



University of Belgrade  
Technical Faculty in Bor

EcoTEK

31<sup>st</sup> International conference

# Ecological Truth & Environmental Research

Editor

Prof. Dr Snežana Šerbula

## PROCEEDINGS

Hotel Sunce, Sokobanja, Serbia  
18–21 June 2024

## PROCEEDINGS

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## **PREFACE**

*The 31<sup>st</sup> international conference Ecological Truth & Environmental Research – EcoTER'24 focuses on showing the latest research findings and innovations in the field of ecology, environmental protection and sustainable development. The conference will be held in Sokobanja (Serbia) in hotel Sunce in the period of 18–21 June 2024.*

*The aim of the conference is to connect the experts in various fields in order to transform attitudes and behaviors in everyday practices, as well as in the industry and economy sector which is essential for achieving the desired changes that our society must undergo.*

*The 31<sup>st</sup> international conference Ecological Truth & Environmental Research – EcoTER'24 is organized by the University of Belgrade, Technical Faculty in Bor, and co-organized by the University of Banja Luka, Faculty of Technology; the University of Montenegro, Faculty of Metallurgy and Technology – Podgorica; the University of Zagreb, Faculty of Metallurgy – Sisak; the University of Pristina, Faculty of Technical Sciences – Kosovska Mitrovica and the Society of Young Researchers – Bor.*

*These Proceedings encompass 119 papers from the authors coming from the universities, research institutes and industries in 15 countries: Brazil, Norway, USA, Spain, Austria, Libya, Italy, Israel, Slovenia, Croatia, Romania, Bulgaria, Montenegro, Bosnia and Herzegovina, North Macedonia, and Serbia. It is a great honor and pleasure to cordially wish a warm welcome to all the participants of the conference.*

*As a part of this year's conference, the 6<sup>th</sup> Student Section – EcoTERS'24 will be held. We appreciate the contribution of the students and their mentors who have also participated in the conference and hope that students will continue to explore and to be curious, since education is a never-ending process, and knowledge is continuously growing.*

*The organization of the EcoTER'24 conference has been financially supported by the Ministry of Science, Technological Development and Innovation of the Republic of Serbia.*

*The support of the Donors and their willingness and ability to cooperate has been of great importance for the success of the EcoTER'24 conference. The organizing committee would like to extend their appreciation and gratitude to the Platinum donors of the conference – Serbia ZiJin Copper doo Bor and HBIS SERBIA, to the Gold donor of the conference – Elixir Group, as well as to the Silver donor of the conference – Serbian Chamber of Engineers.*

*We would like to express our sincere appreciation to all the authors who have contributed to the Proceedings. We would also like to express our gratitude to the members of the scientific, organizing and honorary committees, reviewers, speakers, chairpersons and all the conference participants for their support of the EcoTER'24. Sincere thanks go to all the people who have contributed to the successful organization of the EcoTER'24.*

*Prof. Snežana Šerbula,*

*President of the scientific and organizing committee*



## TABLE OF CONTENTS

### Plenary Lectures

**Branko Bugarski**

ELECTROSTATIC DISPERSION OF POLYMER SOLUTIONS IN THE PRODUCTION OF MICROGEL BEADS CONTAINING BIOCATALYST 1

**Anupama Ghosh, M. R. Del Grande, L. T. Teixeira, S. Letichevsky, C. A. Senna, M. D. Carbajal Ccoyllo, J. F. Chaves e Silva, V. C. Gois de Oliveira, R. N. Correia de Siqueira**

HEAT TREATMENT OF IRON-ADSORBED FUNCTIONALIZED NANOCELLULOSE FIBERS IN ORDER TO SYNTHESIZE HYBRID INORGANIC-CARBON MATERIAL 8

**Alena Bartonova**

ENVIRONMENTAL PROTECTION: WHY IS EUROPE'S AIR (MOSTLY) SO CLEAN? 14

### Invited Lectures

**Nevenka Rajić, J. Pavlović**

APPLICATION OF NATURAL ZEOLITE – CLINOPTILOLITE IN WATER TREATMENT BY ADSORPTION AND PHOTOCATALYSIS 17

**Dušan Nikolić, A. Tasić**

THE EUROPEAN PERCH (*Perca fluviatilis*) AS AN INDICATOR OF OCPs POLLUTION IN DIFFERENT TYPES OF RESERVOIRS IN SERBIA 24

**Jelena Korać Jačić, M. R. Milenković, D. Bartolić**

DEGRADATION OF TETRACYCLINE ANTIBIOTICS IN AQUATIC ENVIRONMENT BY UV IRRADIATION AND FERRIC ION PHOTOLYSIS 30

### Conference Papers

#### Environmental monitoring and impact assessment

**Aleksandra Papludis, S. Alagić, S. Milić, J. Nikolić, I. Zlatanović, S. Jevtović, V. Stankov Jovanović**

NAPHTALENE SCREENING IN BOR'S MUNICIPALITY BASED ON ITS CONCENTRATIONS IN LEAVES AND STEMS OF *Hedera helix* L. 38

**Darko Anđelković, M. Branković**

APPLE PEEL AS A BARRIER TO PESTICIDES MIGRATION INTO DEEPER FRUIT PARTS 43

<b>Darko Anđelković, M. Branković</b>	PERFORMANCES OF QuEChERS BASED GC-MS AND LC-MS/MS METHODS FOR PESTICIDES ANALYSIS IN APPLES	49
<b>Darko Anđelković, M. Branković</b>	COMPARISON OF PESTICIDES STABILITY STORED IN TWO SOLVENTS OF DIFFERENT VISCOSITY	55
<b>Milena Tadić, I. Nikolić, D. Đurović, N. Cupara, J. Vuković</b>	TRihalOMETHANES CONTENT IN HOTEL'S SWIMING POOLS WATER IN A SOUTH OF MONTENEGRO	61
<b>Jelena Vranković, K. Jovičić, V. Đikanović</b>	FIRST LINE DEFENCE ANTIOXIDANT ENZYMES IN <i>Blicca bjoerkna</i> (LINNAEUS, 1758) FROM THE BELGRADE SECTION OF THE DANUBE RIVER	66
<b>Miomir Mikić, R. Marković, V. Marjanović, R. Rajković, M. Jovanović</b>	REcULTIVATION OF RTH FLOTATION TAILINGS IN BOR, SERBIA	71
<b>Miomir Mikić, V. Marjanović, R. Marković, M. Jovanović, R. Rajković</b>	MINING AND THE ENVIRONMENT, ENVIRONMENTAL IMPACT MONITORING PROGRAM FOR FLOTATION TAILING RTH-BOR, SERBIA	77
<b>Vesna Obradović, M. Perović, T. Vučković</b>	EVALUATING CORROSION AND BIOFOULING POTENTIAL BASED ON GROUNDWATER MICROBIOLOGICAL COMPOSITION	83
<b>Vesna Obradović, M. Perović, J. Lekić</b>	EVALUATION OF CORROSION POTENTIAL USING PHYSICO-CHEMICAL WATER QUALITY ASSESSMENT	89
<b>Jelena Čanak Atlagić, A. Marić, K. Jovičić, J. Stanković, V. Đikanović, T. Mitić, M. Raković</b>	QUESTIONING THE RESILIENCE OF THE DANUBE FISH FAUNA UNDER THE PRESSURE OF BELGRADE WASTEWATERS	95
<b>Vladan Marinković, M. Maksimović, M. Jovanović, S. Trujić</b>	MONITORING OF THE STATE OF THE ENVIRONMENT IN THE BOR DISTRICT, GIVEN THROUGH THE EXAMPLE OF THE DISTRIBUTION OF Pb IN THE SOIL LOCATED IN THE IMMEDIATE VICINITY OF THE BOR RIVER	101
<b>Mirjana Ocokoljić, Dj. Petrov, N. Galečić, D. Skočajić, D. Vujičić, J. Čukanović, I. Simović</b>	EFFECTIVENESS OF <i>Photinia × Fraseri</i> 'RED ROBIN' IN THE URBAN LANDSCAPE: TOWARDS OF CLIMATE CHANGE	106
<b>Mirjana Ocokoljić, Dj. Petrov, N. Galečić, D. Skočajić, D. Vujičić, J. Čukanović, I. Simović</b>	<i>Chaenomeles japonica</i> (Thunb.) Lindl. ex Spach IN THE DESIGN OF URBAN PARKS: LEARNING FROM NATURE	113

