lead from aqueous solutions that contain low concentrations of these metals.

**Sažetak**

Silika gel modificiran sa cirkonijum (IV) oksihlorid oktahidratom korišten je za prekoncentriranje hroma i olova prije njihovog određivanja pomoću plamene atomske apsorpcione spektrometrije. Karakteriziranje površine silika gela (aktivirani silika gel) prije i nakon modificiranja određeno je sa Fourier infracrvenom spektroskopijom (FTIR) kako bi se potvrdila imobilizacija cirkonijum (IV) oksihlorid oktahidrata na površini silika gela. Step en zadržavanja, odnosno *recovery* vrijednost analita je određen primjenjujući tehniku kolona. Određeni su eksperimentalni parametri: utjecaj pH vrijednosti uzorka, mase silika gela i prekoncentracioni faktor (volumen uzorka). Pri optimalnim uslovima (pH 9, masa silika gela 250 mg, prekoncentracioni faktor 10) vrijednosti *recovery*-ja su bile 101 % za Cr i 98 % za Pb uz relativnu standardnu devijaciju (RSD) od oko  $\pm 2$  %. Detekcioni limit metode ( $3.3s_b$ ) za hrom iznosi  $41.8 \mu\text{g L}^{-1}$  dok za olovo  $69.7 \mu\text{g L}^{-1}$ . Dobri rezultati *recovery*-ja pokazuju da korištena metoda može biti primjenjena na prekoncentriranje hroma i olova iz vodenih rastvora koji sadrže niske koncentracije ovih metala.



## **Sediment quality assesment in the Bosna River Basin**

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### **Keywords:**

Sediment,  
priority substances,  
heavy metals,  
PCB,  
limit criteria,  
heavy metals

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**Abstract:** Sediment quality assessment is one of the possibilities for elaboration of real picture of risks for water ecosystems. Complex screening of sediment structure and quality in Bosna River Basin was realized in the period of August/November, 2012. Scope of the sediment quality assessment was substances proposed by Directive 2008/105/EC that have tendency to accumulate in sediments plus heavy metals relevant for Danube River Basin (As, Cr, Cu, Zn) and PCB. Only the fraction of sediment with grain size less than 0,063 mm was analyzed because of the bioavailability. Measured data were standardized at 10% content of organic matter and 25 % ratio of lutit fraction. Results of this survey show that the most problematic compounds in Bosna River sediments are heavy metals and polycyclic aromatic hydrocarbons. The most significant contributor of heavy metals is agglomeration Zenica with its industrial surroundings. Agglomeration Sarajevo contributes to this kind of pollution with Hg and Cr. PAH substances mainly fenantrene, antracene, fluorantene and pyrene pose risk to ecosystem along the whole Bosna River. The highest concentrations of PAH was identified in the last monitoring site before the confluence of Bosna River with Sava River.

### **Sažetak**

Procjena kvaliteta sedimenta je jedna od mogućnosti za dobijanje stvarne slike rizika za riječne ekosisteme. Kompleksno snimanje strukture i kvaliteta sedimenta riječnog bazena rijeke Bosne realizovano je u periodu avgust/novembar, 2012. Ispitivanje kvaliteta sedimenta se baziralo na analizi supstanci koje su predložene Direktivom 2008/105/EC i koje imaju tendenciju akumulacije u sedimentu, zatim teški metali relevantni za riječni bazen Dunava (As, Cr, Cu, Zn) i PCB. Analizirana je samo frakcija sedimenta granulacije manje od 0,063 mm, zbog biodostupnosti. Dobivene vrijednosti su normalizirane u odnosu su na sadržaj organske materije od 10 % i sadržaj frakcije gline od 25 %. Rezultati ovog istraživanja ukazali su na teške metale i policiklični aromatski ugljikovodike kao najproblematičnije supstance u sedimentu rijeke Bosne. Zenica zajedno sa svojim industrijskim okruženjem najviše doprinosi koncentraciji teških metala u sedimentu, dok Sarajevo sa svojim industrijskim okruženjem doprinosi ovoj vrsti zagađenja sa Hg i Cr. PAH-ovi, uglavnom, fenantren, antracene, fluorantene i piren uzrokuju rizik po ekosistem duž cijele rijeke Bosne. Najviša koncentracija PAH-ova je identificirana na zadnjoj monitoring tački, neposredno prije ušće rijeke Bosne u rijeku Savu.



