

University of Belgrade
Technical Faculty Bor

PROCEEDINGS

XXIII International Conference Ecological Truth

Editors

Radoje V. Pantovic

Zoran S. Marković

EcoIst '15

Hotel "PUTNIK", Kopaonik, SERBIA
17-20 June 2015

UNIVERSITY OF BELGRADE
TECHNICAL FACULTY BOR



**XXIII International Conference
"ECOLOGICAL TRUTH"**

Eco-Ist'15

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**Radoje V. PANTOVIC
and
Zoran S. MARKOVIC**

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TABLE OF CONTENTS

PLENARY LECTURE

<i>Milinko Zivkovic, B. Spaskovski, S. Stojadinovic, R. Pantovic</i> EFFECTS OF SERVICE SMELTING OF IMPORTED COPPER CONCENTRATE IN THE NEW SMELTER OF RTB BOR.....	1
<i>Dejan Kozelj, V. M. 'Mac' Canby, D. Stojadinovic</i> ENVIRONMENT – SAFETY ASPECTS OF DRILLING ACTIVITIES AS A PART OF GEOLOGICAL EXPLORATION.....	7
<i>Milica Vlahovic, S. Martinovic, T. Volkov Husovic</i> VALORIZATION OF SECONDARY SULFUR FROM OIL REFINING PROCESS FOR SULFUR CONCRETE PRODUCTION.....	12
<i>Dejan V. Stojanovic, M. Stankovic, R. Novcic, S. Komatovic, S. Nestorovic, M. Tomic, A. Konjevic</i> NATURAL SCIENCE RESEARCH IN PROTECTED AREAS OF THE REPUBLIC OF SERBIA.....	29

PROTECTION AND PRESERVATION OF NATURAL RESOURCES

<i>Mihajlo Stankovic</i> GEOPHYTES IN A FOREST FLORA RESERVE ZASAVICA.....	46
<i>Ana Cuculovic, D. Veselinovic</i> ACTIVITY LEVELS OF ¹³⁷ Cs IN DIFFERENT FUNGI SPECIES IN DIFFERENT FUNGI SPECIES IN SERBIA IN THE PERIOD 1999-2013.....	54
<i>Slavka Stankovic, M. Radomirovic, B. Tanaskovski, M. Jovic</i> THE IMPACT OF MAJOR AND MINOR ELEMENTS FROM SEDIMENTS ON THEIR CONTENT IN <i>M. Galloprovincialis</i> FROM THE BOKA KOTORSKA BAY, MONTENEGRO.....	60
<i>Martin Bobinac, S. Andrasev, B. Stajic, A. Bauer Zivkovic</i> STRUCTURAL CHARACTERISTICS OF SILVER LIME AND BLACK LOCUST PLANTATIONS IN DELIBLATO SANDS AREA (SERBIA).....	68
<i>Branko Stajic</i> TREE SPECIES DIVERSITY IN THE STANDS OF BEECH AND VALUABLE BROADLEAVES IN THE AREA OF THE NP "DJERDAP".....	76

Branko Stajic, M. Bobinac, Z. Janjatovic, S. Andrasev, Z. Bakovic
HEIGHT GROWTH OF WHITE ASH (*Fraxinus excelsior L.*) IN THE REGION
MAJDANPECKA DOMENA..... 83

Sinisa Andrasev, S. Roncevic, M. Bobinac, B. Stajic
ELEMENTS OF GROWTH, BIOLOGICAL AND QUALITATIVE STRUCTURE OF
TREES IN VEGETATIVE ORIGIN BLACK LOCUST STANDS ON CHERNOZEM..... 91

Sinisa Andrasev, S. Roncevic, M. Bobinac
THE ASSESSMENT OF SITE CLASSES OF BLACK LOCUST STANDS
IN MU "BAGREMARA"..... 99

TECHNOLOGIES, WASTES RECYCLING AND THE ENVIRONMENT

Mincho Simeonov, H. Ibrishimov, L. Dimitrov
ANALYSIS OF AN INDUCTOR WITH DIFFERENTIATED DOMAINS
OF THE ELECTROMAGNETIC AND THERMAL FIELD..... 106

Lorand Toth, G. A. Gaman, A. Calamar, M. Kovacs, S. Simion
THE NORMALIZATION OF GASEOUS EFFLUENTS POLLUTANTS
FROM LARGE COMBUSTION PLANTS..... 114

Marina Stamenovic, D. Brkic, N. Djordjevic, S. Drmanic, S. Putic
DEVELOPMENT OF METHOD FOR OBTAINING RECYCLED GLASS
FIBERS FROM GRP COMPOSITE MATERIAL..... 121

Darko Bodroza, S. Sladojevic, J. Penavin Skundric, D. Lazic, Lj. Vasiljevic, B. Skundric
STUDY OF BETA ZEOLITE PHYSICAL-CHEMICAL CHARACTERISTICS
BY OBSERVING ADSORPTION OF SOME ACID AND BASE COMPONENTS
FROM AQUEOUS SOLUTIONS..... 128

Dimitrina Koeva, N. Nedelchev, S. Rachev
SENSORLESS FAULT DIAGNOSTIC TECHNIQUES FOR INDUCTION
MACHINES – PART II..... 137

Jakob Lamut, A. Rozman, M. Debelak, B. Lamut, M. Knap
PHASE COMPOSITION AND RECYCLING OF BY-PRODUCTS FROM
STEEL PRODUCTION..... 145

Biljana Jovanovic, I. Andjelovic, M. Popovic, B. Todorovic
REMOVAL OF ARSENIC FROM WASTE METALLURGICAL WATER
FIXING ARSENIC AS CRYSTALLINE SCORODITE..... 151

Mirko Ivkovic, Z. Dragosavljevic, D. Aleksic, Z. Ivkovic
BROWN COAL DEPOSITS OF "SOKO" MINE – USABLE VALUE
OF NATURAL CAPITAL..... 156

