Poly(methyl-methacrylate) (PMMA) has been one of the most widely used polymer material, due to the combination of properties, involving transparency, aesthetics, biocompatibility and, for many applications, adequate mechanical properties. One of the most notable applications have been denture bases. Today, 90% of all dentures are made from PMMA [1].

A special type of these biopolymer materials, autopolymerizing PMMA denture reline resins, have been widely used to provide better retention of removable protheses in cases of alveolar resorption, as well as for denture reparation in case of crack or fracture [2, 13]. However, their mechanical properties are compromised if compared to heat-polymerized materials used for base denture [3]. The main reason is higher unconverted monomer, as reported by various authors [4, 5, 16]. Monomer acts as a microvoid, representing an initial crack causing stress concentration, which makes the material less resistant. There are two methods of decreasing residual monomer content: by heat post-treatment, which can be achieved by immersion in hot water, or by microwave post-irradiation [6-11].

The purpose of this work is to find the optimal parameters for achieving the highest flexural and impact strength of a PMMA autopolymerizing dental resin. These mechanical properties were compared to residual monomer content. The residual monomer (methyl-methacrylate) over 5 %, quite common for an autopolymerizing PMMA dental resin may elicit irritation, inflammation, and an allergic response in oral mucosa, causing a lower biocompatibility [1, 14]. In addition, flexural and impact strength were compared to those of one of the most advanced and expensive PEMA dental repair materials. This material, having a different composition of two components (powder and liquid), does not contain methyl-methacrylate and therefore causes no negative effect on patient's mucosa [12].

The present work was carried out as a part of a continuing programme at the University of Novi Sad, to study the influence of microstructure, composition, and mechanical properties of polymer materials.

Experimental part

Materials used in this study were two commercial autopolymerizing denture resins, known by their trade names Simgal-R (Simgal, Galenika, Belgrade, Serbia) and Akrilat-R (ADA Dental Products, Belgrade, Serbia in collaboration with Dentaurum, Ispringen, Germany). The reference PEMA material used was Ufi-Gel Hard C (Voco, Cuxhaven, Germany). All materials were supplied separately in powder and liquid. When these two components are mixed, the polymerization process is initiated.

Fexural and Impact Strength of Microwave Treated Autopolymerized Poly(Methyl - Methacrylate)
Bremen, Germany). After polymerization, a set of SiC papers (150, 400 and 1200 grit) were used to get the desired shape and dimensions of the samples. Dimensions were verified by a Feinmesszeugfabrik (Suhl, Germany) micrometer, accurate to 0.01 mm at three locations.

Samples were divided in 13 groups, of which the first was left untreated, while the rest were irradiated in a domestic microwave oven with a turntable (Elin MW8020MG with output power of 800 W) at a frequency of 2450 MHz. Specimens were placed on the turntable and exposed to microwave irradiation directly. Power settings were 500, 550, 600 and 650 W, while irradiation time settings were 3, 4 and 5 minutes (power of 500 W and treatment duration of 5 minutes was designated as 500/5). These settings were chosen according to previous results [9 - 11], where higher power and shorter irradiation has shown to be more beneficial than lower power and longer irradiation, at the constant irradiation energy.

Flexural strength was determined using a mechanical tensile testing machine (Toyoseiki AT-L-118B, Tokyo, Japan), with a crosshead speed of 50 mm/min. The test was performed using 3-point bending, with the distance between the supports of 40 mm. Specimens dimensions were 6x2.5x50 mm.

Impact strength was determined using the standard Charpy method (Zwick D-7900, Ulm, Germany), with instrument capacity of 15 J and 160° angle. Specimen dimensions were 6x4x50 mm, without a notch.

The obtained results were statistically analyzed using an one-way analysis of variance (ANOVA), followed by Tukey's test with the significance value of P<0.05.

Differential Scanning Calorimetry (DSC) analysis was performed on TA Instruments Q20 device, to determine glass transitions temperatures as well as enthalpy of polymerisation. The analysis has been run from 20 to 200°C. Enthalpy of polymerization was compared to the results obtained by Fourier Transform Infrared Spectroscopy (FTIR). FTIR analysis was performed on a Thermo Nicolet Nexus 670 FTIR spectrometer (Thermo Electron Corporation, Madison, WI, USA), equipped with a deuterated triglycine sulphate (DTGS) detector. Carbonyl group (C=O) representing residual monomer of each specimen was detected. Each spectrum was obtained by co-addition of 32 scans at 4-cm⁻¹ resolution. All spectra were recorded at room temperature using standard instrument settings.

