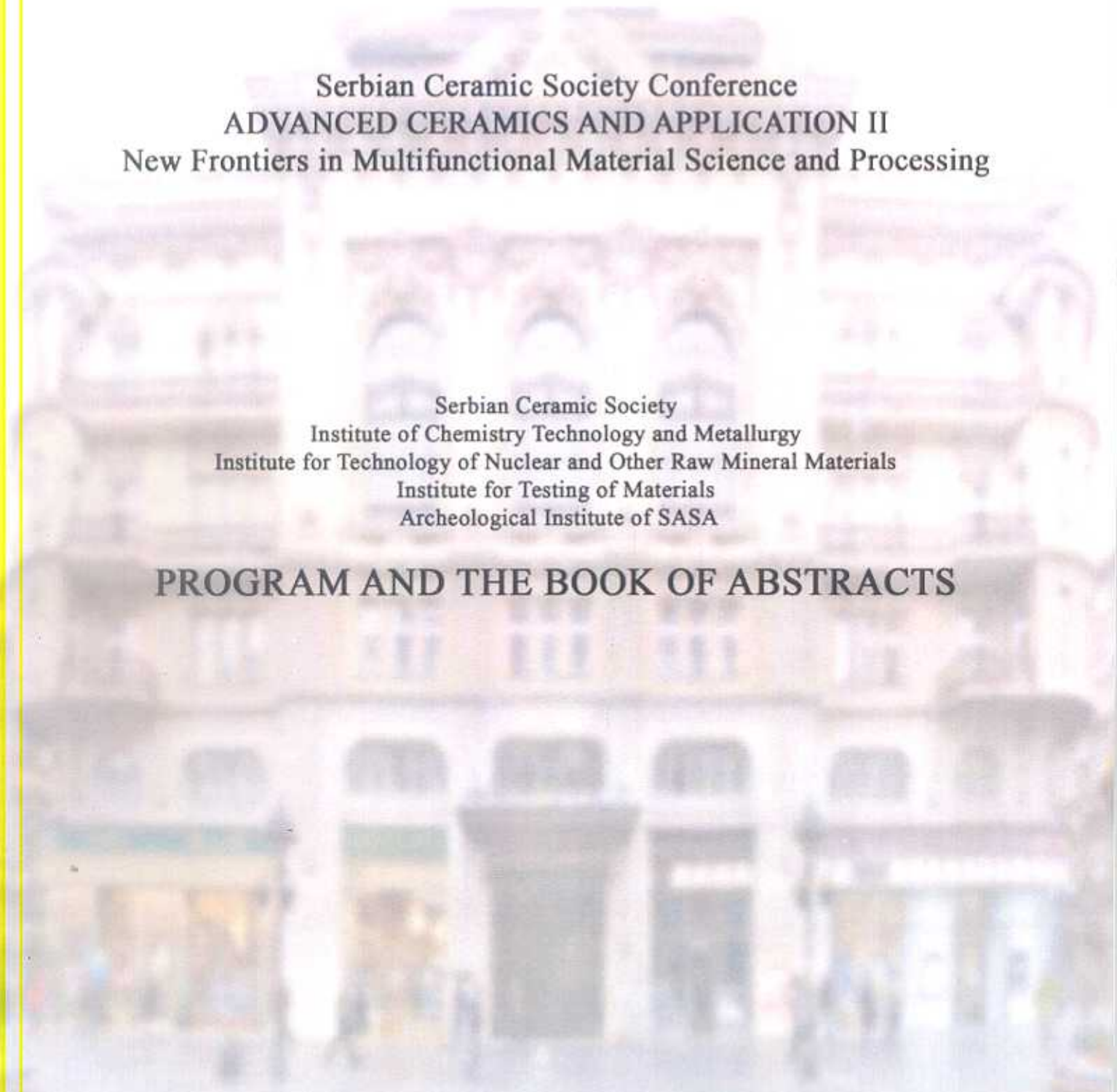
Serbian Ceramic Society Conference

ADVANCED CERAMICS AND APPLICATION II

New Frontiers in Multifunctional Material Science and Processing

Serbian Ceramic Society
Institute of Chemistry Technology and Metallurgy
Institute for Technology of Nuclear and Other Raw Mineral Materials
Institute for Testing of Materials
Archeological Institute of SASA

