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Editor
Milica Vlahović

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FOREWORD

The conditions created by the development of technologies in which modern man lives have led to a complex and paradoxical effect: that by removing obstacles on the way to a more comfortable, simpler, faster and more efficient life and way of working, man also generates numerous misfortunes, attracting dark clouds of threats to the survival of the planet and humanity. The question that concerns and affects all of us - all people, all living beings, systems in which life takes place, large and small, strong and weak - boils down to the problem of the negative impact of man on the environment; this issue invites us to an urgent solution by looking at the causes, proposing solutions, evaluating them, changing approaches and ways of thinking, as well as drawing correct conclusions. Simply put, by adapting nature to one's own needs, man threatens and damages it. That is why, with the joint efforts of all of us, individuals, organizations and states, it is necessary to take all possible measures to immediately prevent the negative effects that are ahead of us.

The importance of renewable sources of electricity, which this international conference focuses on, is noticeable from two angles: the first - it is certain that fossil fuels as a resource will disappear and it is necessary to find alternative sources, the second - the use of renewable energy sources by its essence implies "clean" technology that significantly contributes to reducing CO₂ emissions and thus mitigating climate change and reducing pollution, while encouraging social and economic development in all spheres of life.

The 11th International Conference on Renewable Electrical Power Sources is organized by the Society for Renewable Electrical Power Sources (DOIEE) at SMEITS, with co-organizers: The Institute of Architecture and Urban & Spatial Planning of Serbia (IAUS) and the Chamber of Commerce and Industry of Serbia, with the support of the Ministry of Science, Technological Development and Innovation of the Republic of Serbia.

The registered participants designed their papers according to the given conference topics:

- Energy sources and energy storage;*
- Energy efficiency in the context of use of renewable energy sources (RES);*
- Environment, sustainability and policy;*
- Applications and services.*

Eminent authors - scientists, teachers, experts in this field from fifteen different countries: Algeria, Belgium, Bosnia and Herzegovina, China, Croatia, Greece, Hungary, India, Portugal, Saudi Arabia, Serbia, Slovenia, Spain, the United Arab Emirates, and Ukraine, contributed to the conference through sixty-nine papers that were reviewed by the Scientific Committee of the Conference, and after the review process were accepted for presentation at the conference and for publication in the proceedings.

At the end of this short message and at the beginning of the proceedings I believe that it can be proudly said that scientists, researchers, policy makers and industry experts gathered in one place, in order to exchange experiences and knowledge with the aim of promoting scientific and professional ideas and results of research, technology improvement for the use of RES, promoting the rational use of electricity, affirming and proposing inventive solutions in the field of sustainable sources of electricity.

*Belgrade,
November 2023*

Milica Vlahović

SADRZAJ / CONTENTS

Plenarna predavanja:

1. **IZAZOVI U ELEKTROHEMIJSKOM SKLADIŠTENJU ENERGIJE**
CHALLENGES IN THE ELECTROCHEMICAL ENERGY STORAGE
Branimir N. GRGUR..... 1
2. **POLIANILIN: PROVODNI POLIMER U UREĐAJIMA ZA SKLADIŠTENJE ENERGIJE**
POLYANILINE: CONDUCTIVE POLYMER IN ENERGY STORAGE SYSTEMS
Aleksandra JANOSEVIC LEZAIC 11
3. **ISPITIVANJE KVALITETA EKSPLOZIVNO ZAVAREN OG SPOJA RAZNORODNIH METALA ZA POTENCIJALNU PRIMENU U OBNOVLJIVIM IZVORIMA ENERGIJE**
TESTING THE QUALITY OF EXPLOSIVELY WELDED JOINTS OF DISSIMILAR METALS POTENTIALLY APPLICABLE IN RENEWABLE ENERGY SOURCES
Ana ALIL, Milos LAZAREVIC, Danica BAJIC, Nada ILIC, Tihomir KOVACEVIC, Bogdan NEDIC..... 23
4. **METODE BEZ RAZARANJA I UNAPREĐENJE POUZDANOSTI RADA KULE ZA HLAĐENJE, KAO ASPEKT TEMATIZACIJE OBNOVLJIVIH IZVORA ENERGIJE**
NON-DESTRUCTIVE METHODS AND IMPROVEMENT OF THE COOLING TOWER OPERATION RELIABILITY, AS AN ASPECT OF RENEWABLE ENERGY SOURCES THEMATIZATION
Marko JARIC, Sanja PETRONIC, Nikola BUDIMIR, Zoran STEVIC, Suzana POLIC..... 35

Energetski izvori i skladištenje energije:

1. **ELEKTRIČNA SVOJSTVA TANKIH FILMOVA GO I GO/WPA NA INTERDIGITALNIM ELEKTRODAMA**
ELECTRICAL PROPERTIES OF GO AND GO/WPA THIN FILMS ON INTERDIGITAL ELECTRODES
Zeljko MRAVIK, Milica PEJCIC, Sonja JOVANOVIC, Darija PETKOVIC, Misa STEVIC, Zoran STEVIC, Zoran JOVANOVIC..... 45
2. **MODELOVANJE I SIMULACIJA UREĐAJA ZA NAVODNJAVANJE KAP-PO-KAP**
MODELING AND SIMULATION OF A DEVICE APPLIED FOR LOW-FLOW DRIP IRRIGATION
Noureddine BENSEDIRA, Abdessmad MILLES, Mohammed-Salah AGGOUNE 53
3. **UTICAJ SENKE USLED DENIVELACIJE KROVA NA PROIZVODNJU KROVNE SOLARNE ELEKTRANE IZLAZNE SNAGE 400KW**
THE INFLUENCE OF THE SHADOW CAUSED BY THE SLOPE OF THE ROOF ON THE PRODUCTION OF A ROOF-TOP SOLAR POWER PLANT WITH AN OUTPUT POWER OF 400KW
Marko S. DJUROVIC, Zeljko V. DESPOTOVIC 61

