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**Journal of Silicate Based and Composite Materials** 

#### A TARTALOMBÓL:

- Effect of firing temperature on triaxial electrical porcelain properties made from Tanzania locally sourced ceramic raw materials
- Anisotropic response of the Holzapfel's constitutive model for the lumbar spine considering degenerative conditions
- Preparation of new nano-organoclays from Hexadecylamine, Tetradecylamine and Chalcone with Montmorillonite using ion exchange processes
- Mass transfer of aluminum film from the surface of zeolite on the cathode
- Numerical study of building materials filled by PCM for thermal energy storage
- Internal thread cutting process improvement based on cutting tools treatment by composite powders in a magnetic field



2018/4



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### 2018/4

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# Effect of firing temperature on triaxial electrical porcelain properties made from Tanzania locally sourced ceramic raw materials

**BLASIUS NGAYAKAMO** • The Nelson Mandela African Institution of Science and Technology, Department of Materials Energy Science and Engineering • henryblasius@gmail.com

**S. EUGENE PARK** • The Nelson Mandela African Institution of Science and Technology, Department of Materials Energy Science and Engineering

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#### **Abstract**

The study has investigated the effect of firing temperature during the production of technical triaxial electrical porcelain, for electrical insulation applications using Tanzania locally sourced ceramic raw materials. The green triaxial porcelain samples containing 50 wt% of Pugu kaolin, 35 wt% of Same clay and 15 wt% of feldspar were produced and fired at  $1200\,^{\circ}$ C- $1300\,^{\circ}$ C with a heating rate of  $10\,^{\circ}$ C/min (dwell time of 1.5h) and cooled at  $10\,^{\circ}$ C/min to a room temperature. X-ray diffraction technique was used to investigate phases developed in the triaxial electrical porcelain after firing process. The main crystalline phases revealed were mullite and quartz. The technological properties of the triaxial electrical porcelain such as water absorption, apparent porosity, bulk density, bending and dielectric strength were determined for each porcelain sample fired at high temperature. The optimum physical-mechanical and electrical properties were found at  $1250\,^{\circ}$ C. However, the triaxial electrical porcelain properties were found to decrease with the increase in firing temperature.

Keywords: Firing temperature, triaxial electrical porcelain, physical-mechanical and dielectric properties

Kulcsszavak: Égetési hőmérséklet, triaxiális szigetelő porcelán, fiziko-kémiai és dielektromos jellemzők

#### Blasius NGAYAKAMO

MSc Materials Scientist and Engineer at NM-AIST. Department of Materials Energy Science and Engineering. Fields of interest: ceramic raw materials, flux materials and high voltage porcelain insulators.

#### S. Eugene PARK

Associate Professor and chair, Materials and Energy Science and Engineering at NM-AIST Department of Materials Science and Engineering, Fields of interest: Ceramic and sustainable Energy Engineering.

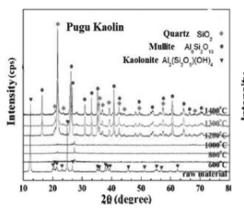
#### 1. Introduction

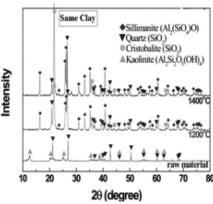
Triaxial electrical porcelain is composed of clay, feldspar which are locally sourced ceramic raw materials and other filler materials such as quartz and alumina. The raw materials play specific roles in influencing the properties and performance of the final products. Clay  $[Al_2Si_2O_5 (OH)_4]$  provides plasticity, quartz  $(SiO_2)$  maintains the shape of the porcelain structure during firing, and feldspar  $[KxNa1-x (AlSi_3) O_8]$  promotes vitrification. The three ceramic raw materials place electrical porcelain in the phase system  $[(K, Na)_2O-Al_2O_3-SiO_2)]$  in terms of oxide hence referred as triaxial porcelain [1, 2]. Traditional ceramic raw materials are the potential candidate materials for the production of triaxial electrical porcelains. The use of traditional ceramics as raw materials instead of industrial chemicals is highly preferred due to the lower price of the raw materials [3].

The properties of triaxial electrical porcelain are contributed to the variations in the composition of the raw materials, the method of production, and the firing temperature adopted [1]. The sintered triaxial porcelain product contains mullite ( $Al_6Si_2O_{13}$ ) and undissolved quartz ( $SiO_2$ ) crystals embedded in glassy phase which result from the liquid phase formed by the melting of feldspar in the raw materials [4].

Therefore the desired properties of triaxial electrical porcelains are achieved particularly during the firing process since the technological properties of clay-based ceramics products depend on firing conditions such as temperature [5, 6]. However, other technological properties which are

evaluated to determine the performance of the ceramic product after firing are water absorption, firing shrinkage and bending strength [6]. During the firing process, the triaxial porcelain body undergoes several phase transitions, during which both composition and structure change significantly which influence triaxial porcelain properties at the end of the firing process [5]. Hence, the properties of the triaxial porcelain are mainly influenced by sufficient development of mullite during firing process since the development of mullite in the porcelain is highly associated with firing temperature of the porcelain which should not be below 1150 to 1200 °C for the mullite forming processes to be completed [5, 7]. Therefore, the development of the physical-mechanical and dielectric properties of porcelain are contributed by each phase developed during firing which depends on the concentration and microstructural attributes which are influenced by temperature and the chemical composition of the raw materials which is an important factor because of its effects on porcelain properties [8-11]. Since the effect of firing temperature on the electrical porcelain properties made from Tanzania locally sourced ceramic raw materials is not reported. Therefore, the work intends to evaluate the effect of firing temperature on the triaxial electrical porcelains made from Tanzania locally sourced ceramic raw materials. However, the study focuses also on the phase changes, surface morphology development as well as the physical-mechanical and dielectric properties of the triaxial electrical porcelain sample due to change in firing temperature.





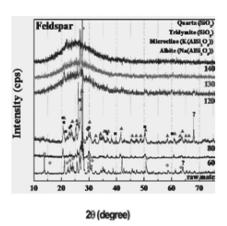


Fig. 1. X-ray diffraction patterns of Pugu kaolin, Same clay and feldspar [16] 1. ábra Pugu kaolin, Same agyag és földpát röntgendiffraktogramjai [16]

#### 2. Experimental procedures

Pugu Kaolin was collected from the Pugu hills, 35 km west of Dar es Salaam, Same clay and feldspar from Same, Kilimanjaro region in the northern zone of Tanzania. The ceramic raw materials were crushed and ball milled to reduce their size. The particle size less than 106 µm was achieved by using sieve shaker Model RX-29-10 digit. The chemical composition of the raw materials was analyzed by using X-Ray Fluorescence (XRF) PANalytical, Model: Minipal4 (PW4030)-Rh X-Ray Tube, 30kV, 0.002mA and the results are presented in *Table 1*. The examination of the surface morphology of the porcelain sample was carried out by Scanning Electron Microscope (SEM) Model: JEOL JSM-6335F having a resolution of 10µm at 2kV. The crystalline phase analysis of the porcelain insulator was analyzed by X-ray diffractometer Model: Bruker D2-PHASER-40Kv/44mA. Six triaxial porcelain samples were produced by varying the composition of the locally sourced materials by 50%wt of Pugu kaolin, 35%wt of same clay and 15%wt of feldspar. The powder mixtures were uniaxially compacted into rectangular shapes at 10 MPa. The porcelain green body samples were seasoned at a room temperature for 5 days and they were oven dried at the temperature of 110 °C for 24 hrs. The sintering of porcelain samples was done at 1200, 1250 and 1300 °C for 1.5 hrs at the ramp rate of 10 °C/min in each firing process. The sintered porcelain bodies were left to cool at 10 °C/min to room temperature and were subjected to physical-mechanical properties and dielectric strength analysis.

#### 3. Results and discussion

#### 3.1 Chemical composition of the raw materials

The chemical compositions of the raw materials in form of their oxides are presented in *Table 1*. The study results reveal that both clays have the higher content of silica and alumina. However, feldspar and Pugu kaolin have a higher content of Hematite (Fe<sub>2</sub>O<sub>3</sub>) compared to Same clay. The literature reports that small amount of coloring oxides such as Fe<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub> less than 0.9% may be accepted for porcelain wares production [12]. However, a considerable high amount of Fe<sub>2</sub>O<sub>3</sub> in Pugu kaolin and feldspar may not be accepted as

they may impart yellowish and reddish color in porcelain wares unless beneficiated. Feldspar has considerable higher alkaline oxide K<sub>2</sub>O than Pugu kaolin and Same clay. During the sintering process, the alkaline oxide K<sub>2</sub>O melts and forms the liquid phase that contributes to densification at higher temperatures due to the formation of the glassy phase. Nevertheless, the quantities of the alkaline oxides depend on the mineralogical nature of the clays and their reactivity during melting of the clay minerals [13]. The alkaline oxides(K<sub>2</sub>O and Na<sub>2</sub>O) play a significant role towards vitrification, phase transformation and mullite grain growth [14, 15].

Oxides	Pugu kaolin	Same clay	Feldspar	
SiO <sub>2</sub>	60.0	60.4	57.1	
Al <sub>2</sub> O <sub>3</sub>	30.3	13.9	14.0	
Fe <sub>2</sub> O <sub>3</sub>	3.95	1.40	3.08	
MnO	0.021	0.00	0.32	
Ca0	0.39	0.00	1.0	
Na <sub>2</sub> O	0.00	0.04	0.20	
K <sub>2</sub> O	2.14	2.6	12.09	

Table 1. Chemical composition of raw materials 1. táblázat Alapanyagok kémiai összetétele

#### 3.2 Mineralogical composition of the raw materials

The X-ray diffraction patterns of the ceramic raw materials before and after firing are presented in *Fig. 1* as reported by [16]. The result shows phase compositions of both Pugu kaolin and Same clay are kaolinite, however, Pugu kaolin showed the development of crystalline phases of mullite and quartz at a temperature of 1400 °C. In addition, Same clay was observed to form cristobalite and sillimanite above 1200 °C. Feldspar contains albite, and microcline, tridymite, and quartz. Since the major components of interests are potassium feldspar (K<sub>2</sub>O·Al<sub>2</sub>O<sub>3</sub>·O·6SiO<sub>2</sub>) sodium feldspar (Na<sub>2</sub>O· Al<sub>2</sub>O<sub>3</sub>·O·6SiO<sub>2</sub>); and lime feldspar (CaO·Al<sub>2</sub>O<sub>3</sub>·O·6SiO<sub>2</sub>). However, the results indicate that feldspar deposit contains a high content of potash feldspar compared to soda feldspar which is also supported by the chemical composition by XRF that is K<sub>2</sub>O is 12.09% while

Na<sub>2</sub>O is only 0.20%. So feldspar deposit is, therefore, a potash feldspar. Feldspar promotes vitrification of the porcelain insulator at the end of the sintering process.

#### 3.2 Characterization of fired triaxial porcelain samples

Fig. 2 presents the results of water absorption, apparent porosity, and bulk density respectively for the porcelain samples versus firing temperature. The figure shows that the best values for physical properties for triaxial electrical porcelain are achieved at the firing temperature of 1250 °C. This might be due to the formation of the liquid phase and densification at this firing range. However, the values of water absorption, apparent porosity, and bulk density were observed to decrease at higher firing temperature. This might be due to the expansion of trapped water bubbles inside the porcelain matrix and change in the composition of the glassy phase [1, 3]. The results of the study are in agreement with the works of [1, 3, 13]. The authors reported that water absorption and bulk density increased due to vitrification and densification of the porcelain samples. However, the physical properties were observed to vary due to the decrease of vitrification range and an increase of firing temperature due to the expansion of trapped water bubbles inside the porcelain sample at high firing temperatures. Generally, the variation of the physical properties of the triaxial electrical porcelain might have been caused by the method of production, chemical and mineralogical properties of the raw materials.

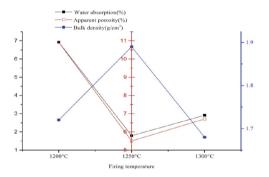


Fig. 2. Physical properties of triaxial electrical porcelain sample versus firing temperature

Fig. 2. Triaxiális szigetelő porcelán fizikai jellemzői az égetési hőmérséklet függvényében

Fig. 3 shows changes in the mechanical strength of triaxial electrical porcelain with firing temperature. The trend shows that the increase of mechanical strength of porcelain sample may be due to increased densification, vitrification and in absence of microcracks. The best mechanical strengths (both bending and compressive strengths) were obtained at 1250 °C. However, the mechanical strengths began to decrease above 1250 °C due to closed pores development and a considerable amount of cracks on the surface of the porcelain samples. The results of the current study are in agreement with the previous studies as reported in the works of Kitouni et al.,[13] and Olupot et al., [1]. The authors have reported that the mechanical strength increases due to increased densification with temperature and tends to decrease due to development of pores at high firing temperature. However, the mechanical strength was found to decrease with

the increase of the firing temperature due expansion of closed pores and microcracks [1]. Hence the mechanical strength of a porcelain sample is strongly dependent on the defects such as pores and cracks [13].

