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UNIVERSITY OF BANJA LUKA
ТЕХНОЛОШКИ ФАКУЛТЕТ
FACULTY OF TECHNOLOGY



MEĐUNARODNI NAUČNI SKUP

XII SAVJETOVANJE

HEMIČARA, TEHNOLOGA I EKOLOGA
REPUBLIKE SRPSKE

ZBORNİK RADOVA

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XII CONFERENCE

OF CHEMISTS, TECHNOLOGISTS
AND ENVIRONMENTALISTS OF
REPUBLIC OF SRPSKA

PROCEEDINGS

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ПОДРУЧНА ПРИВРЕДНА
КОМОРА БАЊА ЛУКА**

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CONTENTS

THEORETICAL AND APPLIED CHEMISTRY	11
PRIMJENA ORGANSKIH AZOTNIH LIGANADA U KONSTRUKCIJI UGLJIKOVIH KVATERNIH CENTARA	12
Jasmin Suljagić	
IDENTIFIKACIJA ORGANSKE SUPSTANCE I ANALIZA SPECIFIČNIH BIOMARKERA U UZORCIMA RIJEKE VRBAS METODOM GC- MS	19
Nemanja Koljančić, Ivan Samelak, Mališa Antić, Branimir Jovančićević, Milica Balaban	
SINTEZA, KARAKTERIZACIJA I ANTIMIKROBNI SCREENING Cu(II) KOMPLEKSA SA MAKROLIDNIM ANTIBIOTIKOM AZITROMICINOM.....	27
Horozić Emir, Cipurković Amira, Ademović Zahida, Hodžić Snježana, Husejnagić Darja, Kolarević Lamija, Bjelošević Demir, Zukić Amila	
KOMPLEKSI BIOGENIH METALA SA BETA-LAKTAMSKIM ANTIBIOTIKOM AMOKSICILINOM: SINTEZA, KARAKTERIZACIJA I <i>IN VITRO</i> ANTIMIKROBNA AKTIVNOST.....	34
Horozić Emir, Cipurković Amira, Ademović Zahida, Zukić Amila, Hodžić Snježana, Husejnagić Darja, Bjelošević Demir, Kolarević Lamija, Ibišević Merima	
ANALIZA BILJNOG POLIMERA KUTINA	42
Snežana Kravić, Zorica Stojanović, Ana Đurović	
STUDIJA INTERAKCIJE M(II) I M(III) IONA SA (7R)-7-((R)-2-AMINO-2-FENILACETAMIDO)-3-METIL-8-OKSO-5-TIA-1-AZABICIKLO[4.2.0]OKT-2-EN-2-KARBOKSILNOM KISELINOM PRIMJENOM FTIR SPEKTROSKOPIJE I STEREO-MIKROSKOPIJE	54
Marić Snježana, Horozić Emir, Cipurković Amira, Bajrić Ermin, Salkić Alma, Ademović Zahida, Ibišević Merima	
TESTIRANJE UČESNIKA U SAOBRAĆAJU NA PRISUSTVO DROGA	59
Mirjana Dragoljić, Vesna Matić, Ljiljana Simurdić, Goran Šmitran	
ISPITIVANJE SORPCIJE RADIOAKTIVNOG JODA (¹³¹I) NA AMINO-FUNKCIONALIZOVANOM MAKROPOROZNOM KOPOLIMERU poli(GMA-co-EGDMA)	68
Aljoša Stanković, Zvezdana Sandić, Ljiljana Suručić	
KVANTNO-HEMIJSKO MODELOVANJE SORPCIJE Cu(II), Cd(II), Co(II) i Ni(II) JONA NA AMINO-FUNKCIONALIZOVANOM MAKROPOROZNOM KOPOLIMERU poly(GMA-co-EGDMA)	76
Ljiljana Suručić, Aleksandra Rakić, Aleksandra Nastasović, Goran Janjić	
SYNTHESIS OF SILVER NANOPARTICLES BY USING PHENYLHYDRAZINE, TRINATRIUM CITRATE, ASCORBIC ACID AND THEIR CHARACTERIZATION	84
Tanja Okolić, Savka Janković, Dijana Jelić	
STRUCTURE, ISOLATION AND APPLICATION OF PLANT POLYMER CUTINE	90
Danijela Rajić, Dragan Tošković, Vesna Gojković, Dario Balaban	
PRAĆENJE ADSORPCIJE METIL VIOLETA NA PRIRODNIM I SINTETIČKIM ALUMOSILIKATIMA	103
J. Penavin Škundrić, Z. Levi, S. Sladojević, R. Petrović, D. Bodroža	
KINETICS OF CERIUM NITRATE THERMAL DECOMPOSITION IN VARIOUS ATMOSPHERES.....	111
Saša Zeljković, Milica Balaban, Tanja Okolić, Dijana Jelić	
BIOLOŠKI AKTIVNI CELULOZNI ZAVOJ SA DIKLOFENAKOM.....	117
Pero Sailović, Branka Rodić Grabovac, Marko Koprena, Mlinarević Vladimir	
CHEMICAL ENGINEERING	124
UTICAJ UDELA TVRDIH SEGMENTA NA TOPLOTNA SVOJSTVA ALIFATIČNIH POLIURETANSKIH ELASTOMERA.....	125
Dejan Kojić, Jelena Pavličević, Milena Špírková, Ayse Aroguz, Mirjana Jovičić, Bojana Ikonić, Jaroslava Budinski-Simendić	
PROIZVODNA PRAKSA I TERMODINAMIČKI PROBLEMI U POGONU ZA OBLIKOVANJE PLASTIKE	132
Vladan Mičić, Ljiljana Tanasić, Mitar Perušić, Dejan Kojić, Jelena Pavličević, Branko Pejović, Nevena Vukić	
UTICAJ VREMENA MLEVENJA KOD HIDRODESTILACIJE PLODA KLEKE NA HEMIJSKI SASTAV ETARSKOG ULJA	140
Vladimir Pavićević, Marko Radović, Svetomir Milojević, Miljana Marković, Marina Stamenović	