4. PROJEKTOVANJE I IZVOĐENJE SOLARNE ELEKTRANE IZLAZNE SNAGE 400KW NA KROVU FABRIČKE HALE "EP BELT"-LOZNICA	
DESIGN AND REALISATION PV ROOF-TOP POWER PLANT 400KW IN THE FACTORY "EP BELT"-LOZNICA	
Zeljko V. DESPOTOVIC, Marko S. DJUROVIC	67
5. PRENAMENA NAPUŠTENIH ILI STARIH NAFTNIH POLJA ZA IZGRADNJU GEOTERMALNIH ELEKTRANA	
THE CONVERSION OF ABANDONED OR MATURE OIL FIELDS INTO GEOTHERMAL POWER PLANT LOCATIONS	
Ivan RAJSL, Sara RAOS	79
6. POBOLJŠANJE SPOSOBNOSTI SAMOIZLEČIVANJA I ŽILAVOSTI MIKROKAPSULA SA TUNG ULJEM DODATKOM GRAFENSKIH NANOPLOCICA I NJIHOVA PRIMENA U EPOKSI SISTEMU	
THE IMPROVEMENT OF SELF-HEALING CAPABILITY AND TOUGHNESS OF MICROCAPSULES WITH TUNG OIL BY THE ADDITION OF GRAPHENE NANOPATELETS AND THEIR APPLICATIONS IN EPOXY SYSTEM	
Natasa TOMIC, Abdullah MUSTAPHA, Maitha ALMHEIRI, Mohamed Nasr SALEH	87
7. MODEL SOLARNOG PANELA SA SOLARNIM TRAGAČEM, UPRAVLJAN POMOĆU ARDUINO UNO MODULA	
MODEL OF THE SOLAR PANEL WITH SOLAR TRACKER CONTROLLED BY THE ARDUINO UNO BOARD	
Ivan TODORIC, Djordje DIHOVICNI, Dragan KRECULJ, Sanja JEVTIC, Nada RATKOVIC KOVACEVIC	93
8. TERMoeLEKTRIČNI EFEKAT KAO IZVOR ENERGIJE U PRUŽNIM ŽELEZNIČKIM APLIKACIJAMA	
THERMOELECTRIC EFFECT AS A SOURCE OF ENERGY IN RAILWAY TRACKSIDE APPLICATIONS	
Sanja JEVTIC, Milesa SREČKOVIĆ, Dragan KRECULJ, Nada RATKOVIĆ KOVACEVIC.....	101
9. POREĐENJE RAZNOVRNIH TIPOVA ENERGIJE OD POKRETNIH VODA	
COMPARISON OF VARIOUS TYPES OF ENERGY FROM MOVING WATERS	
Djordje DIHOVICNI, Dragan KRECULJ, Olga JAKSIC, Nada RATKOVIC KOVACEVIC	107
10. ISPITIVANJE LIF/B SISTEMA KORIŠĆENJEM NEGATIVNOG MODA LDI MS: MOGUĆI SISTEM ZA SKLADIŠTENJE VODONIKA	
INVESTIGATION OF LIF/B SYSTEM USING THE NEGATIVE MODE LDI MS: A POSSIBLE HYDROGEN STORAGE SYSTEM	
Filip VELJKOVIC, Bojan JANKOVIC, Ivana STAJCIC, Milovan STOJILJKOVIC, Marija JANKOVIC, Djordje KAPURAN, Suzana VELICKOVIC	115
11. UŠTEDA ENERGIJE PRILIKOM ELEKTROLITIČKOG DOBIJANJA VODONIKA-POREĐENJE DVOKOMPONENTNIH I TROKOMPONENTNIH JONSKIH AKTIVATORA	
ENERGY SAVINGS IN ELECTROLYTIC HYDROGEN PRODUCTION – COMPARISON OF BINARY AND TERNARY ACTIVATORS	
Sladjana MASLOVARA, Dragana VASIC ANICIJEVIC, Snezana BRKOVIC, Vladimir NIKOLIC, Milica MARCETA.....	119

12. KINETIKA TERMALNE DEGRADACIJE LIGNOCELULOZNOG OTPADA NA BAZI KOŠTICA BRESKVE THERMAL DEGRADATION KINETICS OF LIGNOCELLULOSIC PEACH STONE WASTE Zorica LOPIČIĆ, Anja ANTANASKOVIĆ, Slobodan CVETKOVIC, Vladimir ADAMOVIĆ, Tatjana SOSTARIC, Jelena AVDALOVIC, Mirjana KIJEVCANIN	125
13. THERMAL PROPERTIES OF RAPIDLY SOLIDIFIED Cu-Al-Ni-Mn SHAPE MEMORY ALLOY Borut KOSEC , Milan BIZJAK, Mirko GOJIC, Ales NAGODE, Ivana IVANIC, Blaž KARPE	133
14. PROCENA POTENCIJALA POLJOPRIVREDNO-FOTONAPONSKIH SISTEMA U SRBIJI ASSESSMENT OF THE AGRIVOLTAIC POTENTIAL IN SERBIA Aleksandar IVANCIC, Melita ROGELJ, Bora OBRADOVIC, Slaviša JELISIC.....	139

Energetska efikasnost u kontekstu primene RES:

1. ULOGA KUPCA-PROIZVOĐAČA (PROZJUMERA) U PRIMENI OIEE U SRBIJI: PRE-PREKE I MOGUĆNOSTI THE ROLE OF THE BUYER-PRODUCER (PROSUMER) IN THE IMPLEMENTATION OF RES IN SERBIA: OBSTACLES AND OPPORTUNITIES Marina NENKOVIC-RIZNIC, Borjan BRANKOV, Mila PUCAR, Ana STANOJEVIC.....	147
2. PRIMENA SERIJSKE VEZE KOMPONENTI FREKVENTNO ZAVISNIH KOMPONENTI ISTOG TIPA U SISTEMIMA SA OBNOVLJIVIM IZVORIMA ENERGIJE APPLICATION OF A SERIES CONNECTION OF THE SAME TYPE BANDPASS FREQUENCY DEPENDENT COMPONENTS IN SYSTEMS WITH RENEWABLE ENERGY SOURCES Tykhon SYTNIKOV, Igor PEREKRESTOV, Andrey CHMELECKSKY, Pavlo STUPEN, Valerii SYTNIKOV.....	159
3. SMANJENJE GUBITAKA U DISTRIBUTIVNOJ MREŽI UVAŽAVAJUĆI NESIGURNOST SNAGE OPTEREĆENJA I DISTRIBUIRANE PROIZVODNJE IZ OBNOVLJIVIH IZVORA REDUCTION OF LOSSES IN THE DISTRIBUTION NETWORK CONSIDERING THE UNCERTAINTY OF LOAD AND RENEWABLE DISTRIBUTED GENERATION POWER Nikola KRSTIC, Dragan TASIC, Teodora DENIC.....	165
4. TEHNOLOGIJE ZA PRAĆENJE POLJOPRIVREDNIH ZASADA POMOĆU BESPILOTNIH LETILICA TECHNOLOGIES FOR MONITORING AGRICULTURAL CROPS USING UAV Njegos DRAGOVIC, Milovan VUKOVIC, Snezana UROSEVIC	173
5. MIKRO STEP ELEKTROMOTORNI POGON KONTROLISAN MIKROKONTROLEROM MICRO STEP ELECTRIC DRIVE CONTROLLED BY MICROCONTROLLER Misa STEVIC, Zoran STEVIC, Predrag STOLIC, Ilija RADOVANOVIC, Dejan ILIC, Zoran JOVANOVIĆ.....	181
6. SMART MATERIJALI I SAVREMENI KONTEKST ZA FUNKCIONALIZACIJU OBNOVLJIVIH IZVORA ENERGIJE U GALERIJSKOM PROSTORU SMART MATERIALS AND CONTEMPORARY CONTEXT FOR THE FUNCTIONALIZATION OF RENEWABLE ENERGY SOURCES IN THE GALLERY SPACE Suzana POLIC, Sanja PETRONIC, Marko JARIC.....	185