Dominik Brkic, N. Djordjevic, M. Stamenovic, S. Putic OBTAINING AND CHARACTERIZATION OF MODIFIED NANOCELLULOSE BY FTIR SPECTROSCOPY AND TGA.....	164
Sehval Lapandic THE SIMULATION OF THE BENCHES EXCAVATION IN THE OPEN PIT MINE IN ORDER TO DEFINE SLOPES' STABILITY.....	171
Sasa Stojadinovic, R. Pantovic, D. Jenic, B. Tomic, M. Zikic, M. Denic DEFINITION OF CONTROLLED BLASTING ZONES AT JUZNI REVIR OPEN PIT COPPER MINE MAJDANPEK.....	179
Dragana Randjelovic, S. Jovanovic, N. Mihailovic, R. Sajn THE CONTENT OF MANGANESE IN SOILS AND PLANTS OF BOR MINE OVERBURDEN SITE (SERBIA, SE EUROPE).....	186
Marijana Pantovic, Z. Stevic, M. Rajcic Vujasinovic, I. Radovanovic MONITORING OF ENVIRONMENTAL PARAMETERS BASED ON LABVIEW PACKAGE.....	193
Predrag Stolic, B. Djordjevic, M. Pantovic REDUCING ENVIRONMENTAL IMPACT OF BIG DATA USING SERVER VIRTUALIZATION TECHNOLOGY IN DATA CENTERS.....	198
Vojo Jovanov, B. Anguseva, K. Pantovic, E. Fidancevska DESIGN OF MICROSTRUCTURE OF CERAMICS BASED ON WASTE FLY ASH AND CLAY.....	207
Marina Ravilic, B. Bogdanovic, S. Bajic, S. Lutovac, R. Gacina ANALYSIS OF THE IMPACT OF BLASTING ON CONSTRUCTION FACILITIES AND ENVIRONMENT IN THE PREPARATION OF THE TUNNEL PORTAL SARLAH NEAR PIROT (SOUTH SERBIA).....	212
Vesna Lazarevic, I. Krstic, B. Bijelic, D. Antic POTENTIAL TOXIC EFFECTS OF ZINC IN METAL INDUSTRY.....	219
Ivana Markovic, S. Nestorovic, M. Milenovic, M. Velinovic METAL POWDER PARTICLES AS HEALTH, ENVIRONMENTAL, AND SAFETY HAZARD.....	226
Zaklina Z. Tasic, M. B. Radovanovic, M. B. Petrovic Mihajlovic, A. T. Simonovic, S. M. Milic, M. M. Antonijevic INFLUENCE OF POTASSIUM SORBATE ON ELECTROCHEMICAL BEHAVIOR OF COPPER IN SULFURIC ACID MEDIUM.....	233
Zivce Sarkocevic, M. Bozovic, M. Mistic, B. Stojcetovic STRATEGIES FOR RESTORING THE EXISTING CITY DUMP STATION „SERIJAT“ IN STRPCE.....	240

<i>Dejana Milinkovic, A. Manojlovic, M. Zerajic</i> CEMENT MANUFACTURING TECHNOLOGY AND CO-PROCESSING OF MATERIALS DERIVED FROM WASTE.....	247
<i>Milan I. Cekerevac, Lj. N. Nikolic Bujanovic, M. M. Tomic, M. Z. Zdravkovic, N. H. Popovic</i> THE SUCROSE OXIDATION ON BORON-DOPED DIAMOND IN THE BRINE WATER - CYCLIC VOLTAMMETRY.....	253
<i>Milan I. Cekerevac, Lj. N. Nikolic Bujanovic, M. Z. Zdravkovic, M. M. Tomic, M. Stamenkovic Djokovic, M. V. Simicic</i> THE SUCROSE OXIDATION ON BORON-DOPED DIAMOND IN THE BRINE WATER - POTENTIOSTATIC ELECTROLYSIS.....	260
<i>Danijela Lukic, G. Milojevic Miodragovic, Lj. Torovic, I. Cervenka, S. Bijelovic</i> SILVER NANOPARTICLES AND HUMAN HEALTH RISK.....	267
<i>Dejan Tanikic, R. Pantovic, V. Despotovic, M. Zikic</i> SHAPE MEMORY ALLOYS AND SOME OF THEIR MEDICAL APPLICATIONS.....	274
<i>Miodrag Zikic, M. Pavlovic, V. Milic, N. Vusovic, S. Stojadinovic, D. Tanikic, M. Nikolic, N. Kokalj</i> FEED MATERIAL QUALITY ADJUSTMENTS IN HOLCIM CEMENT PLANT FROM THE ENVIRONMENTAL PROTECTION ASPECT.....	281
<i>Slavica Mihajlovic, V. Jovanovic, D. Radulovic, V. Kasic, Z. Sekulic</i> THE IMPACT OF THE PRODUCTION AND USE OF POLYVINYL CHLORIDE ON THE ENVIRONMENT.....	287
<i>Zivko Sekulic, M. Kragovic, Z. Sekulic, M. Mihailovic, S. Mihajlovic, V. Jovanovic</i> INVESTIGATION OF THE APPLICATION OF THE NATURAL AND WITH LEAD IONS CONTAMINATED ZEOLITE AS AN ADDITION IN PORTLAND CEMENT.....	293
<i>Zorica Lopiccic, M. Stojanovic, J. Milojkovic, M. Mihajlovic, M. Petrovic, T. Sostaric, J. Petrovic</i> INFLUENCE OF PARTICLE SIZE ON BIOSORPTION KINETIC PARAMETERS.....	301
<i>Marija Petrovic, T. Sostaric, M. Stojanovic, J. Milojkovic, M. Mihajlovic, J. Petrovic, M. Stanojevic</i> MODIFIED CORN SILK AS BIOSORBENT FOR Pb(II) IONS REMOVAL FROM AQUEOUS SOLUTION.....	308
<i>Jelena D. Nikolic, A. M. Vujosevic, V. D. Zivanovic, S. D. Matijasevic, V. S. Topalovic, S. N. Zildzovic, S. R. Grujic</i> THE INFLUENCE OF GLASS COMPOSITION ON THE DISSOLUTION RATE.....	314