PROGRAM AND THE BOOK OF ABSTRACTS



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Dear Colleagues, dear friends, we have great pleasure to welcome you to the Advanced Ceramic and Application Conference II organized by the Serbian Ceramic Society in cooperation with the Institute of Chemistry Technology and Metallurgy, Institute for Technology of Nuclear and Other Raw Mineral Materials, Institute for Testing of Materials and Archeological Institute of SASA. This conference brings together researchers from academia and industry to present the latest advances in synthesis and characterization in the field on new ceramic structures. The chosen Conference topics opening the new frontiers in designing of advanced ceramic materials since they cover fundamental theoretical research, modeling and simulation, controlled nanostructured materials synthesis and optimization of the consolidation process, which all together should provide practical realization of the new ideas towards device miniaturization, energy-materials-information integration and preservation of cultural heritage.

Prof. Dr Vojislav Mitić
President of the Serbian Ceramic Society
World Academy Ceramics Member

General Conference topics included:

- Basic Ceramics Science
- Nano-, Bio- and Opto-ceramic Nanotechnologies
- Multifunctional Materials
- Magnetic and Amorphous Materials
- Construction and Eco-ceramic
- Composites, Catalysis, Electro-catalysis
- Artistic Ceramic and Design, Archeological Heritage
- Young Researchers
- **Sintering processes**
  - kinetics
  - thermodinamics
  - microstructure
  - modeling

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P1

Optimization of major oxides content and fired brick properties for various applications

M. Arsenović[1], L. Pezo[2], S. Stanković[3], Z. Radojević[1]

[1]Institute for testing of materials, 11000, Belgrade, Serbia
[2]Institute of general and physical chemistry, 11000, Belgrade, Serbia
[3]Faculty of technology and metallurgy, University of Belgrade, 11000, Belgrade, Serbia

The optimal samples content of major oxides (SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, Na_2O, K_2O, MnO and TiO_2), firing temperature (800–1100 °C) and final properties of tiles, hollow blocks and solid cubes were chosen depending on a final usage of the raw material in heavy clay brick industry. Optimization procedure was performed using Fuzzy Synthetic Evaluation (FSE) algorithm on the basis of previously developed artificial neural networks models that predict compressive strength, water absorption, firing shrinkage, weight loss during firing and volume mass of laboratory products. Trapezoidal membership function is defined by experimentally obtained values and optimal ranges of tested properties. The objective function included all the fired products parameters with equal participation, and its maximum is determined the optimization results. Objective function gained values between 0.6 and 0.7. Solid bricks are proved to be producible of heavy clays containing the highest free SiO_2 and CaO content, by firing at high temperatures. Highly sinterable clays should be used for hollow bricks and the highest quality raw materials in roof tiles production, by firing at 900 °C at laboratory conditions.

P2

Lightweight construction ceramic composites based of pelletized fly ash aggregate

Anja Terzić[1], Zagorka Radojević[1], Ljiljana Miličić[1], Nina Obradović[2], Vladimir Pavlović[2], Ljubica Pavlović[3]

[1]Institute for Materials Testing, Belgrade, Serbia
[2]Institute of Technical Sciences of Serbian Academy of Sciences and Arts, Belgrade, Serbia
[3]Institute for Technology of Nuclear and Other Raw Mineral Materials, Belgrade, Serbia