SYNTHESIS OF CONDUCTIVE BIOBASE POLYMERIC NANOCOMPOSITES	150
Jelena Tanasić, Hasan Pala, Jaroslava Budinski-Simendić, Branka Pilić, Danica Piper, Suzana Cakić, Ivan Ristić	
ZINC REMOVAL FROM BAYER LIQUOR BY USING ALUMINIUM HYDROXIDE WITH SPECIFIC STRUCTURAL PROPERTIES AS CRYSTALLIZATION AGENT.....	155
Đurđa Oljača, Biljana Milovanović, Stefan Pavlović, Radenko Smiljanić, Zoran Obrenović, Radislav Filipović	
KVALITET REZULTATA LABORATORIJSKIH ISPITIVANJA KOD ODREĐIVANJA SASTAVA TEČNOG NAFTNOG GASA METODOM GASNE HROMATOGRAFIJE	160
Mara Jeremić , Tatjana Botić, Pero Dugić, Aleksandra Šinik	
НОВА КОРЕЛАЦИЈА ЕКСЕРГИЈЕ И МАКСИМАЛНОГ ЗАПРЕМИНСКОГ РАДА СА ПРИМИЈЕЊЕНОМ ТЕРМОДИНАМИЧКОМ АНАЛИЗОМ	170
Бранко Пејовић, Митар Перушић, Владан Мићић, Душко Костић, Владимир Дамјановић, Зоран Обреновић	
DETERMINATION OF DEPOSITION RATE OF THE SELECTED METAL COATING BY AUTOCATALYTIC PROCESS	181
Borislav N. Malinović, Srđan Kušljic, Tijana Đuričić, Dajana Dragić	
ISPITIVANJE EFIKASNOSTI INHIBITORA KOROZIJE BAKRA U SIMULIRANOJ RASHLADNOJ VODI.....	189
D. Zorić, B.N. Malinović, T. Đuričić, S. Vranješ	
ELECTRICAL CONDUCTIVITY OF COMPOSITES WITH BIODEGRADABLE MATRICES FILLED WITH GALVANOSTATIC COPPER POWDER.....	194
Zoran Janković, Miroslav M. Pavlović, Marijana R. Pantović Pavlović, Nebojša D. Nikolić, Borislav Malinović, Miomir G. Pavlović	
CHEMICAL TECHNOLOGY.....	203
EFFECT OF SILICA ON THE PROPERTIES OF ELASTOMERIC MATERIALS BASED ON NR/BR/SBR TERNARY RUBBER BLEND	204
Slaviša Jovanović, Vojislav Jovanović, Gordana Marković, Milena Marinović-Cincović, Nevena Vukić, Suzana Samaržija-Jovanović, Jaroslava Budinski-Simendić	
UTICAJ DODATKA PIGMENTA NA SVOJSTVA UMREŽENIH FILMOVA PREMAZA NA OSNOVU SMEŠE ALKIDNE I MELAMINSKE SMOLE.....	211
Mirjana Jovičić, Radojka Milovanović, Jelena Pavličević, Dragan Govedarica, Oskar Bera, Vesna Teofilović, Dejan Kojić	
THERMODYNAMIC MODELLING OF THE PHASE DIAGRAMS FOR Al-Cu15-Mg1-Ti ALLOYS	217
Biljana Zlatičanin, Sandra Kovačević	
THE EFFECTS OF TITANIUM AND BORON CONTENTS ON THE STRUCTURE OF THE Al-Cu15-Mg5 ALLOYS...	225
Biljana Zlatičanin, Sandra Kovačević	
PONAŠANJE CINKA U BAYER - OVOM PROCESU PROIZVODNJE GLINICE	230
Dragica Lazić, Dragana Kešelj, Dragana Blagojević, Dijana Drljača, Radislav Filipović	
UTICAJ KVALITETA SIROVE VODE NA TEHNOLOŠKI PROCES PRIPREME NAPOJNE VODE	238
Duško Đukić, Ljiljana Vukić, Dijana Drljača	
THE SYNTHESIS OF OIL BASED ANTIFOAMING AGENTS.....	252
Vedrana Prorok, Tamara Erceg, Nevena Vukić, Darko Manjenčić, Ivan Ristić	
SYNTHESIS OF POLYURETHANE ELASTOMERIC NANOCOMPOSITES	255
Darko Manjenčić, Ivan Krakovsky, Suzana Cakić, Tanja Radusin, Ivan Ristić	
UTICAJ PRIRODE PROCESNOG ULJA NA SVOJSTVA GUME.....	260
Dragan Govedarica, Novica Sovtić, Oskar Bera, Predrag Kojić, Olga Govedarica, Jelena Pavličević, Mirjana Jovičić	
UTICAJ DODATKA ZnO NA SVOJSTVA UMREŽENIH FILMOVA ALKID/HEKSAMETOKSIMETIL MELAMINSKIH PREMAZA	266
Vedrana Prorok, Sanja Rackov, Tamara Erceg, Branka Pilić, Mirjana Jovičić	
SELEKCIJA TEHNOLOGIJE OBRADJE S OBZIROM NA OPTEREĆENJE OTPADNIH VODA.....	272
Osman Perviz, Samira Hotić	