<b>Mirjana Ocokoljić, J. Čukanović, Dj. Petrov, N. Galečić, D. Skočajić, D. Vujičić, I. Simović</b> <i>Parthenocissus quinquefolia</i> L.: PHENOMONITORING IN BLUE-GREEN INFRASTRUCTURE OF BELGRADE AND NOVI SAD	119
<b>Bojana Tubić, J. Đuknić, K. Zorić, N. Popović, N. Marinković, M. Paunović, M. Raković</b> EFFECTS OF THE IRON GATE DAMS ON THE BENTHIC MACROINVERTEBRATE COMMUNITY	126
<b>Danica Bogdanović, T. Anđelković, I. Kostić Kokić, M. Milovanović</b> GC-MS QUANTITATIVE DETERMINATION OF PHTHALATES IN PVC ARTICLES INTENDED FOR CHILDREN'S USE	132
<b>Danica Bogdanović, T. Anđelković, I. Kostić Kokić, M. Milovanović</b> OPTIMIZATION OF LIQUID-LIQUID PHTHALATES EXTRACTION FROM ARTIFICIAL SALIVA	138
<b>Danica Bogdanović, T. Anđelković, I. Kostić Kokić, M. Milovanović</b> MIGRATION OF DI-2-ETHYLHEXYL PHTHALATE AND DI-N-OCTYL PHTHALATE FROM PVC ARTICLES TO ARTIFICIAL SALIVA	144
<b>Daliborka Stanković, D. Z. Rajković, M. Raković, S. Skorić</b> HAEMOSPORIDIAN PARASITES IN LONG-EARED OWLS WINTERING IN BANAT, SERBIA	150
<b>Nenad Zarić, I. Hotea, A. Lato, M. Zarić, F. Crista</b> UNVEILING PESTICIDE CONTAMINATION IN TRANSBOUNDARY WATERS: A CASE STUDY OF SERBIA AND ROMANIA	156
<b>Nenad Zarić, F. Crista, A. Berbecea, I. Hotea, L. Crista, M. Zarić</b> COMPARATIVE ANALYSIS OF PESTICIDE RESIDUES IN AGRICULTURAL SOILS OF SERBIA AND ROMANIA	160
<b>Milica Veličković, D. Voza</b> THE RELATIONSHIP BETWEEN PM <sub>10</sub> AND METEOROLOGICAL PARAMETERS CLOSE TO THE MINING AREA	164
<b>Biljana Budzakoska Gjoreska, S. Trajanovski</b> MACROZOOBENTHOS COMMUNITY AND ECOLOGICAL STATUS IN PRESVA LAKE (OTESHEVO, STENJE AND EZERANI) IN SPRING 2022	169
<b>Suzana Patcheva, J. Leshoski, E. Veljanoska Sarafiloska</b> PHYTOPLANKTON COMMUNITY AS BIOINDICATOR OF WATER TROPHIC STATE IN LAKE PRESVA	176
<b>Boris Novaković, M. Raković</b> EARLY, LATE AND OUT-BREEDING SEASON BIRD SINGING – EFFECTS OF CLIMATE CHANGE?	183
<b>Boris Novaković, M. Raković</b> THE USE OF HOA (HEMIPTERA-ORTHOPTERA-AVES) INDICATORS TO FORMULATE THE SERBIAN CLIMATE CHANGE INDEX (S <sub>CCI</sub> )	189

<i>Ana Marić, V. Nikolić, D. Škraba Jurlina, V. Sokolović, D. Miličić, T. Karan Žnidaršič, T. Kanjuh, P. Simonović</i>	ASSESSMENT OF NON-NATIVE SPECIES IMPACT ON FISH DIVERSITY IN THE ČELIJE RESERVOIR: IMPLICATIONS FOR CONSERVATION AND MANAGEMENT	194
<i>Ivana Jelić, A. Savić, T. Miljojčić, M. Rajković, M. Janković, N. Sarap, S. Dimović, M. Čurčić, V. Stanić, D. Antonijević, M. Šljivić-Ivanović</i>	THE IMPACTS OF WASTE MATERIALS UTILIZATION IN LIQUID RADIOACTIVE WASTE SOLIDIFICATION BY MORTAR MATRIX	200
<i>Stefan Đorđievski, M. Đukić, A. Petrović, D. Adamović, J. Petrović, Lj. Lekić</i>	INSIGHTS FROM THE DAILY MONITORING OF WATER QUALITY PARAMETERS IN CEROVO RIVER NEAR BOR CITY IN OCTOBER 2023	206
<i>Nataša Kojadinović, S. Đuretanović, A. Milošković, M. Radenković, M. Jakovljević, T. Veličković, M. Nikolić, V. Simić</i>	FISH DIVERSITY ASSESSMENT OF THE IBAR RIVER: A 20-YEAR PERSPECTIVE	212
<i>Milanka Negovanović, L. Kričak, S. Milanović, N. Simić, J. Majstorović</i>	APPLICATION OF EXPANSIVE MORTARS FOR THE FORMATION OF ARTIFICIAL SCREENS DURING BLASTING IN URBAN AREAS	216
<i>Snežana Šerbula, T. Kalinović, A. Radojević, J. Kalinović, J. Jordanović</i>	AIR POLLUTION IN THE BOR REGION FROM 1994 TO 2023	225
<i>Irena Blagajac, I. Samardžić</i>	CAUSES OF FLOODING AND MEASURES TO MITIGATE THE CONSEQUENCES – CASE STUDY OF RAKOVICA MUNICIPALITY (BELGRADE, SERBIA)	231
<b>Urban and industrial ecology</b>		
<i>Žarko Radović, N. Tadić</i>	SIMULATION OF THE EAF DUST RECYCLING	240
<i>Mirko Gojić, S. Kožuh, I. Ivanić, D. Dumenčić</i>	DEVELOPMENT OF METALLURGY AND ENVIRONMENTAL PROTECTION IN THE REPUBLIC OF CROATIA IN THE PERIOD FROM 1900 TO 2020	246
<b>Air, water and soil pollution, prevention and control</b>		
<i>Viša Tasić, T. Apostolovski-Trujić, V. Kamenović, B. Radović, I. Zlatković, N. Ristić, Z. Damnjanović</i>	APPLICATION OF LOW-COST NETWORK FOR URBAN MICROCLIMATE AND AIR QUALITY MONITORING	251