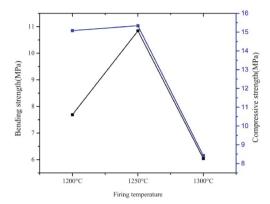


Fig. 3. Variation of mechanical strength of triaxial electrical porcelain sample versus firing temperature

Fig. 3. Triaxiális szigetelő porcelán mechanikai jellemzői az égetési hőmérséklet függvényében

Fig. 4 shows the variation of the dielectric strength of porcelain samples fired at 1200, 1250 and 1300 °C. The trend shows that the dielectric strength increases with an increase in firing temperature and began to decrease with further temperature rise at 1300 °C. The increase of the dielectric strength of electrical porcelains is due to increased vitrification range of the electrical porcelains samples. The results of the current study are also reported by Olupot et al, [1]. The authors evaluated ceramic raw materials from Uganda for electrical porcelain production. The authors obtained the highest dielectric strength of 19kV/mm at 1250 °C. However, above 1250 °C, the samples became more porous due to change in the composition of the glassy phase. The dielectric strength was found to decrease with the increase of firing temperature which affected vitrification range and the dielectric properties of the triaxial electrical porcelain.

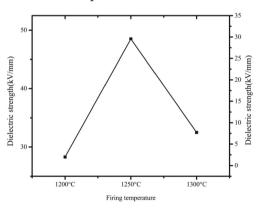


Fig. 4. Variation of dielectric strength of triaxial electrical porcelain sample versus the firing temperature

Fig. 4. Triaxiális szigetelő porcelán dielektromos jellemzői az égetési hőmérséklet függvényében

In *Fig.* 5 the X-diffraction pattern of triaxial electrical porcelain is presented. The diffractogram confirms that the mullite and quartz phases are present in the porcelain insulator. Both phases promote the mechanical and dielectric properties

of the porcelain insulator. However, high peaks of quartz may lead to high amount of glassy phase which may lower the dielectric strength of the porcelain insulators but not the mechanical strength of a porcelain insulator which is affected by microcracks. The high amount glassy phase provides free movement of mobile ions such as Na<sup>+</sup>, K<sup>+</sup>, and Al<sup>3+</sup> which increases the conductivity [17].

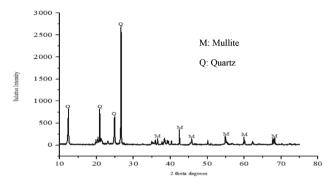


Fig. 5. X-ray diffraction pattern of a porcelain insulator fired at 1250 °C Fig. 5. 1250 °C hőmérsékleten égetett szigetelő porcelán röntgendiffraktogramja

Fig. 6 shows the examination of the surface morphology using the Scanning Electron Microscope (SEM) Model: JEOL JSM-6335F having a resolution of 10nm at 2kV. It was evidenced the densification on the surface of the triaxial electrical porcelain sample after the firing process was completed.

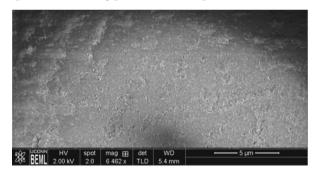


Fig. 6. SEM micrographs showing densification of triaxial electrical porcelain sample at 1250  $^{\circ}\mathrm{C}$ 

Fig. 6. 1250 °C hőmérsékleten égetett triaxiális szigetelő porcelán elektronmikroszkópos felvétele

#### 4. Conclusions

In this research work, the effect of firing temperature on triaxial porcelain samples properties was investigated. At the optimum firing temperature of 1250 °C, the best physical-mechanical and dielectric properties were achieved. However, firing beyond 1250 °C resulted in progressive deterioration of the physical-mechanical and the dielectric properties of the electrical porcelain samples. This might have been caused by the development of microcracks and high content of glassy phase caused by high peaks of quartz. So it is imperative to be aware that, the actual firing temperature and its influence on the triaxial electrical porcelain properties depend on the chemical composition of the materials under study. Therefore, the locally sourced materials need to be evaluated from time to time in order to avoid deviation of the desired triaxial electrical porcelain properties during the firing process.

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# Anisotropic response of the Holzapfel's constitutive model for the lumbar spine considering degenerative conditions

Moustafa Mosbah • Laboratory of numerical and experimental modeling of mechanical phenomena, Department of Mechanical Engineering, University Abd El Hamid Ibn Badis of Mostaganem, Algeria Mohammed Bendoukha • Laboratory of numerical and experimental modeling of mechanical phenomena, Department of Mechanical Engineering, University Abd El Hamid Ibn Badis of Mostaganem, Algeria • bendoukham@vahoo.fr

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#### Abstract

In order to study the risk of disc degeneration under load carriage, an osseo ligamentous FE model of the L4-L5 segment extracted from the validated lumbosacral model L1/S1 was used. Starting from CT images to simplified geometry, many programs and codes were used to simplify the geometry, assemble the mesh and formulate the bony structures of the segment. Abaqus 6.14 was used to pre and post analysis of the FE-model, which included a detailed calibrated model of intervertebral disc (IVD), The range of motion (ROM) curves, the intradiscal pressure, load-strain amount under pure physiological loads were considered for investigation. The model developed in this study considered the holzapfel anisotropy hyperelasticity of the annulus as well as a realistic description of the nucleus geometry, which allowed an improved representation of in vitro and in vivo experimental data during the validation process.

Keywords: intervertebral disc, L4-L5, range of motion finite element model, CT images, intradiscal pressure, Holzapfel anisotropy, hyperelasticity

Kulcsszavak: intervertebrális lemez, L4-L5, mozgásterjedelem végeselemes modellje, CT felvétel, intradiszkális nyomás, Holzapfel anizotrópia, hiperelaszticitás

#### Moustafa MOSBAH

PhD candidate in biomechanics under the supervision of Dr. BENDOUKHA at the laboratory of numerical and experimental modeling of mechanical phenomena. Obtained the licence and Master's degree from the University of Mostaganem in Mechanics.

#### **Mohammed BENDOUKHA**

Research Professor at the University of Mostaganem. Obtained his Engineering degree in Mechanical Engineering from the University of Science and Technology of Oran (USTO). After postgraduation studies he obtained Magister at the USTO IN 1991. Obtained his PhD in tribology speciality in 2010 at the University of Mostaganem, and subsequently his habilitation to direct the research of the same university in 2011. He is active in the field of research at the laboratory of numerical and experimental modeling of mechanical phenomena.

#### 1. Introduction

The disc degeneration is characterized by changes in the morphology and biochemistry of the IVD [16]. These biologic changes of disc degeneration are associated with back pain and other spinal disorders, such as disc herniation, spondylolisthesis, facet arthropathy, and stenosis. FE method is able to simulate a variety of clinical situations in a more way, and has been acting a fully matching partnership based on experimental approaches for spine biomechanics research [12]. The FE-models are widely used to provide an assessment of spine with spinal instrumentation and assist in the design, development and optimization of that spinal instrumentation. Advanced to other methods, these FE-models can predict the changes of stresses and strains in the IVD, vertebrae and ligaments and very comprehensive ROM data [8, 20]. Our main objective is to review the most recent computational studies in the application of FE models that report the issue of research better understanding of low back pain and eventually treat the spinal disorders. Little experiments were performed on intact motion segments (Miller 1986, Lin 1978, Hirsch & Nachemson 1954) and some on FSUs without posterior elements [2, 10, 13]. In the compression load case, the data from Brown 1957 is slightly lower than the generated values and the other experimental data. Some FE-models simulate the effects of degeneration [16], by simply removing some elements from the annulus fibrosus. Where others [18, 19, 20] disclose the influence of geometrical, mechanical, or poromechanical parameters on IVD behavior without showing change in mechanical properties of degenerated IVD. More recently, recent poroelastic finite element model of the lumbar spine was developed to assess spinal response during physiological functions and behavior of degenerative disc [15, 16].

#### 2. Methods and materials

A three-dimensional finite element (FE) model of the L4-5 FSU segment (Fig. 2) by modifying the mechanical properties of the intervertebral disc constituents [3]. All the bony structures including cortical and trabecular bone, whereas facet cartilage layers, annulus ground substance, nucleus pulposus and cartilage endplates were modeled by using solid hexahedral elements. The nucleus pulposus composed 43% of the whole volume of the disc. The symmetric FE-model of the human lumbar disc is developed to investigate the optimized properties required to mimic the intact by matching model predictions to experimental results. Facet joints simulated by a cartilaginous layer extruded in Abaqus along the vectors normal to the inferior face of the superior articular process to the central of thickness of 0.4 mm were modeled to be multi-linear elastic in compression [19] by surfaces-to surface contact with softened contact in the normal direction. [20] An initial typically gap of 0.2 mm was specified as reported [20]. The seven major ligaments were modeled as three dimensional, 4 noded Quad elements (T3D2) and allocated were as piecewise linear functions in stress-strain relationship [7, 8] as shown in *Table*. 1. This formulation allows simulation of changing ligament characteristics with different stress at a different level as a function of stress-strain.

Part	Structure	Young's modulus E (MPa)			Poisson's ratio υ		Element type	Reference
	Trabecular bone	100			0	.2		[8, 14]
	Cortical bone	12,000		0.3			[8]	
	Posterior element	3,500			0.25		C3D8	[8, 14]
Vertebrae	Cartilaginous End plate	23.8			0.4			[20]
	Facet cartilage	11 0.4					[5, 20]	
	Facet contact	Nonlinear soft contact					Contact	[21,22]
Annulus Fibrosus	Healthy	<b>C</b> <sub>10</sub>	C <sub>20</sub>	K1	K2	kappa		
		1.5715	9.2044	12.2	39.7	0.113	C3D8	[22]
	Degenerated	1	8.6	18.56	82.31	0.226		
Nucleus Pulposus	Healthy	Incompressible fluid					F2D4	[6, 19, 21]
	Degenerated	compressible fluid					F3D4	
Ligaments		Nonlinear stress-strain curve S				S4R	[7, 8, 21]	

Table 1. Material properties used in FE Model

The entire model consists of approximately 424 S4R shell elements and 33360 hexahedral elements C3D8

#### 2.1 The intervertebral disc (IVD)

The intervertebral disc was modeled using two separate structure (*Figs. 2* and 5).

#### 2.2 The Nucleus Pulposus (NP)

The Nucleus Pulposus (NP) exhibits a gelatinous core and, therein describes the mutually coupled behavior of both solid deformation and viscoelastic characteristics of fluid flow [7]. An initial hydrostatic pressure of 0.1 MPa was prompted in the fluid filled cavity in the nucleus pulposus to simulate unloaded motion segments (*Fig. 2*) [17]. The healthy nucleus pulposus was modeled as incompressible fluid while the degenerated nucleus was defined as compressible fluid [10]

#### 2.3 The Annulus Fibrosus (AF)

The Annulus Fibrosus (AF) was modeled as an anisotropic continuum structure, with deformation tensor [8], and the material coefficients  $C_{10}$ ,  $C_{20}$ ,  $k_1$ ,  $k_2$  and  $\kappa$  were based on biaxial tension tests performed on healthy and degenerated annuli fibrosi [9]. The progression of degeneration was simulated using three model variants:

$$W_{matrix} = C_{10} (I_1 - 3) + C_{20} (I_1 - 3)^2$$
 (1)

$$W_{fibers} = \frac{k_1}{2k_2} \sum_{\alpha=4,6} \left( e^{k_2 \left[ (1-\kappa)(l_1-3) + \kappa(l_\alpha-1) \right]^2} - 1 \right)$$
 (2)

The human healthy and degenerated intervertebral disc.were modeled using three structures:

- (a) Healthy intervertebral disc (grade I) defined by healthy annulus and healthy nucleus;
- (b) Moderate degeneration disc (grade III) defined by healthy annulus and degenerated nucleus;
- (c) Severe degeneration disc (grade IV) defined by degenerated annulus and degenerated nucleus. The grading is based on the Thompson degeneration scale *Fig. 1*.

