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An Experimental Study of Mechanical Properties and Heat Transfer of Acrylic Composites with Structural and Surface Modified Al₂O₃ Particles

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Abstract:

Thermal properties of acrylate-based composite combined with alumina-based particles were investigated. The composites were made of poly (methyl methacrylate) (PMMA) modified with dimethyl itaconate (DMI) as a matrix. Neat alumina particles (Al₂O₃) and alumina doped with iron oxide (Al₂O₃-Fe) modified with 3-aminopropyl-trimethoxylane (AM) and flax oil fatty acid methyl esters (biodiesel – BD) were used as reinforcement. Thermal conductivity measurements showed that composite with alumina particles with the highest alpha phase content had the highest thermal conductivity values. Mechanical properties (tensile strength, modulus of elasticity and elongation at break) were improved by the addition of modified alumina particles to PMMA/DMI matrix. The thermal properties are the most improved for composite with 3 wt.% Al₂O₃-Fe-AM particles that had the lowest temperature in a crack zone was noticed (10.28 %) compared to PMMA added due to the better stiffness and highest thermal conductivity.

Keywords: Al₂O₃ particles; Reinforcement; Composite materials; Thermal properties.

1. Introduction

Poly (methyl methacrylate) (PMMA) is a transparent and tough plastic material that has wide application in areas where material transparency is important. Inert in the human body, PMMA has shown good biocompatibility. Good mechanical properties retain for a long time. Also, it is used to create denture bases [1], bone cement [2], contact lenses, inhalers [3,4]. It is important to note that PMMA has certain insufficiencies. Potential distortions of residual monomer [5–7] and limitations concerning mechanical properties, especially in terms of modulus of elasticity, impact, fatigue or flexural strength [8,9], might appear as a result of these deficiencies. In a case of diffusion, the residual monomer can leach out from the material and irritate the surrounding tissue. In dentistry itaconic acid its esters are well known.

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They have been broadly used in the production of ionomer cement [10–12]. Itaconic acid is a natural, non-toxic and easily biodegradable compound. It dissolves in water, ethanol and acetone. It is good to know that the itaconates, because of their low toxicity, are used for different purposes in medicine [13]. The addition of itaconate derivate to PMMA decreases the content of residual methyl methacrylate MMA [14,15]. Itaconate derivate and methacrylate have a similar structure and can provide copolymers [6]. However, due to the steric hindrance of the alkyl group, methacrylates are more reactive than the itaconates [6,7]. In order to overcome the drawbacks and limitations of basic PMMA-based materials, the possibility of PMMA modification using the itaconic acid derivatives was investigated. Although the market price of itaconic acid is higher than the price of methacrylic acid, itaconic acid and itaconates are acceptable in terms of ecology and sustainable development [16]. The reason is that itaconic acid is obtained from plants (through enzymatic molasses transformations) while methacrylic acid is obtained from petrochemical sources. Itaconic acid is a dibasic acid and provides more options in the synthesis of esters compared to methacrylic acid. It was found that the addition of diethyl itaconate to PMMA significantly reduces the amount of residual monomer, which makes these materials less toxic. On the other hand, a reduced quantity of residual monomer leads to deterioration of the mechanical properties of the material [15].

The thermal instability is, unfortunately, one of its limitations. Several strategies can be employed to delay its degradation, for example, incorporation of nanoparticles in the polymer matrix. Using different fillers, their physical-mechanical properties can be adjusted. The fillers are added so they can control the shrinkage and mechanical properties of the material [17]. The inorganic particulate fillers often added to polymer composites are micro- or nano-SiO₂, Al₂O₃, Mg (OH)₂ or CaCO₃ particles. They are added to improve their stiffness, strength, modulus and hardness [1,2,18]. Because of the fact that rigid inorganic particles have much higher stiffness comparing to polymer matrices [3,4], adding either micro- or nano particles, stiffness or Young's modulus can be improved. Generally, using fillers that have controlled crystal structure, mechanical properties can be adjusted [19,20]. Alumina (Al₂O₃) or aluminum oxide is a ceramic material that has exceptional mechanical, physical and chemical properties. In nature, it exists as the mineral corundum (Al₂O₃), diaspore (Al₂O₃·H₂O) and gibbsite (Al₂O₃·3H₂O) [21]. Because of their extraordinary chemical properties, alumina is used in composites as reinforcement and as materials for high-temperature applications. Corundum, because of his stability and high elastic modulus, is one of the most used materials in high-temperature applications. In composite materials of particular interest are different structures of aluminum oxide, which can be obtained by thermal treatment [22].