Vladan Kasic, A. Radosavljevic-Mihajlovic, J. Stojanovic, Z. Sekulic, S. Mihajlovic, M. Vukadinovic GEOLOGICAL CHARACTERISTICS OF ZEOLITES FROM IGROS AS RAW MATERIALS FOR USE IN VARIOUS FIELDS OF ECOLOGY.....	320
Dragan S. Radulovic, S. R. Mihajlovic, V. D. Jovanovic, Z. Sekulic OBTAINING FILLERS BASED ON LIMESTONE FROM DEPOSIT "RISTOVA PONTA" – ULCINJ, FOR APPLICATIONS IN VARIOUS INDUSTRIES.....	326
Mirjana Rajcic Vujasinovic, S. P. Dimitrijevic, V. Grekulovic, Z. Stevic, S. B. Dimitrijevic, B. M. Jokic BEHAVIOR OF THE Ag ₄₃ Cu ₃₇ Zn ₂₀ ALLOY IN AERATED SODIUM CHLORIDE SOLUTION.....	334
Milan Gorgievski, N. Strbac, D. Bozic, V. Stankovic THE ADSORPTION OF Cu ²⁺ AND Ni ²⁺ IONS FROM SYNTHETIC SOLUTIONS USING LOW COST BIOSORBENT WHEAT STRAW.....	343
Jelena Milosavljevic, S. Serbula, K. Pantovic PYROLYSIS OF LIGNOCELLULOSIC BIOMASS.....	349
Miodrag Miljkovic, J. Sokolovic, N. Aksic, R. Stanojlovic, R. Pantovic IMPACT OF SELECTION OF MINING UNDERGROUND METHOD OF ORE DEPOSITS ON THE ENVIRONMENT.....	356
Miodrag Miljkovic, J. Sokolovic, B. Babic, S. Perisic, R. Stanojlovic, R. Pantovic PREVENTION, RISK AND INSURANCE OF MINING PRODUCTION SYSTEMS.....	361
Mirjana M. Rajcic Vujasinovic, V. J. Grekulovic, U. S. Stamenkovic, Z. M. Stevic THE INFLUENCE OF BENZOTRIAZOLE ON POTENTIOSTATIC OXYDATION OF AgCu50 ALLOY IN PRESENCE OF CHLORIDES.....	368
Ljubisa Balanovic, D. Zivkovic, N. Strbac, D. Manasijevic, L. Gomidzelovic, A. Mitovski Zn-Al-BASED ECOLOGICAL ALLOYS AND THEIR APPLICATION IN ELECTRONICS.....	374
Jelena Majstorovic, M. Korac, D. Savic, S. Savkovic THE EFFECTS STUDIES OF SHEAR STRENGTH PARAMETERS MORAIN MATERIALS ON ENVIRONMENTAL PROTECTION.....	382
Srba Mladenovic, D. Manasijevic, D. Zivkovic, M. Pantovic THERMAL AND ELECTRICAL PROPERTIES OF THE AS-CAST Sn-RICH ALLOYS.....	388
Gracijan Strainovic, Z. Markovic, I. Profirovic, A. Radulovic THE EFFECT OF PULP DENSITY AND TIME OF FLOTATION ON THE RESULTS OF FLOTATION CONCENTRATION IN INDUSTRIAL CODITIONS.....	393
Nenad Vusovic, R. Pantovic, I. Svrkota, M. Vukovic ENDANGERMENT EVALUATION OF BUILDINGS SITUATED IN LOCAL COMMUNITY „SEVER“ NEAR OPEN PIT BOR.....	396

<i>Katarina R. Mihajlovski, N. R. Radovanovic, M. M. Miljkovic, D. D. Mladenovic, S. I. Dimitrijevic-Brankovic, S. Siler-Marinkovic</i> SUGAR BEET PULP AND MOLASSES AS A SOLID STATE FERMENTATION MEDIA FOR CELLULASE PRODUCTION BY <i>Paenibacillus chitinolyticus</i> CKS1.....	403
<i>Ruzica Micic, A. Jokic, S. Mitic, M. Mitic, M. Cekerevac, Lj. Nikolic Bujanovic, B. Jokic</i> APPLICATION OF ELECTROCHEMICALLY SYNTHESIZED FERRATE(VI) FOR THE REMOVAL OF Pb(II) IONS FROM WATER SAMPLES FOLLOWED BY KINETIC MEASUREMENTS.....	409
<i>Dragoslav Djokic, D. Terzic, J. Milenkovic, T. Vasic, B. Dinic, J. Markovic, R. Stanisavljevic</i> THE POSSIBILITY OF PROCESSING BIODEGRADABLE WASTE IN THE AEROBIC FERMENTER.....	414

ENERGY EFFICIENCY, ENVIRONMENT AND CLIMATE

<i>Marcin Lutynski, S. Lutynska</i> ENVIRONMENTAL CONCERNS OF SHALE GAS PRODUCTION IN POLAND	421
<i>Ladislav Lazic, V. L. Brovkin, A. Varga, J. Kizek</i> REDUCTION OF ENERGY CONSUMPTION AND CO ₂ EMISSIONS THROUGH INCREASE OF COMBUSTION EFFICIENCY	427
<i>Svetlana Nikolic, V. Lazic, L. Mojovic, M. Radetic</i> PRODUCTION OF BIOETHANOL AS A BIOFUEL FROM COTTON FABRICS BY SIMULTANEOUS ENZYMATIC SACCHARIFICATION AND FERMENTATION.....	435
<i>Orhideja Strbac, D. Dobrosavljevic</i> THERMAL IMAGING OF PUBLIC BUILDINGS.....	442
<i>Nada Strbac, D. Zivkovic, M. Mitovski, A. Mitovski, D. Manasijevic, Lj. Balanovic, M. Sokic, M. Rasovic</i> POSSIBILITIES FOR THE IMPROVEMENT OF THERMAL PLANTS EFFICIENCY.....	446
<i>Aleksandra Fedajev, R. Nikolic, I. Svkota</i> RENEWABLE ENERGY SOURCES AND SUSTAINABLE DEVELOPMENT IN TRANSITION COUNTRIES.....	453
<i>Dragan Antic, I. Krstic, A. Djordjevic, V. Lazarevic</i> ENERGY STAR - ENERGY SECURITY MODEL OF TECHNOLOGICAL SYSTEMS.....	461