As coal combustion byproduct fly ash represents a risk for environment: direct ash emission from open land-fills causes pollution of air, soil and water. The solution for this severe pollution problem is fly ash reapplication in various construction ceramic composite materials. Although pelletization of waste powdery material is a known technique in the production of artificial aggregates, it still has not been widely used in construction sector. Here investigated cold-bonded fly ash aggregate was produced in semi-industrial pelletizing device. The fly ash particles were bonded with water-glass (Sodium silicate - Na_2SiO_3) and used as substitution for aggregate in Portland cement based composite. Half of the produced lightweight aggregate was submitted to thermal treatment and afterwards applied in the construction composite in the same ration as in the case of cold-bonded pellets. The performance characteristics of two types of lightweight composites were mutually compared and afterwards correlated with characteristics of normal-weight concrete. Compressive

strength, modulus of elasticity and tensile strength were used as represents of the composites mechanical behavior. Mineral constituents of fly ash pellets were analyzed by means of X-ray diffraction analysis, differential thermal analysis was applied in crystalline phase investigation, and scanning electron microscopy in microstructural analysis. The leaching behavior and environmental impact of hazardous elements were also analyzed. It was concluded that content of potentially toxic elements found in leachate of fly-ash based composites was far below tolerance limit proposed by actual standards for the building materials, characterizing the fly ash non-harmful secondary raw material and enabling its reapplication in building materials industry. Utilizing fly ash to produce quality aggregates should yield significant environmental benefits.

P3

Establishing the model for predicting the moisture and velocity in the critical point during drying of green masonry products

Miloš Vasić[1], Zagorka Radojević[1]

[1]Institute for Materials Testing, Belgrade, Serbia

The aim of this study was to establish the model for predicting the moisture and velocity in the critical point during drying of green masonry products. The raw material was first dried at a temperature of 60°C, and then after cooling to room temperature, was milled down using perforated rolls mill. Milled material was identified and subject to further classical preparation, which precedes the formation on the vacuum presses. Thus prepared sample carried the name - sample A. The starting raw material was mechanically activated for 30 minutes. Thus prepared sample carried the name - sample B. Laboratory samples 120x50x14 mm were formed in a laboratory extruder under a vacuum of 0.8 bar. These samples were used in further experimental work. Drying process was monitored and all process parameters such as: temperature, relative humidity of the drying air, weight changes, linear shrinkage, temperature of the surface and in the centre of test samples were recorded continually.Two mathematical models, based on multi factorial experimental design technique, were set up. The first describes the moisture and the second one the velocity value of the samples B in the critical point as a function of temperature, relative humidity and the velocity of the drying medium.

P4

Slag from metal magnesium production as component in ecologically clean production of various types of construction ceramics

Ljubica Pavlović[1], Anja Terzić[2], Zagorka Aćimović[3], Marko Pavlović[3]

[1]Institute for Technology of Nuclear and Other Mineral Raw Materials, Belgrade, Serbia
[2]Institute for Materials Testing, Belgrade, Serbia
[3]University of Belgrade, Faculty of Technology and Metallurgy, Belgrade, Serbia

In this paper application possibilities of the slag from production of metal magnesium in the plant *Bela Stena, Baljevac*, as an initial component in construction ceramics is investigated. During technological processes of materials manufacturing significant number and quantities of secondary raw materials are being produced. Such by-products are frequently causing number of complex problems connected to their storing, as well as water, air and soil pollution. Majority of these secondary materials can be sorted in the group of useful and reusable materials according to their chemical and mineralogical composition and properties. Secondary raw materials can be applied as basic components in manufacturing processes of number of ceramic building materials. By re-application secondary raw materials are obtaining their significance judging from the economical as well as ecological aspect. During technological process of manufacturing of the metal magnesium, entire specter of by-products occurs. Most of such obtained waste materials can be reused as secondary components in a number of industrial processes. Aim of this investigation is to point out the possibility of slag reusing. Given slag is the by-product from the process of magnesium manufacturing and it can be applied as basic component for number of building materials: building ceramics, building mortars and thermo-insulation mortars. The slag, here investigated, is by-product from magnesium production and it belongs to the four components system Cao-MgO-Al_2O_3-SiO_2. Results of the investigation showed that ceramic based on this secondary material can be used as one of starting components in floor, wall and roof tiles manufacturing. Magnesium production slag application in the composite materials used for production building ceramics is very important because of its economical as well as ecological aspects.