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Abstract

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## **Analysis of trend, seasonality and the relationship between nutrients, oxygen and organic eco-chemical parameters of the Danube in Serbia**

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### **Keywords:**

Danube,  
the spatial and temporal trend,  
linear regression,  
PCA,  
inear regression

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**Abstract:** This work was performed trend analysis and seasonality of the following eco-chemical parameters: ammonium ion, BOD, phosphates, COD, chlorides, nitrates, dissolved oxygen, sulfate, total phosphorus, UV extinction, oxygen saturation on the river Danube. Some of these parameters is visible clearly pronounced linear time trend, with some seasonality can be seen throughout the course, with some parameters there are a large number of outliers, which makes it difficult to register a trend and seasonality types, as well as discontinuities in the time series, which also negatively affect the registration trend and seasonality. PCA (Principal Component Analysis) analysis, we found four factors that are present at the measuring points in the upper reaches of the Danube.

The analysis of the impact can be concluded that the interference can significantly affect the performance of statistical tests and therefore with great attention must choose statistical tests that will be used to select the test that has the best balance of resistance and statistical power. This will get the results on the basis of which they can bring the most authoritative conclusions about the nature and relationships of the tested eco-chemical parameters.

### **Sažetak**

U radu je vršena analiza trenda i sezonalnosti na sledećim eko-hemijskim parametrima: amonijum-jon, BPK, fosfati, HPK, hlorigi, nitrati, rastvoreni kiseonik, sulfati, ukupni fosfor, UV-ekstinkcija, zasićenje kiseonikom na vodotoku Dunava. Kod nekih od navedenih parametara uočljiv je jasno izražen linearni vremenski trend, kod nekih se može uočiti sezonalnost na čitavom toku, kod pojedinih parametara postoji veliki broj ekstremnih vrednosti koji otežava registrovanje trenda i vrste sezonalnosti, kao i diskontinuiteti u vremenskoj seriji koji takođe negativno utiču na registrovanje trenda i sezonalnosti. PCA analizom pronašli smo četiri faktora koja su prisutna na mernim mestima u gornjem toku Dunava. Analizom uticaja može se zaključiti da smetnje mogu bitno da utiču na performanse statističkih testova i zbog toga se s velikom pažnjom moraju birati statistički testovi koji će se koristiti da bi se odabrao test koji ima najbolji odnos otpornosti i statističke moći. Na ovaj način će se dobiti rezultati na osnovu kojih se mogu doneti najmerodavniji zaključci o prirodi i odnosima ispitivanih eko-hemijskih parametara.



## **Removal of natural organic matter from water – application of advanced oxidation processes**

**Habuda-Stanić M., Gašo-Sokač D., Nujić M.**

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**Keywords:**

natural organic matter,  
groundwater,  
NOM removal,  
advanced oxidation processes

**Abstract:** Natural organic matter (NOM) is a complex mixture of organic compounds, mostly fulvic and humic acids, hydrocarbons, proteins and amino acids derived from plant and/or microbial residues. The occurrence of elevated concentration of natural organic matter in water that has been used as source of drinking water, can negatively affect final organoleptic and chemical quality of treated water, especially when chlorine is used as disinfectant, since NOM serve as reactants in the formation of potentially harmful disinfection by-products (DBPs) such as trihalomethanes (THMs), haloacetic acids (HAAs), bromate, chlorate etc. Removal of NOM from water and thus the prevention of DBPs formation can be achieved using following water treatment processes: coagulation/filtration method, ion exchange, adsorption onto activated carbons and membrane filtration while an alternative and most environmentally accepted method for NOM removal represent the application of advanced oxidation processes (AOPs). AOPs imply formation of highly reactive hydroxyl radicals and its reactions caused by usage of ozone, hydrogen peroxide and/or UV light, as well as Fenton's reagent and TiO<sub>2</sub> catalysis. This study reports the latest achievements in NOM removal from water using the AOPs.

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### **Sažetak**

Prirodne organske tvari su kompleksna smjesa organskih spojeva, uglavnom fulvinske i huminske kiseline, ugljikovodika, proteina te aminokiselina biljnog i/ili mikrobiološkog porijekla. Pojava povećanih koncentracija prirodnih organskih tvari u vodama koje se koriste kao sirovina u proizvodnji vode za piće, negativno utječu na organoleptičke karakteristike i kemijsku ispravnost vode za piće, naročito kada se dezinfekcija vode provodi primjenom elementarnog klora pri čemu prirodne organske tvari sudjeluju u reakciji nastanka štetnih dezinfekcijskih nusprodukata kao što su trihalometani, haloacetična kiselina, bromati, klorati itd.