Thermography is a popular non-destructive testing (NDT) method used to detect damage to composite structures. This method is used for specimens up to 15 mm thick because of the propagation of heat through a large thickness result in a poor definition of damage [20]. For thermomechanical analysis of the behavior of many materials, the development of infrared cameras with efficient image processing methods has been successfully used for the last several decades. This non-destructive method enables the measurement of the surface temperature field and its development [23,24]. If the plastic material is deformed, then the temperature will change and the amount and direction of change will depend on the nature and magnitude of the deformation as well as the strength applied. In the deformation process, each component gives a certain kind of thermal effect, so that thermal and mechanical measurements are essential to interpret the mechanical behavior of the composite material [25]. Thermal conductivity is an important parameter in problems involving stable heat transfer because it determines the material, operating temperature level or determines the temperature gradient within this material. This property plays an important role in the performance of materials at high temperatures. Low values of thermal conductivity are required when low heat losses are required. On the other hand, when heat transfer from

one place to another is desirable, then materials with higher thermal conductivity must be selected. However, many factors can affect the thermal conductivity of materials such as temperature, density, porosity, moisture, crystallinity, grain orientation, etc. [26]. The formation and propagation of micro-cracks in materials cause their mechanical, thermal and electrical properties to deteriorate significantly. Crack detection techniques used in polymeric materials are NDT methods such as visual testing, strain measurements [27], computed tomography (CT) scanning, ultrasonic testing, acoustic emission (AE) [28], vibration damage detection techniques [29], electrical impedances and thermographs [30]. The application of NDT depends on the approach of discontinuity, material thickness, depth and type of damage.

The aim of this article was to make a composite material with a matrix made from PMMA/itaconate copolymer and to examine the thermal behavior, depending on the addition of various alumina-based fillers with surface modification. The thermal behavior of polymer composites will be monitored by thermal vision during the tensile testing of notched specimens to obtain an insight into the crack propagation and dependence of temperature in the crack zone on heat conductivity.

2. Materials and Experimental Procedures

2.1 Materials and methods

To produce the sample, the commercial PMMA (Biocril (Galenika AD, Serbia)) and dimethyl-itaconate (DMI) (Sigma-Aldrich) were used. The preparation of PMMA is composed of the two-component system: the first one is a liquid component-methyl methacrylate (MMA) as a monomer and ethylene glycol dimethacrylate (EGDMA) as a modifier, and the second is a powder component-prepolymerized PMMA coated with dibenzoyl peroxide as the initiator. Biocryl, his powder component, has been characterized in the literature as follows: molecular weight is $M_n = 1.27 \times 10^5 \text{ gmol}^{-1}$, $M_w = 3.82 \times 10^5 \text{ gmol}^{-1}$, and the polydispersity index $PI = 3.01$, with a 4.3 wt.% of monomer content and an average particle size of 55 μm [31].

Submicron ferrous oxide doped alumina particles ($\text{Al}_2\text{O}_3\text{-Fe}$), which are used as a reinforcement, were prepared using the sol/gel technique. As an alumina precursor, aluminium hydroxide chloride (Locron L; $\text{Al}_2(\text{OH})_5\text{Cl}\cdot 2.5\text{H}_2\text{O}$), was purchased from Clariant Company in a crystallizes state. The salt, iron chloride ($\text{FeCl}_3\cdot 6\text{H}_2\text{O}$), which was used as a source of iron ions, was obtained from Sigma-Aldrich. Applying the sol-gel technique, the ferrous oxide doped alumina particles were prepared to start from $\text{Al}_2(\text{OH})_5\text{Cl}\cdot 2.5\text{H}_2\text{O}$, $\text{FeCl}_3\cdot 6\text{H}_2\text{O}$, and then as a solvent was used demineralized water. The solution was so prepared that the mass ratio of Al_2O_3 to Fe_2O_3 in the end product would be 90/10. The gel was ground and calcined at 900 °C for 2 h. By a particle size analyzer where the diameters of the particles determined, and they found to be: $d(0.1) = 0.412 \mu\text{m}$, $d(0.5) = 0.608 \mu\text{m}$, $d(0.9) = 1.208 \mu\text{m}$ [27]. By XRD was the crystal structure determined and it was as follows: $\eta\text{-Al}_2\text{O}_3$ (39.4 %), $\kappa\text{-Al}_2\text{O}_3$ (35.1 %) and $\alpha\text{-Al}_2\text{O}_3$ (25.5 %) [19,32,33].