7. BLOCKCHAIN I RANE VIZUELIZACIJE KORIŠĆENJA ENERGIJE VETRA U MUZEJSKIM KOLEKCIJAMA BLOCKCHAIN AND EARLY VISUALIZATION OF THE USE OF WIND ENERGY IN MUSEUMS COLLECTIONS Suzana POLIC	195
8. ENERGETSKA EFIKASNOST U ELEKTRIČNIM VOZILIMA – PREGLED ENERGY EFFICIENCY IN ELECTRIC VEHICLES – AN OVERVIEW Zoran STEVIC, Ilija RADOVANOVIĆ, Predrag STOLIC, Sanja PETRONIC, Marko JARIC, Misa STEVIC, Dejan ILIC.....	203
9. TOPOLOGIJE NEIZOLOVANIH DC-DC KONVERTORA SA POBOLJŠANIM KARAKTERISTIKAMA NON-ISOLATED DC-DC CONVERTERS TOPOLOGIES WITH IMPROVED CHARACTERISTICS Oleksii YAMA, Zoran STEVIC, Oleksandr BONDARENKO	209
10. MOGUĆNOST PRIMENE ULTRAZVUČNE KAVITACIJE U PROCESU PRERADE INDUSTRIJSKIH OTPADNIH VODA POSSIBILITY OF USING ULTRASONIC CAVITATION IN THE PROCESS OF INDUSTRIAL WASTEWATER TREATMENT Sladjana JEZDIMIROVIC, Marina DOJCINOVIC	219
11. ZNAČAJ DISTRIBUCIJE TOPLOTE U SAVREMENIM ENERGETSKI EFIKASNIM ELEKTRIČNIM VOZILIMA IMPORTANCE OF HEAT DISTRIBUTION IN MODERN ENERGY EFFICIENT ELECTRICAL VEHICLES Zoran STEVIC, Borivoje BEGENISIC, Dušan MURGASKI, Luka STAJIC, Sanja PETRONIC, Ilija RADOVANOVIĆ, Suzana POLIC	227
12. PRIMERI PRIMENE VIŠEKRITERIJUMSKOG ODLUČIVANJA U OBLASTI OBNOVLJIVIH IZVORA ENERGIJE EXAMPLES OF THE APPLICATION OF MULTI-CRITERIA DECISION-MAKING IN THE FIELD OF RENEWABLE ENERGY SOURCES Zoran STIRBANOVIC, Dragiša STANUJKIC, Jovica SOKOLOVIC.....	233

Životna sredina, održivost i politika:

1. RAZMATRANJE PRISUSTVA FENANTRENA U OPŠTINI BOR NA BAZI NJEGOVOG SADRŽAJA U LIŠĆU I STABLJKAMA HEDERA HELIX L. A CONSIDERATION OF PHENANTHRENE PRESENCE IN BOR'S MUNICIPALITY BASED ON ITS CONTENT IN LEAVES AND STEMS OF HEDERA HELIX L. Aleksandra D. PAPLUDIS, Slađana C. ALAGIC, Snezana M. MILIC, Jelena S. NIKOLIC, Dragana V. MEDIĆ, Zoran M. STEVIC, Vesna P. STANKOV JOVANOVIĆ.....	239
2. PERSPEKTIVE GRADSKOG VAZDUŠNOG SAOBRAĆAJA U BEOGRADU, SRBIJA PROSPECTS OF URBAN AIR MOBILITY IN BELGRADE, SERBIA Jelena SVORCAN, Djordje CANTRAK, Jelena ANDRIC, Andrea IANIRO.....	245