Uklanjanje prirodnih organskih tvari iz vode te sprječavanje nastanka dezinfekcijskih nusprodukata moguće je postići slijedećim procesima obrade voda: koagulacijom s filtracijom, ionskom izmjenom, adsorpcijom na aktivne ugljene te membranskom filtracijom, no ekološki najprihvatljiviji način uklanjanja prirodnih organskih tvari predstavlja primjena naprednih oksidacijskih procesa (eng. *advanced oxidation processes*, AOPs). Napredni oksidacijski procesi podrazumijevaju reakcije u kojima nastaju i reagiraju reaktivni hidroksilni radikali primjenom ozona, vodikova peroksida i/ili UV svjetla te Fentonova reagensa ili fotokatalitički aktivnog titanijeva dioksida. U ovom radu biti će prikazana najnovija dostignuća u području uklanjanja prirodnih organskih tvari iz vode primjenom naprednih oksidacijskih procesa.



## The Application of Microfluidics for Anionic Surfactant Determination

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### Keywords:

anionic surfactant,  
dimethyldioctadecylammonium-  
tetraphenylborate,  
FIA/SIA,  
microfluidic,  
potentiometric.

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**Abstract:** Anionic surfactants (AS) are represented with 58% of the total global production of surfactants. Standard methods for determination of AS in wastewater and commercial products are MBAS (Methylene Blue Active Substances), and two-phase titration. Because of the shortcomings that both standard methods have, there is a need for replacing them. Potentiometric measurements with the ion-selective electrode (ISE) solved most of the problems of standard methods. Microfluidics methods offer new possibilities in determining AS exploiting the ability to control molecules in space and time. Flow injection analysis (FIA) and Sequential injection analysis (SIA) using the screen-printed microsensor with incorporated new ionic associate (dimethyldioctadecylammonium-tetraphenylborate, DDA-TPB) as a detector, were used for determination of the AS. The measured electromotive force (EMF) is a function of the AS concentration in the sample. Sodium dodecyl sulfate (NaDDS) and sodium dodecyl benzene sulfonate (NaDBS) were used for calibration. The real samples are commercial products and industrial wastewater with different contents of AS. The sensor showed a sub-Nernstian slope for both anionic surfactants. Measurements were performed with adjusted ionic strength and pH. The results showed good agreement with those obtained using the standard methods.

### Sažetak

Anionski tenzidi (AS) su s 58% zastupljeni u ukupnoj svjetskoj proizvodnji tenzida. Standardna metoda za određivanje AS u otpadnim vodama je spektrofotometrijska MBAS (*Methylene blue active substances*) metoda dok se za određivanje AS u komercijalnim proizvodima koristi titracija u dvije faze. Zbog nedostataka koje imaju obje standardne metode, postoji potreba za iznalaženjem novih metoda koje bi ih mogle zamijeniti. Potenciometrijska mjerenja ionsko-selektivnom elektrodom s tekućom membranom (ISE) rješavaju većinu problema standardnih metoda. Mikrofluidičke metode nude nove mogućnosti u određivanju AS iskorištavajući mogućnost kontrole molekula u vremenu i prostoru. Metoda injektiranjem u protok (FIA) i sekvencijska injekcijska analiza (SIA) uz korištenje *screen-printed* mikrosenzora s novim ionskim asocijatom (dimetildioktadecilamonijevim-tetrafenilboratom, DDA-TPB) kao detektorom, korištena je za određivanje AS. FIA/SIA sustav je kućne izrade kao i program kojim je sustav vođen. Izmjerena elektromotorna sila (EMS) u funkciji je koncentracije AS u uzorku. Natrijev dodecil sulfat (NaDDS) i natrijev dodecil benzensulfonat (NaDBS) upotrebljeni su za kalibraciju, a realni uzorci su uzorci komercijalnih proizvoda i industrijske otpadne vode s različitim sadržajem AS. Senzor je pokazao sub-Nerstovski nagib za oba anionska tenzida. Mjerenja su izvedena uz podešenu ionsku jakost i pH vrijednost. Dobiveni rezultati pokazali su dobro slaganje s rezultatima dobivenim standardnim metodama.