The modification of the particles took place in two ways: with 3-aminopropyl trimethoxy silane (AM) and methyl esters of linseed oil fatty acids (biodiesel – BD) [30]. Prepared particles modified with AM and BD were denoted as $\text{Al}_2\text{O}_3\text{-AM}$, $\text{Al}_2\text{O}_3\text{-BD}$, $\text{Al}_2\text{O}_3\text{-Fe-AM}$, and $\text{Al}_2\text{O}_3\text{-Fe-BD}$.

2.2 Preparation of PMMA/Dimethyl Itaconate Dental Composites Reinforced with Alumina Particles

The matrix is made of PMMA and 5 wt.% DMI by adding DMI to the liquid monomer and then PMMA powder added to the liquid. Composite materials were prepared by

adding alumina particles to the monomer and dispersed in an ultrasonic bath for 30 minutes to avoid agglomeration and to obtain a good dispersion. This is followed by the addition of the powder component (63 wt.%) into the liquid. It is stirred for 5 minutes after being transformed into a paste that has been put into a mold. The mold made of aluminium alloy was closed and heated at 70 °C for 1 hour, after which the polymerization was completed at 100 °C within 30 minutes. Using 1, 3 and 5 wt.% of different alumina particles (samples prepared with iron oxide and alumina nanoparticles with two surface modifications), samples were prepared. A polymer matrix consisting of PMMA and DMI is designated PMMA/DMI (hereinafter DMI).

2.3 Characterization Methods

The morphologies of the composites with modified alumina-based particles were examined using a field emission scanning electron microscope (FESEM), MIRA3 TESCAN, operated at 3 kV.

Instron Testing Machine (model number 6025) T was the testing machine used for the tensile test. For the tension test, the specimen had standard dimensions. The cross-section was in the testing area of 2 x 5 mm and the length was 5 cm. To measure the modulus of elasticity of the composite was used they obtained data. Notches of the specimens used for monitoring of crack propagation on samples 60 x 12 x 3 mm were 4 mm deep with angle 30°. The specific heat conductivity was measured with a specially prepared apparatus adapted to the size and shape of the samples (Fig. 1).

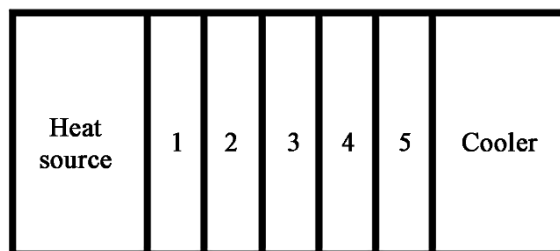


Fig. 1. A sketch of the apparatus for measuring specific heat conductivity (1 - Conductor of heat (aluminum); 2 - a material known to stp; 3 - conductor of heat (aluminum); 4 - sample; 5 - conductor of heat (aluminum)).

Apart from neglecting the edge effects (which was considered in the construction), it can be considered that the heat flux through the elements of the apparatus is the same. Then the measurement procedure for specific thermal conductivity is reduced to the measurement of temperature drops on the reference element (2) and the sample (4). In case the glass is selected as a reper, the specific heat conductivity of the sample in W/(mK) is obtained:

$$\lambda = 0.84 \frac{T_1 - T_3}{T_3 - T_5} \quad (1)$$

To measure the temperature of the elements in the described apparatus, a digital thermionic camera FLIR E6 was used, based on a non-cooled microbolometer detector. It forms a thermal image by measuring the infrared radiation of a particular body or the entire scene. The software that the camera contains the necessary correction when converting the thermal image into an appropriate thermogram, which represents an approximation of the exact temperature of the recorded object, or the temperature distribution in the scene. Further processing (postprocessing) is done with the help of professional software FLIR Tools + (Fig. 2.).