NOVA GENERACIJA BIORAZGRADIVIH HIDRAULIČNIH FLUIDA FORMULISANIH NA BAZI SINTETSKIH ESTARA.....	279
Marica Dugić, Branko Despotović, Novak Damjanović, Pero Dugić	
MIKROMEHANIČKA ANALIZA STAKLO-POLIESTER KOMPOZITNOG MATERIJALA (±45°) NAKON UDARNOG OPTEREĆENJA.....	288
Aleksandra Jelić, Vanja Mališić, Vladimir Pavićević, Marina Stamenović, Slaviša Putić	
BIOTECHNOLOGY.....	295
UTICAJ KOREKCIJE NEKIH PARAMETARA NA FERMENTACIJU MEDOVINE UZ PRIMJENU GOMPERTZ MODELA.....	296
Zvezdana Kisin, Saša Papuga, Ana Velemir, Aleksandar Savić	
ПОРЕЂЕЊЕ КВАЛИТЕТА ВОЋНИХ РАКИЈА ОД ШЉИВА И ЈАБУКА ДОБИЈЕНИХ СПОНТАНИМ ФЕРМЕНТАЦИЈАМА У ЦИЉУ ЊИХОВЕ СТАНДАРДИЗАЦИЈЕ.....	305
Слободанка Михајловић, Ана Велемир, Александар Савић	
EFFECT OF DIFFERENT REACTION PARAMETERS ON LIPASE-CATALYZED ESTERIFICATION OF NARINGIN AND ESCULIN	312
Ana Milivojević, Marija Ćorović, Milica Carević, Katarina Banjanac, Dejan Bezbradica	
UTICAJ NAČINA EKSTRAKCIJE, VRSTE OTAPALA I NAČINA PREDOBRADE BILJNOG MATERIJALA BILJKE <i>MELISSA OFFICINALIS</i> L. NA ANTIMIKROBNU AKTIVNOST	319
Azra Bakrač, Muamer Hadžić, Halid Makić, Aida Džaferović, Vildana Jogić, Subha Džafić	
FERMENTATION OF BUCKWHEAT HONEY WITH THE ADDITION OF CHOCOLATE PRODUCT AFTER EIGHT	328
Nikolina Marković, Ana Velemir, Aleksandar Savić	
FOOD TECHNOLOGY.....	340
DOBIJANJE DESTILATA ŠLJIVE SORTE POŽEGAČE	341
Miljojka Mijailović, Mladena Jakšić, Svetomir Milojević, Miljana Marković	
UTICAJ RAŽEVOG KISELOG TIJESTA FERMENTISANOG SA <i>LACTOBACILLUS PLANTARUM</i> NA SVOJSTVA HLJEBA	349
Amel Selimović, Dijana Milićević, Amra Selimović, Ramzija Cvrk, Tijana Brčina, Halid Junuzović, Huriya Alibašić, Amela Jašić	
FIZIČKOHEMIJSKE OSOBINE ACIDOFILNOG MLJEKA UZ DODATAK RAZLIČITIH PROTEINSKIH SUPLEMENATA	359
Milka Stijepić, Jovana Glušac, Dragica Đurđević-Milošević, Vesna Kalaba	
ANALIZA PILEĆEG MESA SA RAZLIČITIM TRETANIMA ISHRANE	369
Džaferović Aida, Ekrem Pehlić, Samira Dedić, Kemal Salkić	
ESTIMATION OF QUALITY PARAMETERS OF FERMENTED MILK PRODUCTS DURING STORAGE.....	376
Suzana Jahić, Aida Džaferović, Amra Musić	
UTICAJ POSTUPAKA ODMRZAVANJA NA PROMJENU BOJE SVINJSKOG MESA (<i>M. Longissimus dorsi</i>).....	384
Danica Savanović, Radoslav Grujić, Jovo Savanović	
APPLICATION OF PULSED LIGHT FOR DECONTAMINATION OF FOOD PACKAGING MATERIALS.....	393
Svetlana Pelemiš, Vesna Gojković, Danica Savanović, Radoslav Grujić	
GLIADIN PROTEINS EXTRACTION FROM WHEAT FLOUR WITH ETHANOL – DETERMINATION OF THE OPTIMUM SOLVENT CONCENTRATION	398
Vesna Gojković Cvjetković, Radoslav Grujić, Danica Savanović	
GLIADIN CHROMATOGRAPHIC SEPARATION FROM WHEAT FLOUR EXTRACT – DETERMINATION OF OPTIMUM COLUMN TEMPERATURE.....	406
Vesna Gojković Cvjetković, Željka Marjanović-Balaban, Danijela Rajić, Svetlana Pelemiš	
HEMIJSKI SASTAV I ANTIOKSIDATIVNA AKTIVNOST ULJA DOBIJENIH IZ SJEMENKI	
<i>Rubus idaeus</i> L. i <i>Rubus fruticosus</i> L.	414
Staniša Latinović, Nataša Ivković, Senad Krivdić, Ladislav Vasilišin, Goran Vučić, Zoran Kukrić	
UTICAJ NAČINA EKSTRAKCIJE, VRSTE OTAPALA I NAČINA PREDOBRADE UZORKA NA ANTIOKSIDATIVNU AKTIVNOST BILJKE <i>SEMPERVIVUM TECTORUM</i>	421