3. ULOGA SINERGIJE RUDARSKIH I RAČUNARSKIH TEHNOLOGIJA U PROCESU TRANZICIJE KA OBNOVLJIVIM IZVORIMA ELEKTRIČNE ENERGIJE	
THE ROLE OF THE SYNERGY OF MINING AND COMPUTER TECHNOLOGIES IN THE PROCESS OF TRANSITION TO RENEWABLE ELECTRICAL POWER SOURCES	
Predrag STOLIC, Ilija RADOVANOVIĆ, Zoran STEVIC, Dejan PETROVIC.....	253
4. ODRŽIVOST REŠENJA ZASNOVANIH NA OBNOVLJIVIM IZVORIMA ELEKTRIČNE ENERGIJE – INFORMATIČKI PRISTUP	
SUSTAINABILITY OF SOLUTIONS BASED ON RENEWABLE SOURCES OF ELECTRICITY - ICT APPROACH	
Predrag STOLIC, Ilija RADOVANOVIĆ, Zoran STEVIC	261
5. CHATGPT, MATERIJALI I OBNOVLJIVI IZVORI ENERGIJE: JEDAN NEELABORIRANI PROSTOR	
CHATGPT, MATERIALS AND RENEWABLE ENERGY SOURCES: ONE UNREALIZED SPACE	
Suzana POLIC, Sanja PETRONIC, Marko JARIC.....	269
6. ANALIZA STRUKTURE OŠTEĆENJA GRAĐEVINSKIH KONSTRUKCIJA NA OSNOVU ODREĐIVANJA FRAKCIONOG SASTAVA OSTATAKA	
ANALYSIS OF THE STRUCTURE OF BUILDING STRUCTURE FAILURES BASED ON THE DETERMINATION OF THE FRACTIONAL COMPOSITION OF DEBRIS	
Valerija CHORNA, Elena PONOMARYOVA, Sergey SHATOV, Liliia DRUZHININA.....	279
7. UTICAJ EFEKTA STAKLENE BAŠTE NA KLIMATSKE PROMENE	
THE INFLUENCE OF THE GLASS GARDEN EFFECT ON CLIMATE CHANGES	
Sladjana JEZDIMIROVIC, Marina DOJCINOVIC	287
8. PRIMENA TEHNOLOGIJE 3D ŠTAMPE BETONA U REPUBLICI SRBIJI	
APPLICATION OF 3D CONCRETE PRINTING TECHNOLOGY IN SERBIA	
Stefan Z. MITROVIC, Ivan IGNJATOVIC.....	295
9. ULOGA VODOPROPUSNIH PROIZVODA U POPLOČAVANJU URBANIH SREDINA U SVETLU ODRŽIVOG KORIŠĆENJA RESURSA	
THE ROLE OF PERMEABLE PRODUCTS IN THE PAVING OF URBAN ENVIRONMENT IN THE LIGHT OF SUSTAINABLE USE OF RESOURCES	
Marina ASKRABIC, Aleksandar RADEVIC, Aleksandar SAVIC	301
10. OTPADNO STAKLO KATODNIH CEVI U PRIPREMI BETONA – POVEĆAVANJE ODRŽIVOSTI	
CATHODE RAY TUBE WASTE GLASS IN CONCRETE PREPARATION – INCREASING SUSTAINABILITY	
Ivana JELIĆ, Aleksandar SAVIC, Tatjana MILJOJIC, Marija SLJIVIC-IVANOVIC, Marija JANKOVIC, Slavko DIMOVIC, Dimitrije ZAKIC, Dragi ANTONIJEVIC	309
11. DOPRINOS STUDIJI VEGETACIJSKOG POKRIVAČA: STUDIJA SLUČAJA ZELENIH POVRŠINA U GRADU HRAOUA (ALŽIR)	
CONTRIBUTION TO THE STUDY OF VEGETATION COVER: A CASE STUDY OF GREEN SPACES IN THE CITY OF HRAOUA (ALGERIA)	
Mostafia BOUGHALEM, Mourad ARABI, Abdoukadr TOURE, Khadidja BOUKAROUBA, Farida OUZAL	317

12. TRANZICIJA KA OBNOVLJIVIM IZVORIMA ENERGIJE, DEKARBONIZACIJA I PROMENE U ENERGETSKOM SEKTORU KOJE UTIČU NA RADNIKE U TRADICIONALNIM INDUSTRIJAMA TRANSITION TO RENEWABLE ENERGY SOURCES, DECARBONIZATION, AND CHANGES IN THE ENERGY SECTOR AFFECTING WORKERS IN TRADITIONAL INDUSTRIES	
Miloš CURCIC	323

Aplikacije:

1. IMPLEMENTACIJA SOLARNE ELEKTRANE SNAGE 200 KWP NA RAVNOM KROVU U PARAĆINU IMPLEMENTATION OF 200 KWP SOLAR POWER PLANT ON A FLAT ROOF IN PARAĆIN Bosko IVANKOVIC, Zoran LAZAREVIC, Ilija RADOVANOVIC, Misa STEVIC, Predrag STOLIC, Dejan ILIĆ, Zoran STEVIC	329
2. FIZIČKO-HEMIJSKA KARAKTERIZACIJA ŠTAMPANIH PLOČA PHYSICO-CHEMICAL CHARACTERIZATION OF PCBs Silvana B. DIMITRIJEVIC, Aleksandra T. IVANOVIC, Srdjana MAGDALINOVIC, Stefan S. DJORDJIJEVSKI, Stevan P. DIMITRIJEVIC	333
3. DEALLOYING PDNI5 LEGURE U 0.5M SULFATNOJ KISELINI DEALLOYING OF PDNI5 ALLOY IN 0.5M SULFURIC ACID Stevan P. DIMITRIJEVIC, Silvana B. DIMITRIJEVIC, Aleksandra T. IVANOVIC, Renata KOVACEVIC	341
4. SAGOREVANJE OTPADNOG TERMOBARIČNOG EKSPLOZIVA POD KONTROLISANIM USLOVIMA KAO IZVOR ENERGIJE COMBUSTION OF WASTE THERMOBARIC EXPLOSIVE UNDER CONTROLLED CONDITIONS AS A SOURCE OF ENERGY Danica BAJIC, Mirjana KRSTOVIC, Mladen TIMOTIJEVIC, Bojana FIDANOVSKI	351
5. INTERAKCIJE LASERA OD INTERESA ZA MATERIJALE U SISTEMIMA I KOMPONENTAMA U TRANSFORMACIJI ENERGIJE U LINEARNOM I NELINEARNOM OPSEGU LASER INTERACTION OF INTEREST FOR MATERIALS IN SYSTEMS AND COMPONENTS IN ENERGY TRANSFORMATION IN LINEAR AND NONLINEAR RANGES Mileša SRECKOVIC, Aleksandar BUGARINOVIC, Milanka PECANAC, Zoran KARASTOJKOVIC, Milovan JANIĆIJEVIC, Aleksander KOVACEVIC, Stanko OSTOJIC, Nenad IVANOVIC	359
6. DETEKCIJA MELASE LAŽNIH DATULA INFRACRVENOM SPEKTROKOPIJOM PRIMENOM HIJERARHIJSKE KLASIFIKACIJE DETECTION OF DATE MOLASSES ADULTERATED BY INFRARED SPECTROSCOPY USING ASCENDING HIERARCHICAL CLASSIFICATION Samir CHERIGUI, Ilyes CHIKHI, Hadj FAYÇAL DERGAL, Ferial CHELLALI, Hanane CHAKER	369
7. DETEKCIJA FALSIFIKOVANJA MELASE GROŽĐA FIZIKO-HEMIJSKIM PARAMETRIMA DETECTION OF ADULTERATION OF GRAPE MOLASSES BY PHYSICO-CHEMICAL PARAMETERS Samir CHERIGUI, Ilyes CHIKHI, Hadj FAYÇAL DERGAL, Ferial CHELLALI, Hanane CHAKER	373
8. SENZOR SALINITETA ZASNOVAN NA HEKSAGONALNOM FOTONOM KRISTALNOM VLAKNU SALINITY SENSOR BASED ON A HEXAGONAL PHOTONIC CRYSTAL FIBER Ilhem MIREĐ, Hicham CHIKH-BLED.....	377