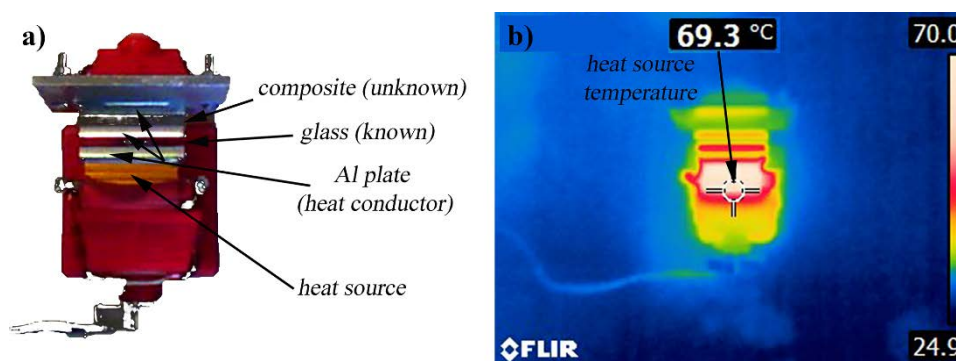


Fig. 2. Heat conductivity measurements: a) experimental setup, b) thermo photograph.

3. Results and Discussion

The results obtained from the thermal conductivity measurements are presented in Table I.

Tab. I Thermal conductivity of PMMA/DMI matrix and obtained composites.

Sample	λ [W/m·K]	Sample	λ [W/m·K]
PMMA/DMI	0.236		
1 wt. % Al ₂ O ₃ -AM	0.602	1 wt. % Al ₂ O ₃ -Fe-AM	0.679
3 wt. % Al ₂ O ₃ -AM	0.542	3 wt. % Al ₂ O ₃ -Fe-AM	0.690
5 wt. % Al ₂ O ₃ -AM	0.701	5 wt. % Al ₂ O ₃ -Fe-AM	0.789
1 wt. % Al ₂ O ₃ -BD	0.596	1 wt. % Al ₂ O ₃ -Fe-BD	0.668
3 wt. % Al ₂ O ₃ -BD	0.634	3 wt. % Al ₂ O ₃ -Fe-BD	0.671
5 wt. % Al ₂ O ₃ -BD	0.637	5 wt. % Al ₂ O ₃ -Fe-BD	0.674

The key role in the prediction of the thermal stability is thermal conductivity. That is the ability of a material to conduct the heat rather than absorb it. It is known, that the reinforcement, for instance, metal dioxide nanoparticles such as alumina, titanium dioxide and etc. can modify the thermal stability and lower the thermal degradation of a polymer [25]. The thermal conductivity of the PMMA is increased by increasing the temperature of material up to the glass transition temperature (T_g), after which thermal conductivity decreases (above T_g) [34]. Thus, fillers can decrease flammability and enhance thermal stability. The addition of Al₂O₃ in the matrix increases thermal conductivity. Due to the crystalline difference between neat polymer and ceramic particulate filled polymer composites, it appears this difference in values. It was observed that the crystallinity of composite polymers increases by the addition of Al₂O₃ particles, as Al₂O₃ particles show more thermal conductivity than the matrix polymer [35]. Table I presents a notable increase in thermal conductivity by the addition of fillers when compared to PMMA. The addition of particles in 1 wt.% contributes to the increase of heat conductivity where the lowest value was found in composite with 1 wt.% Al₂O₃-Fe-AM in comparison with the PMMA/DMI. Further addition of particles increases thermal conductivity due to the increase of component of higher thermal conductivity (PMMA 0.167-0.25 W/mK [36] and alumina 12-38.5 W/mK [37]). It may be noticed that the conductivity is higher with Al₂O₃-Fe particles due to the difference in chemical structure, dense crystal structure with a higher content of alpha phase that contributes to better thermal conductivity.

The presence of BD on alumina surface decreases thermal conductivity when compared to modification with AM and impedes further increase in particle share.

Tab. II Tensile properties of PMMA matrix and obtained composites.