AGRICULTURE: AGRIBUSINESS, AGROENGINEERING AND ORGANIC FOOD PRODUCTION

Snezana Devic, L. Kuresevic, M. Cocic
MINERALOGICAL CHARACTERIZATION OF THE ZEOLITIC TUFFS
AND ASPECTS OF ITS APPLICATION IN AGRICULTURE..... 471

*Slobodan Krnjajic, A. Radojkovic, M. Radovic, I. Kostic, M. Kostic, J. Mitrovic,
Z. Brankovic, G. Brankovic*
INSECTICIDE EFFECT OF NON-TOXIC INORGANIC POWDERS AGAINST
BEAN WEEVIL (*Acanthoscelides obtectus*)..... 480

Gordana Drazic, M. Arandjelovic, V. Popovic, J. Ikanovic
ECOREMEDIATION – THE CONCEPT OF SUSTAINABLE MANAGEMENT
OF NATURAL RESOURCES IN AGRICULTURE..... 486

Bozidar Mihajlovic, S. Kirin, O. Milosevic
DEVELOPEMENT ORGANIC FOOD PRODUCTION IS PARAMOUNT
FACTOR VS GENETICAL MODIFICATION FOOD (GMO)..... 492

Gordana Dozet, G. Cvijanovic, M. Vasic, N. Djuric, S. Jaksic, V. Djukic
EFFECT OF MICROBIAL FERTILIZER APPLICATION ON YIELD OF BEAN
(*Phaseolus vulgaris L.*) IN ORGANIC PRODUCTION SYSTEM..... 501

Gorica Cvijanovic, J. Marinkovic, G. Dozet, V. Djukic, N. Djuric, M. Cvijanovic
MICROORGANISMS INDICATORS OF THE BALANCE IN
THE AGRO-ECOLOGICAL SISTEM..... 508

*Nemanja Barac, S. Skrivanj, D. Manojlovic, Z. Bukumiric, G. Trajkovic,
M. Barac, R. Petrovic, A. Corac*
HEAVY METALS FRACTIONATION IN AGRICULTURAL SOILS
FROM THE IBAR RIVER VALLEY (SOUTHERN SERBIA):
BIOACCUMULATION BY *Solanum tuberosum L.*..... 514

URBAN ECOLOGY

Dejan Filipovic, Z. Radosavljevic, R. Colic, H. Müller, E. Rajic, S. Ivanovic
IMPROVEMENT OF SEA IMPLEMENTATION IN URBAN PLANNING IN SERBIA..... 522

Snezana Serbula, M. Nikolic, A. Radojevic, S. Manasijevic, N. Davitkov
EFFECT OF SO₂ ON THE QUALITY OF AMBIENT AIR IN BOR..... 530

Jovana Petrovic, S. Vicentic, N. Stavretovic
ANALYSIS OF THE BIOLOGICAL SPECTRUM OF FLORA
OF THE QUAY IN NOVI SAD..... 535

<i>Jovana Petrovic, J. Tomicevic-Dubljevic, N. Stavretovic</i> SPATIAL USE AND THE NEEDS OF VISITORS OF PARK-FOREST KOSUTNJAK IN BELGRADE.....	541
<i>Nenad Stavretovic, J. Petrovic</i> LAWNS OF THE PARK IN BANOVO BRDO	548
<i>Nenad Stavretovic, J. Petrovic</i> LAWNS OF THE RESIDENTIAL AREAS OF BELGRADE (The suburbs Cerak and Vidikovac).....	554
<i>Vesna Radinovic, S. Stamenkovic</i> AIR QUALITY MONITORING BY USING EPIPHYTIC LICHENS IN THE URBAN PART OF PIROT (SOUTHEASTERN SERBIA) 2002– 2014.....	559
<i>Milos Stanojevic, Dj. Stojicic, M. Ocokoljic</i> OVERVIEW OF THE RECONSTRUCTION OF SQUARE OF JOVAN SARIC IN KRALJEVO CITY.....	564
<i>Vesna Mila Z. Colic Damjanovic, A. J. Vuja</i> NEW URBAN REALITY: HYBRID MEGALOPOLIS.....	570
<i>Jasmina Jaksic</i> REGIONAL LANDSCAPE PROPOSAL CONCEPT FOR PROGRAM GUIDELINES AND USAGE OF THE MUNICIPALITY OF ULCINJ IN REPUBLIC OF MONTENEGRO.....	576
<i>Marina Pesic, V. Ristic Vakanjac, M. Antonijevic, B. Vakanjac, N. Markovic</i> GOOD MONITORING AS A PRECONDITION FOR HIGH DRINKING WATER QUALITY: CASE STUDY OF ZLOT WATER SUPPLY SOURCES (BOR, SERBIA).....	583
<i>Ljiljana Nikolic Bujanovic, M. Cekerevac, M. Tomic, M. Zdravkovic, V. Jeftic, M. Stamenkovic Djokovic</i> POSSIBILITY OF ARSENIC REMOVAL BY FERRATE(VI) IN THE TREATMENT OF RAW DRINKING WATER.....	590
<i>Svetlana Nikolic, L. Mojovic, V. Vujacic, B. Nikolic, D. Marinovic</i> THE QUALITY OF RIVER MLAVA IN POZAREVAC MUNICIPALITY.....	597
WATER SUPPLY AND PROTECTION	
<i>Violeta Cibulic, S. Mrazovac Kurilic, N. Staletovic, L. Stamenkovic</i> POLLUTION OF THE SAVA RIVER FROM ENTRANCE IN SERBIA TO CONFLUENCE IN THE DANUBE RIVER.....	603
<i>Savo Perendic, D. Ciric, B. Stakic, J. Sokolovic</i> APPLICATION OF THE FILTER-ANTRACIT® FOR DRINKING WATER PURIFICATION.....	610