P5

Alumina as raw material in production of ceramic materials: the changes of alumina crystal structure by mechanical activation procedure

Ljubiša Andrić[1], Anja Terzić[2], Ljubica Pavlović[1], Zagorka Aćimović[3], Marko Pavlović[3]

[1]Institute for Technology of Nuclear and Other Mineral Raw Materials, Belgrade, Serbia
[2]Institute for Materials Testing, Belgrade, Serbia
[3]University of Belgrade, Faculty of Technology and Metallurgy, Belgrade, Serbia

In this paper mechano-chemical activation of alumina and the influence of the activation process on the change of the alumina crystal structure is presented. This study is based on investigation of kinetics and mechanism of the particle formation together with the phase transformations occurring in the process. Theoretical principles of operation of high-energy mechano-activators, such as vibration and planetary mechano-activators during the mechano-

chemical activation of alumina were used in the investigation, along with their dependance on selected experimental conditions, detailed investigation of the possibility to obtain high-grade αAl_2O_3 alumina in the form of micron-sized, non-agglomerated particles by the method of mechano-chemical activation combined with heat treatment, and starting from the γAl2O3 alumina modification after the Bayern process. The elements that are necessary for determination of operation of high-energy mechano-activators, particularly vibration and planetary ones, were determined by detailed investigation of the alumina mechano-chemical activation; also, conditions required to define the both technological and production parameters of mechano-chemical activation are fulfilled, too. Based on investigated parameters and theoretical consideration of the alumina mechano-chemical activation, together with its influences on change of the alumina crystal structure by usage of mechano-chemical activators with advanced construction and contemporary instrumental techniques used in determination and observation of the most important physical, chemical and thermic characteristics, kinetic model which is the basis for a quick and efficient determination of above parameters in order to optimize and automatise mechano-chemical activation processes was developed.

P6

The luminescent properties of yttrium oxyapatite doped with Eu^{3+} ions

V. Jokanović, B. Čolović, N. Jović

Institute of Nuclear Sciences Vinča, Mike Petrovica Alasa 12-14, Belgrade, Serbia

The luminescent properties of yttrium oxyapatites doped with europium ions, synthesized by reflux method, were investigated in this paper. This low temperature procedure of synthesis gave, by the first time, pure oxyapatite phase as ideal host material for incorporation of any rare earth ion. The SEM investigations showed a very specific rope-like morphology of the obtained powder. The structure of yttrium apatite with incorporated Eu^{3+} ions, as luminescent active centers, had shown the most important reflections of hexagonal phase of yttrium oxide silicate - yttrium oxyapatite; obtained as a consequence of the Y^{3+} ion stabilization. The alternative positions of Eu^{3+} ions in crystal lattice of oxyapatite influenced its specific luminescent properties, induced by various crystal environments of Eu^{3+} ions which substitute calcium or yttrium ions. The deep analysis of the europium emission spectra showed that it is possible to refine positions of Eu^{3+} ions inside the oxyapatite crystal cell.

P7

Synthesis of the glass-ceramics based on basalt

Marko Pavlović[1], Snežana Grujić[1], Anja Terzić[2], Ljubiša Andrić[3]

[1]University of Belgrade, Faculty of Technology and Metallurgy, Belgrade, Serbia
[2]Institut for Materials Testing, Belgrade, Serbia
[3]Institute for Technology of Nuclear and Other Raw Mineral Materials, Belgrade, Serbia