Halid Makić, Husejin Keran, Azra Bakrač, Samira Dedić, Kemal Salkić, Adijana Jaganjac-Mehadžić,
Almedina Komić

QUALITY CONTROL AND FOOD SAFETY	429
KVALITETA MEDA NA TRŽIŠTU BOSNE I HERCEGOVINE	430
Džemil Hajrić, Dragan Brenjo, Katica Arar, Drago Sando, Nijaz Bajramović	
SIMULTANO ODREĐIVANJE SADRŽAJA JONSKIH VRSTA U LEKOVITIM I AROMATIČNIM ČAJEVIMA	439
Nikola Filipović, Jelena Božović, Antonije Onjia, Dragana Z. Živojinović	
ANALIZA OMEGA-3 MASNIH KISELINA U DIJETETSKIM SUPLEMENTIMA NA BAZI RIBLJEG ULJA	450
Snežana Kravić, Zorica Stojanović, Ana Đurović, Zvonimir Suturović, Tanja Brezo	
EFIKASNOST SISTEMA UPRAVLJANJA BEZBJEDNOŠĆU HRANE U PROIZVODNJI PEKARSKIH PROIZVODA	458
Brane Novaković, Radoslav Grujić	
POTVRĐIVANJE PRISUSTVA AFLATOKSINA M1 U SIROVOM MLIJEKU METODOM NA LC-MS/MS.....	466
Biljana Pećanac, Jelena Aničić, Milijana Golić, Slobodan Dojčinović, Željko Sladojević	
ANTIOKSIDATIVNA AKTIVNOST KOMERCIJALNE KAFE SA TRŽIŠTA BOSNE I HERCEGOVINE	475
Božana Odžaković, Natalija Džinić, Zoran Kukrić, Slavica Grujić	
MULTIELEMENTNA ANALIZA CRNOG, ZELENOG, BILJNIH I VOĆNIH ČAJEVA PRIMENOMICP-OES METODE.....	483
Jelena Božović, Nikola Filiović, Antonije Onjia, Dragana Z. Živojinović	
TEXTILE TECHNOLOGY	494
ANTIMIKROBNA AKTIVNOST BIOMEDICINSKIH TEKSTILNIH MATERIJALA OBRADENIH ETARSKIM ULJIMA SIBIRSKJE JELE (<i>Abies Sibirica</i>)	495
Ljiljana Sretković, Adela Medović	
KEY FACTOR INTERACTIONS DURING EXTRUSION INDUCING FIBRILLATED POLYPROPYLENE FOIL STRIPS FINENESS.....	504
Nina Đapić	
UTICAJ NESELEKTIVNE OKSIDACIJE VODONIK-PEROKSIDOM NA SORPCIONA SVOJSTVA VLAKANA KONOPLJE.....	513
Jovana Ž. Milanović, Matea Korica, Mirjana M. Kostić	
CORPORATE SOCIAL RESPONSIBILITY OF COMPANIES INDUSTRY TEXTILE AND CLOTHES IN THE REPUBLIC OF SERBIA	520
Snežana Urošević, Milovan Vuković, Gordana Kokeza, Nada Štrbac	
UTICAJ STRATEGIJSKOG PLANIRANJA NA EFIKASNOST I EFEKTIVNOST PREDUZEĆA U TEKSTILNOJ INDUSTRIJI.....	529
Miloš Sorak, Miroslav Dragić	
UTICAJ PROCESA BOJENJA EKSTRAKTIMA BILJKE <i>Achillea millefolium L.</i> NA SORPCIONA SVOJSTVA PAMUČNIH PLETENINA	539
Jovana Ž. Milanović, Predrag M. Milanović, Snežana B. Stanković, Dragana R. Grujić	
PRIMARNI POSTUPAK OBRADJE SIROVIH KOŽA	549
Danijela Kovačević, Marina Stamenović, Vladimir Pavićević	
BIO-INSPIRISANI TEKSTILNI MATERIJALI SPECIJALNIH SVOJSTAVA	553
Branislava B. Lazić, Biljana B. Popović, Milan M. Gligorićević	
PRIMENE PAMETNOG TEKSTILA.....	569
Branislava B. Lazić, Biljana B. Popović, Snežana Poznanović	
UTICAJ NESELEKTIVNIH OKSIDACIONIH SREDSTAVA NA SADRŽAJ UVEDENIH FUNKCIONALNIH GRUPA I SORPCIONA SVOJSTVA VLAKANA KONOPLJE	585
Jovana Z. Milanovic, Matea Korica, Mirjana M. Kostic	
GRAPHIC TECHNOLOGY AND DESIGN.....	593
ANALIZA DIGITALNE ŠTAMPE I TRENDVI U NJENOM RAZVOJU.....	594

Nemanja Kašiković, Mladen Stančić, Dragoljub Novaković, Gojko Vladić, Saša Petrović