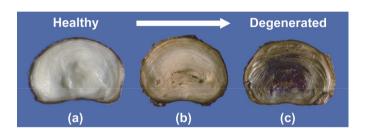


Fig. 1. (a) Healthy intervertebral discs; (b) moderate degeneration; (c) severe degeneration [23]

 ábra (a) Egészséges intervertebrális lemez; (b) közepes mértékű károsodás; (c) súlvos károsodás [23]

#### 3. Model formulation

The algorithms proposed in the present study is implemented into Abaqus as a User Material UMAT code embedded in an Python and Intel Fortran Compiler environment to simulate the biomechanical behavior of Annulus. This numerical approach consists on Holzapfel's hyperelastic constitutive models of the Annulus and a constitutive model of the seven major Ligaments, which were implemented in Abaqus via subroutines. The coefficients employed in all three conditions are listed in Table 1. While the material properties and in situ nuclear pressure were altered, osteophytes, local tears or similar macroscopic abnormalities were not modeled. Rather than attempting to simulate these defects of an independent individual and random nature, the focus was kept on the effects of measured changes in tissue behavior.

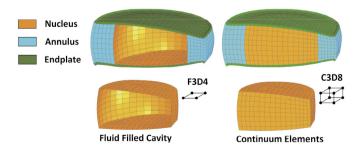


Fig. 2. Finite Element Model of the Intervertebral Disc IVD 2. ábra Intervertebrális lemez IVD végeselemes modellje

<sup>1.</sup> táblázat A végeselemes modellben használt anyagjellemzők

#### 4. Loading and Boundary Conditions

The IVD model comprises the intervertebral disc and the adjacent vertebral bodies. The lower vertebral body was rigidly fixed. Pure unconstrained axial compressive of 2000 N was applied to the upper vertebral body to the reference point which coupled all points on upper surface of the disc. The resulting stress-strain distribution at the center of the disc were estimate and the intradiscal Pressure IDP was predicted using the cavity point. Theoretically speaking, we used the concept of nucleus pulposus pressure measurement; by means of in vitro experiments on vertebra-disc-vertebra preparation whither, we found that the incompressible fluid pressure within the nucleus is directly related to the axial compression applied to the disc. The lower portion of the L5 vertebral body and its inferior facets were rigidly fixed. The load was applied through a reference node constrained to the upper surface of the L4 vertebrae using Abaqus Coupling Constraints elements (CCE). The L4/L5 Functional Spinal Unit (FSU) move in six different directions (six degrees of freedom (DOF)). A pure bending moment was incrementally increased 0, 1, 2.5, 5, 7.5, 10 Nm and applied to the model in all three planes of motion.

#### 5. Results

#### 5.1. ROM results

The finite element model predicted an increase in hydrostatic stress in the middle regions of the annulus by nearly seven fold. The intradiscal pressure IDP is considered to be very significant component to approximate internal stress behavior of the intervertebral disc. The in vitro measurements of the nucleus pressure in human intervertebral discs have shown proportion between IDP pressure and applied Load on the superior surface motion (ROM) in all loading directions except for lateral bending comparing to experimental.

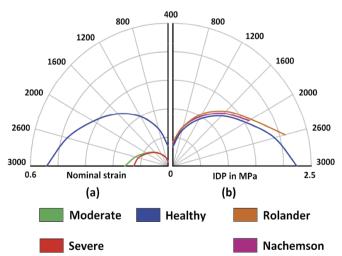


Fig. 3. (a) Comparison of intradiscal pressure (IDP) of the Healthy model with experimental results (b) Comparison of nominal strain of fibers against the Load (N) of Healthy, Moderate and Severe models

 ábra (a) Kísérleti intradiszkális nyomás (IDP) összehasonlítása az egészséges modell alapján számított értékkel; (b) Szálmegnyúlás és terhelőerő összehasonlítása egészséges, közepes és súlyos modellben The greatest changes between Healthy and Moderate and Severe models were predicted in axial rotation by 30 % and 36 %. In lateral bending, a 27% increase in ROM was predicted in the Severe scenario (with respect to the Healthy condition), while the change in the Moderate scenario was approximately 32% (*Fig. 4*).

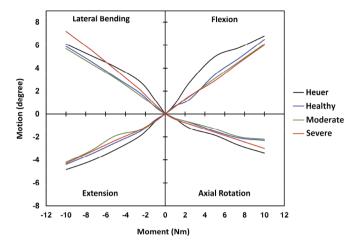


Fig. 4. ROM of L4-L5 FSU 4. ábra L4-L5 funkcionális szegmentális egység (FSU) mozgásterjedelme (ROM)

#### 5.2. Stress and strain results

The analysis of the stress results allowed us to prove the most loaded region. As a most critical region for of spinal diseases, the presence of disc degeneration under physiological load steer to the alteration of the spine comportment and the pain function. The results for the body functions (Figs. 5-6) indicate that the superior values of the stresses were situated at posterior region which reaches stress of 2.9 MPa for healthy disc and 2.4 MPa for the degenerated disc; as expected, the results highlight that the most risky load condition corresponds to posterior side. For the degenerated nucleus the results presented in Moderate degeneration a decreased stress according to strain decreasing comparing to Healthy IVD. Furthermore, the magnitude of stress predictions were reduced in the anterior region of the disc while they increased in the posterior region as the simulated degeneration progressed. In all cases, the highest stresses were concentrated in the posterior region of the disc in extension loading. The highest strain were concentrated in the posterolateral region of the disc in lateral bending loading as degeneration progressed (Healthy-Moderate-Severe). The compressive strain prediction in the annulus increased in anterior extremities in extension and in the posterior extremities in flexion Fig. 5.

#### 6. Discussion

The aim of this work was to construct an accurate FE-model to characterize the mechanical behavior of healthy and degenerated IVD. This model takes into account the IVD nature and also the clear preferential orientation of the collagen fibers in the annulus. The reduction in nonlinearity behavior with degeneration could suggest a diminished compaction effect of the degenerate tissues at large deformations which could be related to structural changes in annulus. The obtained

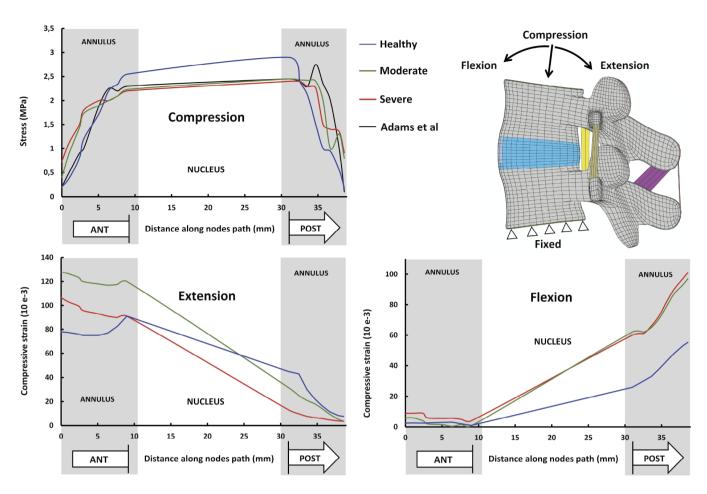


Fig. 5. Axial stress distribution and compressive strain predictions obtained from FE Models in the mid-height plane of the IVD from posterior to anterior midline under Compression (2000 N), Flexion and Extension (10 Nm).

5. ábra Tengelyírányú feszültségeloszlás és összenyomódás a végeselemes modell alapján az intervertebrális lemez középsíkjában, a posteriorból az anteriorba, nyomás (2000 N), hajlítás és megnyúlás (10 Nm) hatására

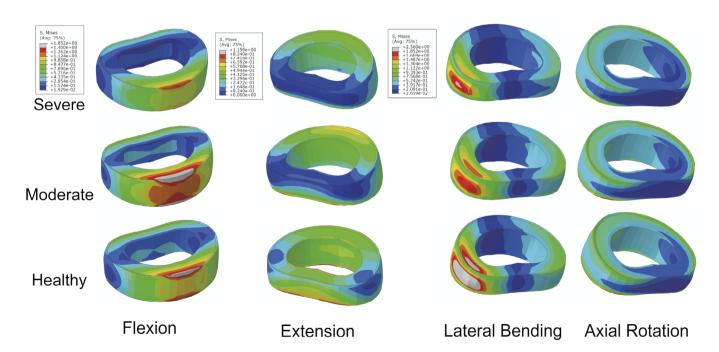


Fig. 6. Von-Mises Stress distribution obtained from FE Models: Healthy, Moderate, Severe 6. ábra Von-Mises feszültségeloszlás a végeselemes modell alapján egészséges, közepes és súlyos modellben

results are also in agreement with the experimental behavior [9, 20]. Experimental results, showed an increase of the elastic modulus in the toe part, thus there is a big role of fibers therefore when the fibers act there is no additional stiffening. In this analysis it has been obtained that the degeneration affects the biomechanics of this FSU; in particular an increase of the ROM in extension and partial decrease in flexion and lateral bending has been noticed.

#### 7. Conclusions

In conclusion, the developed FE-model is capable of generating the mechanical behavior of normal and degenerated NP tissue with a favorable approximation. But on the degeneration of the other segments is a very complex procedure which should be identified in the degeneration of the AF tissue with accurate mechanical properties. Furthermore, the current work provides a qualitative analysis of the influence of single level disc degeneration on the mechanics of the segments under flexion/extension, lateral bending and axial rotation moments. It has been seen that degeneration modified the degree of motion and loading of the degenerated level. These changes could increase the risk of progression of degeneration to the nearest segments of the spine. The results obtained with the FE model seem to be in good agreement with invitro data as showed in Figs. 2 to 4. Additionally, nonlinear analytical functions used in the IVD ligaments facet joint may be used as input data to perform more accurate computational simulations of the full lumbosacral spine.

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#### Preparation of new nanoorganoclays from Hexadecylamine, Tetradecylamine and Chalcone with Montmorillonite using ion exchange processes

MANAR GHYATH ABD-ALMUTALIB AL-MOSAWY • Department of Chemistry, Faculty of Science, University of Kufa

EMAD ABBAS JAFFAR AL-MULLA - College of Health and Medical Techniques,

Al-Furat Al-Awsat Technical University ■ almullaemad@gmail.com

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#### **Abstract**

This study reports the effect of three new organic cations including hexadecylamine (HDA), tetradecylamine (TDA) and 1-(4-aminophenyl)-3-(4-chlorophenyl)prop-2-en-1-one, chalcone (CH) on the basal spacing of the montmorillonite clay to MMT modification. Fourier Transform infrared spectroscopy (FTIR) was used to evaluate the incorporation of these cations in the MMT. X-ray diffraction technique was utilized to indicate the basal spacing of the treated clay as a measure of susceptibility of new organoclays. The FTIR and XRD results shown that the three new organic cations were successfully incorporated in the montmorillonite clay.

Keywords: sodium montmorillonite, modification, surfactant, organic cation

Kulcsszavak: nátrium montmorillonit, modifikálás, felületaktív anyag, szerves kation

#### Manar Ghvath ABD-ALMUTALIB AL-MOSAWY

is a PhD student at Department of Chemistry,
Faculty of Science, University of Kufa, Iraq;
She has received her MSc from Faculty of
Education for Girls, University of Kufa, Iraq in
2010. Main fields of interest: organic synthesis,
nanomaterials, biopolymer nano-composites. She
has published many scientific papers in citation
and non-citation indexed journals.

#### **Emad Abbas Jaffar AL-MULLA**

is an Asst. Prof., College of Health and Medical Techniques, Al-Furat Al-Awsat Technical University, Iraq; He has received his PhD from University Putra Malaysia, Malaysia in 2010, He was a Post Doctoral researcher at the same University from April 2012 to April 2013; main fields of interest: bioorganic synthesis, nanomaterials, biopolymer nano-composites; He has more than 50 papers in Scopus and ISI journals; his H-Index = 14 according to Scopus database.