*Katarina Zoric, N. Popovic, J. Djuknic, S. Andjus, M. Ilic, M. Kracun-Kolarevic,
N. Marinkovic, J. Canak Atlagic, M. Paunovic*
WATER QUALITY ASSESSMENT BASED ON MACROINVERTEBRATES – SMALL
HILLY STREAMS IN THE CARPATHIAN-BALKAN REGION OF EASTERN SERBIA... 614

*Katarina Zoric, B. Vasiljevic, J. Tomovic, A. Atanackovic, B. Tubic, N. Popovic,
M. Rakovic, B. Novakovic, M. Paunovic*
ALLOCHTHONOUS MACROINVERTEBRATE SPECIES OF THE SERBIAN
STRETCH OF THE TISA RIVER..... 621

ECOLOGICAL MANAGEMENT (LOW, ECONOMY AND STANDARDIZATION)

Velimir Komadinic, S. Manasijevic
THE SPECIFICS OF RISK ASSESSMENT FOR SMALL AND
MEDIUM-SIZED ENTERPRISES..... 625

Milana Pisaric
HABITATS CONSERVATION IN CASE LAW OF COURT OF EUROPEAN UNION..... 633

Milan Martinovic
CONSERVATION AND LAND PLANNING..... 641

Miodrag Miljkovic, R. Pantovic, M. Zivkovic, D. Labovic
MODELS FOR CALCULATION OF MACHINE OUTPUT AND PRODUCTION COSTS... 646

Vesela Radovic, E. Arabska
PREVENTING CONTAMINATION IN DISASTERS - FROM THEORY TO
THE PRACTICE IN SERBIA..... 653

Jetena Velimirovic
SERBIAN WATER MANAGEMENT AND EUROPEAN INTEGRATION..... 661

ECOLOGICAL ETHICS AND ECOLOGICAL EDUCATION

Marijana Demajo
ANALYSIS OF PUBLIC OPINION TOWARDS THE RECENTLY PROCLAIMED
NATURE PARK "RUSANDA"-VOJVODINA, SERBIA..... 666

Toni Gjorgiev, G. Stoilovski
ECOLOGY THROUGH THE PRISM OF GLOBALIZATION..... 671

Slobodan Popovic, M. Vidakovic, A. Majstorovic, B. Martinovic, J. Toskovic
SOCIALLY RESPONSIBLE CONDUCT OF SERBIAN IN THE USE DISPOSED WASTE. 675

Miljan S. Ilic
IMPORTANCE OF GEOGRAPHY IN EDUCATION OF YOUNG PEOPLE WITH
SPECIAL EMPHASIS ON THE TEACHING CONTENT OF THE ENVIRONMENT..... 681

<i>Milan Martinovic</i> DEVELOPING ENVIRONMENTAL AWARENESS.....	687
--	-----

<i>Marina Vukin, M. Kelember, B. Crnoseljanski</i> EDUCATIONAL ROLE OF THE "ARBORETUM OF THE FACULTY OF FORESTRY" - EXAMPLE OF GOOD INCLUSIVE PRACTICE.....	692
---	-----

ECO TOURISM AND SUSTAINABLE DEVELOPMENT

<i>Marius Kovacs, G. A. Gaman, A. Calamar, L. Toth, S. Simion, I. Eisler</i> GAS MONITORING IN THE ENVIRONMENT, FOLLOWING A FIRE IN AN UNDERGROUND TOURISTIC FACILITY.....	699
--	-----

<i>Cipriana Sava</i> TOURISM ACTIVITY AND SHALE GAS EXPLOITATION IN BUZIAS, THE SPA RESORT IN TIMIS COUNTY, ROMANIA.....	707
--	-----

<i>Suzana Gavrilovic, D. Skocajic</i> IMPORTANCE OF LANDSCAPE CHARACTER IN THE PROCESS OF ESTABLISHMENT OF EDUCATIONAL (NATURE) TRAILS IN FUNCTION OF ECOTOURISM.....	714
--	-----

<i>Nikola Ristic, V. Secerov, B. Lukic, D. Filipovic</i> STRATEGIC PLANNING OF TRANSPORT INFRASTRUCTURE AS BASIS OF ECOTOURISM DEVELOPMENT IN NEGOTIN.....	720
--	-----

<i>Tijana Bogdanovic, D. Avramovic</i> DJAVOLJA VAROS – A NATURAL PHENOMENON.....	727
--	-----

<i>Andjelina Maric, A. Mihajlovic, M. Vidosavljevic</i> POSSIBILITY OF TOURIST VALORIZATION OF VLASINA LAKE.....	735
---	-----

<i>Snezana Urosevic, D. Karabasevic, M. Maksimovic</i> THE CONCEPT OF SUSTAINABLE DEVELOPMENT OF RURAL TOURISM.....	741
--	-----

PREVENTIVE MEDICINE AND ECOLOGY

<i>Ljubica Zupunski, V Spasic Jokic, V Gordanic</i> SENSITIVITY ANALYSIS IN RADIOGENIC CANCER RISK ASSESSMENT.....	749
---	-----

<i>Dusko Djukanovic, I. Svrkota, R. Pantovic, R. Milanovic</i> FURTHER DEVELOPMENT OF OIL SHALE INDUSTRY IN SERBIA.....	755
--	-----

<i>Marina Birovljev, V. Cvetanovski</i> TOXIC VIGILANCE DATA ON POISONING IN SERBIA.....	763
---	-----

<i>Aleksandra Stankovic</i> THE ASSESSMENT OF HEALTH RISKS FROM MEDICAL WASTE GENERATED IN DENTAL PRACTICES.....	768
<i>Aleksandra Stankovic, M Nikolic</i> INFLUENCE OF EXPOSURE TO AIR POLLUTION ON USE PRIMARY HEALTH CARE AT WOMEN.....	773
<i>Konstansa Lazarevic, D. Bogdanovic</i> THE INFLUENCE OF NUTRITION ON THE TOXICITY OF POLYCHLORINATED BIPHENYLS	778
<i>Stefan Denda, J. Stojanovic</i> THE MEDICAL-GEOGRAPHICAL ASPECTS OF ENDEMIC NEPHROPATHYIN THE MUNICIPALITY OF LAZAREVAC.....	784
AUTORS' INDEX.....	793