9. NAPREDAK U FOTONSKIM KRISTALNIM VLAKNAMA: METODE PROIZVODNJE I PRIMENA ŠIROKOG SPEKTRA	
ADVANCEMENTS IN PHOTONIC CRYSTAL FIBER: FABRICATION METHODS AND BROAD-SPECTRUM APPLICATIONS	
Mohammed DEBBAL, Hicham CHIKH-BLED, Mouweffeq BOUREGAA, Mohammed CHAMSE EDDINE OUADAH	385
10. ENERGETSKA EFIKASNOST PREDIZOLOVANIH PLASTICNIH CEVI	
ENERGY EFFICIENCIES OF PRE-INSULATING PLASTIC PIPES	
Vasilis ZOIDIS.....	393
11. STATISTIČKO MODELOVANJE NEKIH EKOLOŠKI PRIHVATLJIVIH LEGURA NA BAZI BAKRA	
STATISTICAL MODELING OF SOME ENVIRONMENTALLY-FRIENDLY COPPER-BASED ALLOYS	
Aleksandra T. IVANOVIC, Silvana B. DIMITRIJEVIC, Stevan P. DIMITRIJEVIC, Branka B. PETKOVIC.....	403
12. SPEKTROSKOPSKA ANALIZA NATRIJUM KARBONATA	
SPECTROSCOPY ANALYSIS OF ACTIVATED SODIUM CARBONATE	
Natasa DJORDJEVIC, Milica VLAHOVIC, Slavica MIHAJLOVIC, Nenad VUSOVIC, Srdjan MATIJASEVIC	409
13. ANALIZA PERFORMANSI KRUŽNOG FOTONSKOG KRISTALNOG VLAKNA ZA TERAHERC APLIKACIJE	
PERFORMANCE ANALYSIS OF CIRCULAR PHOTONIC CRYSTAL FIBER FOR TERAHERTZ APPLICATIONS	
Mohammed CHAMSE EDDINE OUADAH, Mohammed DEBBAL, Assia AHLEM HARRAT, Hicham CHIKH-BLED, Mouweffeq BOUREGAA	415
14. POSTUPAK IZRADE POLIMERNOG KALUPA ZA ISPITIVANJE NA ISTEZANJE BIKOMPOZITNIH MATERIJALA	
POLYMER MOULD MANUFACTURING FOR TENSILE TESTING OF BIOCUMPOSITE MATERIALS	
Marija BALTIC, Milica IVANOVIC, Igor STAMENKOVIC, Miloš VORKAPIC, Aleksandar SIMONOVIC	421
15. HABANJE Ti-6Al-4V NANOKOMPOZITA SA DISPERGOVANIM ZrO₂ DOBIJENOG MEHANIČKIM LEGIRANJEM I SPARK PLAZMA SINTEROVANJEM	
WEAR BEHAVIOR OF ZrO ₂ DISPERSED Ti-6Al-4V ALLOY NANOCOMPOSITES PREPARED BY MECHANICAL ALLOYING AND SPARK PLASMA SINTERING	
R. KARUNANITHI, M. PRASHANTH, M. KAMARAJ, S. SIVASANKARAN	427
16. PROIZVODNJA NISKOLEGIRANOG Cr-Mo-Ni ČELIKA U ELEKTROLUČNOJ PEĆI	
PRODUCTION OF LOW ALLOY Cr-Mo-Ni STEEL IN ELECTRIC ARC FURNACE	
M. GOJIC, M. DUNDJER, S. KOZUH, I. IVANIC, D. DUMENCIC	435
17. NUMERIČKA SIMULACIJA I DIZAJN SPOJNICA OD FOTONSKIH KRISTALNIH VLAKNA ZA SEPARACIJU TALASNIH DUŽINA	
NUMERICAL SIMULATION AND DESIGN OF A PHOTONIC CRYSTAL FIBER COUPLER FOR WAVELENGTH SEPARATION	
Assia AHLEM HARRAT, Mohammed CHAMSE EDDINE OUADAH, Mohammed DEBBAL.....	445