#### 1. Introduction

Sodium montmorillonite (MMT) is a naturally occurring clay mineral characterized by relatively low negative charge of its aluminosilicate layers as a result of octahedral substitutions of Al by Mg (II), Fe (II) or other divalent metal ions in a central octahedral sheet (Meleshyn, Bunnenberg, 2006). This negative charge is compensated by inorganic cations such as Na+, K+, Ca<sup>2+</sup>and Mg<sup>2+</sup> in the interlayer space while these inorganic cations can be exchanged by other cations (Chen et al., 2008). However, cation-exchange reaction have been traditionally exploited as an effective method to replace these inorganic ions with organic cationic surfactant molecules, which intercalate into the clay gallery, resulting in expansion of the interlayer spacing and leading to an increase in the basal spacing. These organic cations render the surface of the clay mineral hydrophobic, leading to the increase of the clay wettability and providing favourable interactions with organic molecules. Both organic-modified and unmodified clays have been used for different industrial applications such as rheological additives, thickeners in coating products glues, plastisols, drilling fluids and cosmetics (Jaynes, Boyd, 1991). Recently, clays were used in the field of materials science such as solid phase polymeric nanocomposites. In the 1990's, the use of organically modified clays in polymer-clay nanocomposites has attracted researchers into this area of materials science and technology (Zhang, Wilkie, 2003). The organic modification of clay minerals leads to a decrease in surface energy making clays compatible with polymers. The surface energy of clay minerals and polymer can be determined from contact angle measurements (Maiti, Bhowmick, 2005; Stretz et al., 2005). Studies reported that melt processed nylon 6-clay nanocomposites was prepared using organoclay. Based on X-ray diffraction analysis, various arrangement of alkyl chains in organoclays HDA proposed by Lagaly (1986). Novel organo-montmorillonites have been synthesized and characterized using different ammonium compounds (Rajkrian et al., 2008; Arroyo et al., 2003). Series of anion-cation surfactants modified organoclays were prepared by incorporating of cationic surfactant hexadecyltrimethyl ammonium bromide and anionic surfactant, sodium dodecylsulfonate to montmorillonite (Chen et al., 2008; Rathanawan et al., 2001). In this study, three different ammonium; HDA, TDA and CH were used to modify the compatibility of montmorillonite clay with polymer. These organo-montmorillonites can be used in both medical and industrial applications as polymer nanocomposite.

#### 2. Experimental

#### 2.1. Materials

Sodium montmorillonite, Hexadecylamine and Tetradecylamine obtained from Sigma Aldrich, Germany were used. Hydrochloric acid was obtained from J.T. Baker, USA. p-aminoacetophenone and p-chlorobenzaldehyde were obtained from Fluka. Sodium hydroxide was obtained from B.D.H.

#### 2.2. Preparation of CH (Sadiq et al., 2015)

0.5 g (1 mmol) of p-aminoacetophenone with 1 mmol p-chlorobenzaldehyde were mixed, then 0.5 mL of 10% NaOH aqueous solution and 5 mL of 99% ethanol were added to the mixture. The mixture was stirred at room temperature for 4 hrs. The crude mixture was poured in to ice water and then

acidified the product with 10% HCl solution. The solid formed was filtered then washed with ethanol and water at a ratio of 10:5 mL. Recrystallization from 99% ethanol afforded (67% yield, yellow solid, m.p 160–165 °C).

#### 2.3. Preparation of organoclays (OMMTs)

Organoclay was prepared with a cationic exchange process, where Na in the MMT was exchanged with the alkylammonium ion, which was prepared applying the procedure reported by Al-Mulla et al. (2011) in an aqueous solution. Sodium montmorillonite (Na-MMT) 4.00 g was stirred vigorously in 600 mL of hot distilled water for 1 h to form a clay suspension. 4.50 g of HDA, TDA and CH, which HDA been dissolved separately in 400 mL of hot water with 16.00 mL of concentrated hydrochloric acid were added into the clay suspension. After being stirred vigorously for 1 h at 80 °C, the organoclay suspension was filtered and washed with distilled water until no chloride was detected with a 1.0 M silver nitrate solution. It was then dried at 60 °C for 72 h. The dried organoclay was ground until the particle size was 100µm (Al-Mulla et al., 2010a; 2010b). Structure of HDA, TDA and CH is shown in Fig. 1.

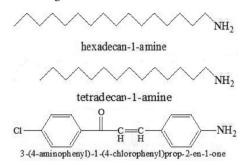


Fig. 1. Structures of the three organic cations 1. ábra A három szerves kation szerkezete

#### 2.4. Characterization

Organoclays were characterized using two different techniques including X-ray diffraction and Fourier Transform Infrared Spectroscopy.

#### 2.4.1. X-ray diffraction (XRD) analysis

X-ray diffraction (XRD) study was carried out using a Shimadzu XRD 6000 diffractometer with Cu K radiation (k = 0.15406 nm). The diffractogram was scanned in the ranges from 2° to 10° at a scan rate of 1°/min.

#### 2.4.2. Fourier Transform Infrared (FTIR) spectroscopy

The FTIR spectra of the blend samples were recorded by the FTIR spectrophotometer (Perkin Elmer FT-IR-Spectrum BX, USA) using KBr disc technique.

#### 3. Results and discussion

#### 3.1. XRD analysis

The alkyl ammonium cation exchange enable the conversion of the hydrophilic interior clay surface into the hydrophobic surface and consequently increase the layer distance as well

(Phua et al., 2013). Na-MMT was surface treated with HDA, TDA and CH as intercalation agents through cation exchange process. The cationic head groups of the intercalation agent molecule would preferentially reside at the layer surface and the tail of the compound will radiate away from the surface. The presence of these chains in the galleries makes the originally hydrophilic silicate to organophilic and thus, increase the layer-to-layer spacing of Na-MMT (Al-Mulla et al., 2009). The obtained HDA-MMT, TDA-MMT and CH-MMT were studied using XRD measurements in the 2Θ ranges from 2° to 10°. Na-MMT shows a d001 diffraction peak at  $2\Theta = 6.91^{\circ}$  which, assigns to the interlayer distance of the natural montmorillonite with a basal spacing of 1.27 nm (Agag, Takeichi, 2001). Fig. 2 reveals XRD the basal spacing (d001 value) increase from 1.27, 1.51, 1.57 and 1.78 nm for Na-MMT, HDA-MMT, TDA-MMT and CH -MMT, respectively.

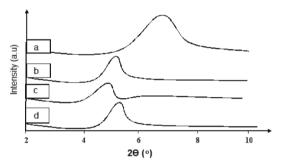


Fig. 2. The XRD patterns of (a) Na-MMT, (b) HDA-MMT, (c) TDA-MMT and (d) CH-MMT

2. ábra Röntgendiffraktogramok; (a) Na-MMT, (b) HDA-MMT, (c) TDA-MMT és

HDA, TDA, CH have been incorporated into the Na-MMT galleries to products organoclays. The basal spacings and angles of these organoclays were shown Table 1. Thus, XRD result indicate that organoclays are successfully intercalated into the silicate layer.

Sample	Exchanged cation	20 (°)	d(001) spacing (nm)
Montmorillonite	Na <sup>+</sup>	6.91	1.27
HDA	C <sub>16</sub> H <sub>32</sub> NH <sub>3</sub> <sup>+</sup> (HDA <sup>+</sup> )	5.64	1.57
TDA	C <sub>14</sub> H <sub>28</sub> NH <sub>3</sub> <sup>+</sup> (TDA <sup>+</sup> )	4.96	1.78
СН	C <sub>15</sub> H <sub>9</sub> CIO NH <sub>3</sub> <sup>+</sup> (CH <sup>+</sup> )	5.86	1.51

Table 1. Diffraction angle and basal spacing of montmorillonite and modified montmorillonite with different organic cations

1. táblázat Diffrakciós szög és rácssík távolság; montmorillonit és különböző szerves kationokkal modifikált montmorillonit

#### 3.2. FTIR spectroscopy

FTIR spectra are a useful technique to verify the presence of HDA<sup>+</sup>, TDA<sup>+</sup> and CH<sup>+</sup> in the clay. *Fig. 3* shows the FTIR spectra of Na-MMT, pure HDA and HDA-MMT, *Fig. 4* shows the FTIR spectra of Na-MMT, pure TDA and TDA-MMT, *Fig. 5* shows the FTIR spectra of Na-MMT, pure CH and CH-MMT. The infrared spectrum of the Na-MMT shows two peaks, which correspond to Si-O stretching at 1033 cm<sup>-1</sup> and interlayer water deformation vibration at 1632 cm<sup>-1</sup> (Guo *et al.*, 2006). The band at 3625 cm<sup>-1</sup> results from the O-H

stretching vibration and the band at 3043 cm<sup>-1</sup> results from the CH= stretching vibration back to compound CH. The peaks observed at 2924-2854 cm<sup>-1</sup>, 2926-2852 cm<sup>-1</sup> and 2850-2920 cm<sup>-1</sup> correspond to the presence of the C-H asymmetric and symmetric stretching vibration for HDA-MMT, TDA-MMT and CH-MMT, respectively. The IR spectrum reveals a weak incorporate of the compound CH in the clay (*Fig. 5.c*).

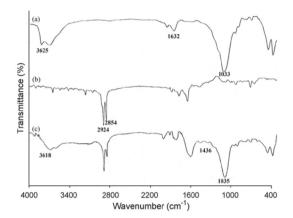


Fig. 3. FTIR spectra of (a) Na-MMT, (b) pure HDA and (c) HDA-MMT 3. ábra FTIR spektrumok; (a) Na-MMT, (b) tiszta HDA és (c) HDA-MMT

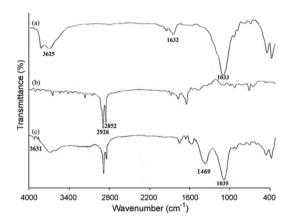


Fig. 4. FTIR spectra of (a) Na-MMT, (b) pure TDA and (c) TDA-MMT 4. ábra FTIR spektrumok; (a) Na-MMT, (b) tiszta TDA és (c) TDA-MMT

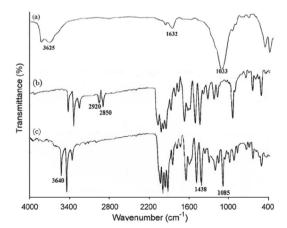


Fig. 5. FTIR spectra of (a) Na-MMT, (b) pure CH and (c) CH-MMT 5. ábra FTIR spektrumok; (a) Na-MMT, (b) tiszta CH és (c) CH-MMT

In the spectra of HDA-MMT, TDA-MMT and CH-MMT, the absorption bands 3618, 3631 and 3640 cm<sup>-1</sup> indicate the

presence of free O-H stretching and at 1035, 1033 and 1085 cm<sup>-1</sup> correspond to C-N stretching, respectively (Ramachandran *et al.*, 2007). In addition to the bands of the original Na-MMT the band at 1436, 1469 and 1438 cm<sup>-1</sup> suggest the existence of the ammonium ion. Therefore, these indicate that HDA<sup>+</sup>, TDA<sup>+</sup> and CH<sup>+</sup> were intercalated in the silicate layers. This suggests that HDA, TDA and CH are sorbed into the silicate layers of Na-MMT.

#### 4. Conclusions

Three new ammonium cations including HDA, TDA and CH were used to modify montmorillonite clay in an attempt to create susceptible clay to polymers. The organoclays were characterized using FTIR and XRD based on results of this study. Based on results of this study, the following conclusions can be drawn:

- These ammonium cations can be successfully incorporated in the montmorillonite clay.
- The basal spacing of the montmorillonite clay increases as a result of incorporating HDA, TDA and CH.
- The IR spectrum showed that there is a weak incorporate of the compound CH in the clay.
- The new organoclay can be used to produce polymer nanocomposites.

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Abd-Almutalib Al-Mosawy, Manar Ghyath – Al-Mulla, Emad Abbas Jaffar: Preparation of new nanoorganoclays from Hexadecylamine, Tetradecylamine and Chalcone with Montmorillonite using ion exchange processes

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- Crystallography, mineralogy and modelling
- · Environment and geological processes
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## Mass transfer of aluminum film from the surface of zeolite on the cathode

N. N. LEBEDEVA • Institute for Problems, Baku State University • nnlebedeva@gmail.com

V. I. ORBUKH • Institute for Problems, Baku State University • orbukh@ramblero.ru

G. M. EYVAZOVA • Institute for Problems, Baku State University • eygoncha@mail.com

N. H. DARVISHOV • Institute for Problems, Baku State University • n\_darvishov@mail.ru

CH. G. AKHUNDOV • Institute for Problems, Baku State University • ch.axundov@mail.ru

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#### **Abstract**

In the present study, the phenomenon of mass transfer of an aluminum film deposited on the zeolite surface onto a clamping electrode was observed. The proposed four-layer model explains the very fact of mass transfer (inhomogeneous over the cross section of the structure) in the absence of anomalously large electric fields at the cathode.