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INVESTIGATION OF THE APPLICATION OF THE NATURAL AND
WITH LEAD IONS CONTAMINATED ZEOLITE AS AN ADDITION
IN PORTLAND CEMENT

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ABSTRACT

In presented paper results of the removal of lead ions from water solutions by the natural zeolite (clinoptillite) and application of such contaminated zeolite materials as a supplement for standard additions portland cement are showed. In this investigations, the natural zeolite (clinoptilolite) with particle size 100% - 43 μm was used. The experiments of lead ions adsorption showed that the maximum adsorption capacity of the natural zeolite was 66 mg Pb^{2+}/g . After adsorption experiments the saturated zeolite with lead ions was used as a supplement to portland cement. Investigations of the mechanical properties of the cement with the addition, natural zeolite and zeolite saturated with lead ions showed that this material can be used as a supplement for portland cement up to 30%.

Key words: Natural zeolite, Pb zeolite, Portland cement, Flexural strengths, Compressive strength.

INTRODUCTION

Natural zeolites are crystalline hydrated aluminosilicate minerals of alkaline and alkaline earth cations, which are very widespread in nature. However, only clinoptilolite, mordenite, filipsite, chabazide, erionite, ferrierite and analcite are in sufficient amount spread in the nature so their exploitation cost effective [1]. Due to the unique properties that they have: high selective and high cation exchange capacity, high degree of crystallinity, uniform channels of molecular dimensions, a high degree of hydration and easy dehydration, low density, high absorption capacity for ions of heavy metals (lead, copper, zinc, etc.) natural zeolites found wide application:

- catalysts in many reactions,
- such as molecular sieves and cation exchangers [2]

- Adsorption of impurities in the waste water
- adsorbents for radionuclides - ^{137}Cs [3]
- ammonia adsorbents [4,5]
- adsorbents of heavy metals ions - Pb, Cu, Zn [6]
- material for recultivation of degraded lands (ash thermal power plants, flotation tailings)
- material in pharmacy and veterinary medicine
- feed additive in veterinary medicine for the prevention of digestive problems in young animals [7].

In order to improve properties of natural zeolite (clinoptilolite), various methods of modification of the starting minerals were applied [8]. Such modified zeolites can be used as:

- adsorbent of mycotoxins that are found in animal food such as: aflatoxins, zearalenone, ochratoxin A, ergot alkaloids [2]
- tool for removing impurities from the water [9 - 11]; weakly polar organic molecules; inorganic anions - sulfate, phosphate, chromate;
- decontaminant land-radionuclide-uranyl ion [12; 13] cesium etc.

From an environmental point of view, it is possible to use zeolites in certain ecosystems:

- 1 Air: adsorbent moisture, gases and unpleasant odors in the stables (NH_3 , CO_2)
- 2 Water: adsorbent cations, anions, heavy metals, low polar organic molecules
- 3 Land: adsorbent radionuclides; tool for re-cultivation of land
- 4 Feed: adsorbent of mycotoxins (zearalenone, aflatoxin B1, B2 and G2; vomitoxin, ochratoxin, T-2, DAS -diacetoksisiciprenol), feed additive for the prevention of diarrhea and regulating the pH.

However, storing and keeping of once used zeolite is still not fully resolved, and very low number of studies are engaged in solving this problem. The aim of this paper is to investigate the potential use of natural zeolite (clinoptilolite) contaminated with lead ions in the cement industry as the replacement of standard additions in portland cement.

EXPERIMENTAL PART

Material. In this investigations, natural zeolite originating from Vranjska Banja, Serbia and Portland cement PC 42.5R from Beočin, Serbia were used. Chemical compositions of starting raw materials are given in table 1.

Methods. Adsorption isotherm for lead ions removal by the natural zeolite was obtained according following procedure: 1 g of the natural zeolite was mixed with 50 ml of aqueous solution contaminated with different concentrations of lead ions (350-7200 mg/l). The suspensions were mixed 24 h, at a stirring speed of 350 min^{-1} . The initial pH was not adjusted and only was only measured and was in range from 2.8 for the maximum lead ions concentration (7200 mg / l), to 4.3 for the lowest concentration of lead ions (350 mg / l). After reaction time, suspensions were centrifuged for 10 min at $10\,000 \text{ min}^{-1}$. Then, using the atomic absorption spectrophotometry (AAS) and the

instrument "Aanalysis 300" starting and non adsorbed concentrations of lead ions were determined. The amounts of adsorbed ions of lead were determined from the difference between the initial and the concentration of ions in the filtrate.

To obtain with lead ions contaminated zeolite which will be used as an addition to Portland cement, concentration of lead ions equal to the maximum adsorption capacity obtained by adsorption isotherm was used. The procedure for obtaining contaminated zeolites was:

Natural zeolite was milled to reach the grain size 100% of $-43 \mu\text{m}$, and afterwards mixed with PbNO_3 under following procedure: 400 g of $\text{Pb}(\text{NO}_3)_2$ was dissolved in 120 l of distilled water. Then, 3.5 kg of natural zeolite was added in lead ions contaminated water, so the concentration of lead ions in the solution was approximately equal to the maximum adsorption capacity obtained by adsorption isotherm (66 mg/g). Reaction time was 7 days with occasional mixing. After 7 days dewatering, filtration, drying and disintegration of sample was done. Finally, dry zeolite contaminated with lead ions (PbZ) was obtained. Determined adsorbed amount of lead ions was $\sim 60 \text{ mgPb}^{2+}/\text{g}$ which is very close to the maximum adsorption capacity of natural zeolite obtained from adsorption isotherm ($66 \text{ mgPb}^{2+}/\text{g}$).