18. FOTOKATALITIČKA DEGRADACIJA KONGO CRVENE BOJE KORIŠĆENJEM KOMPOZITA UIO-66 METALO-ORGANSKIH MREŽNIH STRUKTURA I METALNIH OKSIDA PHOTOCATALYTIC DEGRADATION OF CONGO RED DYE USING UIO-66 MOF-METAL OXIDES COMPOSITES Dimitrije PETROVIC, Marija EGERIC, Radojka VUJASIN, Yi-nan WU, Fengting LI, Ljiljana MATOVIC, Aleksandar DEVEČERSKI	451
19. EKSPERIMENTALNA OPTIČKA ANALIZA OTPORNOSTI NA LOM NERĐAJUĆEG ČELIKA EXPERIMENTAL OPTICAL ANALYSIS OF STAINLESS STEEL FRACTURE BEHAVIOUR Katarina COLIC	461
20. OPTIMIZOVANI PRORAČUN ČELIČNIH HALA NA DEJSTVO POŽARA OPTIMIZED FIRE DESIGN FOR STEEL PORTA-FRAMED SHEDS Filip LJUBINKOVIĆ, Luís LAÍM, Aldina SANTIAGO	469
21. HIDROFOBIZACIJA KALCITA STEARINSKOM KISELINOM MOKRIM POSTUPKOM HYDROPHOBIZATION OF CALCITE BY WET METHOD USING STEARIC ACID Slavica MIHAJLOVIC, Nataša DJORDJEVIC, Vladan KASIC, Srdjan MATIJASEVIC.....	479
22. INDEX ZA PROCENU STRUKTURALNE EFIKASNOSTI ČELIČNIH RAMOVA INDEX FOR THE ASSESSMENT OF STRUCTURAL EFFICIENCY OF STEEL PORTAL FRAMES Filip LJUBINKOVIC, Luís Simões da SILVA	485
23. RAZVOJ APARATURE ZA IN SITU ISPITIVANJE ANKERA NOSACA SOLARNIH PANELE DEVELOPMENT OF THE APPARATUS FOR IN SITU TESTING OF SOLAR PANEL RACKING ANCHORS Gordana BROČETA, Aleksandar SAVIC, Milica VLAHOVIC, Sanja MARTINOVIC, Tatjana VOLKOV HUSOVIC.....	495
24. POVEĆANJE EFIKASNOSTI DOBIJANJA BIOGASA I NJEGOVOG KORIŠĆENJA U POSTROJENJU ZA TRETMAN KOMUNALNIH OTPADNIH VODA INCREASING THE EFFICIENCY OF BIOGAS PRODUCING AND ITS UTILIZATION IN THE MUNICIPAL WASTEWATER TREATMENT PLANT Darja ZARKOVIC, Milica VLAHOVIC, Bilyana ISZITY	503
25. ISPITIVANJE MORFOLOGIJE SUMPOR-POLIMERNOG KOMPOZITA MORPHOLOGY INVESTIGATION OF SULFUR-POLYMER COMPOSITE Milica VLAHOVIC, Kong FAH TEE, Aleksandar SAVIC, Nataša DJORDJEVIC, Slavica MIHAJLOVIC, Tatjana VOLKOV HUSOVIC, Nenad VUSOVIC	513
26. PRIMENA VARENJA, TVRDOG I MEKOG LEMLJENJA U IZRADI SOLARNIH SISTEMA APPLICATION OF WELDING, BRAZING AND SOLDERING IN SOLAR SYSTEMS MANUFACTURING Zoran KARASTOJKOVIC, Milesa SRECKOVIC, Misa STEVIC	521
27. ŠTETNI EFEKTI LEGURA ZA LEMLJENJE IZ ŠTAMPANIH KOLA PRILIKOM ZAJEDNIČKOG TOPLJENJA SA GVOZDENIM I ČELIČNIM DELOVIMA HARMFULL EFFECTS OF SOLDERING ALLOYS FROM PRINTED CIRCUITS WHEN MELTED TOGETHER WITH IRON&STEEL COMPONENTS Zoran KARASTOJKOVIC, Ognjen RISTIC, Misa STEVIC	529

HIDROFOBIZACIJA KALCITA STEARINSKOM KISELINOM MOKRIM POSTUPKOM

HYDROPHOBIZATION OF CALCITE BY WET METHOD USING STEARIC ACID

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Apstrakt

U ovom radu su prikazani rezultati modifikovanja kalcita stearinskom kiselinom mokrim postupkom. U eksperimentima je kao polazni materijal korišćen prirodni krečnjak sa visokim sadržajem kalcita (većim od 95 %). Površina kalcita je modifikovana različitim količinama stearinske kiseline - 1, 2, 3 i 4 %. Proizvodi su okarakterisani termičkom analizom (DTA/TGA), polarizacionom mikroskopom, IR analizom i „floating“ testom. Rezultati su pokazali da se hidrofobnost površine kalcita povećava sa povećanjem količine stearinske kiseline na površini kalcita i da je hidrofobnost dostigla preko 99 % kada je koncentracija stearinske kiseline bila 2 %. Termička analiza je pokazala da se pri početnim koncentracijama stearinske kiseline od 2 % molekuli surfaktanta hemisorbuju na površini kalcita.

Ključne reči: kalcit, stearinska kiselina, mokri postupak, hidrofobnost.

Abstract

In this paper, the results of the modification of calcite with stearic acid using the wet method are presented. In the experiments, natural limestone with a high content of calcite (greater than 95 %) was used as the starting material. The calcite surface was modified with different amounts of stearic acid - 1, 2, 3 and 4 %. The products were characterized by thermal analysis (DTA/TGA), polarization microscopy, IR analysis and by the evaluation of a floating test. The results of the floating test showed that hydrophobicity of the calcite surface increased with increasing the amount of stearic acid on the calcite surface and that the hydrophobicity reached over 99 % when the concentration of stearic acid was 2 %. Thermal analysis showed that at initial concentrations of stearic acid of 2 %, surfactant molecules are chemisorbed at calcite surface.

Key words: calcite, stearic acid, wet method, hydrophobicity.

1 Introduction

By modification of the calcite surface with surfactants, the limestone employed for PVC production obtains properties that enable the manipulation in the production process, due to the fact that it does not adsorb humidity, and, thus improves blending with PVC. As a result of this process homogeneous mixture is obtained [1].

From this point of view, investigation of the phenomena and mechanism of surface modification of calcite, in order to determine the optimal parameters for obtaining surface-modified calcite, represents the subject of interest of much research [2-5]. The product of the reaction between stearic acid and the calcite surface is a monolayer of hydrophobic organic molecules, which has a strong effect on the final characteristics of the composite because it represents an interphase between the two phases of heterogeneous materials [1].

It is generally accepted that carboxylic acids or their salts react with calcium carbonate to produce a layer of the corresponding calcium salt (calcium stearate in the case of stearic acid) at the mineral surface. Papirer and Fekete found that only one molecule of stearic acid is associated with a Ca^{2+} ion, indicating that the alkyl chains in the monolayer are vertically oriented to the surface of the calcite mineral [6, 7]. The vertical orientation of alkyl chains was confirmed by X-ray analysis. They also showed that applying of stearic acid in concentrations higher than necessary to obtain a monolayer chemisorbed at the calcite surface leads to the formation of multi-layers of physisorbed stearic acid at the mineral surface. It is well known that thermogravimetric analysis (TGA) could be used to confirm the presence of an organic phase at the calcite surface, although it is not used for determining the amount of acid required for the optimal cover of the calcite mineral surface.

Commonly used calcite coating techniques, on a laboratory scale, include dry coating and the wet method. This paper reports the results of investigating the feasibility of the calcite surface modification with stearic acid by the wet method.

2 Experimental procedure

Materials

The natural limestone used in this work was obtained from “Banjakomerc”, Serbia. This product is used as filler in the production of PVC elements and pipelines. Stearic acid, a fatty acid, was used as the surfactant for the modification of the calcite surface. Chloroform was used as the organic solvent for stearic acid dissolution.