Sample	Tensile stress (σ_t), MPa	Elongation at break (ε_t), %	Modulus of elasticity (E_t), MPa
PMMA/DMI	39.5	7.88	800.0
1 wt. % Al ₂ O ₃ -AM	48.1	7.58	762.8
3 wt. % Al ₂ O ₃ -AM	49.0	11.9	838.3
5 wt. % Al ₂ O ₃ -AM	35.4	5.09	549.5
1 wt. % Al ₂ O ₃ -BD	38.4	9.50	569.2
3 wt. % Al ₂ O ₃ -BD	46.3	12.2	590.2
5 wt. % Al ₂ O ₃ -BD	25.4	8.85	473.1
1 wt. % Al ₂ O ₃ -Fe-AM	37.0	6.45	896.7
3 wt. % Al ₂ O ₃ -Fe-AM	47.9	6.59	1092.7
5 wt. % Al ₂ O ₃ -Fe-AM	35.7	4.96	825.4
1 wt. % Al ₂ O ₃ -Fe-BD	37.9	9.49	747.4
3 wt. % Al ₂ O ₃ -Fe-BD	39.1	9.50	776.5
5 wt. % Al ₂ O ₃ -Fe-BD	29.3	8.76	629.6

In order to investigate the amount of alumina particles on tensile characteristics, mechanical testing was performed. The values of tensile stress at break (σ_t), elongation at break (ε_t) and tensile modulus (E_t) are shown in Table II. Results indicate that in all specimens with the incorporation of 3 wt.% particles caused a significant increase of σ_t , ε_t and E_t . This increment may be prescribed to the effective dispersion of Al₂O₃-Fe particles, meaning that a higher amount of intermolecular (dipole/dipole) interactions between surface hydroxyl groups of particles and polar pendant groups of PMMA/DMI matrix was established [31]. From the data presented, the addition of 3 wt.% Al₂O₃-Fe-AM gave the material having the best modulus of elasticity that was improved by 26.79 % compared to PMMA. The results suggest that the composites reinforced with particles having AM surface modification show better compatibility in the PMMA/DMI matrix than particles with BD. Longer aliphatic chains of BD contributes to the increase of elongation at break of composites when compared to particles with AM modification.

The surface of a cracked surface after tensile testing was analyzed by scanning electron microscope, and obtained images are presented in Fig. 3.

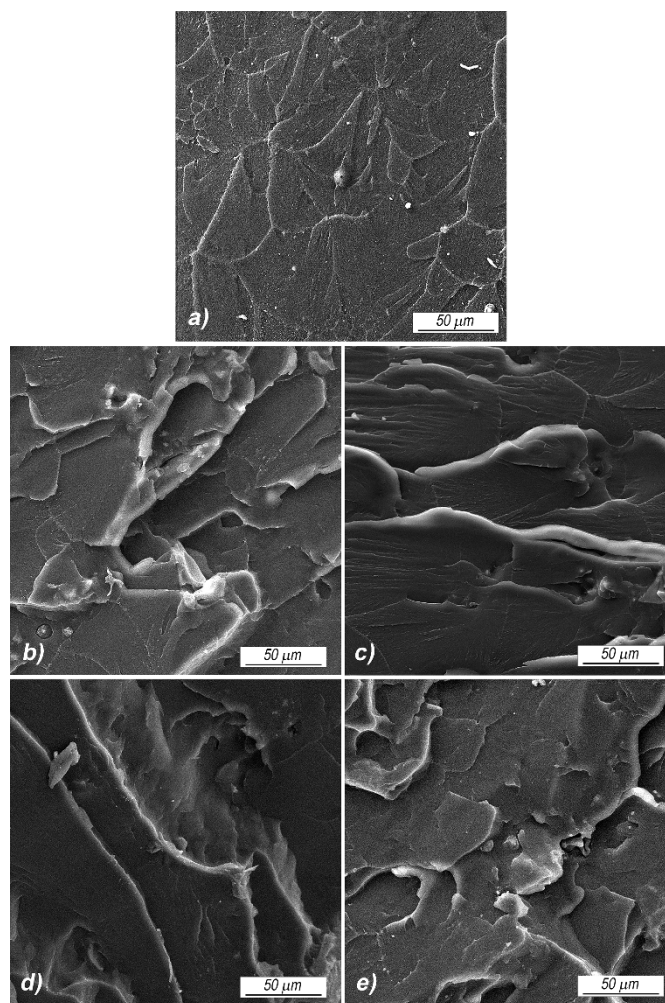


Fig. 3. FE-SEM images of the fracture surface of the specimen: a) PMMA/DMI and composites reinforced with b) 5 wt.% Al_2O_3 -AM, c) 5 wt.% Al_2O_3 -BD, d) 5 wt.% Al_2O_3 -Fe-AM and e) 5 wt.% Al_2O_3 -Fe-BD.