Afterwards, mixing of Portland cement with zeolite: 10% (PC10Z), 30% (PC30Z), 50% (PC50Z), and 10% of Pb-zeolite (PC10PbZ); 30% of Pb-zeolite (PC30PbZ) and 50% of Pb-zeolite (PC50PbZ) in laboratory mill without balls was performed in duration of 10 minutes. On such obtained samples of cement with addition of zeolite and on starting sample (cement PC42.5R) standard physico-mechanical testing have been performed according to European Normative EN197:

- Determination of the specific surface according to Blaine (EN 196-6) (Blaine apparatus was used).
- Determination of standard consistency (EN196-3) (Vicat apparatus was used).
- Determination of the setting time of the cement dough (EN 196-3) (Vicat apparatus was used with the rod replaced by a needle)
- Determination of the compressive strength (EN 196-1).

Thusly, the following cements with additions are obtained:

PC42.5R
PC+10%-PZ = PC10Z
PC+30%-PZ = PC30Z
PC+50%-PZ = PC50Z
PC+10%PbZ = PC10PbZ
PC+30%PbZ = PC30PbZ
PC+50%PbZ = PC50PbZ

All stated cements had mass of 4000 g.

RESULTS AND DISCUSSION

Results of adsorption of lead ions from water by using natural zeolite

To determine the maximum adsorption capacity of natural zeolite for lead ions, at first adsorption isotherm was determined. This part of experiments was important, in order to define the conditions for obtaining the contaminated zeolite which will be used as an addition to portland cement.

Results of adsorption of lead ions on the natural zeolite, with particle size -43 μm are given in Figure 1. Figure 1a showed the adsorption isotherm, which is obtained as a dependence of the amount of adsorbed lead ions in mg/g vs. equilibrium concentration of pollutant in the solution after adsorption on zeolite in mg/l. Figure 1b showed dependence of the adsorption percent of lead ions vs. its initial concentration. From Figure 1a, it can be seen that, under applied experimental conditions, with increasing initial concentration of pollutants increases the concentration of adsorbed ions of lead.

The results showed in Figure 1a are fitted using the Langmuir and the Freundlich adsorption model. Mathematical forms of this two isotherms are given in equations 1 and 2.

$$\text{Langmuir isotherm: } c_{ads} = \frac{abc_e}{1 + bc_e} \quad (1)$$

$$\text{Freundlich isotherm: } c_{ads} = bc_e^\beta, \quad (2)$$

where c_{ads} is the concentration of the adsorbed lead ions (mg/g), c_e is the equilibrium concentration of lead ions in the filtrate (mg / l), a is the maximum adsorbed lead ions concentration of adsorbed per gram of adsorbent, b is the adsorption equilibrium constant of the surface (l/mg), and the β is factor heterogeneity and determined affinity of the adsorbent according to adsorbate.

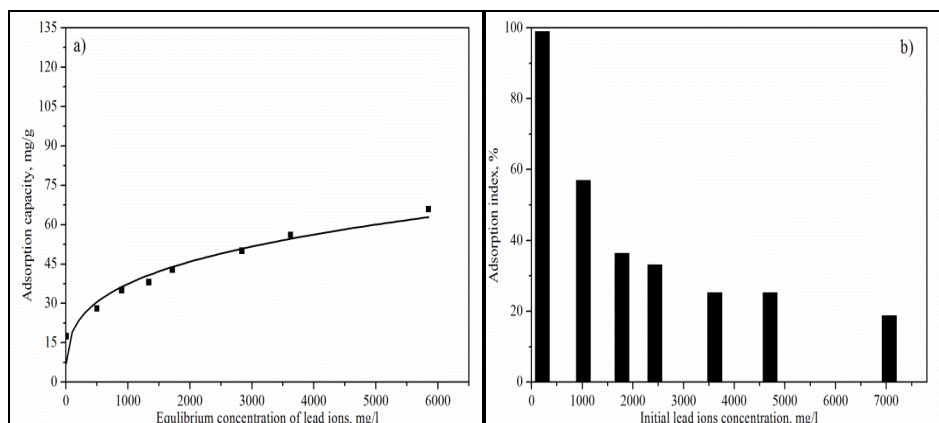


Figure 1. a) Adsorption isotherms of lead ions on natural zeolite; b) The dependency index sorption of lead in the function of the starting concentration of lead ions in solution

For results showed in Figure 1 the best fit of experimental data was obtained by the Freundlich adsorption model ($r^2 > 0.93$) indicating a complex mechanism of binding ions lead to the natural zeolite. The maximum amount of adsorbed lead ions was 66 mg/g.

Also, we measured the pH of the suspension after the reaction time. For all initial concentrations of lead ions equilibrium pH were higher than initial, but, both, initial and equilibrium pH, decreased with increasing initial concentration of lead ions. Measuring of the equilibrium pH showed that final pH, for all initial concentrations of lead ions was lower than pH 6.0 what according to [14] mining that lead was in cationic, and dominantly Pb^{2+} form, i.e. precipitation of lead in form of hydroxide did not occur. These investigations showed that the natural zeolite (clinoptilolite) from the Vranjska banja, Serbia has a good adsorption capacity for binding lead ions from water and aqueous solutions. However, because storage of waste material (zeolite contaminated with lead ions) is still not solved, we did the second part of experiments, and investigated opportunity of using that material as an additive in Portland cement production instead of the existing standard additions.

Results of the application of the natural zeolite contaminated with lead ions as an additive in portland cement

The influence of zeolite and saturated zeolite on Portland cement quality was monitored by determining of the chemical composition, physical and mechanical characteristics.

The results of the chemical analysis of all portland cement mixes are given in the Table 1, while in the Table 2 are presented the results of physical properties of PC42.5R and PC with addition of zeolite and contaminated in various mass ratios.

As can be seen (Table 1) the content of PbO in the PbZ was 2.22. When such zeolite is added into PC in mass ratio 10 %, the 0.29 PbO is obtained (PC10PbZ). By increasing of the PbZ content in the PC, the content of PbO also increased. Thus, with 50 % content of Pb zeolite and 50 % PC 42.5R, there was 1.08 % of PbO in the PC50PbZ sample. In our previous paper [15] it was found that lead ions are connected via strong forces with natural zeolite and there is no desorption of its ions. Because of that, the Pb leaching test has not been performed in this investigation.