Keywords: zeolite, mass transfer, clinoptilolite, zeolite pores, zeolite framework Kulcsszavak: zeolit, tömegáram, klinoptilolit, zeolit pórusok, zeolit vázszerkezet

#### **Nelli N. LEBEDEVA**

Graduated at Baku State University in 1960 with a specialty in Physics of Semiconductors and Dielectrics. Field of interests: physical phenomena inhomogeneous system 1) ferroelectrics-semiconductor; 2) photoconductors-plasma of a gas discharge; 3) dielectrics (zeolites) -conductors (graphite, silicon Ag Cu)

#### Vladimir I. ORBUKH

Graduated at Baku State University in 1971
with a specialty in Physics of Semiconductors
and Dielectrics. Field of interests: theory
of physical phenomena inhomogeneous
system 1) ferroelectrics-semiconductor; 2)
photoconductors- plasma of a gas discharge;
3) dielectrics (zeolites) –conductors (graphite,
silicon, Ag, Cu)

#### Goncha M. EYVAZOVA

Graduated at Baku State University (BSU) department of Physics in 1974. Completed her PhD degree under the supervision of Prof. A. M.

Mamedov at BSU. Now she is leading scientific researcher in Nano Center, Department of Physics, BSU. Her current research interests include the synthesis of nanostructured metal chalcogenides, various nanoparticles, and investigation of optical and electrical properties.

#### Namig H. DARVISHOV

Graduated at Baku State University in 1974 with a specialty in Physics of Semiconductors and Dielectrics. Field of interests: 1) optical; 2) ferroelectrics-semiconductor; 3) photoconductors-plasma

#### Chingiz G. AKHUNDOV

Graduated at Baku State University in 1976 with a specialty in Physics of Semiconductors and Dielectrics. Field of interests: physical phenomena inhomogeneous system 1) ferroelectrics-semiconductor; 2) photoconductors-plasma of a gas discharge; 3) dielectrics (zeolites) - conductors (graphite, silicon, Ag, Cu)

Analysis of data on the structure and properties allows us to consider this zeolite not only as a promising sorbent, but also as an object on which it is possible to investigate poroemission of electrons, electron multiplication and gas discharge in pores [3], dielectric and electrical properties [4]. If the sorption properties of the zeolite are determined by the size and configuration of the pores, then its electrical properties are determined by the composition of the substance in the pore space [5]. On the basis of X-ray and spectral chemical analysis, we established that the natural zeolite we are studying belongs to high-silica zeolites of the clinoptilolite type [6]. The content of the channels, which is an off-frame subsystem, is positively charged Na +, K +, Mg +, Ca + ions that compensate for the negative charge of the framework, as well as a large number of molecules of H<sub>2</sub>O-coordination water.

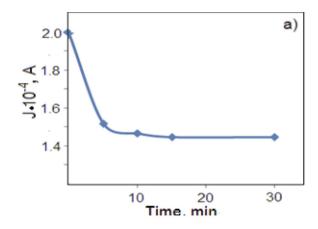
Water plays an important role in ensuring the stability of the clinoptilolite framework and, as suggested, leads to an increase in the mobility of ions in the pore space. Loosely bound cations and water molecules in pores of zeolites lead to high values of the dielectric constant at low frequencies and a significant dependence of the electrophysical properties of seolites on frequency and temperature. The ability of cations to diffuse through a large open zeolite structure makes it possible to obtain high ionic conductivity in zeolites. In recent years, many attempts have been made to investigate the factors that control ion transport in an external electric field [7]. These

#### 1. Introduction

Zeolites are aqueous aluminosilicates, the infinite aluminosilicate frame of which is formed by the joint of the common vertices of  $[SiO_4]^4$  and  $[AlO_4]^5$  tetrahedra, having interconnected cavities occupied by large ions and water molecules [1]. The loosely bound cations and water molecules in the pores of the zeolites are characterized by considerable mobility, which allows ion exchange and reversibility of the dehydration without affecting the aluminosilicate rigid framework. Due to this property zeolites have extremely wide scope of use in industry and agriculture.

Currently, more than 45 types of natural zeolites are known, of which the most common are clinoptilolite, heylandite, phillipsite, lomonite, mordenite, erionite, chabazite, ferrierite, analcime. Clinoptilolite is defined as a series of zeolitic minerals having a clear structural topology of heylandite (HEU) and a Si/Al ratio of 4.0.

The structural topology of the tetrahedral HEU lattice has been extensively studied and has a C2/m symmetry with oblate channels bounded by ten-membered channels (7.5×3.1A) and eight-membered tetrahedral rings (4.6×3.6 A) parallel to the C axis. The additional eight-membered circular channels (4.7×2.8 A) are parallel [100] and [102] and intersect with the preceding channels within [010], forming a system of two-dimensional parallel channels to [010], which are responsible for the layered structure [1, 2].



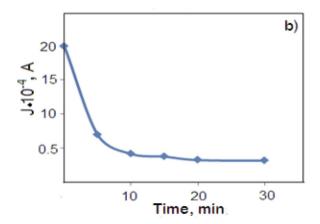


Fig. 1. The time dependence of the current at a negative (a) and a positive (b) electrode potential

1. ábra Áramerősség időfüggése (a) negatív és
(b) pozitív elektródpotenciál esetén

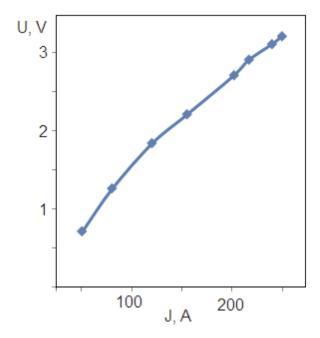


Fig. 2. The current-voltage characteristic of a plate with aluminum sputtered on one side 2. ábra Áramerősség-feszültség karakterisztika egy oldalán alumíniummal bevont lemez esetén

studies are focused mainly on the effect of temperature, the degree of hydration, the nature of the metallic cations on the conductivity, measured at alternating voltage.

In the present work, a mass transfer of a metal film deposited on the surface of a plate of natural zeolite to a negatively charged electrode is observed when a sufficiently weak constant electric field is applied.

The processes of mass transfer in the electrode-sample contact take place during welding, when the welding current is turned on, as the pre-electrode region of the heating is heated, the plastic deformation resistance decreases, respectively, the actual contact areas expand and the processes of mutual diffusion of metals develop. The mass transfer rate is controlled by the temperature in contact, the residence time of metals at elevated temperatures, the state of the surfaces of parts and electrodes, and the physical properties of the metals to be welded

Another mechanism of, mass transfer is field desorption and field ionization [8]. The desorption theory and field evaporation consider the case of emission of positive ions in a strong electric field. Theoretical estimates of the desorption intensity for various metals are in the range 1428-288 Mv/sm.

All the above conditions are not implemented in our case.

Materials: The natural zeolite clinoptilolite was used as an object of study: syngonia-monoclinic, space symmetry group C2/m; the unit cell parameters: a = 1.761, b = 1.780, c = 0.741 nm,  $\beta$  = 115.2°. For the experiment from a bulk monoblock of natural zeolite clinoptilolite, the chemical composition of which, confirmed by the X-ray analysis, contained: Al $_2$ O $_3$ -11.36, SiO $_2$ -67.84, Na $_2$ O - 1.25, MgO - 0.49, P $_2$ O $_5$ - 0.11, SO $_3$ -0.03, K $_2$ O - 3.01, CaO - 0.29, TiO $_2$ - 0.08, MnO - 0.078, Fe $_2$ O $_3$ -1.19, KJ - 11.64, the plates with the size (15×10×2)  $10^{-3}$  were cut.

Thermal sputtering of aluminum on the ground surface (15×10) of a  $10^{-3}$ sm zeolite plate was carried out in VUP in a vacuum of  $10^{-3}$ torr.

#### 2. Measurements

The plate with aluminum deposited on one side was placed in a cassette, where it was clamped between two polished copper electrodes. The electrodes were supplied with a constant voltage (U = 250V). The time dependence of the current was measured when the electrode pressed to aluminum was a cathode (*Fig. 1.a*) and an anode (*Fig. 1.b*).

In both polarities, the currents slowly decrease with time and after 30 minutes they reach saturation. After achieving a stable current  $(10^{-4}\text{A})$ , the I-V characteristic was measured (*Fig. 2*).

#### 3. Discussion

It was found that as a result of the current passing through the zeolite plate, the aluminum film on its surface can pass to the electrode. Such an aluminum mass transfer occurs only when the electrode is a cathode. It is important to note that the transfer of aluminum to the clamping electrode is not carried out over the entire area of the film. To explain the observed phenomenon, we will start from the four-layer model: a zeolite plate-an aluminum film on it-a thin air gap-a clamping electrode. The conductivity of the zeolite is carried out by positive ions moving in the pores of zeolite. In our model, the zeolite ions, moving toward the cathode, fall on the surface of the aluminum film, where they are neutralized due to the metallic conductivity of aluminum. Simultaneously, on the opposite side of the film (from the side of the air gap) the same positive charge arises. In other words, the charge of positive ions of the zeolite that have approached the aluminum film is converted to the same positive charge of aluminum ions on the opposite side of the film. The next stage is the recombination of these positive aluminum ions with electrons from the clamping cathode. This can happen in two ways: either electrons from the clamping electrode will transfer to the surface of the aluminum film, or positive ions of aluminum will transfer to the clamping electrode. The first of these mechanisms is more traditional and is realized when the metal film is a structure close to a single crystal, which provides a high binding energy of the ion with the film. The second mechanism is realized in the case of strongly amorphous films, which are disordered agglomerates with low binding energy of the ion with film. Such films are formed in those cases when the film is deposited on a non-crystalline (and, in general, unordered) surface. In our case, this is exactly the case, as can be seen from Fig. 3, which presents a photograph of the surface of the zeolite plate on which aluminum was deposited.

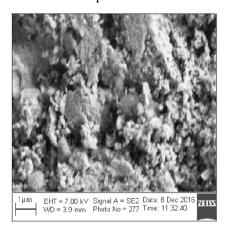


Fig. 3. Morphology of the surface of the zeolite plate 3. ábra Zeolit lemez felületi morfológiája

#### 4. Conclusions

In this paper we show the possibility of mass transfer in the absence of anomalously large electric fields. This is possible due to the large positive charge on the metal amorphous film, which arise, from the neutralization of positive ions of the zeolite.

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Workshop

## Qualitative and Quantitative Analysis of Clays and Clay Minerals

Inst. of Geography und Geology, University of Greifswald, Germany

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organized by Georg Grathoff and Laurence Warr (Grathoff@uni-greifswald.de)



## Numerical study of building materials filled by PCM for thermal energy storage

Youcef Oussama SOUCI • Laboratory of Numerical and Experimental Modelling of Mechanical Phenomena, University Abd El Hamid Ibn Badis of Mostaganem, Algeria • souci\_youcef@yahoo.fr

**SAMIR HOUAT •** Laboratory of Numerical and Experimental Modelling of Mechanical Phenomena, University Abd El Hamid Ibn Badis of Mostaganem, Algeria

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#### **Abstract**

Phase change material (PCMs) is used to equilibrate the temperature fluctuation and to store energy in several practical application areas such as electronics, automobile industry and also buildings. In this paper, an example of the application of phase change material in the building application is investigated. The physical problem of phase change is solved by using a numerical study based on the finite element method. The PCM selected in this study, is incorporated directly within the building materials for improving its capacity of thermal storage. Two different building materials are selected according to their usual applying as construction materials for residential buildings. The numerical results have been checked and validated with an experimental model and the error is estimated.

The results reveal the potential of PCM in the enhancement on the thermal inertia of the wall and effectively reduced the fluctuation of the interior surface temperature. In addition, with the increasing filling amount of PCM, the thermal response of the building material will be more effective through improving its thermal insulation and stored more of energy as a latent heat.

Keywords: Building materials, PCM, energy storage, finite element method Kulcsszavak: Építőanyagok, PCM, energia tárolás, véges elemes eljárás

#### 1. Introduction

As demand in thermal comfort of the buildings rises, the energy consumption is correspondingly increasing. For example, in Algeria, the energy consumption of buildings has increased by 22% the last three years. Housing and tertiary buildings are responsible for the consumption of approximately 43% of all energies [1].

At present, the thermal energy storage systems (TESS) are becoming more popular with regard to its incorporation in building materials, which in turn will reduce the dependence on fossil fuels for environmental and economic reasons [2]. The energy content in latent heat allows the PCM to transform its phase (from liquid to solid or vice versa) that permit it to store or release energy according to the required thermal loads [3]. Relying on the phase changing state, we note the three different cases by which the PCMs are characterized (solid-solid / solid-liquid / liquid-gas) [4]. Moreover, the solid-liquid phase changing is most suitable for the storage of thermal energy, where PCMs are clustered in three types: organic, inorganic and eutectic [5-7].