<i>Nebojša Tadić, Ž. Radović, A. Knežević</i>	
ANALYSIS OF THE INFLUENCE OF NATURAL GAS COMPOSITION AND EXCESS AIR COEFFICIENT ON COMBUSTION PRODUCTS	258
<i>Aleksandar Jovanović, N. Knežević, M. Bugarčić, J. Petrović, M. Sokić, M. Stevanović, A. Marinković</i>	
INVESTIGATION OF MULTI-CYCLE USAGE OF NANOPHOTOCATALYSTS IN DEGRADATION OF THIOPHANATE-METHYL	265
<i>Vesna Obradović, M. Perović, P. Pajić</i>	
PHYSICO-CHEMICAL AND MICROBIAL ANALYSIS IN SELECTED GROUNDWATER IN SERBIA	270
<i>Silvia Dimova, K. Zaharieva, O. Dimitrov, P. D. Petrov, H. Penchev</i>	
METHATHESIS SYNTHESIZED OLIGOMERIC POLYPHENYLACETYLENE AS STERIC STABILIZER OF CARBON NANOTUBES/PLANT EXTRACT SYNTHESIZED ZINC OXIDE HYBRIDS	276
<i>Miljan Marković, M. Gorgievski, N. Štrbac, V. Grekulović, M. Marković, K. Božinović, D. Jovanović</i>	
EQUILIBRIUM ANALYSIS OF COPPER IONS BIOSORPTION ONTO HAZELNUT SHELLS	282
<i>Vesna M. Marjanović, R. Marković, D. Božić</i>	
CALCULATION OF CALCIUM OXIDE CONSUMPTION IN THE MINE WASTEWATER TREATMENT FROM INACTIVE OPEN PITS OF THE COPPER MINE	287
<i>Marina Marković, M. Gorgievski, N. Štrbac, V. Grekulović, M. Marković, M. Zdravković, D. Jovanović</i>	
THERMODYNAMIC ANALYSIS AND INFLUENCE OF THE pH VALUE ON THE BIOSORPTION OF COPPER IONS ONTO HAZELNUT SHELLS	294
<i>Jelena Korać Jačić, D. Bartolić, M.R. Milenković</i>	
THE IMPACT OF FERROUS AND FERRIC IONS ON DEGRADATION OF ANTIHYPERTENSIVE DRUG DIHYDRALAZINE IN IRON-BASED FLOCCULATION AND COAGULATION METHODS FOR WASTE WATER TREATMENT	299
<i>Berina Sejdinović</i>	
OILY WASTEWATER	305
<i>Vesela Radović, S. Krnjajić, S. Stanković, V. Tomić, G. Knežević</i>	
ENVIRONMENTAL RISKS CAUSED BY THE POLLUTION FROM AGRICULTURAL PLASTICS – A BRIEF STATE OF ART	311
<i>Marija Koprivica, J. Dimitrijević, J. Petrović, M. Ercegović, M. Simić</i>	
COMPARISON BETWEEN HYDROCHAR AND ITS ALKALI MODIFIED FORM IN THE REMOVAL OF Cd(II) IONS FROM AQUEOUS SOLUTION	317

<i>Milena Pijović Radovanović, M. Seović, I. Perović, N. Zdolšek, J. Georgijević, P. Laušević, S. Brković</i>	
EFFICIENT REMOVAL OF RHODAMINE B FROM AQUEOUS SOLUTIONS USING CARBONIZED WASTE CAR TIRES: CHARACTERIZATION AND ADSORPTION STUDIES	323
<i>Svetlana Butulija, J. Maletaškić, B. Todorović, G. Branković, A. Krstić, R. Mihailović, B. Matović</i>	
SYNTHESIS, CHARACTERIZATION AND ADSORPTION POTENTIAL OF CORN COB-DERIVED ACTIVATED CARBON	329
<i>Vladan Nedelkovski, S. Stanković, D. Medić, D. Buzdugan, I. Hulka, S. Milić, M. Radovanović</i>	
PHOTOCATALYTIC PROPERTIES OF C-ZnO NANOPARTICLES SYNTHESIZED <i>via</i> MECHANOCHEMICAL METHOD	335
<i>Aleksandar Zdravković, M. Nikolić, D. Marković Nikolić, D. Stojadinović, G. Petković, T. Nikolić</i>	
EQUILIBRIUM AND THERMODYNAMICS OF NITRATE SORPTION BY MODIFIED ZEOLITE FROM AQUEOUS SOLUTION	341
<i>Aleksandar Zdravković, M. Nikolić, D. Marković Nikolić, D. Stojadinović, I. Ristić, T. Nikolić</i>	
POTENTIAL USAGE OF OAT STRAW FOR ANIONS REMOVAL FROM WATER: A KINETIC STUDY	348
<i>Aleksandar Zdravković, M. Nikolić, A. Pavlović, D. Marković Nikolić, G. Petković, T. Nikolić</i>	
ULTRASOUND-ASSISTED EXTRACTION OF ACETAMIPRID FROM POLLUTED SOIL	354
<i>Katerina Zaharieva, B. Barbov</i>	
PLANT-MEDIATED SYNTHESIS AND PHOTOCATALYTIC INVESTIGATIONS OF CeO <sub>2</sub> -ZnO COMPOSITES	358
<i>Milena Milošević, M. Abdualatif Abduarahman, M. M. Vuksanović, Z. Veličković, N. Knežević, B. Najdanović, A. Marinković</i>	
CELLULOSE BASED MEMBRANE FOR CATIONIC POLLUTANTS REMOVAL FROM WATER	363
<i>Milena Milošević, A. Marinković, M. M. Vuksanović, Z. Veličković, I. Đuričković, B. Najdanović, N. Knežević</i>	
HEMP MODIFIED WITH BETAINE AS A GREEN AND EFFICIENT ADSORBENT FOR REMOVAL OF ANIONIC DYES FROM WATER	369
<i>Nevena Surudžić, M. Spasojević, M. Crnoglavac Popović, M. Stanišić, R. Prodanović, O. Prodanović</i>	
PHENOL REMOVAL FROM WASTEWATER WITH HORSERADISH PEROXIDASE IMMOBILIZED BY PERIODATE METHOD ONTO NOVEL MACROPOROUS POLY(GMA-CO-EGDMA) CARRIERS	375