Table 1. Chemical composition of zeolites and portland cements

Oxide, %	SiO ₂	Al ₂ O ₃	CaO	MgO	Fe ₂ O ₃	K ₂ O	Na ₂ O	TiO ₂	SO ₃	MnO	PbO	LoI
Zeolite samples												
PbZ	65.12	13.50	3.47	0.39	2.25	1.15	1.07	-	-	-	2.22	10.83
Cement samples												
PC 42.5R	24.02	6.03	59.60	2.03	1.48	0.76	0.34	0.346	3.4	0.087	0.0065	1.89
PC10Z	29.40	6.46	48.87	1.84	2.25	0.68	0.32	0.17	3.13	0.080	0.0043	6.75
PC30Z	33.11	7.89	43.73	1.64	2.23	0.81	0.45	0.17	2.70	0.066	0.0038	7.20
PC50Z	40.60	9.34	34.41	1.40	2.23	0.89	0.36	0.17	2.28	0.053	0.0033	7.92
PC10PbZ	28.10	6.71	53.87	2.00	1.58	0.82	0.55	0.646	2.80	0.084	0.29	2.75
PC30PbZ	34.83	8.02	43.66	1.91	1.57	1.04	0.71	0.346	2.68	0.071	0.72	4.44
PC50PbZ	40.75	9.54	34.98	1.80	1.53	1.01	0.88	0.329	1.88	0.054	1.08	6.16

Since the initial zeolite sample had grain size $-43 \mu\text{m}$, when this sample is mixed with Portland cement the specific surface area significantly increased what can be seen in Table 2. Standard consistency (SK, %) of all investigated cements is higher than SK of PC (29.0%), and was in the range from 31.6 % for PC10PbZ to 44.0 % for PC50FeZ.

Table 2. Results of physico-mechanical properties of the investigated cements

Sample	Fineness, %	Sp, cm^2/g	SK, %	Setting time, min		
				Start of the setting	End of the setting	
PC42.5R	0.6	3940	29.0	220	270	
PC10Z						
PC30Z						
PC50Z						
PC10PbZ	0.05	4880	31.6	190	250	
PC30PbZ	0.06	6960	37.0	270	340	
PC50PbZ	0.1	9840	36.8	190	250	
Sample	Flexural strength, MPa			Compressive strength, MPa		
	2 days	7 days	28 days	2 days	7 days	28 days
PC42.5R	6.4±0,8	8.4±0,6	9.6±0,8	33.0±0,7	49.0±0,8	61.1±1.4
PC10Z	1.8±0,1	5.4±0,2	10.3±0,4	5.3±0,1	22.5±1,8	58.9±0.6
PC30Z	2.1±0,1	6.1±0,1	9.7±0,5	7.5±0,2	29.8±0,3	57.4±1.5
PC50Z	2.0±0,1	5.8±1,1	7.8±0,5	6.7±0,3	26.9±1,1	50.5±1.9
PC42.5R	6.4±0.8	8.4±0.6	9.6±0.8	33.0±0.7	49.0±0.8	61.1±1.4
PC10PbZ	6.2±0.4	7.5±0.7	9.2±0.2	25.2±0.8	41.2±0.9	63.3±2.1
PC30PbZ	4.3±0.1	5.9±0.2	8.5±0.5	17.0±0.4	31.3±0.7	57.3±0.6
PC50PbZ	2.4±0.1	4.2±0.6	6.9±0.3	10.0±0.6	23.4±0.1	44.9±1.5

According to the Point 17 of Regulation [15] which concerns cement PC 42.R and similar cements (according to the SRPS EN 196-3), initiation of setting time for cement PC 42.5 R must be ≥ 60 min. All investigated cements are fulfilling this condition. However, the shortest setting time (initiation and ending) is noticed in the case of PC30Z (80/100 min) and PC50Z (80/130 min). The longest setting time is obtained in the case of PC30PbZ (270/340 min), which is longer time period than PC setting time for 50 min (initiation), i.e. 70 min – end of setting.

Table 2 show values of flexural strength and compressive strength of Portland cement and cement with addition natural and saturated zeolite for 2, 7, 28 days of hydration. Based on these results it can be seen that values of flexural strength and compressive strength depend on the mass content of addition used in Portland cement. Strengths of investigated cements are decreasing with the increasing of saturated zeolite content in the PC for all investigation periods (2, 7 and 28 days). However, the decrease of the strength was after 2 days of hydration, lower for 7 days, and the lowest for 28 days.

According to the EN 197 Standard, cement marked as CAM I should have compressive strength 42.5 MPa. Compressive strengths of newly-formed cements with addition of Pb ion saturated zeolite after 28 days of hydration are mostly above these values. It can be said that all used zeolites can be applied as additives for Portland cement in the quantity up to 30 %. Namely, values of compressive strength (after 28

days of solidification) for all cements with 30 % of additive are high (PC30PbZ – 57.3 MPa and PC30Z – 57.4 MPa) in comparison with requested minimum - 42.5 MPa.

CONCLUSION

Based on the results obtained from adsorption isotherms, it can be seen that increasing of the concentration of the lead ions in the water solutions the concentration of adsorbed lead ions increased. The maximum amount of lead ions adsorbed on zeolite under certain experimental conditions was 66 mg Pb²⁺/g.

Results of using zeolite saturated with lead ions as the addition for portland cement, instead of the more common additives, showed that this material can be used as Supplementary Cementitious Materials (SCM). Since the flexural strengths (after 28 days of solidification) for the samples obtained by addition of 50 wt. % zeolite into Portland cement are in the range 7 - 8 MPa, they can be considered as low values. The optimal mass content of zeolite or with lead ions contaminated zeolites in Portland cement should not exceed 30 %. Since values of compressive strength (after 28 days of solidification) for all cements with 30 % of additive are higher (PC30PbZ – 57.3 MPa and PC30Z – 57.4 MPa) in comparison with requested minimum - 42.5 MPa, it may be concluded that used zeolite (lead ions saturated) can be applied as additives for Portland cement in the quantity up to 30 wt. %.

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