Visual examination of the treated specimens was performed in order to check if there occurred any surface changes, as the result of exposure to microwave irradiation. Fracture surfaces were examined by JEOL JSM-6460LV (JEOL Ltd., Tokyo, Japan) scanning electron microscope (SEM), operating at 25 kV. The specimens were coated with gold, using Balltec SCD-005 coating device.

### Results and discussion

Flexural strength and impact strength obtained for Simgal-R, Akrilat-R, and Ufi-Gel Hard C are shown in tables 1 and 2, respectively. For more convenience, the results are presented by charts as well, figures 1-4.

From tables 1 and 2, and figures 1-4, it can be seen that as the power and time increase, flexural and impact strength increase too. Furthermore, statistical analysis ANOVA followed by Tukey test (tables 1 and 2), shown that statistically significant differences occur mostly on samples treated at higher power and time settings. It can be seen that, although both are PMMA, there are differences between mechanical properties of Simgal-R and Akrilat-R.
In untreated condition, flexural and impact strength are higher for Akrilat-R. The same trend is noticed for maximum values in treated condition. Compared to Ufi-Gel Hard C, Simgal-R and Akrilat-R have higher mechanical properties, especially flexural strength. The main reason is that Ufi-Gel Hard C is basically a PEMA.

A typical DSC curve is shown in figure 5. Glass transition temperatures and enthalpies of transition are shown in table 3.

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From table 3, it can be seen that glass transition temperatures are all in the region from 51.98 and 62.75°C.
However, enthalpies of polymerization values are smaller as the irradiation power and time increase. Furthermore, at 650 W and 3, 4 and 5 min, polymerization does not occur, indicating that the tested material does not contain monomer (table 3, fig. 5).

FTIR results are shown in table 4, while representative FTIR curves are shown on figure 6. The results of FTIR analysis shown that methyl-methacrylate monomer content decreases, as the power and time microwave irradiation settings increase. This is in good agreement with the results of enthalpy of polymerization obtained by DSC analysis.

Visual examination of samples from both materials revealed that at irradiation power of 650 W and 5 min time, the surface of the samples becomes rough and irregular, figure 7. Furthermore, the sample becomes bent and the color is changed, making this power/time setting unacceptable from the aesthetics and patient mouth fitting points of view. This is the result of excessive heating from the turntable, which limits the available irradiation parameters to 650 W and 4 min.

The fracture mode obtained by SEM observation shows a typical brittle rupture by "quasi-cleavage" mechanism, figure 8. The observation indicated that fracture mode is not gradually influenced by microwave irradiation. However, fracture mode of Simgal-R and Akrilat-R is similar, and different from Ufi-Gel Hard C, which is expected, since they belong to a different type of polymer materials.

**Conclusions**

According to obtained results, some conclusions can be drawn.

Microwave post-polymerization treatment increases both flexural and impact strengths of both tested PMMA reline resins.

The rise in tested mechanical properties is the result of decreasing methyl-methacrylate monomer content, by the additional heat generated through microwave irradiation. Residual monomer acts as empty space, or a microvoid, which, under load may propagate and cause fracture.

The highest irradiation parameters, power of 650 W and 5 min time induce an excessive heat, that may cause unwanted changes in sample surface texture and bending. This is unacceptable from the point of view of aesthetics and patient’s mouth fitting.

Optimal microwave parameters, for achieving the highest flexural strength are: 650/3 for Simgal-R and 650/4 for Akrilat-R. For impact strength, the optimal parameters are: 650/4 for both Simgal-R and Akrilat-R.
Lower methyl-methacrylate content for microwave treated samples makes them more biocompatible, by reducing the risk of irritation and allergic reaction. The achieved monomer content makes them suitable for all but the most sensitive patients, offering considerably higher flexural and impact strength at lower cost than the most modern materials, such as Ufi-Gel Hard C.

By applying microwave post polymerization treatment, the resulting product may become more durable and user-friendly, while still having a lower cost.

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