## **Simultaneous preconcentration of Cu, Fe and Mn on solid sulphur from river water samples prior to determination by FAAS**

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**Keywords:**

solid sulphur,  
preconcentration,  
metals,  
FAAS.

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**Abstract:** In the present study a simple and fairly rapid solid phase extraction (SPE) procedure has been applied. A mini-column loaded with sulphur powder as a nontoxic and low cost adsorbent for simultaneous preconcentration of copper, iron and manganese prior to determination by flame atomic absorption spectrometry, was used. The recoveries for the analytes under the optimum analytical conditions (pH 8; preconcentration factors of 10; flow rate 1 mL min<sup>-1</sup>; amount of adsorbent 2.5 g) from solutions of known low metal concentrations were 104%, 109% and 90% for Cu, Fe and Mn, respectively. Furthermore, the adsorbed metal ions were eluted from the column by 1 mol L<sup>-1</sup> of HNO<sub>3</sub> in methanol. The described procedure was tested on real water samples of Vogošća river. The used method has been successfully applied to the preconcentration and determination of trace Cu, Fe and Mn from river water samples. After preconcentration, the concentrations of Cu and Mn less than 0.05 µg mL<sup>-1</sup>, and Fe less than 0.5 µg mL<sup>-1</sup> were determined in river water samples.

### **Sažetak**

U ovom radu primijenjen je jednostavan i relativno brz postupak ekstrakcije na čvrstom adsorbentu. Korištena je mini kolona punjena sumporom u prahu kao netoksičnim i lako dostupnim adsorbentom za simultano prekoncentriranje Cu, Fe i Mn prije njihovog određivanja atomskom apsorpcionom spektrometrijom - plamenom tehnikom. Dobivene vrijednosti *recovery*-ja analita pri optimalnim analitičkim uslovima (pH 8; prekoncentracioni faktor 10; brzina protoka uzorka 1 mL min<sup>-1</sup>; količina adsorbenta 2.5 g), iz rastvora poznate niske koncentracije metala, bile su 104%, 109% i 90% za Cu, Fe i Mn, respektivno. Adsorbovani metalni joni su eluirani iz kolone sa 1 mol L<sup>-1</sup> HNO<sub>3</sub> u metanolu. Prethodno opisana procedura testirana je na realnim uzorcima vode Vogošćanske rijeke. Korištena metoda uspješno je primijenjena za prekoncentriranje i određivanje tragova Cu, Fe i Mn iz uzoraka riječne vode. Nakon prekoncentriranja određena je koncentracija Cu i Mn niža od 0.05 µg mL<sup>-1</sup>, odnosno Fe niža od 0.5 µg mL<sup>-1</sup> u uzorcima riječne vode.





## **Determination of Total Iron Content in Black Tea**

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### **Keywords:**

iron,  
black tea,  
spectrophotometry,  
mineral digestion

**Abstract:** The aim of this work was the assessment of total iron (Fe) content in some black tea brands by using a mineral digestion and Spectrophotometric method. In 4 samples of black tea from different manufactures and with 3 parallel were prepared by digestion and oxidation with a mixture of sulphuric and nitric acid. The total Fe content in analyzed black tea varies from 21 mg Fe/kg to 37,6 mg Fe/kg. The used spectrophotometric method is simple and sensitive method that can be applied for the determination of total Fe content in plant material. Results of this analysis suggest regular consumption of black tea as a significant natural source of organic-trace elements Fe for metabolic needs and their benefits on human health.

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### **Sažetak**

Cilj ovog rada je procjena ukupnog sadržaja željeza (Fe) u nekim brendiranim crnim čajevima primjenom mineralne digestije i spektrofotometrijske metode. 4 uzorka crnog čaja različitih proizvođača i u 3 paralelke pripremljeni su digestijom i oksidacijom s smjesom sumporne i nitratne kisleine. Sadržaj ukupnog željeza u uzorcima crnog čaja varirao je od 21 mg Fe/kg do 37,6 mg Fe/kg. Primjenjena metoda spektrofotometrije je jednostavna i osjetljiva i može biti primjenjena za određivanje ukupnog sadržaja željeza u biljnom materijalu. Rezultati ove analize sugerišu na redovnu konzumaciju crnog čaja kao značajnog prirodnog izvora organskog željeza u tragovima za metaboličke potrebe i njegove benefite na ljudsko zdravlje.