PREGLED MOGUĆNOSTI PRIMENE TEHNOLOGIJA ADITIVNE PROIZVODNJE ZA IZDRADU ALATA U GRAFIČKOJ INDUSTRIJI.....602

Gojko Vladić, Bojan Banjanin, Nemanja Kašiković, Gordana Delić, Magdolna Pal

QUALITY ANALYSIS OF PAD PRINTED LINE ELEMENTS IN DEPENDENCE OF DOCTOR BLADE TRAJECTORY611

Sandra Dedijer, Magdolna Pál, Živko Pavlović, Ivana Tomić, Gojko Vladić

UTICAJ PARAMETARA ŠTAMPE NA SPOSOBNOST ZADRŽAVANJA VODE ŠTAMPANIH PLETENINA.....617

Mladen Stančić, Đorđe Vujčić, Branka Ružičić, Dragana Grujić

MATERIJALI NA OSNOVU POLILAKTIDA ZA PRIMENU U AMBALAŽNOJ INDUSTRIJI: AKCENAT NA ŠTAMPARSKIM SVOJSTVIMA.....626

Nevena Vukić, Dejan Kojić, Ljiljana Tanasić, Vesna Teofilović, Tamara Erceg, Borislav Simendić, Ivan Ristić

ENVIRONMENTAL ENGINEERING632

POSSIBILITIES OF THE APPLICATION OF THE INOVATIVE ARCHITECTURAL DECISION ANALYSIS MODEL – ADAM WITHIN THE ENVIRONMENTAL ISSUES633

Miljan Šunjević, Boris Obrovski, Sonja Dmitrašinović, Mirjana Vojinović Miloradov

MULTIDISCIPLINARY CONSIDERATIONS IN THE MANAGEMENT OF EMERGING ENVIRONMENTAL QUALITY ISSUES.....638

Dejan Vasovic, Goran Janackovic, Jelena Malenovic Nikolic, Lidija Milosevic, Zarko Vranjanac

SADRŽAJ METALA OLOVA U VODOTOKU RIJEKE DREŽANKE644

Sejit Bobar

PRIMJER RJEŠAVANJA PROBLEMA BIORAZGRADLJIVOG OTPADA PRIMJENOM KONCEPTA ČISTIJE PROIZVODNJE.....649

Dajana Dragić, Saša Papuga, Alekandar Savić

OTHERS.....656

CAMPANULACEAE FAMILY PRESENCE DURING NEW HOLOCENE PERIOD IN ELBASANI TOWN – MIDDLE ALBANIA657

Dr. Admir Jançe, PhD. Anila Jançe

PALEOPALYNOLOGICAL DISSIPATION OF *RHAMNUS* TYPE, IN ELBASAN CITY – ALBANIA662

Dr. Admir Jançe, PhD. Anila Jançe

DAILY MICROBIAL AIR POLLUTION IN ELBASAN CITY – ALBANIA666

PhD. Anila Jançe, Prof. As. Dr. Valentin Bogoev, Dr. Admir Jançe

MICROBIAL CONTAMINATION CAUSED BY THE PRESENCE OF MOSSES AND BACTERIA IN THE AIR OF ELBASANI TOWN – MIDDLE ALBANIA671

PhD. Anila Jançe, Prof. As. Dr. Valentin Bogoev, Dr. Admir Jançe.

S P O N S O R S.....676

Original scientific article – Originalni naučni rad

EFFECT OF SILICA ON THE PROPERTIES OF ELASTOMERIC MATERIALS BASED ON NR/BR/SBR TERNARY RUBBER BLEND

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Abstract

The effects of pyrogenous silica on the mechanical properties of elastomeric materials based on natural rubber (NR), polybutadiene rubber (PB) and styrene-butadiene rubber (SBR) are reported. For sample preparations the content of network precursor was constant (25:25:50), but the content of filler was varied (0, 40, 60, 80, 100 phr). Curing behavior was assessed using oscillating disc rheometer. Results indicated that the minimum torque and maximum torque increase with increasing filler loading in the compounds, whereas scorch time shows a decreasing trend. Cure time of obtained composites increases with increasing filler loading. Mechanical properties were evaluated before and after thermal ageing (during 72h or 168h at 100°C) of obtained composite materials. Incorporation of silica has improved the tensile modulus. However, elongation at break exhibited a different trend. For tensile strength, optimum values were obtained at 60 phr silica content

Key words: elastomers, mechanical properties, silica, thermal ageing

INTRODUCTION

The use of blends of rubber is widespread, the purpose being to obtain a balance of properties, including cost, which one elastomer alone cannot supply. Synthetic rubbers may, therefore, be added to each other as binary mixtures to improve the inferior properties of one of the components (THONGPINA, 2013). Elastomeric materials based on polyisoprene rubber (NR) have a certain advantages, such as flexibility. Nevertheless, some of its properties fall short in certain applications, such as oil resistance, air permeability, ozone resistance, compression set, and thermal aging resistance. Polybutadiene is classified as general purpose rubbers intended for the manufacture of tires and general mechanical products. However, one type of rubber may not possess all the physical properties desired in a finished product. For example, in tire tread the high abrasion resistance under certain conditions conferred by the use of BR is desirable, but the poor road holding and rib tearing properties are not, hence blends of BR with NR and (SBR) styrene-butadiene rubber are employed. Compatibility is the fundamental property, deciding the practical utility of a polymer blend (Ngudsuntear, 2014). If the two elastomers in a blend are incompatible, it will exist in the form of two separate phases and the cured blend will show inferior properties (Marković, 2015). SBR is widely used as one of the network precursor for automotive tires, wire and cable applications due to its high elongation, but unfortunately it has low elastic modulus and durability and needs some additives such as; antioxidants, accelerators,

