

TESTING OF THE FRACTURE RESISTANCE OF THE HEAT-CURING DENTURE BASE ACRYLIC RESIN

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Abstract: For many years, poly-methyl methacrylate has been used as a material of choice for making the denture base, thanks to its good and desirable performances, such as: simplicity in work, possibility of reparation, aesthetics and affordable price. Considering to its insufficient hardness and fracture resistance, there is a tendency to improve the mechanical properties of the material, by changing its basic composition. The aim of the research was to determine the fracture resistance of the heat-curing denture base acrylic resin materials.

Materials and methods: For the research ,20 samples of the 2 heat-curing acrylics had been prepared, standard ones and reinforced acrylic resin material. After the storage in the saline for 15 days, measurements of the fracture resistance were performed by using the universal testing device. The data were statistically processed using the Student's t-test for independent samples.

Results: By measuring the flexural strength and deflection at breakage, it has been proven that there was, statistically, a significant difference of the flexural strength between reinforced (179.91-248.72MPa) and standard heat-curing acrylics (183.25-200.74MPa). The deflection at breakage showed approximately the same values for both materials (1.0-1.4mm; 1.0-1.5mm).

Conclusion: By enhancing the polymer, the mechanical properties of the denture base acrylic resin materials will be improved, primarily, higher fracture resistance, that means that these technologies need to be improved.

Keywords: heat-curing acrylic resin, fracture resistance, deflection at breakage, complete denture.

1. INTRODUCTION

One of the most commonly used dental materials in dental prosthetics is polymethyl methacrylate (PMMA), especially as a material of first choice for making the denture base for the reason of possessing good properties, such as: simplicity of work technology, possibility of reparation and relining, good possibilities of polishing, stability in the oral environment, satisfactory aesthetics and affordable prices [1]. Ideally, the material for making denture base will have appropriate mechanical properties such as hardness, strength, fracture resistance, elasticity

modulus, wear resistance, thermal and impact load [2]. However, the PMMA material is characterized by insufficient strength and fracture resistance and significant polymerization contraction [3].

Strength represents a resistance to the force and can be defined as tensile strength, compressive strength, flexural strength and tensile strength [4]. PMMA is characterized by high compressive strength, greater than it can develop during mastication. Important parameters are tensile and flexural strength [5]. Flexural strength is important because, when flexing the denture, depending on the direction of force, a combination of material compression on

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one side occurs, and the stretching of the material on the opposite side. Due to less tensile strength, the fracture of the material will occur on the stretching side. This is of particular importance when the dentures are made from fragile materials, such as PMMA, which has considerably greater compressive strength than the tensile, so that the fracture of the material is due to stretching [5,6].

Johnston et al. have shown that in the period of several years after the surrender of complete dentures in 68% of cases there is a fracture of the denture. [3] In the study of *Darbar*, 33% of the total number of complete denture repairs is loss of tooth, while 29% belongs to fractures in the area of the midline of the maxillary complete denture base [1].

Complete denture fracture is often found in clinical practice with the most common localization in the midline of the maxillary complete denture [7]. The reasons for denture fractures are multiple and most commonly associated with the method of manufacturing, the presence of residual monomers, the type of polymerization procedure, the presence of cracks and the poor stability of the dentures, or simply the fatigue of the material over the time [2,7].

Isma Lisa Ali in her 2008 study showed that light and heat-polymerizing PMMA showed significantly higher values of surface hardness, tensile strength, and modulus of elasticity compared to autopolymerizing PMMA [8].

Material fatigue is one of the most important mechanical properties of the material, which is based on the fact that material breakage occurs at lower load stresses due to cyclic repetition. The estimated time to use mobile complete and partial denture should be 5 years, after which fatigue and cracking occur even at low loads [5].

Fractures of complete dentures occur in intra-oral (extensive mastication forces, inadequate occlusal plane, strong attachment of the upper and lower lip frenulas, errors in the applied occlusion concept) and extraoral conditions (fallout of dentures during coughing, or simply placing dentures onto a hard surface) [7].

The literature data suggest that there are attempts to improve the mechanical properties of PMMA in order to reduce the incidence of denture fractures. For this purpose, the chemical correction of the polymer base by the addition of polyethylene glycol dimethacrylate or the reinforcement of the material by the addition of fibers of different origin has been attempted [9].