The applications of PCM can be extended to several disciplines such as medicine, sports and construction [8-9]. The integration of PCM in building materials such as concrete, ceramics, glass and hollow brick [10-16] is a way to enhance the storage capacity of building envelope and then to rationalize the use of renewable and non-renewable energies.

These studies have requested an improvement in thermal inertia, in order to store thermal energy [17]. The most effective method is using PCMs in both cooling and passive and active heating of the building.

#### Youcef Oussama SOUCI

PhD candidate in mechanical engineering under the supervision of Prof. Houat Samir at the laboratory of numerical and experimental modelling of mechanical phenomena. Obtained the licence and Master's degree in civil engineering from the University of sciences end technology of Oran –(USTO) Algeria.

#### **Samir HOUAT**

Professor at the University of Mostaganem (UMAB) of Algeria and head of numerical and experimental modelling of mechanical phenomena laboratory. He is the author of several scientific publications. He has been a member of the technical committees on several national and international congress, seminar and journals.

The ways by which the PCMs are incorporated, namely the direct incorporation, immersion and encapsulation have been analyzed by Hawes and Feldman [18].

Soaresa et al [19], provided a comprehensive review on the previous research addressing the passive latent heat thermal energy storage systems with PCM in buildings and their related performance. The study covers different characteristics, thermal properties, and selection criteria of PCM. The experiments and the numerical modelling of heat transfer with PCM and different dynamic simulations of energy building with PCM are reported. Finally, the life cycle assessments, both environmental and economic, were discussed.

Castell et al [20], experimentally tested the PCM with two types of typical construction material. The study showed that using the PCM reduces the peak temperature by 1°C and the electrical energy consumption by 15%. Alawadhi [21], one of the few researchers who studied numerically the thermal analysis of two-dimensional model of common building bricks with cylindrical holes integrated PCM to reduce the heat flow by storing energy from outdoor space in a hot climate during the day. This energy was retrieved during night time by solidification of PCM. The study focused on investigating different types of PCM at different melting temperatures, different PCM quantities at different brick's spatial locations. The study reported up to 17.55% savings in energy.

Many studies have investigated the use of PCM in buildings and showed that the PCM can remarkably improve the building energy performance. But few studies have examined the comparison between building materials when using PCM and its practical application.

At the present paper, to determine the influence of the use of the thermal energy system in building applications, a numerical study based on the finite element method has been carried out. To reach this objective, two different construction materials which were usually used in Algeria have been selected. The purpose of this study is to evaluate the total energy storage of these two materials, when filled by PCM, by analyzing and comparing their energetic performance.

#### 2. Materials and methods

#### 2.1 Material descriptions

Two building materials filled by PCM are carried out. The first one (M1) is the hollow brick and the second material (M2) is the breeze block as shown in *Fig. 1*. The hollow brick used consisting of 8 internal empty holes which have a parallelepiped section  $(3.5 \times 3.5 \text{ cm}^2)$ . The total dimensions of this material are thickness = 10 cm, width = 20 cm and length 30 cm. For the breeze block is consists of 6 parallelepiped holes  $(7 \times 14 \text{ cm}^2)$  and its total dimensions are thickness = 20 cm, width = 50 cm and length = 20 cm.



Fig. 1. Left: Hollow brick; Right: Breeze block 1. ábra Bal: üreges tégla, jobb: falazóblokk

The PCM selected and used in the present work is the organic paraffin n-octadecane with a phase change temperature of PCM at the main peak is 27.6 °C and the latent heat is about 243.5 kJ/kg [17]. This PCM was chosen because of its compatibility with closed container made of plastic in order to prevent its leakage at the liquid state and also because they are considered non corrosive and are chemically inert. The thermo physical characteristics of hollow brick, breeze block and PCM are shown in *Table 1*.

Material	Density (kg/m³)	Thermal Conductivity (W/m·K)	Heat capacity (J/kg·K)
PCM (solid)	865	0.358	1934
PCM (liquid)	780	0.148	2196
Hollow Brick	1600	0.7	840
Breeze Block	1300	1.05	648

Table 1. Characteristics of PCM and building materials used 1. táblázat PCM és falazóblokkok jellemzői

#### 2.2 Governing equations and boundary conditions

In order to simplify the mathematical model, many assumptions are made in this numerical analysis:

- Heat transfer process across the building material with PCM is unsteady and is treated as a one-dimensional problem (because these building material represent elements of a wall which its thickness is smaller than the other dimensions).
- The PCM material used is pure, homogeneous and isotropic.
- All thermo-physical properties of the building materials were kept constant except the conductivity and specific heat of PCM may be different in the liquid and solid phases.
- The natural convection of the PCM during the melting process and the super-cooling effect during the freezing process can be neglected when the temperature difference between the initial conditions and boundary is small [22].
- The interface layers between the building material and the PCM are homogeneous with perfect contact between them, which means that contact resistance is neglected.
- The surrounding radiation is neglected.

The transient enthalpy equation is given by Eq. (1):

$$\rho \frac{\partial \mathbf{h}}{\partial t} = \lambda \frac{\partial^2 \mathbf{T}}{\partial \mathbf{x}^2} \tag{1}$$

For the Non-PCM layer, the enthalpy (KJ/kg) is given only by the sensible enthalpy as shown in Eq. (2) and for The PCM layer, the enthalpy (KJ/kg) is computed in Eq. (3) as the sum of the sensible enthalpy and the latent heat.

$$h = h_{ref} + \int_{T_{ref}}^{T} C_p \, \partial T \tag{2}$$

$$h = h_{ref} + \int_{T_{ref}}^{T} C_p \, \partial T + f L \tag{3}$$

Where  $h_{ref}$  is the reference enthalpy at the reference temperature  $T_{ref}$  f is the liquid fraction and L is the latent heat of PCM (KJ/kg). The liquid fraction of PCM is defined as:

$$f = \begin{cases} 0, & if \ T < T_m \quad (solid) \\ 1, & if \ T > T_m \quad (liquid) \end{cases}$$
 (4)

Where  $T_m$  is the melting temperature of PCM.

For both building materials with and without PCM, Eq. (5) is used to calculate the energy stored by unit of surface E(t):

$$E(t) = \int_0^t (\varphi_{in,s} - \varphi_{ext,s}) dt$$
 (5)

 $\phi_{\text{in,s}},~\phi_{\text{ext,s}}$  are the inside and the outside surface heat flux, respectively.

The boundary condition at the indoor and the outdoor surfaces:

$$-\lambda_{b} \frac{\partial T_{b}}{\partial x} = h_{c,j} (T_{j} - T_{j,s})$$
 (6)

$$\left\{ \begin{array}{l} h_{c,j} = h_{c,in} \text{ , } T_j = T_{in} \text{ , } T_{j,s} = T_{in,s} \\ h_{c,j} = h_{c,ext} \text{ , } T_j = T_{ext} \text{ , } T_{j,s} = T_{ext,s} \text{; } x = w \end{array} \right.$$

The numerical values of the convective heat transfer coefficient can be assumed as in [23]  $h_{c,ext} = 20 \text{ W/m}^2 \, ^{\circ}\text{C}$  and  $h_{c,in} = 10 \text{ W/m}^2 \, ^{\circ}\text{C}$ .

At the building material/PCM interfaces:

$$\lambda_{\text{pcm}} \frac{\partial T_{\text{pcm}}}{\partial n} = -\lambda_{\text{b}} \frac{\partial T_{\text{b}}}{\partial n} \tag{7}$$

Where n is the coordinate normal to the surface of the PCM cavity.

#### 3. Numerical validation

Before exhibiting the obtained results, the numerical code is validated by comparison with an experimental solution available in the literature [24]. The experimental setup depends on testing a hollow slab incorporated by PCM. The PCM selected is characterized by a melting temperature equal to 27 °C. At the bottom surface of the slab, a linear increasing is imposed in temperature of 20 °C to 35 °C for one hour (Rate of rise = 1.25 °C/h) and then maintains the temperature at 35 °C for 6 hours. Their top surface is in direct contact with the atmosphere of the laboratory room  $T_{\rm in} = 0$  °C.

A study on mesh type and size has been carried out for this validation. It has been selected a triangular mesh with an element number equal to 2428 and the convergence criterion for temperature used is 10<sup>-4</sup>.

Fig. 2 shows the comparison between our numerical results with the results that were obtained in the experimental on the variation in the temperature of the bottom and top surfaces of the slabs. For the validation study, the Maximum Error calculated by Eq. (8) is used to assess the difference between experimental and numerical results.

Maximum Error (%)= 
$$\max_{1.2...n} \left\{ \left| \frac{T_{exp} - T_{num}}{T_{exp}} \times 100 \right| \right\}$$
 (8)

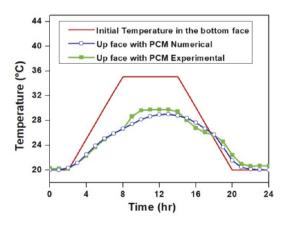


Fig. 2. Temperature at the interfaces of the floor panel 2. ábra Hőmérséklet a födémpanel felületén

As shown in *Fig. 2*, the numerical result agrees well with the experimental measurements with the maximum error of (5%). Three reasons can explain the slight discrepancy between simulation and experimental data. Firstly, the quality of the insulation material that has been used in the experiment to isolate the lateral faces of the slab is not perfect to provide the same performance as that used in the numerical simulation. Secondly, in the experimental study, it has been found that the melting temperature is not equal to the solidification temperature of the PCM. Thus, in the numerical study, this

difference between both temperatures could create a sort of imprecision in the selection of the  $\delta T$  value (phase transition zone). Finally, the possibility that the effect of the natural convection in the experimental case is not neglected and it can be integrated into the liquid state of the PCM what is different from the hypotheses that have been taken in the simulation model.

#### 4. Results and discussion

The test results about the influence of the PCM on the thermal storage capacity of two different building materials have been summarized in *Figs. 3* to 5.

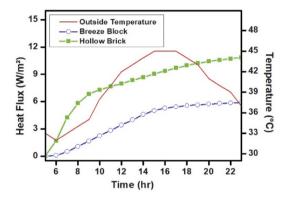


Fig. 3. Heat flux at the indoor surface for both materials with PCM 3. ábra Hőáram mindkét PCM anyaggal kitöltött blokk esetén beltéri felületen

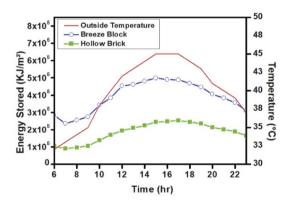


Fig. 4. Energy stored per unit of surface as a function of time 4. ábra Felületre vonatkoztatott hőtárolás az idő függvényében

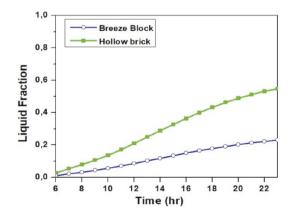


Fig. 5. Liquid Fraction for both materials with PCM 5. ábra Folyadék fázis mindkét PCM anyaggal kitöltött blokkra vonatkozóan

The finite element method is utilized to solve the physical problem. For both building materials selected, it has been used a Free quadrilateral element to mesh the computational domain. The software used at this numerical study offers the possibility to configure an automatic mesh with different element sizes. The choice of the type of mesh is based on the study of the influence of the number of elements on two physical parameters namely the average temperature of model and the liquid fraction of PCM. The results of this study are shown in *Table 2*.

	Number of elements		Tempe (°0		Liquid Fraction	
Material  Mesh size	Breeze Block	Hollow brick	Breeze Block	Hollow brick	Breeze Block	Hollow brick
Fine	305	160	27.78	29.09	0.232	0.549
Intermediate	1000	276	27.79	29.06	0.229	0.547
Extremely fine	6976	1375	27.79	29.03	0.228	0.543

Table 2. Effect of the mesh size on the temperature and the liquid fraction of PCM 2. táblázat Hálóméret hatása a hőmérsékletre és a PCM folyadék fázisára

The observation of the results shows the independence of the number of meshes on the values of the temperature and the liquid fraction of PCM. Similarly, beyond a finer mesh, the increase in the number of elements becomes insensitive and it will not necessarily change the solution but it requires a longer calculation time. Therefore, after various mesh tests, it has adopted a Structured quadrilateral mesh with an element number equal to 800 for the hollow brick and 1000 element number for the breeze block.