softeners and fillers to improve its properties (Camargo, 2009). The phenomenon of reinforcement of elastomers is of great importance for the structuring of materials in new technologies. The nanoparticle addition to the elastomers based on different network leads to an increase in the modulus and to an improvement of key properties such as tensile strength, elongation, as well as abrasion resistance (Nawawi, 2012). The silica surface hydroxyl groups including isolated, vicinal, and geminal silanols play a key role in most of the applicative properties (Samaržija-Jovanović, 2013). The presence of silanol groups on the silica surface induces particle–particle interaction which tends to favor filler agglomeration in the rubber matrix. This highly polar surface makes it poor compatible with most rubbers due to weak rubber–filler interactions. The addition of nanoparticles influences the cross-linking regime as well, especially in some types of rubbers. Reinforcement of elastomers is a particularly complex process if material contains more than one type of network precursor. By creating a multi-phase system, characteristics of individual phases can be partly preserved or significantly changed due to the influence of intermolecular interaction (Marković, 2012). Materials which have a satisfactory thermal stability and mechanical properties required in the specific exploitation conditions are obtained by addition of the optimum content of active fillers. If elastomers are compressed over a long time, it loses its capability to return to its original thickness. The compression set values are expressed as a percentage. The lower the value, the better material resists permanent deformation under a given deflection. In this study the goal was to determine the influence of silica on the properties of sulfur cured elastomeric materials based NR/BR/SBR ternary rubber blends which has been widely used in *rubber* industry, particularly in tire *applications*.

MATERIAL AND METHODS OF WORK

Polyisoprene rubber, NR SMR-20 (0.92 g/cm³) was supplied by Malaysia; polybutadiene rubber, BR SKD N (0.91 g/cm³), with 94% of 1,4 *cis* content, Mooney viscosity MI (1+4) 100°C=44 M – was supplied by Njižnjekamsk (Russia); Styrene butadiene rubber, SBR Europa Intol 1783 (density=0.94g/cm³), is an emulsion styrene-butadiene rubber with 23.5% bound styrene, obtained by cold polymerization extended with 37.5% RAE oil, was supplied by Versalis (Italy). News 175G, Wuxi (China)(ρ = 2.0 g/cm³) was used as nano silica had 22 nm average size of primary particles Content of filler was 0, 20, 40, 60, 80, and 100 phr. The curing system was: N-cyclohexyl-2-benzothiazolsulfonamide *-CBS((1,4 phr); diphenyl guanidine, DPG, (1 phr); N-(cyclohexylthio)phthalimide, CTP 100 (0.2 phr) and sulfur (2 phr). In rubber compounds the network precursor ratio was 25/25/50 (w/w/w). Content of zinc oxide was 3 phr. The stearic acid content was 2 phr. Naphthenic process oil is used throughout a mix of rubber compounds (content 10 phr). These facilitate in rising the dispersion of fillers and flow characteristics of the compound throughout more process All samples are mixed in a laboratory mixer K-0 INTERMIX (Francis Shaw), volume 1 L, and laboratory roll mill (14201-Buzuluk Komarov) at a speed of rollers n1/n2 = 17.4/14 and a temperature between 60-70°C, according to the procedure ASTM D318489. The sheeted rubber compound was conditioned at 23 ± 2° C for 24 h prior to crosslinking behavior assessment at 160 °C using Monsanto Moving Die Rheometer (model 100S). All test specimens were compression molded at 160 °C during the determined optimum curing time (t_{c90}). The scorch time, t_{s2} is the time to 2 units of torque increase above minimum torque. The optimum cure time, t_{c90} is the time to 90% of maximum torque evaluated from the following expression:

$$M_{t90} = (M_h - M_1) \times 0,9 + M_1 \quad (1)$$

where M_h is the maximum torque, and M_{t90} a new torque reading corresponding to 90% cure in the rubber were determined from the cure traces generated at 160 ± 2 °C by oscillating disc

rheometer at an angular displacement of ± 3 and a frequency of 1.7 Hz. The cure rate index (CRI) is the measure of rate of vulcanization based on the difference between optimum cure time of vulcanization t_{c90} and the scorch time t_{s2} . It can be calculated from the relation:

$$\text{CRI} = 1/t_{c90} - t_{s2} \times 100 \quad (2)$$

The sheets were cut and vulcanized in polished molds in a press at 160 C and after that were cut into dumbbell-shaped specimens (five replicates from each sample) for the assessment of mechanical properties using an electronic tensile testing machine (Zwick 1425, Germany) at speed of 500 mm/min. Samples of at least 0.12 mm in thickness with flat surface were cut for hardness measurements according ASTM D 2240 using durometer of model 306L type. The unit of hardness is expressed in (Shore A). The compression set was assessed using the measurement according ASTM D395 procedure. Test procedures of aging consist in exposing samples to the effect of harmful factors in a given time interval.

RESULTS AND DISCUSSION

Crosslinking of rubber macromolecules represent topologically critical phenomenon, when the ensemble of chains forms a three-dimensional network. In order to design the compounds formulation the representative combination of network structure should be managed for specific exploitation conditions of obtained materials. In the case of nano silica as reinforcing filler the strong filler/filler interactions resulting from polar surface functional groups such as siloxane are believed to be primarily responsible for the mechanical properties. The influence of silica loading on the crosslinking characteristics of the compounds based on NR/BR/SBR (25/25/50) rubber blends is show in the Table 1.