## **Analysis of the soil in the vicinity of the mine "Bužim" - northwestern part of Bosnia and Herzegovina**

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### **Keywords:**

Soil,  
X-ray analysis of soil,  
Minerals,  
Identification of minerals,  
Manganese mine "Bužim"

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**Abstract:** In order to determine the individual Mn species, the investigations of soil were performed in the area of Bužim in the northwestern part of Bosnia and Herzegovina. Samples were taken at seven locations, at the manganese mine and its surrounding area. It was determined that the chemical properties of soil considerably affect the distribution of the individual species of Mn in the soil. The acidity of soil may cause Mn oxide-minerals to dissolve and release a significant amount of Mn ions in soil, therefore increasing the toxicity of soil for many plants. The method of X-ray analysis was used in our investigation to determine the mineral content of the studied soils. Based on these studies and chemical analysis, we were able to determine the presence of certain species of Mn.

All samples were scanned in an automatic X-ray goniometer with CuK $\alpha$  radiation and monochromatic graphite, whereat there was recorded the area of 2-64 ° in 2  $\Theta$ .

By using X-ray analysis, it was established a form of manganese presence in the soil or mineral content consisting of Mn, and the presence of other minerals such as quartz, albite, mica-muscovite, microcline, chlorite and kaolin minerals.

### **Sažetak**

U cilju određivanja pojedinih specija Mn vršena su istraživanja tla na području Bužima u sjeverozapadnom dijelu Bosne i Hercegovine. Uzorci su uzeti na sedam lokaliteta, i to unutar rudnika mangana i u njegovoj okolini. Utvrđeno je da hemijska svojstva tla utječu na raspodjelu pojedinih specija Mn u tlu. Kiselost tla može uzrokovati da se mineralni oksidi Mn otope i otpuštaju dovoljno iona mangana u tla i na taj način čini tla toksičnijim za mnoge biljke. Za naša istraživanja smo koristili metodu rentgenske analize da bi odredili mineralni sadržaj istraživanog tla. Na osnovu tih istraživanja i hemijskih analiza mogli smo utvrditi prisustvo pojedinih specija Mn. Svi uzorci su snimani na automatskom rentgenskom goniometru uz CuK $\alpha$  zračenje i grafitni monohromator, pri čemu je snimano područje od 2-64° u 2  $\Theta$ . Rentgenskom analizom ustanovljen je oblik mangana prisutan u tlu odnosno mineralni sadržaj u čijem sastavu je Mn te prisustvo drugih minerala kao što je kvarc, albit, liskun-muskovit, mikroklin, klorit i kaolinski minerali.



## **Validation of method for the determination of mercury in the auxiliary substances Azorubine 21% and Azorubine 85% using Cold-vapor Atomic Absorption Spectrometry**

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### **Keywords:**

Azorubine,  
Hg determination,  
Cold-vapor Atomic  
Absorption Spectrometry,  
microwave acid  
digestion

**Abstract:** Heavy metals, such as Mercury (Hg), are sometimes found in pharmaceuticals. Although the concentration of those elements is very low, their control is very important because of its toxicity. Permissible concentration of Mercury (Hg) in Azorubine 21% and Azorubine 85% is prescribed by the Directive of the European Commission concerning the specific purity criteria on food coloring. As the Pharmaceutical company "Bosnalijek" uses auxiliary substances such Azorubine 21% and Azorubine 85% in its production process, the quality of these substances is monitored prior to usage. The focus of this paper is on validating precise and accurate methods of Hg determination in auxiliary substances mentioned above, by Cold-vapor Atomic Absorption Spectrometry after microwave acid digestion of solid samples. The best results at measuring concentration of mercury were achieved using the acid digestion of mixing of test samples with 1 mL MQ water + 1 mL 65% HNO<sub>3</sub> + 1 mL 70% HClO<sub>4</sub> + 5 mL 96% H<sub>2</sub>SO<sub>4</sub> and digesting it for 30 min. on 1000 W. The concentration of Hg in the sample is determined by Cold-vapor Atomic Absorption Spectrometry with sodium borohydride as a reducing agent. The method was successfully validated and can be applied for the determination of Hg in solid samples of Azorubine 21% and Azorubine 85%, with value of recovery factor of 95% - 104% and 96-105%, respectively.