Modification of the calcite surface

The modification of the calcite was performed using the wet method according to the modified procedure published by Rezaei Gomari [3]: a 10 % aqueous suspension of limestone was treated with an appropriate amount of stearic acid dissolved in chloroform. The concentrations of stearic acid used for coating of the calcite surface were 1, 2, 3, and 4 %. Each suspension was stirred at 4000 rpm, for 15 min, at 50 °C. After the reaction, the suspensions were centrifuged at 10000 rpm for 10 min, washed with distilled water and the obtained products were dried at 60 °C.

Characterization methods

a) Particle size. The granulometric composition of the starting material was determined using a COULTER MULTISIZER. A dilute suspension of the sample in an electrolyte (1 % NaCl) was prepared. Before the granulometric analysis, the sample was dispersed by intensive mixing in a lab mixer for 15 min.

b) Chemical analysis. AAS PERKIN ELMER 703 was used for the determination of the chemical composition of the starting limestone.

c) Microscopic analysis. For identification of the minerals present in starting limestone, qualitative mineral analysis was performed on a polarized light microscope by the immersion method (immersion in xylene for starting limestone, and in water for surface-modified calcite).

d) Determination of the active ratio of the modified calcites. The optimal amount of stearic acid required for the calcite surface modification was determined by the floating test [8] which represents the ratio of the floated product to the overall weight of the sample after mixing in water and vigorous stirring. This ratio, called active is expressed as:

$$I_0 = M_p / (M_p + M_t) \times 100$$

where I_0 is an active ratio (%), M_p is the mass of the floated product and M_t is the mass of the non-floated product.

d) Thermal analysis. Thermal analysis was performed using a NETZESCH 409 EP instrument. The starting calcite as well as surface modified sample were tested in the temperature range from 20 to 1000 °C, at a heating rate of 10 °C/min, under an air atmosphere.

e) IR spectroscopy. The IR spectra were recorded on a HEWLETT PACKARD instrument using the KBr pellet technique, by mixing 1 % of the sample with 99 % of KBr. The samples were investigated in the wavenumber range of 4000 – 2500 cm⁻¹.

3 Results and discussion

The granulometric composition of the starting limestone is given in Table 1. The upper size of the starting limestone was $\approx 10 \mu\text{m}$ and the average diameter of $D_{50} \approx 5 \mu\text{m}$.

Table 1. Granulometric composition of starting limestone

Size range, μm	M, %
-18 + 15	0.80
-15 + 10	5.20
-10 + 8	15.80
-8 + 6	31.30
-6 + 5	31.60
-5 + 3	10.87
-3 + 0	4.33
Total	100.00

The chemical composition of the starting limestone (“Banjakomerc”) was: CaO - 55.07 %, SiO₂ - 0.57 %, MgO - 0.62 %, Al₂O₃ - 0.038 %, Fe₂O₃ - 0.015 % and loss on ignition - 43.55 %. Based on the amount of CaO, the calculated amount of CaCO₃ was 98.29 %.

Mineralogical composition of the investigated limestone was: calcite, opal, wollastonite, getitlimonite, apatite, rutile, and vesuvianite. The sample was mainly in crystal forms (an exception was opal). The main component of the starting sample was calcite (over 95 %), then opal and limonitegetit, which could be observed free or as a cream on the calcite surface, wollastonite and vesuvianite, together with apatite and rutile, were present in traces.

In order to investigate the effect of the stearic acid concentration at the calcite surface on the hydrophobicity of the obtained products, the floating test was performed and the active ratio of each product was determined. The results are presented in Figure 1.

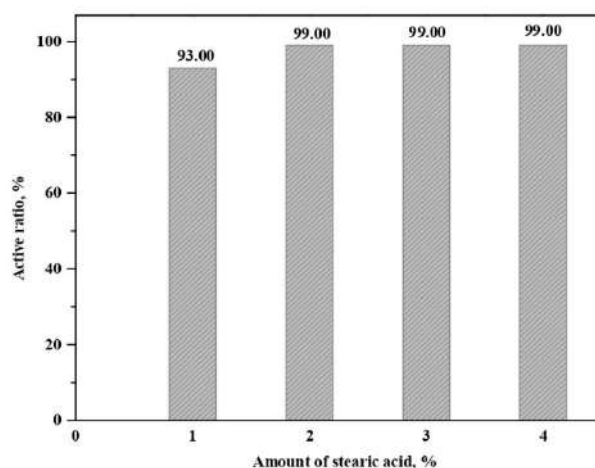


Figure 1. The effect the stearic acid concentration on the active ratio of obtained products

The results presented in Figure 1, showed that with 2 % stearic acid, the active ratio of 99.90 % was achieved and it remained constant with further increase of the stearic acid amount. Hence, 2 % stearic acid was sufficient to obtain a completely hydrophobic calcite surface. Due to this fact, the sample was further characterized by thermal analysis and polarization microscopy.

The thermal (DTA and TG) curves of the starting calcite and the calcite modified with 2 % stearic acid are shown in Figure 2.