<i>Miljana Radović Vučić, N. Velinov, J. Mitrović, S. Najdanović, M. Petrović, M. Kostić, A. Bojić</i>	MODIFIED ACTIVATED WOOD SAWDUST AS GREEN ENVIRONMENTAL-FRIENDLY CATALYST FOR TREATMENT OF PHARMACEUTICAL EFFLUENT	381
<i>Jelena Mitrović, M. Radović Vučić, N. Velinov, S. Najdanović, M. Kostić, M. Petrović, A. Bojić</i>	ADVANCE OXIDATION OF TEXTILE DYE BY ACTIVATED HYDROGEN PEROXIDE WITH UV-C LIGHT	387
<b>Protection and preservation of natural resources</b>		
<i>Gordana Šekularac, M. Aksić, T. Dimitrijević, M. Ratknić, N. Gudžić</i>	QUANTIFYING SOIL EROSION OF THE TOM'S BROOK CATCHMENT (WESTERN SERBIA)	393
<i>Gordana Šekularac, M. Aksić, T. Dimitrijević, S. Gudžić, N. Gudžić, D. Gračak, M. Grčak, M. Ratknić</i>	EFFECT OF IRRIGATION RATE ON THE ONSET INTENSITY OF GREY MOULD AND LATE BLIGHT IN GREEN HOUSE TOMATOES	399
<i>Tatjana Dimitrijević, M. Ratknić, G. Šekularac, M. Aksić</i>	INFLUENCE OF SOIL TYPE ON MEAN TREE HEIGHTS OF FIR TREES IN A 40-YEAR PROVENANCE TRIAL	406
<i>Dragana Božić, Lj. Avramović, V. Trifunović, R. Marković, Z. Stevanović, V. Marjanović, E. Požega</i>	AGITATION LEACHING OF FLOTATION TAILINGS AT THE PILOT PLANT	412
<i>Ivana Kerkez Janković, D. Vilić, M. Nonić, J. Devetaković, M. Šijačić-Nikolić</i>	FOREST FRUIT SPECIES OF URBAN FOREST "KOŠUTNJAK" (SERBIA) – GENEPOOL ASSESSMENT AND CONSERVATION	418
<i>Boris Novaković, N. Paskaš, M. Raković</i>	NEW DATA ON THE DISTRIBUTION OF AQUATIC BEETLES IN SERBIA	424
<i>Matej Fike, M. Pezdevšek, A. Roger</i>	COMPARING FROST PROTECTION STRATEGIES FOR SUSTAINABLE AGRICULTURE IN SLOVENIA	430
<i>Filip Maksimović, M. Nonić, D. Vilotić, I. Kerkez Janković, M. Šijačić-Nikolić</i>	GENE POOL OF FOREST FRUIT TREES IN THE PROTECTED AREA OF THE NATURAL MONUMENT "KOŠUTNJAK FOREST" – THEN AND NOW	435
<i>Dragana Medić, S. Milić, N. Milošević, M. Nujkić, M. Pešić, V. Nedelkovski, S. Stanković</i>	APPLICATION OF THE SHRINKING CORE MODEL IN THE LEACHING PROCESS OF $\text{LiNiMnCoO}_2$	441

## Ecotoxicology and environmental safety

<b>Branko Matovic, J. Maletaskic, S. Butulija, S. Petrovic, B. Todorovic</b> IMMOBILIZATION OF LEAD USING CERIA CRYSTAL STRUCTURE	448
<b>Dragana Medić, S. Milić, N. Milošević, M. Nujkić, S. Alagić, A. Cvetković, A. Papludis</b> CAUSES AND POSSIBLE CONSEQUENCES OF THERMAL RUNAWAY IN LITHIUM-ION BATTERIES	454
<b>Nena Velinov, M. Radović Vučić, J. Mitrović, M. Petrović, S. Najdanović, D. Bojić, A. Bojić</b> KINETIC AND EQUILIBRIUM STUDIES OF CHROMIUM SORPTION USING ULTRASONICALLY MODIFIED WOOD SAWDUST BY ALUMINA	460
<b>Hazardous materials and green technologies</b>	
<b>Uroš Stamenković, I. Marković, V. Čosović, B. Markoli</b> THE INFLUENCE OF AGEING PARAMETERS ON MICROHARDNESS, ELECTRICAL CONDUCTIVITY AND MICROSTRUCTURE OF SOME Al-Mg-Si ALLOYS	466
<b>Marija Simić, D. Aćimović, B. Savić Rosić, M. Ječmenica Dučić, K. Stojanović, D. Maksin, T. Brdarić</b> KINETIC STUDY OF DEGRADATION BISPHENOL A BY FENTON PROCESS	472
<b>Danka Aćimović, K. Stojanović, M. Simić, B. Savić Rosić, Z. Vranješ, M. Ječmenica Dučić, T. Brdarić</b> DETECTION OF BISPHENOL A INTERMEDIATES DURING FENTON PROCESS AND PREDICTION OF REACTION PATHWAYS	476
<b>Tanja Brdarić, D. Aćimović, B. Savić Rosić, K. Stojanović, M. Simić, Z. Vranješ, M. Ječmenica Dučić</b> ADVANCED OXIDATION PROCESSES (AOPs) FOR WASTEWATER TREATMENT: BIBLIOMETRIC STUDY	480
<b>Vanja Trifunović, S. Milić, Lj. Avramović</b> POSSIBILITY OF ZINC AND CADMIUM RECOVERY FROM HAZARDOUS INDUSTRIAL WASTE – EAF DUST	486
<b>Sandra Bulatović, N. Nedić, T. Tadić, B. Marković, A. Nastasović</b> MAGNETIC BIOSORBENT BASED ON THE <i>Ambrosia arthemisiifolia</i> FOR ADSORPTION OF MALACHITE GREEN FROM WATER	491
<b>Milan Nedeljković, S. Mladenović, J. Petrović, M. Mitrović</b> STUDIES OF THE INFLUENCE OF GRAPHENE NANOSHEETS ON THE WETTABILITY OF ECO-FRIENDLY SOLDER ALLOYS	497

<i>Ana Simonović, M. Petrović Mihajlović, M. Radovanović, Ž. Tasić, M. Antonijević</i> ELECTROCHEMICAL SENSORS FOR DETERMINATION OF ANTIBIOTICS	502
<i>Sonja Stanković, V. Nedelkovski, D. Buzdugan, I. Hulka, M. Gorgievski, S. Milić, M. Radovanović</i> INFLUENCE OF CALCINATION TEMPERATURE ON THE MORPHOLOGY, CHEMICAL COMPOSITION, AND STRUCTURE OF ZnO NANOPARTICLES	508
<b>Human and ecological risk assessment</b>	
<i>Milena Tadić, I. Nikolić, D. Đurović, J. Vuković, N. Cupara</i> CHILDREN HEALTH RISK ASSESSMENT OF TRIHALOMETHANES CONTENT IN HOTEL'S SWIMMING POOL WATER IN MONTENEGRO	515
<i>Miljan Bigović, D. Đurović, Lj. Ivanović, M. Blagojević, A. Orahovac</i> HEALTH RISK ASSESSMENT OF ACRYLAMIDE IN POTATO CHIPS FROM MONTENEGRIN MARKET	520
<i>Vesna Djikanović, K. Jovičić, J. S. Vranković, M. Dimitrijević, S. Kovačević, N. Pankov, B. Miljanović</i> ACCUMULATION OF HEAVY METALS AND HUMAN HEALTH RISK ASSESSMENT <i>via</i> THE CONSUMPTION OF FRESHWATER FISH <i>Esox lucius</i>	524
<b>Agriculture: nutrition, organic food and health impacts</b>	
<i>Vitaly Erukhimovitch, M. Huleihel</i> OPTIMIZATION OF PREPARATION PROCEDURES FOR FUNGAL INFECTED PLANTS BY FTIR ANALYSES	531
<i>Mahmoud Huleihel, V. Erukhimovitch</i> POSSIBLE USE OF FOURIER–TRANSFORM INFRARED (FTIR) MICROSCOPY FOR IDENTIFICATION OF FUNGAL PHYTO–PATHOGENS	536
<i>Ana Čučulović, J. Stanojković, R. Čučulović</i> RADIOACTIVITY IN SAMPLES OF IMPORTED MINERAL FERTILIZER ANALYZED IN THE PERIOD 2020–2022	541
<i>Nenad Zarić, M. Zarić</i> METAL CONTENTS IN VEGETABLES ORIGINATING FROM COAL FIRED THERMAL POWER PLANTS REGION	547
<b>Alternative energy: efficiency and environmental policy</b>	
<i>Snežana Brković, N. Zdolšek, I. Perović, M. Seović, P. Laušević, J. Georgijević, M. Čebela</i> ENHANCING OXYGEN EVOLUTION: THE ELECTROCATALYTIC POWER OF Ag-DOPED BISMUTH FERRITE	552