The two materials selected subject to an external air temperature of a day time as an example of real conditions. Constant temperature is applied as the indoor air temperature  $T_{\rm in} = 25$  °C.

As it is shown in Fig. 3, a comparison of the heat flux at the indoor surface between the two building materials is investigated. In the light of the results shown in this figure, the level of the heat flux obtained by the hollow brick is higher than that obtained with the breeze block. One of the significant reasons that lead to this result can be the difference between the PCM amounts inserted within both materials. The amount of PCM in the breeze block is about 83% larger than that used in the hollow brick due to the different geometries between the two materials. Therefore, the hollow brick-PCM system will not benefit a lot from its high latent heat because when a small quantity of PCM is used, this will leads to accelerate the melting/freezing process and making PCM most time in a deactivated mode. On the other hand, for the breeze block with PCM, most of heat will be stored as latent heat rather than as sensible heat due to the large energy needed by PCM in order to complete its phase change process. As a result, more energy will be absorbed by the material instead of being transmitted to the inner side.

Fig. 4 presents the quantity of heat per surface unit as a function of time, which is calculated by using Eq. (5). It can

be seen that the thermal energy stored in the breeze block with PCM is larger than that stored in the hollow brick.

At the end of the day, it has been found that the energy stored as latent heat by the PCM placed inside the breeze block is about  $2.29\times10^6$  KJ/m<sup>2</sup>. While, for the PCM inside the hollow brick, it has stored a small quantity of latent heat which is around  $10^6$  KJ/m<sup>2</sup>.

This result is compatible with what has been mentioned above. As it is observed from the obtained values that as the amount of PCM increases, the demand to store more latent heat becomes important in order to complete the phase change process.

To aid in understanding these results in *Fig. 4*, it has been evaluated the liquid fraction as is shown in *Fig. 5*. It can be seen that more than 50% of the PCM inserted in the hollow brick has converted to the liquid state. In turn, only 25% of the PCM in the breeze block has changed its phase. This finding reveals that most of the energy is stored as a sensible heat in the breeze block with PCM due to the large amount of PCM that has been remained most of the time at its solid state. Thus, it has the opportunity to store more of latent heat and this leads to reduce the heat gain. Due to this fact, using a building material which is capable to contain a larger amount of PCM is more effective to weak the effect of outdoor temperature fluctuation on indoor thermal environment and that by enhancing its thermal insulation effect.

#### 5. Conclusions

A numerical model was established to evaluate the potential of the thermal energy stored when it's applied in the building application. The proposed solution to achieve this is the incorporation of a phase change material in the construction elements. The numerical code is validated with an experimental model in the literature. It has been seen that the numerical results agreed well with the experimental results. Two different building materials are selected as an example to test its capacity of stored energy as a latent heat. Comparing the results from both materials, it is observed that the PCM addition, produces a reduction in the fluctuation of the heat flux at the inner surface of the breeze block more than the hollow brick. It has been explaining this result from the fact that there is a direct correlation between the increases in the amount of PCM and reduced heat flux at the inner surface.

It is observed that for the same test conditions, the breeze block with PCM stores 2 times more energy as a latent heat per unit surface than the hollow brick. This difference is due to the ability of PCM to complete its phase change rapidly. However, using a great quantity of PCM leads to delay the melt/solidification process so that the PCM could benefit more broadly from its potential to store the latent heat. Therefore, in practice and under similar conditions of this study, it is recommended to select a building material that can receive as much PCM as possible. This is because the increasing of the PCM amount has a positive effect on improving the thermal inertia of the building material.

#### Nomenclature

<sup>C</sup>p: Specific heat capacity (kJ/kg·°C)

f: Liquid fractionh: Enthalpy (kJ/kg)

h<sub>c</sub>: Convective heat transfer coefficient

L: Latent heat of fusion, (kJ/kg)  $Q_s$ : Solar flux radiation (W/m<sup>2</sup>)

*T* : Temperature (°C)

*t* : Time (h)

w: Thickness of wall (m)

#### **Greek symbols**

k: Thermal conductivity (W/m·°C)

P: Density (kg/m³)

#### **Subscripts**

b: Building material

m: Melting s: Surface Ref: Reference num: Numerical exp: Experiment in: Interior ext: Exterior

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# Internal thread cutting process improvement based on cutting tools treatment by composite powders in a magnetic field

JÜRI OLT • Chair of Biosystems Engineering, Institute of Technology, Estonian University of Life Sciences • jyri.olt@emu.

VIACHESLAV V. MAKSAROV • Department of Mechanical Engineering, Saint-Petersburg Mining University • maks78.54@mail.ru

ALEKSANDR I. KEKSIN - Department of Mechanical Engineering,

Saint-Petersburg Mining University • keksin.a@mail.ru

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#### **Abstract**

This article deals with the subject of using iron and titanium carbide (Fe + TiC) based composite powder for treatment of cutting tools in a magnetic field with the purpose of improving internal thread cutting process. As a result of studies performed by us, it has been established that composite powder grain size influences the condition of the contact surfaces of cutting tool teeth and process efficiency. Also a relationship has been discovered between the changes in internal thread roughness and the condition of thread tap teeth contact surfaces pretreated in a magnetic field by Fe + TiC composite powder having various grain sizes.

Keywords: internal thread, thread cutting, thread surface roughness, thread tap, magnetic-abrasive finishing, tooth contact surfaces condition, composite powder

Kulcsszavak: belső menet, menetfúrás, menet felületi érdessége, menetfúró, mágneses-abrazív felületkezelés, fog kontakt felület minősége, kompozit por

#### Jüri OLT. DSc.

Professor at Estonian University of Life Sciences.

He is Head of Department of Production
Engineering from 2005. His main research area
are fundamentals of production enginering,
materials cutting and design of technological
machinery.

#### Viacheslav MAKSAROV, DSc.

Professor at Saint-Petersburg Mining
University. He is Head of Department of
Mechanical Engineering from 2012 and Dean
of Electromechanical Faculty from 2015.
Specializes in the field of dynamics of machining
technological systems.

#### Aleksandr KEKSIN, PhD.

Associate professor at Saint-Petersburg Mining University. Specializes in the field of materials science and technology of construction materials.

#### 1. Introduction

Fatigue breakdown of thread joints operating under dynamic loads causes emergency and unscheduled machine shutdowns, resulting in increased equipment downtime and financial costs. Fatigue breakdown of thread joints is often encountered in practice; therefore, it forces production workers and researches to think over possible ways of solving this problem time and again.

Quite a wide range of design measures intended to increase fatigue resistance is proposed nowadays. Design assurance is a necessary measure; however, it is not sufficient. Technological measures that ensure necessary quality characteristics of mating thread surfaces also play a great role in fatigue strength improvement [4]. For example, Yakushev found out in his studies [15] that forming thread surface roughness with parameter  $R_{\rm a}=0.63$  –1.25  $\mu m$  increases fatigue strength of a thread joint several times as compared to a threaded joint with surfaces roughness parameter  $R_{\rm a}=2.5$  –  $3.2~\mu m$ .

At present, no significant complications arise as the exterior thread surface microrelief is formed. However, internal thread is quite a different story: it is rather difficult to ensure roughness parameter  $R_a \leq 1.6~\mu m$  on its flanks. It is especially difficult with items made of corrosion resistant materials that are very often used in power engineering. In this regard, the studies performed by these authors are aimed at forming internal thread surfaces in workpieces corrosion resistant materials so that internal thread flanks roughness would be within  $R_a=0.63-1.25~\mu m$ .

The most common process of forming a internal thread profile is the internal thread tapping process [3, 10, 12]. In spite of its popularity, it has significant drawbacks connected with ensuring the quality indicator specified above.

The technological reduction of internal thread surface roughness  $Ra < 1.6 \mu m$  during tapping is inseparably associated to the improvement of the condition of cutting tool teeth contact surfaces that can be effected by performing finishing operations. Magnetic-abrasive finishing (MAF) [6, 7] is a promising method of forming various conditions (rounding radius, roughness and microhardness) of tooth contact surfaces of a thread tap.

Magnetic-abrasive finishing [1, 5, 13-14, 17] is essentially abrasive effect made of a workpiece (a thread tap) (*Fig. 1*) by ferromagnetic powder abrasive mass compacted by magnetic field [6-9, 11, 16]. Using MAF as final machining of thread taps allows to remove the previous defective layer and to form a new, hardened one, to round off the cutting edges to the necessary limits, as well as to reduce the roughness of cutting teeth contact surfaces in a relatively short space of time (t = 60 - 210 s). As an example, it is presented in *Fig. 2* photographs of contact surfaces of a thread tap teeth before and after magnetic-abrasive finishing [7].

A substantial change in the condition of thread tap teeth contact surfaces is caused by specific features of magnetic-abrasive finishing, namely oriented and selective abrasive microcutting and microsmoothing [7]. Magnetic-abrasive powder having a high cutting power and enhanced magnetic properties plays a major role in microcutting and microsmoothing. However, it is not possible to ensure them at the same time for grains of a homogeneous material. Therefore, the sphere of application of magnetic-abrasive powders made of homogeneous materials is limited to machining of soft materials. It is possible to attain high hardness and good magnetic properties by creating powder materials in which every grain is a composition from ferromagnetic base and hard non-magnetic inclusions.

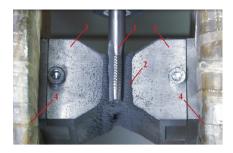


Fig. 1. Magnetic-abrasive finishing of a thread tap: 1) – the tap; 2) – magnetic-abrasive powder; 3) – pole tips; 4) – solenoids.
 1. ábra Menetfúrá mágneses-abrazív felületkezelése: 1) fúrászerszám. 2) mágnes

1. ábra Menetfúró mágneses-abrazív felületkezelése: 1) fúrószerszám, 2) mágnesesabrazív por, 3) elektródavégek, 4) tekercsek

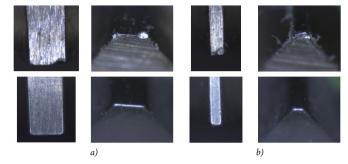


Fig. 2. Thread tap teeth before (the upper row) and after (the lower row) magneticabrasive finishing: a and b) the 4th tooth at the inlet section; c and d) the 6th tooth at the calibrating section

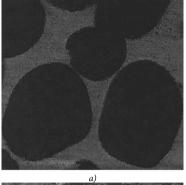
2. ábra Menetfog a mágneses-abrazív felületkezelés előtt (felső sor) és után (alsó sor): a és b) a negyedik fog a beömlési szelvényben, c és d) a hatodik fog a kalibrálási szelvényben

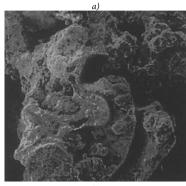
The most efficient composite magnetic-abrasive powder is a composite based on iron and titanium carbide (Fe + TiC) [1], a final product of chemical interaction resulting from combined heating of mixtures Fe + C + Ti. The presence of iron in the process of synthesis does not impede generation of titanium carbide in the temperature range from 1400 to 1600 °C. However, a rise in temperature brings about a reduction in titanium carbides content in the composition because of their dissolution [1].

Analysis of the Fe + TiC composite powder microstructure (Fig. 3.a) demonstrates that dark rounded spots (globules) of titanium carbides are observed against the light background of iron matrix. The globules differ in size insignificantly; they are distributed evenly throughout the matrix field. The size of the globules depends on the carbide phase concentration in the composition, i.e. grains grow larger as carbide concentration increases. The rounding of globules is caused by a limited capacity of titanium carbides dissolution in iron and the striving of every system to take the form that is the most suitable for it [1].

Powder particles are shaped as irregular polyhedrons (*Fig. 3.c*) with protruding carbide grains being abrasive cutting areas. The surface morphology of a high-melting abrasive in ferromagnetic matrix, its size and shape are presented in *Fig. 3.b*.

The microstructure of powder consists of carbides in the form of spherical, flakey and cast inclusions in iron matrix. The microhardness of ferromagnetic matrix is within the range of 2,100-3,400 MPa and that of Ti-C is 30,000-31,000 MPa. High microhardness of titanium carbide, a low level of its interaction with iron at high heating temperatures and, as a consequence, good magnetic properties of powder allow to ensure intense removal of base metal and an improved quality of the machined surface [1].