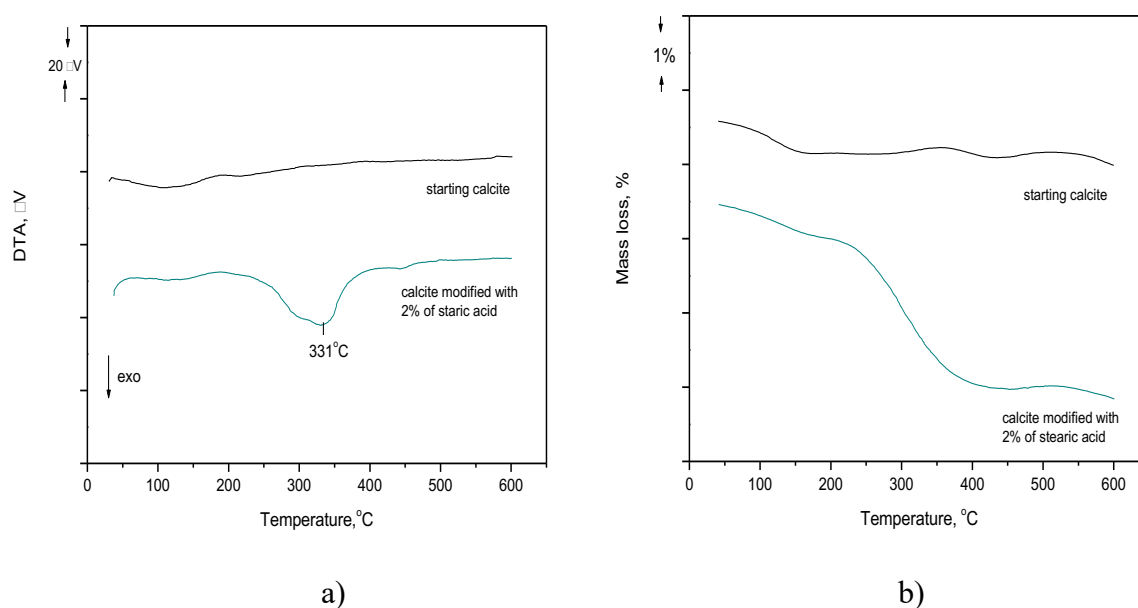


Figure 2. Thermal: DTA (a) and TG curves (b) of the starting calcite and calcite modified with 2 % stearic acid

According to the DTA curves in Figure 2a, mass losses in two temperature intervals were observed: the first in the range from 20 to 200 °C and the second from 200 to 400 °C. The mass loss in the first temperature range is assigned to the desorption of weakly bonded water, which was found in both the starting and the modified samples. In the second temperature interval, oxidation of the organic component was detected, which was followed by an exothermic peak on the DTA diagram. Since the starting sample did not contain an organic phase, an exothermic peak was not found on the DTA curve. On the contrary, on the DTA curve of the sample modified with 2 % stearic acid, an exothermic maximum at 331 °C was detected as a result of oxidation of the organic substance present at the calcite surface. The appearance of an exothermic peak in a temperature range of 200–400 °C, and the absence of peaks on the DTA curve of calcite modified with 2 % stearic acid in the first temperature range, indicated that organic substance was chemisorbed at the calcite surface.

The combustion of the organic component was determined by a mass loss. As seen from the TG curves in Figure 2b, the starting sample exhibited insignificant mass loss because it did not contain an organic component. Furthermore, the highest mass loss was found above 200 °C for the sample modified with 2 % stearic acid, due to the oxidation of the organic component, while the mass loss from 0 to 200 °C resulted in a loss of moisture. The mass loss for the starting calcite and calcite modified with 2 % stearic acid is presented in Table 2.

Table 2. Mass loss from TG curves for starting calcite and calcite modified with 2 % stearic acid

Concentration of stearic acid, %	Mass loss, %			
	0-200 °C	200-400 °C	400-600 °C	20-600 °C
-	0.40	0.07	0.12	0.59
2	0.50	1.94	0.21	2.65

Microphotographs of the starting calcite and calcite modified with 2 % stearic acid are presented in Figure 3.

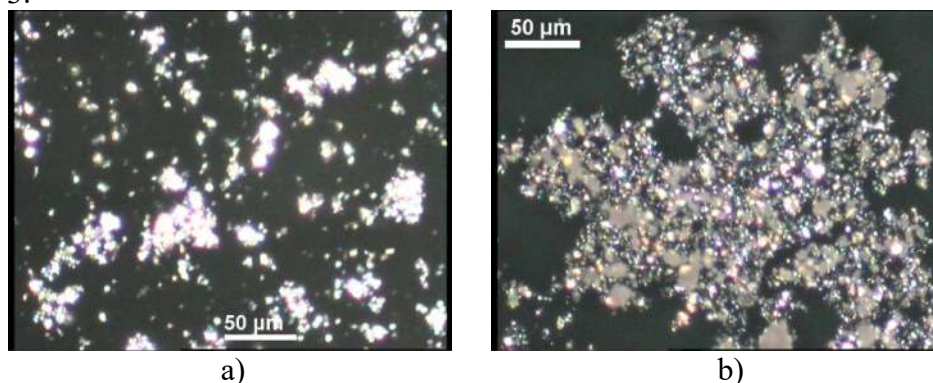


Figure 3. Microphotographs of starting calcite (a), calcite modified with 2 % stearic acid (b)

Compared to the starting sample (Figure 3a), the microphotograph of modified calcite (Figure 3b) indicates that in the presence of water as an immersion liquid, particles of coated calcite tend to concentrate, and thus agglomerates are clearly visible. These results may be another evidence that even with 2 % stearic acid high hydrophobicity of the coated product was achieved.

The starting calcite as well as calcite modified with 3 % stearic acid were additionally characterized by IR spectroscopy. The obtained IR spectra are shown in Figure 4.

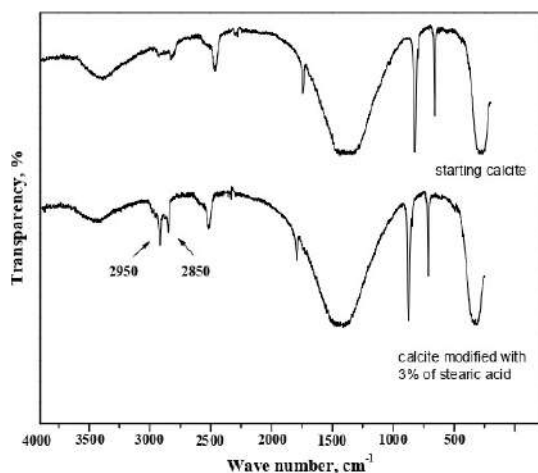


Figure 4. IR spectra of the starting calcite and calcite modified with 3 % stearic acid

According to Figure 4, two intensive bands at 2950 cm^{-1} and 2850 cm^{-1} observed for the calcite modified with 3 % stearic acid, represent asymmetric and symmetric stretching vibrations of

the C-CH₂ bonds of stearic acid. As expected, these peaks are not present in the IR spectra of the started calcite, since it does not have an organic phase.

4 Conclusions

The results presented in this paper show that the hydrophobic product is obtained by modification of the calcite surface with stearic acid using the wet method. The floating test results indicated that 2 % stearic acid was sufficient to obtain the satisfactory hydrophobicity of the product. Thermal analysis showed that at initial concentrations of stearic acid of 2 %, surfactant molecules are chemisorbed at the calcite surface. The hydrophobic material obtained by simple modification can find important applications as a filler in the polymer industry.

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