In this paper, results and investigation of possibility of sythesis of the glass-ceramics based on basalt are presented. Glass-ceramic due to its specific properties (high hardness, abrasion resistance, good chemical consistency, etc.) has wide area of application in mining, civil engineering and building industry, metallurgy, for manufacturing of parts of equipment or devices, constructing materials, etc. For obtaining of the glass-ceramic basalt from *Vrelo, Kopaonik* deposit was used. A series of melting on various temperatures from 1150 to 1300°C were performed. Synthesis parameters, temperature, melting interval, and cooling rate were defined. Selection of composition of glassy mixture was performed in order to obtain satisfactory quality. On thus obtained products various conditions of thermal treatment were performed and optimal regime was determined: gradual heating up to 850°C in time intervals of 1.5 h and progressive cooling gave good physico-mechanical properties. Structure of glass-ceramic samples obtained by melting of basalt after thermal processing is crypto–crystal accompanied by appearance of small low-crystallized aggregates. The structure also contains bubbles filled with either air of glass. These are initial results of investigation of application of basalt from Serbian deposits for obtaining glass-ceramics. These products are not carcinogenic and they can successfully substitute materials such as: asbest or various metallic materials. Technology applied in manufacturing of basalt is ecologically clean, which is of high importance from economical, ecological and energetically point of view.

P8

The influence of Ni on the performance of Al, Fe, Ni pillared bentonite based electrodes in electrooxidation of phenol

T. Mudrinić, Z. Mojović A. Milutinović-Nikolić, M. Žunić, P. Banković, A. Ivanović-Šašić, D. Jovanović

University of Belgrade - Institute of Chemistry, Technology and Metallurgy, Center for Catalysis and Chemical Engineering, Njegoševa 12, 11000 Belgrade, Republic of Serbia

Composite Nafion-clay modified glassy carbon electrodes (GCEs) were tested in the electrooxidation of phenol in acidic media. The clay originated from Mečji Do, a seldom investigated bentonite deposit in Serbia. Raw clay was Na-enriched and pillared with different species. The following pillared clays (PILCs) were obtained: Al90Fe10-PILC, Al85Fe10Ni5-PILC and Al90Fe5Ni5-PILC, where numbers represent the molar percentage of each species in the pillaring solution. The aim of this investigation was to test the influence of Ni on the electrochemical performance of PILC bearing electrodes. The obtained materials were characterized by X-ray diffraction, FTIR spectroscopy and X-ray photoelectron spectroscopy. The cyclic

P10

Chromite based refractory coatings used in expandable patterns casting of Fe-C alloys

Zagorka Aćimović[1], Anja Terzić[2], Ljubiša Andrić[3], Ljubica Pavlović[3], Marko Pavlović[1]

[1]University of Belgrade, Faculty of Technology and Metallurgy, Belgrade, Serbia
[2]Institute for Materials Testing, Belgrade, Serbia
[3]Institute for Technology of Nuclear and Other Mineral Raw Materials, Belgrade, Serbia

A possibility to develop new chromite-based refractory coatings for casting applications has been investigated in this paper. The results of monitoring of synthesis and characterization procedure and finally application of the refractory coatings showed that sediment stability of coating suspension was crucial parameter for the quality of the coating. Optimization of the coating composition with the controlled rheologic properties was achieved by application of different coating components, particularly by application of a new suspension agent and by alteration of coating production procedure. Chromite was applied as filler. The chromite sample was tested by X-ray diffraction analysis, diffraction thermal analysis and scanning electron microscopy. The shape and grain size were analyzed with program package OZARIA 2.5. It was shown that application of this type of water-alcohol-based lining had a positive influence on surface quality, structural and mechanical properties of the castings of Fe-C alloys obtained by casting into sand molds, according to the method of expandable patterns.