Table 1. Determined crosslinking characteristics of rubber compounds based on NR/BR/SBR and different content of silica

Silica content (phr)	ML (dNm)	MH (dNm)	t_{s1} (min)	t_{c90} (min)	CRI (min^{-1})
0	0.49	5.28	0.65	1.03	263.16
20	0.83	6.38	0.63	1.07	227.27
40	1.53	8.22	0.49	1.37	113.64
60	10.57	22.42	0.47	1.36	112.36
80	19.51	32.3	0.44	1.26	121.95
100	21.60	48.68	0.22	1.04	121.95

Generally, acidic compounds retard the crosslinking of rubber compounds. For this reason, precipitated silica, which contains a large number of acidic silanol (Si-OH) groups, is not added without an activator in elastomeric materials based on natural rubber. The surface of silica is acidic and therefore the strong hydrogen bonds with functional groups at network precursor are forming. For materials based on SBR due to the existence of phenyl groups the hydrogen bonding of the silica silanol group are influencing the strong silica/SBR interactions. Silica particles also can adsorb molecules of polar curative agents, rendering the deactivation of crosslinking process. Physical ageing which occurs as the gradual process may greatly affect many applicative properties depending on ageing temperature. Heat aging stability is a substantial for use in severe conditions. Retention of mechanical properties after accelerated heat aging is a specification for such long-term uses. Values of elongation at break are explained by the nature of filler (mainly the shape of the filler particles), but a very important factor is the bond strength between matrix and filler, which reduces the mobility of the polymer phase (better "wetting" of filler rubber macromolecules), the dispersion of fillers and agglomerates of filler particles. Tensile strength is a complex function depending on crosslinks type, concentration of elastically active network chains and chemical structure of

used network precursor. It is well known that if rubber is deformed by an external force, part of the input energy is stored elastically in the chains and is available (released upon crack growth) as a driving force for fracturing. The remaining energy is dissipated through molecular motions by heat; and as such, is made unavailable to break the chains. At higher crosslinking densities the chains motions become restricted, and the network is incapable of dissipating as much energy. The tensile strength increased with silica content. The optimal rubber reinforcing it is assumed that all filler agglomerates are dispersed to the aggregate. Generally silica particles tend to agglomerate due to formation of hydrogen bond between the hydroxyl group interactions and thereby enhanced the tendency for agglomeration. At lower loading of silica the nano particles are well dispersed and thereby increased the surface area for interaction. When silica content increases, the values of the elongation at break and hardness values increase. Rubbers are susceptible to oxidative aging because of unsaturated carbon-carbon double bonds in the backbone. Elevated temperatures usually promote oxidative ageing. In some elastomeric materials the tensile strength increases after ageing as a result of the further process of cross-linking. It was found that the sulfur of polysulfide -C-S_x-C- bond lead to further cross linking. The resistance of the elastomeric materials to thermal aging is considered as an essential requirement for the product service life. The ageing process is defined by a set of irreversible physical and chemical changes in the material. Tensile strength is reduced and for longer ageing process, the greater are the changes. However, elastomeric materials thermal ageing can be very complex due to the two competitive processes taking place simultaneously: cross-linking and chain scission. Crosslinking will lead to an increase in the elastic modulus and a consequent decrease in the extensibility of the material, whereas chain scission will result in the loss of the elastic modulus. In sulfur cured elastomers the bond energy between the sulfur and the polymer backbone greatly differ. There are three types of crosslinks: polysulfidic, disulfidic, and mono-sulfidic. For rubbers cross-linked with sulfur systems under elevated temperature, conversion of polysulfide bonds in mono-sulphidic is observed. The reaction is followed by separation of low molecular weight groups such as hydrogen sulfide, sulfur dioxide, and carbon disulfide. The tensile strength of prepared materials before and after thermal ageing (100°C for 72 h and 168 h) are shown in the Figure 1. The influence of silica content on the elongation at break before and after ageing is shown in the Figure 2. We can observe that there is a marginal decrease in the tensile strength and elongation at break after aging during 72 h and 168 h for samples which contain 80 phr and 100 phr silica. The reductions of the properties are due to partial crosslinking of the elastomer backbone and degradation of the rubber taking place upon ageing. Values of elongation at break decrease with ageing time increase. Elongation at break decreases with tightening of the conditions of accelerated ageing process. The influence of silica content on the hardness before and after ageing of prepared elastomeric materials is shown in the Figure 3. It was determined that hardness for all composite materials increases with tightening of the conditions of accelerated ageing process. Well-dispersed nano particles are barrier for heat transfer through the material, thereby preventing fast degradation and are acting as a mass transport barrier to oxygen and volatile decomposition products.

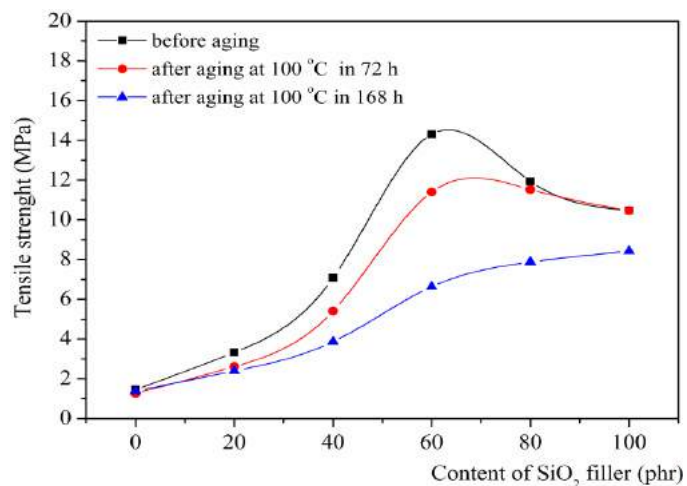


Figure 1. The influence of silica content on tensile strength of NR/BR/SBR composites before and after ageing during 72h or 168 h at 100 °C.