SEM image of polymer matrix PMMA/DMI (Fig. 3a) shows relatively smooth surface while the composites (Fig. 3 b-e) exhibited similar morphology with higher roughness, which may result from increased resistance to fracture due to reinforcement. It is evident in the images that the specimens with the PMMA/DMI and with the 5 wt.% Al_2O_3 -BD+PMMA/DMI displayed a smoother and more compact structure than another specimen with 5 wt.% Al_2O_3 -AM+PMMA/DMI but with notable defects as particle clusters. In this specimen's group, it was noticed that the particles have formed bunch-shaped structures inside the resin matrix. That means that the AM surface modification of the particles contributed to the homogeneous dispersion of the particles in the matrix and improved interface compatibility.

A thermal imaging camera was used to monitor the change in material temperature in the fracture region during material tensile testing. Thermography is based on the use of thermal energy and its absorption and dispersion in the sample. Infrared thermography is a contactless optical imaging technique for the detection of cracks and micro-cracks. The temperature distribution clearly indicates the shape and location of the damaged area. Thermographs of defective composites show anisotropy of heat flow [38]. Thermo grams of the samples during the tensile test were shown in Fig. 4.

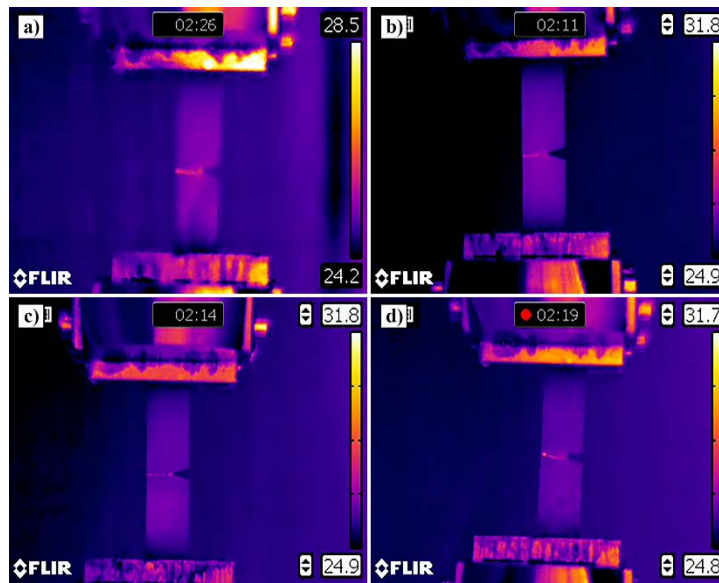


Fig. 4. Thermo grams of the samples during the tensile test: a) 5 wt.% Al_2O_3 -AM, b) 5 wt.% Al_2O_3 -BD, c) 5 wt.% Al_2O_3 -Fe-AM and d) 5 wt.% Al_2O_3 -Fe-BD

When elastic-plastic material undergoes plastic deformation, part of the deformation dissipates in the form of heat and an increase in temperature can occur if the deformation occurs rapidly relative to the time it takes for the deformation to occur. This is especially true for polymeric materials because the heat generated during localized deformation does not have time to conduct the surrounding areas. Polymers are susceptible to thermal effects because they exhibit greater rigidity and variations in strength with respect to lesser temperature changes [39]. When the thermal conductivity is increasing, the fracture along the entire sample is better distributed. No large amount of heat is generated around the fracture [40].