P11

Dilatometric Analysis of Mechanically Activated $SrTiO_3$ Powder

J. Živojinović[1*], D. Kosanović[1], N. Obradović[1], A. Peleš[1], N. Labus[1], S. Filipović[2], V. B. Pavlović[1], M. Mitrić[2], M. M. Ristić[3]

[1]Institute of Technical Sciences of the Serbian Academy of Sciences and Arts, Knez Mihailova 35/IV, 11000 Belgrade, Serbia
[2]Vinča Institute of Nuclear Sciences, Condensed Matter Physics Laboratory, Belgrade, Serbia
[3]Serbian Academy of Sciences and Arts, Knez Mihailova 35, 11000 Belgrade, Serbia

Strontium-titanate powder was mechanically activated in a planetary ball mill for 0, 5, 10, 30, 60, 90 and 120 minutes. Non-isothermal sintering of non-activated and activated $SrTiO_3$ powder samples in the temperature interval from 50 to 1300 ^{0}C with three different heating rates (10, 15 and 20 ^{0}C/min) was investigated on a dilatometer. X-ray powder diffraction and scanning electron microscopy (SEM) were used to determine the phase composition, lattice microstrains and microstructure morphology of the samples. XRD results showed the presence of new phases: $SrCO_3$ (strontuim-carbonate) and TiO_2 (anatase) after 30 minutes of mechanical activation.

P33

Possibility of use waste materials from floating plant Feldspar ujanovac in ceramical industry after removing the surplus of iron

Dragan S. Radulović, Slavica R. Mihajlović

Institute for Technology of Nuclear and other Mineral Raw Materials, Belgrade

In this work is given a scheme of the results of determining the conditions under which the waste material from plant for flotation "Feldspar" Bujanovac can become the start as row material for the ceramic industry. Also, in this paper is given a review of results determining conditions of thickening and filtration of waste material from the flotation plant "Feldspar" – Bujanovac.

Tests of possibilities of thickening and filtration of the non-magnetic fraction of waste material sample of the class -0,063 mm after magnetic separation in laboratory conditions have been carried out within this research. Based on dewatering testing, time of thickening and diagram review of the results have been determined.

P34

Recycling of waste gypsum and its repeated usage in the civil engneering and ceramic industry

Dragan S. Radulović[1], Anja Terzić[2], Ljubica Pavlović[1], Velimir Antanasković[1],

[1]Institute for Technology of Nuclear and Other Mineral Raw Materials, Belgrade, Serbia
[2]Institute for Materials Testing, Belgrade, Serbia

This paper shows result of investigation of possibilities of recycling waste gypsum and finding the adequate appliances of revitalized gypsum in the ceramic industry and civil engineering. The gypsum which is used in the ceramic industry is very fine material. It has high content of semi-hydrate gypsum. After usage of molds in the process of the production of sanitary-ware, the gypsum in the form of dihydrate become useless. The gypsum for production of molds could be reused thanks to development of this, suggested technology. This is the way for supstitution imported component. Appliance of revitalized secondary gypsum is highly important in both following aspects: economical and environmental.

P35

The ceramic materials based on Ag doped zeolite

A. Radosavljević-Mihajlović[1], M. Omaraševič[1], A. Došen[1], N. Đordević[2]

[1]Vinča Institute of Nuclear Sciences, University of Belgrade, Belgrade, Serbia
[2]Institute for Technology of Nuclear and Other Mineral Raw Materials, Belgrade, Serbia

The method of the thermal treatment of cation exchanged zeolites (ZTIT) is shown as very acceptable for synthesis of alkaline earth and alkaline framework aluminosilicates. The type and valence state of extraframework cations give rise to recrystallization of amorphous substances to different framework topologies. The fully exchanged Ag^+ - forms of these zeolites were prepared after several successive exchanges from 0.21 M $AgNO_3$. The ion exchanged samples were thermally treated in the temperature range between 900 to 1300 oC for 1h.