<i>Nebojša Potkonjak, Đ. Čokeša, M. Marković</i>	NONLINERA PHENOMENA DURING VOLTAMMETRIC MEASUREMENT OF COPPER CORROSION	558
<i>Mirjana Marković, Đ. Čokeša, N. Potkonjak</i>	EVALUATION OF THE HYDROGEN DIFFUSION COEFFICIENT IN METAL HYDRIDE BATTERIES	562
<b>Greenhouse effect and global climate change</b>		
<i>Slobodan Milutinović, T. Radenović, S. Živković</i>	FORESTS UNDER THREAT: IMPLICATIONS OF CLIMATE CHANGE ON SERBIAN WOODLANDS	566
<i>Danijela Nikolić, S. Jovanović, Z. Đorđević, D. Končalović, V. Vukašinović</i>	GLOBAL WARMING – TREND ANALYSIS IN THE REPUBLIC OF SERBIA	574
<b>Sustainable development and green economy</b>		
<i>Dragana Randelović, A. Jovanović, B. Marković, M. Sokić</i>	CONTRIBUTION OF THE INSTITUTE FOR TECHNOLOGY OF NUCLEAR AND OTHER MINERAL RAW MATERIALS TO THE SDGs – TOWARDS INTERNATIONAL DECADE OF SCIENCE FOR SUSTAINABLE DEVELOPMENT	580
<i>Veljko V. Savić, J. D. Nikolić, V. Topalović, M. S. Djošić, M. Marković, S. Matijašević, S. Grujić</i>	CHEMICAL DURABILITY EVALUATION OF SINTERED FLY ASH BASED GLASS	586
<i>Stefan Mitrović, S. Brković, M. Seović, N. Zdolšek, P. Laušević, J. Georgijević, I. Perović</i>	RECYCLING ELECTRONIC WASTE CPUs FOR ENHANCED HYDROGEN AND OXYGEN EVOLUTION: AN ECO-FRIENDLY LEACHING APPROACH	593
<i>Adrijana Jevtić, D. Riznić, M. Vuković</i>	BRAND MANAGEMENT AND SOCIO-ECONOMIC ASPECTS OF ADAPTATION TO CLIMATE CHANGES	598
<i>Ana Radojević, J. Jordanović, T. Kalinović, J. Kalinović, S. Šerbula</i>	PROSPECTS OF SUSTAINABLE UTILIZATION OF FOOD WASTE	606
<i>Maja Bogdanović, I. Blagajac</i>	DECENTRALIZATION OF THE URBAN TOURIST ZONE OF ZLATIBOR	613

## Environmental biology

- Sladana Popović, N. Nikolić, Ž. Savković, M. Stupar, D. Predojević, A. Anđelković, O. Jakovljević**  
ISOLATION AND CULTIVATION OF CHROOCOCCUS (CYANOBACTERIA) FROM AEROPHYTIC BIOFILM IN STOPIĆ CAVE 621
- Tamara Mitić, J. Čanak Atlagić, J. Tomović, J. Stanković, D. Mrdak, D. Škraba Jurlina, A. Marić**  
MORPHOMETRIC STUDY OF EUROPEAN BULLHEAD *Cottus gobio* FROM DIFFERENT DRAINAGE POPULATIONS 626
- Jelena Đuknić, N. Popović, B. Vasiljević, B. Tubić, S. Andjus, M. Ilić, M. Paunović**  
ECOLOGICAL POTENTIAL OF THE DANUBE RIVER THROUGH SERBIA BASED ON BIOLOGICAL QUALITY ELEMENTS 632
- Sladana Popović, G. Subakov Simić, S. Stanković, D. Lazić**  
*Chlorella vulgaris* GROWTH IN SMALL OPEN CULTIVATION SYSTEMS 638
- Olga Jakovljević, S. Popović, D. Predojević**  
EPIPHYTIC DIATOMS AS TOOL IN BIOINDICATION OF LAKE PALIĆ 643
- Mihailo Jovanović, J. Paunković**  
IMPROVING PALEOENVIRONMENTAL RECONSTRUCTIONS BASED ON SMALL VERTEBRATES IN THE BALKANS 648
- Jovana Damjanović, M. Milković, A. Mišćević, M. Šćiban, V. Lakušić, M. Stanković**  
SUPPLEMENT TO THE LIST OF ENTOMOFAUNA FROM THE RESEARCH ACTIONS AND CAMPS OF SRSBE “JOSIF PANČIĆ” AT SRN ZASAVICA 654
- Mihajlo Stanković**  
“LIVING FOSSILS” IN THE CRASH FAUNA OF THE ZASAVICA SPECIAL NATURE RESERVE 662

## Environmental and material flow management

- Nataša Knežević, A. Jovanović, M. Vuksanović, M. Savić, M. Milošević, A. Marinković**  
DEGRADATION OF DYE CRYSTAL VIOLET RELEASED FROM THE TEXTILE INDUSTRY 669
- Milenko Jovanović, D. Kržanović, E. Požega, V. Marinković, M. Mikić**  
APPLICATION AND ENVIRONMENTAL SUITABILITY OF HYBRID GEOGRIDS 674
- Miroslav Drljača**  
MODERN APPROACH TO SUPPLY CHAIN BASED ON CIRCULAR ECONOMY PRINCIPLES 681

<i>Isidora Berežni, T. Marinković, N. Stanisavljević, M. Muhadinović, B. Batinić</i> ASSESSMENT OF THE MUNICIPAL SOLID WASTE MANAGEMENT – CASE STUDY: NOVI SAD (SERBIA)	687
---	-----

<i>Ljubiša Balanović, D. Manasijević, I. Marković, U. Stamenković, K. Božinović</i> CALCULATION OF THERMODYNAMIC PROPERTIES Al-Ga-Sn TERNARY ALLOY USING GENERAL SOLUTION MODEL	693
---	-----

### Life-Cycle-Analysis (LCA)

<i>Danijela Nikolić, S. Jovanović, D. Mikić, Z. Đorđević</i> LIFE CYCLE ASSESMENT OF THE HAIR DRYER WITH ECO-it SOFTWARE	701
--	-----

## Student Section – EcoTERS'24

<i>Students: Sofija Kostić, Aleksa Marjanović (Serbia)</i> <i>Mentor: Maja Nujkić (Serbia)</i> SOME ASPECTS OF THE APPLICATION OF METAL-ORGANIC FRAMEWORKS	709
---	-----

<i>Student: Jelena Janković (Serbia)</i> <i>Mentor: Maja Nujkić (Serbia)</i> MECHANISMS OF CADMIUM UPTAKE INTO THE PLANT	711
--	-----

<i>Student: Jovana Kumbrijanović (Serbia)</i> <i>Mentors: Maja Nujkić, Sonja Stanković (Serbia)</i> COAGULATION PROCESS AND APPLICATION OF NEW ECOLOGICAL COAGULANTS	713
---	-----

<i>Student: Lazar Cvetković (Serbia)</i> <i>Mentors: Maja Nujkić, Tanja Kalinović, Jelena Kalinović (Serbia)</i> SOME APPLICATION ASPECTS OF THE MATERIALS BASED ON THE GREEN MAGNESIUM OXIDE ECOLOGICAL COAGULANTS	715
--	-----

<i>Students: Milena Radivojević, Kristina Konstadinović (Serbia)</i> <i>Mentors: Maja Nujkić, Dragana Medić (Serbia)</i> RECYCLING OF USED LITHIUM-ION BATTERIES	717
--	-----