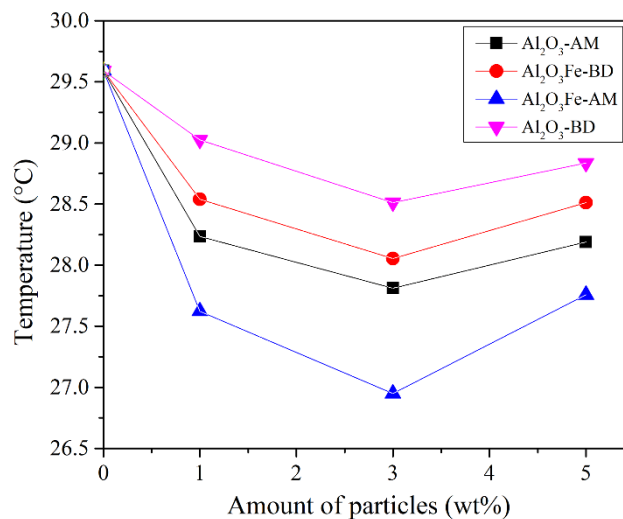


Fig. 5. Temperature – Amount of particle curves measured in the crack zone during a tensile test of composites.

The thermogravimetric curves of PMMA and PMMA composites are presented in Fig. 5. The values show the temperature values at the cracking initiation point. It is noticed that all samples show the same trend. Temperature values of all composites that contained 3

wt.% is in decrease. With the samples of 5 wt.% composites an increase in temperature was observed due to the agglomeration of particles and deterioration of mechanical properties. The thermal stability of PMMA is most improved in the specimen where Al₂O₃-Fe-AM was added due to the better stiffness (higher modulus of elasticity) and thermal conductivity which significantly lowered the temperature. The largest decrease of temperature in a crack zone was noticed for composite with 3 wt.% Al₂O₃-Fe-AM (10.28 %) compared to PMMA.

4. Conclusion

Based on the results of this study, thermal conductivity, modulus of elasticity and the strengths were improved by adding alumina-based reinforcement to the matrix, which was made from PMMA itaconate copolymer. Aluminum oxide-based particles were used as reinforcement with a modified organosilane-3-aminopropyl-trimethoxylane (AM) surface and in the second stage with fatty acid methyl esters (biodiesel-BD). Surface modification particles establish a better bond with the polymer matrix through covalent and hydrogen bonds and dipole-dipole interactions. The efficient dispersion of particles in the matrix also improves the transmission of the load through the matrix, which is followed by a thermionic camera. Thermo grams during a tensile test indicate differences in temperatures occurring in the fracture zone of composite materials. The temperature in the fracture zone is closely related to the efficiency of the load transfer and the temperature conductivity of the material itself. The mechanical properties of the material are improved by adding inorganic particles based on the activated surface of aluminum oxide, thereby creating new composite material with improved mechanical and thermal properties.

The results show significant improvement in the thermal conductivity of composites with the addition of all types of alumina-based particles. Modulus of elasticity is also being improved. The best results are obtained by the 3 wt.% Al₂O₃-Fe-AM. A higher particle ratio indicates the occurrence of agglomeration of particles that are not well dispersed in the matrix. Agglomerates represent centers where the mechanical properties of a composite are reduced.

Acknowledgments

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Сажетак: Истражена су топлотна својства композита на бази акрилата у комбинацији са алуминијум оксидним честицама (Al_2O_3). Матрицу композита представља поли (метилметакрилат) (ПММА) модификован диметил итаконатом (ДМИ). Као ојачање коришћене су чисте Al_2O_3 честице и Al_2O_3 честице допиране гвожђе оксидом (Al_2O_3-Fe) модификоване 3-аминопропил-триметоксианом (АМ) и метил-естрима масних киселина ланеног уља (биодизел-БД). Мерења топлотне проводљивости показала су да композити са честицама Al_2O_3 са највишим садржајем α – фазе има највеће вредности топлотне проводљивости. Механичка својства (затезна чврстоћа, модул еластичности и прекидно издужење) побољшана су додатком модификованих честица Al_2O_3 у ПММА/ДМИ матрицу. Топлотна својства су највише побољшана за композит са 3 мас.% $Al_2O_3-Fe-AM$ честице које су имале најнижу температуру у зони пукотина (10,28 %) у поређењу са додатком ПММА због боље крутости и највише топлотне проводљивости.

Кључне речи: Al_2O_3 честице, ојачање, композитни материјали, термичка својства.

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