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### **Sažetak**

Teški metali, kao što je živa (Hg), ponekad se mogu naći u farmaceutskim proizvodima. Iako je njihova koncentracija veoma niska, praćenje istih je veoma važno zbog njene toksičnosti. Dozvoljena koncentracija žive (Hg) u Azorubine 21% i Azorubine 85% je propisana Direktivom Europske komisije u vezi s posebnim kriterijima čistoće boja za hranu. Kako farmaceutska kompanija "Bosnalijek" koristi pomoćne supstance Azorubin 21% i Azorubin 85% u svom proizvodnom procesu prati se njihov kvalitet prije upotrebe. Fokus ovog rada je na validaciji precizne i tačne metode određivanja Hg u Azorubinu 21% i Azorubinu 85%, koristeći tehniku hladnih para atomske apsorpcione spektrometrije nakon mikrotalasne kiseline digestije čvrstih uzoraka. Najbolji rezultati pri mjerenju sadržaja žive su postignuti nakon mikrotalasne digestije ispitnih uzoraka smjesom koja sadrži 1 mL MQ vode + 1 mL 65% HNO<sub>3</sub> + 1 mL 70% HClO<sub>4</sub> + 5 mL 96% H<sub>2</sub>SO<sub>4</sub> u periodu od 30 min. na 1000 W. Koncentracija Hg u uzorku je nakon toga određena tehnikom hladnih para atomske apsorpcione spektrometrije uz natrij borohidrid kao rededucens. Metoda je uspješno validirana i može se primjenjivati za određivanje Hg u čvrstom uzorku Azorubina 21% i Azorubina 85%, uz vrijednost *recovery*-ja faktora od 95% -104% i 96-105%, redom.



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## **Content of selected toxic and essential metals in muscle tissue and gills of *Oncorhynchus mykiss* (Walbaum, 1792) from stream and fishpond from Bugojno (Bosnia and Herzegovina)**

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**Keywords:**

toxic metals,  
essential metals,  
fish, gills,  
muscle tissue,  
fishpond,  
stream.

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**Abstract:** In this study toxic and essential metal concentrations were determined in muscle tissue and gills of trout (*Oncorhynchus mykiss*, Walbaum, 1792) from stream and fishpond from Bugojno. The concentrations of metals Cd, Co, Cr, Fe, Mn, Pb, Ni, Zn, Ca, Mg and Na were determined by flame atomic absorption spectrometry (FAAS). The obtained results showed that concentrations of toxic metals in muscle tissue did not exceed maximum allowable concentrations (MACs) prescribed by EU legislation while the MACs values of metals in gills are not defined by this legislation. Concentrations of metals in gills were higher than the concentrations in fish muscle tissue for most analyzed metals. Thus, the concentration of Fe in gills of the trout from the stream is 15, and Zn in gills of the trout from the fishpond is 21 times higher than the content of Fe and Zn in muscle tissue. The analysis confirmed that the concentration of metals in fish is in positive correlation to the concentration of metals in water. The concentrations of toxic metals were higher in trout from the fishpond, while the concentrations of essential metals (except Zn) were lower in relation to the trout from the stream, which could be related to their nutrition.

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### **Sažetak**

U ovom istraživanju određena je koncentracija toksičnih i esencijalnih metala u mesu i škragama Kalifornijske pastrmke (*Oncorhynchus mykiss*) iz ribnjaka i potoka sa lokaliteta Bugojna. Za određivanje koncentracije metala Cd, Co, Cr, Fe, Mn, Pb, Ni, Zn, Ca, Mg i Na korištena je atomska apsorpciona spektrometrija-plamena tehnika (FAAS). Dobivene koncentracije toksičnih metala u mesu ribe ne prelaze maksimalno dozvoljene koncentracije prema legislativi EU (Evropske Unije), dok iste u škragama nisu propisane ovom legislativom. Koncentracije metala u škragama su veće u odnosu na koncentracije u mesu ribe za većinu analiziranih metala. Tako je koncentracija Fe u škragama pastrmke iz potoka 15, a Zn u škragama pastrmke iz ribnjaka 21 puta veća u odnosu na sadržaj Fe odnosno Zn u mesu. Analizom je potvrđeno da je koncentracija metala u ribama u pozitivnoj korelaciji sa koncentracijom metala u vodi. Koncentracije toksičnih metala su više kod pastrmke iz ribnjaka dok je koncentracija esencijalnih metala (sa izuzetkom Zn) niža u odnosu na ribe iz potoka što se može povezati sa načinom ishrane.