<i>Student: Milica Denić (Serbia)</i> <i>Mentor: Ana Radojević (Serbia)</i> MEDICAL WASTE ISSUES RELATED TO COVID-19 PANDEMIC	719
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<i>Student: Sara M. Pantović (Serbia)</i> <i>Mentor: Enisa S. Selimović (Serbia)</i> PRESENCE OF TOXIC AND POTENTIALLY TOXIC ELEMENTS IN SOME DOMESTIC FRUIT FROM THE PEŠTER PLATEAU, SJENICA, SERBIA	721
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<i>Student: Milena Stanković (Serbia)</i> <i>Mentor: Ljiljana Stanojević (Serbia)</i>	CHEMICAL COMPOSITION OF ESSENTIAL OIL ISOLATED FROM FRESH AND DRY LEAVES OF <i>Geranium robertianum</i> L.	723
<i>Student: Nikola Petrović (Serbia)</i> <i>Mentor: Ana Simonović (Serbia)</i>	TOXIC EFFECTS OF PETROLEUM DERIVATIVES ON LIVING ORGANISMS FROM CONTAMINATED SOILS	725
<i>Students: Anja Antanasković, Nevena Ilić (Serbia)</i> <i>Mentors: Milan Milivojević, Suzana Dimitrijević-Branković, Zorica Lopičić, Nikola Vuković (Serbia)</i>	ENZYME IMMOBILIZATION ON MODIFIED BIOMASS: OPTIMIZATION AND CHARACTERIZATION	727
<i>Student: Milena Balabanović (Serbia)</i> <i>Mentor: Ana Radojević (Serbia)</i>	BIOLOGICAL TREATMENT OF THE BIODEGRADABLE WASTE	729
<i>Student: Natalija Stojanović (Serbia)</i> <i>Mentors: Maja Nujkić, Vladan Nedelkovski (Serbia)</i>	ADSORPTION MATERIALS BASED ON NANOPARTICLES FOR THE REMOVAL OF ARSENIC FROM WASTEWATER	731
<i>Student: Jelena Vesković (Serbia)</i> <i>Mentor: Antonije Onjia (Serbia)</i>	HEALTH RISK ASSESSMENT OF RARE EARTH ELEMENTS IN GROUNDWATER NEAR A THERMAL POWER PLANT	733
<i>Students: Vladimir Topalović, Anja Antanasković, Veljko Savić (Serbia)</i> <i>Mentors: Marija Djošić, Zorica Lopičić, Ana Vujošević, Jelena Nikolić (Serbia)</i>	EFFECT OF PHOSPHATE GLASS AND BIOCHAR ON ROSE GROWTH	735
<i>Student: Aleksandra Milenković (Serbia)</i> <i>Mentor: Ljiljana Stanojević (Serbia)</i>	THE REDUCING POWER OF BLACK PEPPER ( <i>Piper nigrum</i> L.) ESSENTIAL OIL HYDRODISTILLATION FRACTIONS	737
<i>Student: Marija Tasić (Serbia)</i> <i>Mentor: Dragan Cvetiković (Serbia)</i>	ENVIRONMENTAL METHOD OF GOLD NANOPARTICLES SYNTHESIS AND THEIR CHARACTERIZATION	739
<i>Student: Marija Stanković (Serbia)</i> <i>Mentor: Jelena Kalinović (Serbia)</i>	PURIFICATION METHODS FOR POLLUTED AIR	741
<i>Student: Marija Stanković (Serbia)</i> <i>Mentor: Jelena Kalinović (Serbia)</i>	PURIFICATION OF INDUSTRIAL WASTEWATER	743

<i>Students: Željka Nikolić, Nebojša Radović (Serbia)</i> <i>Mentor: Olga Tešović (Serbia)</i>	
RISKS OF CHLORINE EXPOSURE IN HOUSEHOLD CLEANING: A CALL FOR AWARENESS AND PREVENTION	745
<i>Students: Željka Nikolić, Nebojša Radović (Serbia)</i> <i>Mentor: Olga Tešović (Serbia)</i>	
IS THERE A NEED TO INFORM CITIZENS MORE DIRECTLY ABOUT THE HANDLING OF HOUSEHOLD HAZARDOUS WASTE?	747
<i>Students: Nataša Simonović, Tamara Milosavljević (Serbia)</i> <i>Mentors: Jelena Stanojević, Ljiljana Stanojević, Jelena Zvezdanović, Dragan Cvetković (Serbia)</i>	
SOLID WASTE FROM HYDRODISTILLATION OF HERNIARIAE HERBA ( <i>Herniaria glabra</i> L.) AS A POTENTIAL SOURCE OF ANTIOXIDANTS	749
<i>Students: Aleksa Vizi, Nebojša Radović, Željka Nikolić, Stefan Lekić (Serbia)</i> <i>Mentors: Goran Roglić, Ksenija Stojanović, Vele Tešević (Serbia)</i>	
SUSTAINABLE SOLUTIONS IN ANALYTICAL CHEMISTRY: COMBINING OF INSTRUMENTAL TECHNIQUES AND ENVIRONMENTAL-FRIENDLY NATURAL INDICATORS FOR CLASSICAL VOLUMETRY	751
<i>Students: Aleksa Vizi, Nebojša Radović, Željka Nikolić (Serbia)</i> <i>Mentors: Ivan Kojić, Ksenija Stojanović (Serbia)</i>	
EFFICIENT DETERMINATION OF UNDECYLENIC ACID CONTENT IN PHARMACEUTICAL PRODUCTS: A NOVEL SIMPLE APPROACH	753
<i>Student: Andrijana Miletić (Serbia)</i> <i>Mentor: Antonije Onjia (Serbia)</i>	
HEALTH RISK ASSESSMENT OF POTENTIALLY TOXIC ELEMENTS IN AGRICULTURAL SOIL OF BRANIČEVO DISTRICT	755
<i>Student: Jelena Obradovic (Serbia)</i> <i>Mentor: Antonije Onjia (Serbia)</i>	
DISTRIBUTION OF PM <sub>2.5</sub> , CO <sub>2</sub> , HCHO, AND TVOC IN AIR IN A HIGH SCHOOL CLASSROOM	757
<i>Student: Gordan Mišić (Serbia)</i> <i>Mentors: Ana Radojević, Jelena Jordanović (Serbia)</i>	
TOXICOLOGICAL EFFECTS OF MICRO- AND NANO-PLASTICS ON HUMAN HEALTH	759
<i>Student: Anđela Bogdanović (Serbia)</i> <i>Mentor: Marija Petrović Mihajlović (Serbia)</i>	
MAGNESIUM AND ITS ALLOYS	761
<b>Author index</b>	<b>763</b>



## ELECTROCHEMICAL SENSORS FOR DETERMINATION OF ANTIBIOTICS

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### Abstract

*In today's pharmaceutical practise, antibiotics are used to treat bacterial infections because they are easy to use, inexpensive and effective. For this reason, they are very often prescribed for the treatment of humans and animals. However, the continuous and long-term use of antibiotics has led to the need to detect and determine their presence in a large number of different samples using appropriate sensors. However, electrodes used for electrochemical analysis can perform poorly as their surface can be easily contaminated by analytes or intermediates of electrochemical reactions. To solve these problems, many researchers have focused on modifying the relevant electrodes to achieve better performance.*

**Keywords:** electrochemical sensor, electrodes, antibiotic.

### INTRODUCTION

Since their discovery until today, antibiotics have played a very important role in reducing bacterial growth or suppressing bacteria. Due to the uncontrolled use of antibiotics, they can be found in canned food, milk, meat and waste water. The antibiotic residues in wastewater from pharmaceutical waste cause side effects, even a low concentration leads to serious consequences for the environment and human health [1]. Therefore, there is a need to find simple, precise and economical methods for their detection in various samples. Recently, the methods investigated, as fast and accurate, are electrochemical methods and the use of suitable electrodes as electrochemical sensors. Electrochemical methods are characterised by high sensitivity, high accuracy and trace detection. Great attention of researchers is paid to the search for new electrodes that have a large surface area, good electrical conductivity and high electronic transport, which has been demonstrated by carbon-based electrodes such as carbon nanomaterials, carbon nanotubes, graphene (and its derivatives) [2–5]. Metal-based electrodes also showed good results as electrochemical sensors for the detection of antibiotics [1,6]. The paper presented research in which very efficient electrochemical sensors for the detection and determination of some types of antibiotics were found.