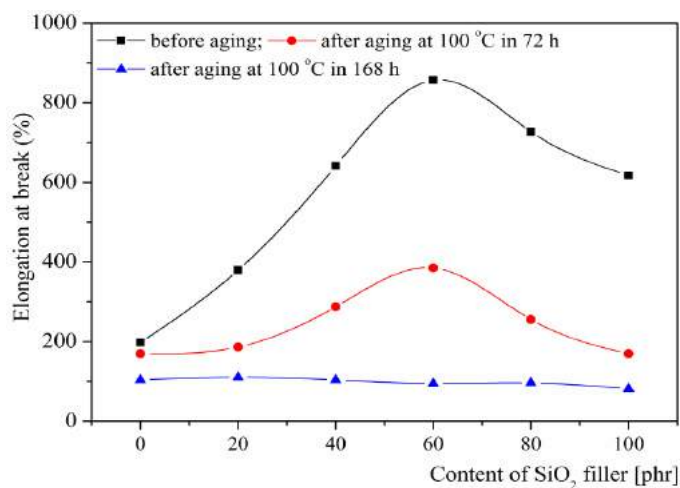


Figure 2. The influence of silica content on the elongation at break before and after ageing during 72h or 168 h at 100 °C for composites based on NR/BR/SBR.

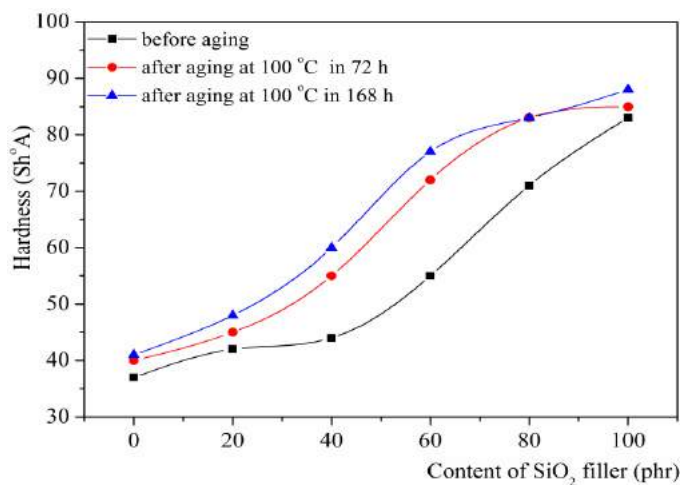


Figure 3. The effect of silica content on hardness of composites based on NR/BR/SBR ternary rubber blends after ageing at 100 °C during 72h or 168 h.

In the figure 4 is shown the influence of silica content on the compression set of prepared materials. This characteristic of materials is important as spontaneous stress release of the elastomeric materials and is the indicator what could occur due to the external forces or internal pressure changes during elastomeric product exploitation.

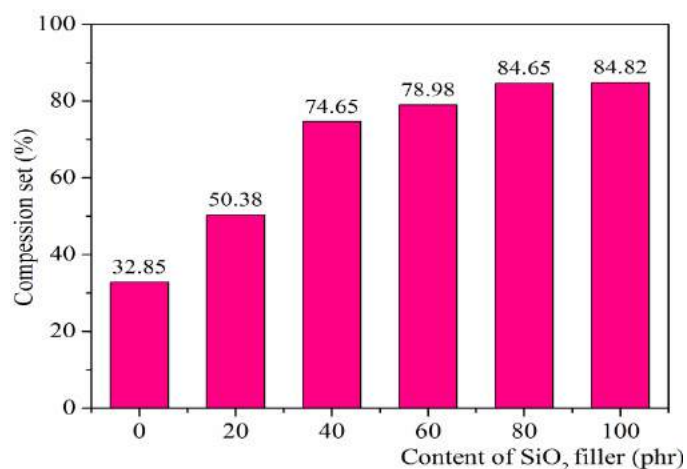


Figure 4. The influence of silica content on compression set of materials based on NR/BR/SBR ternary rubber blend

It was determined that the compression set of prepared elastomeric materials increases linearly with the content of used silica. The introduction of reinforcing fillers into the rubber blends reduces its elasticity, which in turn increases the compression set. This performance is attributed to non-crosslinked areas that do not contribute to the permanent network structure and relax during the compression stage. This permanent deformation is of particular concern when elastomers are used to for seals fabrication. Decreasing seal force has the potential to create leaks over time

CONCLUSIONS

The aim of this work was to assess the influence of silica nano particles on ageing of elastomeric materials based on NR/BR/SBR ternary rubber blends in order to find the best formulation of rubber compounds. Elastomeric materials based on this three network precursor are used for tire tread fabrication. Thermal stability and ageing is the primary characteristics for processing and application of elastomeric products. It was assessed that after ageing during 72h or 168 h at 100 °C the mechanical properties of prepared materials decreases (changes in the morphology; degradation of rubber; changes in interaction between components at elevated temperatures). The resistance of the elastomeric materials to thermal ageing is considered as an essential requirement for the service life of elastomeric materials. When silica content increases, the values of the elongation at break and hardness values increase. It was determined that the compression set increases linearly with nano-silica content. It was estimated that values of the compression set obtained for 25% of deformation are appropriate for application of prepared materials.

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