## **Validation of an AAS-VGA method for determination of arsenic in titanium dioxide**

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### **Keywords:**

Validation,  
Atomic Absorption  
Spectrophotometry Vapor Generation  
Accessory (AAS-VGA),  
arsenic,  
titanium dioxide.

**Abstract:** In analytical laboratories atomic absorption spectrophotometry measurements are frequently used for determination of metal content in some raw materials for drug production. Atomic absorption spectrophotometry with vapour generation assembly (AAS-VGA) is well known technique for the trace analysis of arsenic. The aim of this study was to provide better and more accurate qualitative and quantitative analysis of arsenic in the raw material like Titanium dioxide, and validation of this method was performed thorough the following tests: selectivity, linearity, LOD, LOQ, accuracy, precision, intermediate precision and robustness. Measurements of the absorbance were done by using AAS Varian 240 FS – VGA instrument. Furthermore, standard solutions of arsenic in different concentration - target concentration of arsenic was 5 ppm, spiked solutions, test solutions and blank were prepared for the analysis. For validation of this method the literature of vendor and Ph.Eur. was used. The results are satisfactory and are included into the set limits: selectivity-complies; linearity- $r:0,9986$ ; LOD: $0,7\text{ppm}$ ; LOQ: $2,0\text{ppm}$ ; accuracy: $104,7\%-111,5\%$ ; precision-CV: $0,68\%$ ; intermediate precision-CV: $0,69\%$ ; robustness-solution stability D: $3,8\%$ , method parameters CV:  $0,87\%$ . Validated method is reliable and suitable for analysis of arsenic in titanium dioxide.

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### **Sažetak**

U analitičkim laboratorijama atomska apsorpciona spektrofotometrija često se koristi za određivanje prisustva metala u materijalima za farmaceutsku upotrebu. Atomska apsorpciona spektrofotometrija sa dodatkom VGA jedinice (AAS-VGA) dobro je poznata tehnika za analizu tragova arsena. Cilj ovog istraživanja bio je pružiti bolju i tačniju kvalitativnu i kvantitativnu analizu arsena u materijalima poput titan dioksida, i validacija ove metode je provedena ispitivanjem sljedećih parametara: selektivnost, linearnost, LOD, LOQ, tačnost, preciznost, intermedijarna preciznost i robusnost. Mjerenja apsorbanca su izvedena pomocu AAS Varian 240 FS - VGA instrumenta. Nadalje, za analizu su pripremljene standardne otopine arsena različitih koncentracija - ciljane koncentracija arsena je 5 ppm, otopine standardnog dodatka, otopine probe i slijepe probe. Za validaciju ove metode koristena je literatura proizvidjaca Varian, i vazece Ph.Eur. Rezultati su zadovoljavajuci, i ulaze u zadane granice: selektivnost – odgovara; linearnost –  $r:0,9986$ ; LOD: $0,7\text{ppm}$ ; LOQ: $2,0\text{ppm}$ ; tačnost: $104,7\%-111,5\%$ ; preciznost – CV: $0,68\%$ ; intermedijarna preciznost – CV: $0,69\%$ ; robusnost – stabilnost otopine D: $3,8\%$ , parametri metode CV:  $0,87\%$  Provjerena metoda je pouzdana i pogodna za analizu arsena u titan dioksidu.



## **Kinetic and Flow Injection Spectrophotometric Determination of N-Acetylcysteine ethyl ester Based on the Reduction of Copper(II)- neocuproine**

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### **Keywords:**

N-Acetylcysteine ethyl ester,  
Kinetic spectrophotometry,  
Fixed time method,  
Flow injection.