### RESULTS AND DISCUSSION

#### Carbon based electrochemical sensors for detection of antibiotics -composites

Dehdashtian and her team [7] investigated sensitive and selective sensor based on carbon paste electrode which was modified by Sodium Montmorillonite nanoclay. This composite was developed as an electrochemical sensor for antibiotic Cefotaxime. The researchers used

the composite to study the electrooxidation of Cefotaxime using cyclic voltammetry and differential pulse voltammetry. The morphology of the modified and unmodified electrode was characterized by scanning electron microscopy. Results of the investigation that current oxidation peak on the modified carbon paste electrode was significantly improved compared to unmodified carbon paste electrode. Modified carbon electrode exhibited linear response over two linear concentration ranges from 0.5 to 40nM and 40 to 2400nM Cefotaxime. Detection limit of modified carbon electrode was 0.1nM. Investigation confirmed that this sensor can be applied for monitoring of Cefotaxime in urine and serum samples.

Iron cation exchanged montmorillonite modified glassy carbon electrode (Fe-Mt/GCE) via a direct voltametric method was used as electrochemical sensor for detection of antibiotic Tetracycline hydrochloride [8]. Modified electrode showed sensitivity of 27.1  $\mu\text{A}/\text{Mcm}^2$ . The Fe-Mt/GCE exhibits higher response current intensity in the rapid scanning process in regard to traditional electrode, which should be contributed to the larger surface area and more active sites of the modified electrode for the oxidation reaction of Tetracycline hydrochloride.

Yu and his colleges developed ultra-sensitive graphene and copper sensor, Gr/Cu, supported on glassy carbon (GCE/Gr/Cu) [9] for the determination of Levofloxacin. The composite give very good conducting properties and impressive sensitivity of 19.7  $\mu\text{A}/\mu\text{Mcm}^2$ . The achieved detection limit of Levofloxacin was 11.86nM. Good selectivity was proved in the presence of various ions typically found in water and other drug molecules. Recovery values were between 97.7 and 103.8% in tap water.

The presence of  $\beta$ -lactam antibiotics in milk samples is of particular concern and represents an unavoidable contaminant of milk. Therefore, it was necessary to develop a sufficiently precise and sensitive method for rapid  $\beta$ -lactam antibiotics detection. Specific electrochemical receptor sensor based on the graphene/thionine (GO/TH) composite was established. The mechanism of the electrochemical receptor sensor was a direct competitive inhibition of the binding of horseradish peroxidase-labeled ampicillin (HRP-AMP) to the mutant BlaR-CTD protein by free-lactam antibiotics. Then, horseradish peroxidase (HRP) catalyzed the hydrolysis of the substrate hydrogen peroxide ( $\text{H}_2\text{O}_2$ ), which produced an electrochemical signal. Experimental data showed that this method gives possibility for quantitative detection of Cefquinome from 0.1 to 8  $\mu\text{g}/\text{L}$  and the limit detection was 0.16  $\mu\text{g}/\text{L}$ , which represents a much smaller value than the maximum residue limit of 5 $\mu\text{g}/\text{L}$  set by the European Union. The limit of detection of spiked milk samples with cefalexin, cefquinome, cefotafur, penicillin G and ampicillin were 14.88  $\mu\text{g}/\text{L}$ , 2.46  $\mu\text{g}/\text{L}$ , 17.16  $\mu\text{g}/\text{L}$ , 0.06  $\mu\text{g}/\text{L}$ , 0.21  $\mu\text{g}/\text{L}$  and the limits of quantitation (LOQ) were 36.09  $\mu\text{g}/\text{L}$ , 5.40  $\mu\text{g}/\text{L}$ , 41.45  $\mu\text{g}/\text{L}$ , 0.13  $\mu\text{g}/\text{L}$ , 0.42  $\mu\text{g}/\text{L}$ , respectively. Investigated electrochemical sensor showed that it can be successfully applied for the assessment of  $\beta$ -lactam antibiotics in milk, and the obtained results were confirmed by the application of chromatography-tandem mass spectrometry. The obtained results point to conclusion that electrochemical receptor sensor had a favourable recovery of 84.89–102.44% [10].

### **Carbon based electrochemical sensors for detection of antibiotics -nanocomposites**

Due to their great potential in sensitivity and selectivity in the determination of pharmaceutical products, among other things, and antibiotics in different samples, electrochemical sensors play an increasingly important role, especially electrochemical

sensors produced with different electrode modifications. Guo *et al.* [11] synthesized three-dimensional porous MXene-carbon nanofiber (CNF) nanocomposites by assembling environmentally friendly and inexpensive bacterial cellulose gel sheets as a carbon source with novel two-dimensional MXenes nanoplate clusters and pyrolyzing the composite for determination of Chloramphenicol. The presented research results show that MXene/CNF nanocomposites give excellent electrical conductivity, large specific surface area, abundant active sites, and excellent electrochemical properties. New synthesized electrochemical sensor MXene/CNF for determination Chloramphenicol achieved a good linear response in concentration range of Chloramphenicol from 0.03–25 $\mu$ M and with a low detection limit of 9nM. These investigators confirmed that the sensor demonstrated good repeatability and reproducibility with relative standard deviations of 2.94 and 3.29%, respectively. This sensor was successfully applied for detection of Chloramphenicol in real samples as milk and jasmine tea beverage. Roushani *et al.* [12] also develop an electrochemical sensor for the selective determination of Chloramphenicol. This group of scientist prepared an electrochemical sensor by modifying GCE with nanocomposite of (3-Aminopropyl) triethoxysilane, graphene oxide and silver nanoparticles. The method used during research was differential pulse voltammetry. The prepared sensor showed good electrochemical performances: linear response in concentration range of 10 pM/L to 0.2  $\mu$ M/L; detection limit of 3.3 pM/L. Electrochemical sensor for determination of Chloramphenicol developed by fabricating CGE with a nanocomposite of graphene oxide and zinc oxide showed excellent analytical performance with detection limit of 0.01 $\mu$ mol/L and linearity over a concentration range of 0.2 to 7.2  $\mu$ M/L. Electrochemical method using during the research was differential pulse voltammetry [12,13].

In order to increase the sensitivity of Levofloxacin detection nanocomposite-modified glassy carbon electrode ( $\text{Ti}_3\text{AlC}_{2\text{max}}$  AND/GCE) featured in as sensor [14]. Great electrocatalytic activity for the oxidation of Levofloxacin is exhibited by the Activated Nanodiamond (AND) and  $\text{Ti}_3\text{AlC}_2$  max phase ( $\text{Ti}_3\text{AlC}_{2\text{max}}$ ) nanocomposite-modified glassy carbon electrode ( $\text{Ti}_3\text{AlC}_{2\text{max}}$  AND/GCE). This modified electrode performed high selectivity and sensitivity with limits of detection of Levofloxacin 20.47nM. Research confirms the fact that the novel sensor can be used as a content monitoring sensor of Levofloxacin in milk yogurt and cheese samples with high reliability and accuracy.