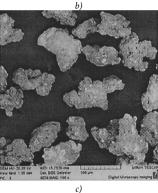


Fig. 3. Composite magnetic-abrasive powder based on iron and titanium carbide (Fe + TiC): a) powder microstructure; b) particle morphology; c) the shape of grains [1]

3. ábra Vas és titánkarbid (Fe + TiC) alapú kompozit mágneses-abrazív por: a) por mikroszerkezet, b) szemcse morfológia, c) szemcsealak [1]

Thus, the purpose of the studies carried out by these authors is to improve the internal thread tapping technology through preliminary treatment of the thread tap using the MAF method with the help of Fe + TiC composite powder.

#### 2. Materials and experimental methods

The magnetic-abrasive finishing of the thread tap was carried out on a specially designed plant located in the CNC machines laboratory at the department of mechanical engineering of the Saint Petersburg Mining University. The MAF device was based on a CNC milling machine that provides all working movements necessary for the machining process.

 $M16\times2$  thread taps made of R6M5 quick-cutting steel were taken as pilot prototypes. A composite powder based on iron and titanium carbide (Fe + TiC) having various grain sizes  $\Delta=160$  –  $315~\mu m$  was used for forming a magnetic-abrasive brush in the work zone of the electromagnetic system. The following constant process factors were adopted for the magnetic-

abrasive finishing of the thread taps: magnetic flux density B=0.6 T; polishing time t=210 s; the thread tap rotation frequency in the work space of the electromagnetic system n=475 rpm; thread tap advance along the pole tips S=120 mm/min; machining gap between the pole tips and the thread tap  $\delta=1$  mm. The following parameters were used to control the condition of the contact surfaces of the thread tap teeth: cutting edge rounding radius  $\rho$ ,  $\mu$ m; roughness of the flanks, the front and the rear surfaces of the cutting teeth  $Ra_1$ ,  $Ra_2$  and, respectively  $Ra_3$ ,  $\mu$ m; microhardness of the rear surfaces of teeth HV, MPa; the amount of material being removed Q, g.

The rounding radius of the cutting edges, the roughness of the flanks, the front and the rear surfaces of the thread tap cutting teeth were controlled with the help of a Hommel Tester T8000 profilograph-profilometer; microhardness was measured with the help of a PMT-3M microhardness tester; the amount of removed material, with the help of a VLTE 310 type scale.

The studies of internal thread cutting technology were carried out using a Trens SN 32/750 thread-cutting lathe. The workpieces undergoing the machining were made of 08Kh18N10T grade corrosion resistant material (metal thickness 40 mm). M16×2 mm thread taps were taken as cutting tools. Theses taps were preliminarily subjected to MAF with the purpose of creating various conditions of the contact surfaces of the tool teeth.

After cutting internal thread in a workpiece made of a corrosion resistant material using thread taps preliminarily subjected to MAF under various machining conditions, the roughness of the flanks of the internal thread profile was controlled. Roughness was controlled using a Hommel Tester T8000 profilograph-profilometer.

The experimental data on the roughness of internal thread surfaces were subjected to statistical analysis in the Statistica 10 software environment.

#### 3. Results and discussion

As a result of the studies of magnetic-abrasive finishing of thread taps using Fe + TiC composite powder, it has been established that the magnetic-abrasive powder grain size  $\Delta$  influences the condition of thread tap teeth contact surfaces, characterized by the cutting edge rounding radius  $\rho$ , the roughness of the cutting teeth flanks Ra<sub>1</sub>, that of their front Ra<sub>2</sub> and rear Ra<sub>3</sub> surfaces, the microhardness of cutting teeth rear surfaces Hv, and at the machining efficiency expressed in the amount of removed material Q (Fig. 4).

Analysis of the dependency curves presented in Fig. 4 allows to conclude that within the range of Fe + TiC composite powder grain sizes under study ( $\Delta=160-315~\mu m$ ), the thread tap cutting edges rounding radius after MAF varies within the range  $\rho=40...51~\mu m$ , the roughness of tooth flanks Ra<sub>1</sub> = 0.068...0,073  $\mu m$ , the roughness of tooth front surfaces Ra<sub>2</sub> = 0.09 - 0.11  $\mu m$ , the roughness of tooth rear surfaces Ra<sub>3</sub> = 0.095...0,149  $\mu m$ , the microhardness of tooth rear surfaces Hv = 9,722 - 11,162 MPa and the amount of removed material is within the range Q = 6...8 g.

The studies of thread taps MAF with the use of Fe + TiC composite powder having various grain size bear witness to a

substantial improvement of the condition of cutting tool teeth contact surfaces in view of the fact that originally the cutting edge had defects resulting from previous machining (*Fig. 2*), the tooth flank roughness was equal to  $R_{a1}=0.19~\mu m$ , the tooth front surface roughness  $R_{a2}=0.26~\mu m$ , the tooth rear surface roughness  $R_{a2}=0.26~\mu m$  and the tooth rear surface microhardness HV=7.507~MPa.

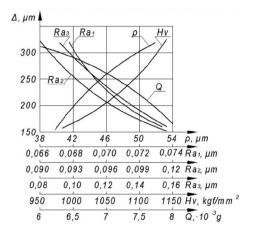


Fig. 4. Fe + TiC composite powder grain size vs. the condition of thread tap teeth contact surfaces and the MAF efficiency curves:  $\rho$  – cutting edge rounding radius;  $R_{a1}$  – tooth flank roughness;  $R_{a2}$  – tooth front surface roughness;  $R_{a3}$  – tooth rear surface roughness;  $H_{v}$  – rear tooth surface microhardness; Q – the amount of removed material

4. ábra Fe + TiC kompozit por szemcseméret és a fúrószerszám kontak felületének állapota, MAF hatékonysági görbék: ρ – vágóél lekerekítési rádiusz; R<sub>al</sub> – fog profil érdessége; R<sub>a2</sub> – fog frontfelület érdessége; R<sub>a3</sub> – fog hátfelület érdessége; Hv – fog hátfelület mikrokeménysége; Q – leválasztott anyagmennyiség

After the MAF of thread taps using Fe + TiC composite powder, experimental studies of internal thread cutting were performed. The thread taps used in this studies were previously machined by composite powder having various grain sizes in a magnetic field, and a dependency has been established between a change in internal thread roughness and the condition of thread tap teeth contact surfaces. Since condition is understood as a totality of microgeometrical parameters of contact surfaces of teeth, i.e. the cutting edge rounding radius, the roughness of cutting teeth flanks, front and rear surfaces, the microhardness of teeth rear surfaces and it is not possible to detect the dependency between the influence of each of these parameters on the internal thread roughness since during the MAF they are formed simultaneously, it was decided to plot the dependency curve in the following way: plot the thread roughness values on the ordinate axis and the composite powder grain size values that changed during forming of various conditions of thread tap teeth contact surfaces during the MAF, on the abscissa axis.

The dependency between changes in internal thread roughness and the condition of thread tap teeth contact surfaces presented in *Fig. 5* allows to conclude that an increase in composite powder grain size from 160 to 315  $\mu$ m during the MAF of thread taps is accompanied in the subsequent process of internal thread cutting using these taps by increased roughness of the profile of internal thread cut in workpieces made of 08Kh18N10T grade corrosion resistance material from 0.85 to 1.28  $\mu$ m (Ra parameter). It should be noted that the roughness of internal thread cut by a thread tap that was not previously subjected to the MAF is 1.77  $\mu$ m. The roughness of thread cut

by a previously MAF machined thread tap is reduced at least 1.4 times irrespective of composite powder grain size  $\Delta = 160 - 315$  µm. This confirms the efficiency of using this finishing method within the framework of the parameters range under study.

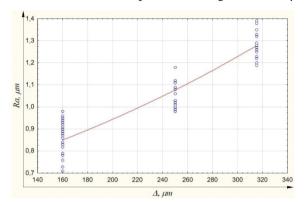


Fig. 5. Dependency between changes in internal thread roughness (Ra) and the condition of thread tap teeth contact surfaces formed due to varying composite powder grain size

5. ábra Kompozit por szemcseméretének hatása a fúrószerszám fog kontakt felületének állapota és a belső menet felületi érdessége (Ra) közötti kapcsolatra

The studies of the MAF of thread taps using Fe + TiC having various grain size have shown that thread tap cutting edges rounding radius changes within the range of  $\rho$  = 40 – 51  $\mu m$  (Fig. 4). Since the cutting depth during thread tapping a <  $\rho$ , in this case the deterioration of internal thread roughness  $R_a$  parameter in workpieces made of 08Kh18N10T grade corrosion resistant material from 0.85 to 1.25  $\mu m$  can be explained by elastic deformation of a workpiece surface layer in the process of cutting that has an adverse effect on surface microrelief formation.

Experimental data analysis allowed to conclude that within the Fe + TiC composite powder grain size range under study ( $\Delta$  = 160 – 315  $\mu$ m) the most efficient MAF method for thread taps machining is using powder grain size  $\Delta$  = 160  $\mu$ m at magnetic flux density B = 0.6 T and finishing time t = 210 s.

#### 4. Conclusions

In conclusion, it can be said that using magnetic-abrasive finishing (MAF) during the machining of thread taps for the improvement of technology of internal thread surface cutting in workpieces made of 08Kh18N10T grade corrosion resistant material will allow to reduce the roughness of internal thread surfaces from Ra = 1.77  $\mu m$  (thread cutting by a standard thread tap) to Ra = 1.28 – 0.85  $\mu m$  (depending on the Fe + TiC composite powder grain size used during the MAF of thread taps).

Thus, the purpose of studies set by the authors (improvement of internal thread cutting technology by means of preliminary MAF of thread tap teeth contact surfaces) can be considered as achieved since internal thread profile flank roughness Ra < 1.6  $\mu m$  and within the range of Ra = 0.63 – 1.25  $\mu m$  in workpieces made of a corrosion resistant material is ensured.

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# Euro Building Materials & Construction Technologies Conferences

June 20-21, 2019 | Stockholm, Sweden



#### About Conference

The fundamental subject of Building Materials & Construction Technologies 2018 is "Commencing New Techniques for Efficient Structures & Dwellings". This unique opportunity that we extend to our speakers and attendees is not being offered by any other conference organizers. Through this the abstracts and research profiles of our speakers and organizing committee members getting global visibility which is an additional feature that you would be receiving in addition to networking opportunities before, during and after the conference.

#### **BUILDING MATERIALS 2019 salient features**

- · Meet Academia and Industry visionaries to get inspired
- · Expand your knowledge and find solutions to problems
- Knowledge, Benchmarking and Networking offered at one place
- · Forge connections and for global networking
- Highly Organized and Structured Scientific programs
- Poster presentations and world class exhibitions
- Meet with new vendors and suppliers

#### **How Conference Series LLC Ltd Conferences differs from others?**

- 3000+ Conferences across the globe in 30+ countries all through the year
- Over 25 Million+ Visitors and 20000+ Unique Visitors per conference
- Participation by Stalwarts from various international societies
- Internationally renowned speakers and scientists representation
- · Career guidance for early career researchers and students
- · Interesting scientific deliberations and discussions
- Perfect platform for Global Networking

#### Goals

- The conference program emphasizes evidence-based practice, educational innovation, practical application, and peer to peer networking and collaboration. The goals of the conference is to provide a transformative professional development experience through
- Bringing together the world's scientific experts to catalyze and advance scientific knowledge about Building Materials & Construction Technology new techniques, and promote and enhance scientific collaborations around the world.
- Bringing together community leaders, scientists, and policy leaders to promote and enhance the knowledge towards improving the material both durable and economical. Thereby reducing the risk factor for living beings.

#### **Objectives**

- After participating in this meeting, attendees should be able to
- Discuss emerging issues in new Building Material techniques
- Discuss and apply recent research findings related to Eco-friendly building materials
- Reflect on the place of critical distance in Building Materials & Construction Technologies

#### **Major Sessions**

- Durability Enhancing Products
- Recycled Aggregates
- Green Building Materials Sustainable Construction
- Waste Reduction
- Biodegradable waste
- Structural Loads
- Load Cases
- Design of Shear Walls
- Plans for urban and rural subdivisions
- Determining & implementing zoning laws
- Elastic deformation
- Chemical Reaction
- Plant & Equipment management
- Infamous disasters in engineering
- Bearing capacity of Soil
- Bridge Launching Techniques













Theme: Commencing New Techniques for Efficient Structures & Dwellings

Organized by the Italian Ceramic Society www.icers.it - info@icers.it





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## XVICONFERENCE AND EXHIBITION OF THE EUROPEAN CERAMIC SOCIETY



Organizing Secretariat



AIM Group International Florence Office info@ecers2019.org