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**Abstract:** Kinetic spectrophotometric and flow injection methods of analysis were developed for the determination of a novel lipophilic cell-permeable cysteine derivative N-Acetylcysteine ethyl ester (NACET). The proposed methods are based on a reduction reaction of Cu(II)-neocuproine reagent to Cu(I)-neocuproine by NACET in a Britton-Robinson buffer solution. The absorbance of the generated Cu(I)-neocuproine complex is measured at 458 nm. In the kinetic procedure a fixed time method was utilized for the construction of calibration graphs, and the recorded peak height was used as a quantitative variable in the flow injection procedure. A linear calibration graph was obtained from  $1.0 \times 10^{-6}$  to  $1.0 \times 10^{-4}$  mol L<sup>-1</sup> of NACET by the use of the kinetic spectrophotometric method where the detection limit was  $4.7 \times 10^{-8}$  mol L<sup>-1</sup>. The linearity of the proposed flow injection method was obtained over the range  $2.0 \times 10^{-6}$  mol L<sup>-1</sup> –  $6.0 \times 10^{-5}$  mol L<sup>-1</sup> NACET, where the limit of detection was  $2.7 \times 10^{-7}$  mol L<sup>-1</sup>.

### **Sažetak**

Razvijene su kinetičke i FIA spektrofotometrijske metode analize određivanja novog lipofilnog spoja N-acetilcistein etil estera (NACET). Predložene metode se temelje na reakciji redukcije Cu(II)-neokuproina pomoću NACET-a u otopini Britton-Robinsonovog pufera pri čemu nastaje Cu(I)-neokuproin. Apsorbancija nastalog Cu(I)-neokuproin kompleksa je mjerena pri 458 nm. Kod kinetičkog postupka metoda određenog vremena se koristi za konstrukciju pravca umjeravanja, dok je zabilježena visina pika korištena kao kvantitativna varijabla kod protočne analize injektiranjem. Pravac umjeravanja je zabilježen kinetičkom spektrofotometrijskom metodom u području koncentracija NACET-a od  $1,0 \times 10^{-6}$  do  $1,0 \times 10^{-4}$  mol L<sup>-1</sup> gdje je granica dokazivanja  $4,7 \times 10^{-8}$  mol L<sup>-1</sup>. Predloženom FIA metodom zabilježena je linearnost u rasponu koncentracija od  $2,0 \times 10^{-6}$  mol L<sup>-1</sup> do  $6,0 \times 10^{-5}$  mol L<sup>-1</sup>, s granicom dokazivanja  $2,7 \times 10^{-7}$  mol L<sup>-1</sup>.



## **A Study for the Method Transfer of Lisinopril dyhydrate and Hydrochlorotiazide from HPLC to RRLC**

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### **Keywords:**

HPLC (High performance liquid chromatography),  
RRLC (Rapid resolution liquid chromatography)

**Abstract:** Increasing demands for faster and more economical analysis initiated method development and validation on RRLC, an advanced form of conventional HPLC. A time and solvent consuming HPLC method for lisinopril dihydrate and hydrochlorothiazide assay (and determination of the related substances) was transferred and validated as a new RRLC method by using the new technique, i.e., the RRLC system with the short column (100 mm in length) and the low particle size of the packing (2.7  $\mu\text{m}$  in diameter).

Validation of methods consisted of an analysis of the following parameters: selectivity, linearity, limit of detection, limit of quantization, accuracy, precision and robustness of the method using the device RRLC Agilent 1200. The results are good in relation to its borders, so that it can be considered that the method is reliable, and its use in practice has led to significant savings in time and consumables.

The long-term investment benefit for the company are a small number of the instruments (savings in the initial investment), the small space needed for the instruments, the lowered costs of annual maintenance and calibration of the instrument, the lowered energy consumption, etc.

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### **Sažetak**

Sve veći zahtjevi za bržu i ekonomičniju analizu inicirali su razvoj i validaciju metoda na RRLC. HPLC metoda za određivanje sadržaja lizinopril dihidrata i hidrohlorotiazida i srodnih supstanci, transferirana je i validirana kao nova RRLC metoda, uz korištenje nove tehnike, odnosno RRLC sistema sa kraćom kolonom (dužina 100 mm) i dijametrom kolone od 2,7  $\mu\text{m}$ . Validacija metode sastojala se od analize sljedećih parametara: selektivnost, linearnost, limit detekcije, limit kvantizacije, tačnost, preciznost i robustnost metode, uz korištenje uređaja RRLC Agilent 1200. Dobiveni su dobri rezultati u odnosu na postavljene granice, tako da se može smatrati da je metoda pouzdana, a njena upotreba u praksi dovela je do znatne uštede u vremenu i potrošnom materijalu u laboratoriji. Dugoročno, benefit je i u uštedi na broju potrebnih instrumenata i troškovima za njihovo održavanje i kalibraciju instrumenata.