The highly sensitive electrode was fabricated based on cobalt oxide nanorods integrated hexagonal boron nitride-modified glassy carbon electrode ( $\text{CoCo}_2\text{O}_4$  NRs/h-BN/GCE) for the determination of veterinary antibiotic drug ronidazole (RNZ) [1]. The  $\text{CoCo}_2\text{O}_4$  nanorods were prepared by cetyl trimethyl ammonium bromide (CTAB) assisted hydrothermal method and the  $\text{CoCo}_2\text{O}_4$  NRs/h-BN composite was prepared using an ultra-sonication/stirring process. Modified electrode nanocomposite  $\text{CoCo}_2\text{O}_4$  NRs/h-BN showed lower LOD of 3nM and highest sensitivity of 5.845  $\mu\text{A}/\mu\text{Mcm}^2$  for RNZ determination.

Babulal [15] and co-workers were successfully synthesized ytterbium molybdate nano petals (YbMO) with porous carbon (PC) nanocomposite (NC) using ultrasonication method for monitoring the levels of Metronidazole [15]. Obtained modified electrode YbMO/PC NC/GCE exhibited larger reduction peak current and lower potential compared to other modified electrodes. The YbMO/PC NC/GCE performed following results: exceptional

sensitivity of  $3.28 \mu\text{A}/\mu\text{Mcm}^2$ ; wide linear range 0.01–10.61 and 20.61–1630.61  $\mu\text{M}$ ; and low detection limit (LOD) of 0.006  $\mu\text{M}$ . Modified YbMO/PC NC/GCE was used for detection of Metronidazole in serum (101%), urine (97.9%), tablets (99.15%), lake (99.1%), and river water (99.8%).

The antibiotic Oxytetracycline (OTC) has a significant role in increasing food production because it is effectively and efficiently used in the fight against bacterial infections in crops and livestock. That's why antibiotics Oxytetracycline entered in wide application in agriculture and animal husbandry. However, their wide application results in a bad impact on people's health and the environment in general. So the goal is to identify the appropriate agent for the detection of these antibiotics, which are also environmentally friendly. Therefore, Mliki *et al.* [16] report the fabrication of cobalt-doped ZnO/GO nanocomposites for OTC sensors using a simple and environmentally friendly method that does not require toxic solvents. The successful preparation of cobalt-doped ZnO/GO nanocomposites and the determination of the surface area, structure, morphological features, chemical composition and purity of the nanocomposites were demonstrated by contact angle measurements, X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS) and UV–Vis. The electrochemical methods used were cyclic voltammetry (CV), electrochemical impedance spectroscopy and differential pulse voltammetry. The ZnO/GO nanocomposites showed a linear response in concentration ranges from  $10^{-12}$  M to  $10^{-7}$  M, and an impressive detection limit of  $1.6 \cdot 10^{-13}$  M was achieved. Among others, this sensor showed very good performances and remarkable selectivity, proving its usefulness for the detection of OTC traces in real milk samples.

Due to the increasing use of the antibiotic Ciprofloxacin, which leads to an increase in endogenous resistance and numerous health risks, Jiwanti *et al.* [17] present a novel electrochemical sensor in the form of a screen-printed electrode (SPE) reinforced with a novel rGO-SnO<sub>2</sub> nanocomposite. The electrochemical method used to investigate the behaviour of the electrochemical sensor was square-wave voltammetry. The investigated electrochemical sensor showed unparalleled sensitivity and accuracy in the determination of Ciprofloxacin content. The results obtained indicate the superior performance of the SPE/rGO-SnO<sub>2</sub> electrode. The SPE/rGO-SnO<sub>2</sub> electrode exhibited the largest active surface area ( $0.0252 \text{ cm}^2$ ), which enabled faster electron transfer. In addition, the SPE/rGO-SnO<sub>2</sub> electrode showed an impressively low detection limit of 2.03  $\mu\text{M}$  in a concentration range of 30–100  $\mu\text{M}$  for Ciprofloxacin, setting a new benchmark for the sensitivity of 9.348  $\mu\text{A}/\mu\text{M}$  in the detection of Ciprofloxacin. The investigated electrode also showed its good properties in real samples such as river water and milk with remarkable recovery rates of 101.2 and 97.7%, respectively. In view of the research results, the SPE/rGO-SnO<sub>2</sub> electrode proved to be a promising and effective tool for ciprofloxacin detection and determination [17].

Metronidazole is an antibiotic which has a highly harmful impact on human health and domestic animals that's why it's very important to avoid environmental pollution [18]. The hydrothermal technique has been utilized to prepare Nickel Tungsten oxide (NiWO<sub>4</sub>) nanosheets which were anchored with the carbon nanofiber (CNF) to produce CNF/NiWO<sub>4</sub> nanocomposite, the nanocomposite was coated on the glassy carbon electrode (GCE) and

applied to the electrochemical detection of MTD. For detection of electrochemical behavior of the GCE/CNF/NiWO<sub>4</sub> electrochemical impedance spectroscopy (EIS), cyclic voltammetry studies (CV), and differential pulse voltammetry (DPV) were used. The investigated electrode performed following results: a low detection limit of 0.4 μM, a wide linear response range of 0.01–650 μM, and a higher sensitivity of 0.274 μA/μMcm<sup>2</sup>. Prepared electrode was applied for detection of Metronidazole in tap water and urine samples with acceptable recoveries [18].

## CONCLUSION

Antibiotics are very useful drugs for the treatment of bacterial infections, but recently their use has been intensified so that they can be found in samples of milk, meat, water, pharmaceutical waters, etc. In addition, prolonged exposure to antibiotics through the consumption of products containing antibiotics or exposure to untreated waste containing antibiotics can lead to various health problems. Therefore, it is very important to find suitable sensors for the detection of antibiotics in different samples. Electrochemical sensors for the detection of antibiotics in various samples provided particularly good results, showing good selectivity and sensitivity, and their performance was particularly improved by suitable modifications.

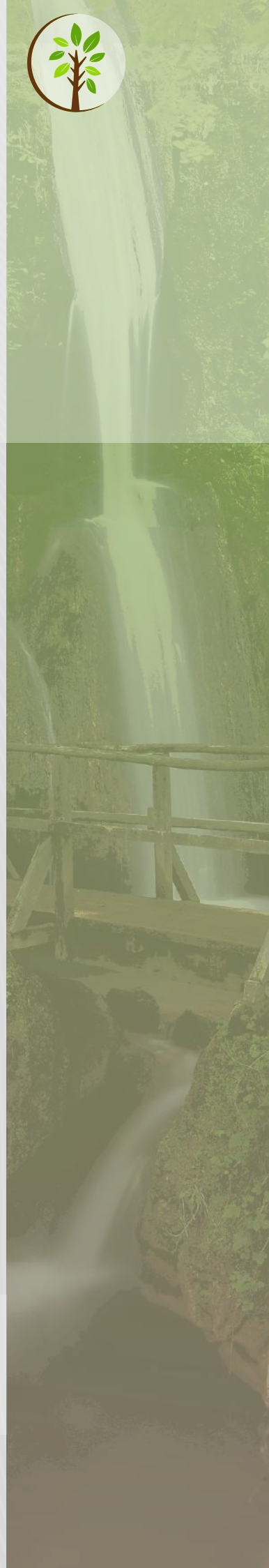
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