P36

Investigation of construction ceramic from objects of cultural and historical heritage

Zagorka Radojević, Anja Terzić, Ivana Delić Nikolić

Institute for Materials Testing, Belgrade, Serbia

Gothic-Romanic monastery on the location of Novi Rakovac, Gradina is significant site from aspect of historical and cultural heritage of the Republic of Serbia. Exact date of building of the monastery can not be clearly identified. Also, it is probable that during monastery "life" additional changes of building construction elements and, even, some reparation works have been performed. However, today, the monastery building is mostly in ruin. Thus, investigation has been carried out in order to make restoration and renovation plans. First step was to mark sampling locations on the building and its elements which would not make further harm or damage to the monument. Afterwards, sampling of stone, mortar and brick specimens has been performed. All specimens were well preserved and carefully stored for its continuity and compactness would be maintained for further laboratory testing. Thus, results were utmost exact and precise and, furthermore, plans for restoration and renovation could be established. Namely, basing on the obtained results from investigations of given materials, new materials, which resemble old ones, could be designed and applied in restoration process. This paper presents results of investigation conducted on brick samples. Applied investigation is mostly engaged with textural characteristics of material in question. Reason for such choice of investigated properties is the fact that first task given to newly designed brick, which should replace old, original brick in the monument, is to aesthetically fit in the building conception. In relation to building functionality, other properties such are compressive strength; water absorption and adhesiveness were investigated. Thus, future durability of the renovated historical and cultural monument could be satisfied.

P37

Facade ceramic tiles: nicrostructural analysis of superficial defects

Zagorka Radojević, Anja Terzić

Institute for Materials Testing, Belgrade, Serbia

This investigation was conducted with an aim to determine nature and cause of defects appearing on the glazed facade ceramic tiles. The results of the investigation of incusions visible surface of ceramic tiles are presented in this paper. Glazed ceramic tiles, were previously in use, namely they were embedded on the exterior of a facility. Influenced by atmosphere, defects appeared on visible surface of glazed ceramic tiles. Defects were shaped as dark-brown dots surrounded by yellow halo. Investigation was conducted on the tiles previously embedded on the façade, as well as on new, previously not used ceramic tiles. With an aim to obtain answer on the question of defects origin, investigation was conducted in accordance with standard SRPS ISO 10545: determination of dimensions and surface quality; determination of moisture expansion; determination of chemical resistance; determination of resistance to stains. Microstructural analyses were conducted by means of optical microscope and scanning electron microscope coupled with energy dispersive spectrometer device. Analysis of results highlighted possibility of defects cause being carbon and iron as impurity, both present in raw material used for glaze production.

P38

Biocompatibility screening of biomaterial based on porous apatite with a film of alginate polymer

Aleksić Milena[1], Rajković Jelena[1], Vasiljević Perica[1], Đorđević Ljubiša[1], Miljković Miroslav[2], Najman Stevo[2], Jokanović Vukoman[3]

[1]Department of Biology and Ecology, Faculty of Science and Mathematics, University of Niš, Serbia
[2] Institute of Biomedical Research, Medical Faculty, University of Niš, Niš, Serbia
[3] Vinča Institute of Nuclear Sciences, Lab of Radiation Physics and Chemistry, Belgrade, Serbia

New generation of biomaterials is identified as a priority area with emphasis on ability to last longer and be better adapted to the environment of the human body. The primary requirement is biocompatibility of the biomaterials. This study was conducted in order to examine biomaterial based on porous apatite with a thin film of alginate polymer (PA + A). *In vivo* test and *in vitro* hemolytic assay were used in order to assess the biocompatibility. PA + A was implanted in Wistar rats subcutaneously in the interscapular region and intramuscularly in muscle gastrocnemius at two, four and eight weeks. After this period, the rats were euthanized and the implants with surrounding tissue were surgically removed from rats and prepared for light microscopy and scanning electron microscopy analysis (SEM). Histopathologic and SEM analysis showed the presence of multinuclear giant cells, fibroblasts and increased collagen production which should be related as inflammatory response. The results of the hemolytic assay showed that the studied biomaterial had low hemolysis. These findings suggests that the biomaterial is compatible with blood, but the inflammatory reaction in tissue to the biomaterial indicates that it is necessary to perform further tests.