Some studies have examined the effect of the addition of glass, carbon or polyethylene fibers [10]. Research has also been carried out regarding the addition of various inorganic substances. However, the

problem with the addition of inorganic substances is related to the fact that the biocompatibility of the base material can be compromised and in the patient exhibit some of the signs of oral irritation [11].

The addition of ZrO₂ in order to improve the mechanics of PMMA, is considered significant for the reason of the biocompatibility of the material, possessing high fracture resistance and the possibility of significant reinforcement of the material by creating a new generation of acrylic ceramic matrix [12].

Asar have shown that the addition of 2% ZrO₂ to PMMA achieves a maximum resistance value of 6.55 kJ / m² fracture. However, some other authors suggest that the addition of 20wt% ZrO₂ results in a fall in resistance to PMMA breakdown and surface hardness by 3-6% [13].

Some studies have also presented attempts to introduce alternative polymers as a selection material for the development of a denture base such as polyamide, epoxy resin, polystyrene or vinyl acrylic. With all attempts, an ideal material for making a complete denture has not been found and designed.

In order to improve the antimicrobial properties of the PMMA base material, TiO₂ and SiO₂ were added which also showed a significant effect on the value of the tensile strength of PMMA which was directly correlated with the amount of added nanoparticles [14].

The most commonly used method of testing flexural properties of denture base material is the three-point flexural test, adopted by international standards for polymer materials, including ISO 1567: 1999, Dentistry-Denture base polymers. [15] This method is successfully applied in the fields of fracture resistance research, elasticity modulus and fracture energy of various denture base materials [16].

Because conventional acrylate is still the most commonly used in dental practice, the purpose of this research was to measure the fracture resistance of the standard heat-polymerized acrylic based on PMMA and the reinforced heat-curing acrylic resin, to examine whether reinforcement of the acrylic resin achieves better mechanical properties, above all a higher fracture resistance which is otherwise considered the main disadvantage of complete dentures.

2. MATERIAL AND METHODS

Two materials from the group of heat-curing acrylic resin, Triplex hot (Ivoclar Vivadent AG, Schaan, Liechtenstein) and Superacryl plus (Spofidental A.S., Jičín, Czech Republic) were used in the

study. Of each material, 10 samples were made of dimensions 50x9x4mm. Waxed models of the samples are converted to acrylic samples using standard procedures of moulding technique and heat polymerization (for 45 minutes at 100 ° C in a water bath) (Figure 1, 2, 3).

After the finishing of the polymerisation procedure, acrylic samples were polished with sandpaper (Figure 4, Figure 5).



Figure 1. Vax model



Figure 2. Placing of vax samples in dental flask



Figure 3. Dental flask before applying of acrylic material



Figure 4. Acrylic samples before polishing

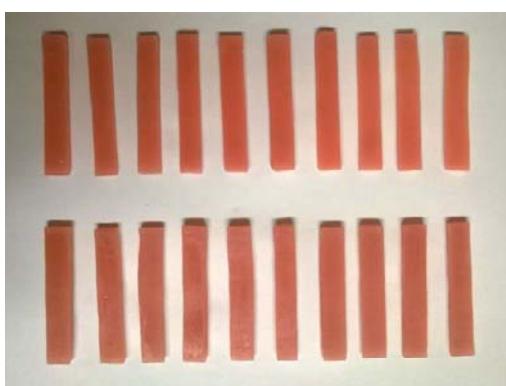


Figure 5. Acrylic samples. Triplex Hot and Superacryl plus

The samples were stored in physiological saline at room temperature for 15 days in order to simulate the conditions of the oral environment (Figure 6).



Figure 6. Samples in physiological saline

The final dimensions of the acrylic samples are shown in Table 1.

Table 1. Acrylic samples dimensions.

		Samples dimensions (mm)									
		I	II	III	IV	V	VI	VII	VIII	IX	X
Triplex hot	width	8,0	8,8	8,3	8,7	8,8	8,3	8,4	8,5	8,6	9,0
	height	4,3	4,4	4,4	4,3	4,3	4,3	4,0	4,4	4,1	4,0
Superacryl plus	width	8,2	8,9	9,3	9,5	9,1	8,0	8,6	9,1	9,1	8,9
	height	4,0	4,0	4,2	4,1	4,1	4,1	4,0	4,4	4,4	4,2

The measurement of fracture resistance was performed at the Universal Testing Device (Instron, Model 1122 Reconditioned, Norwood, Massachusetts USA) (Figure 7). Each sample is placed on two supports. The velocity of the jaw is adjusted to 1mm/min, while the support range is set to 30mm.

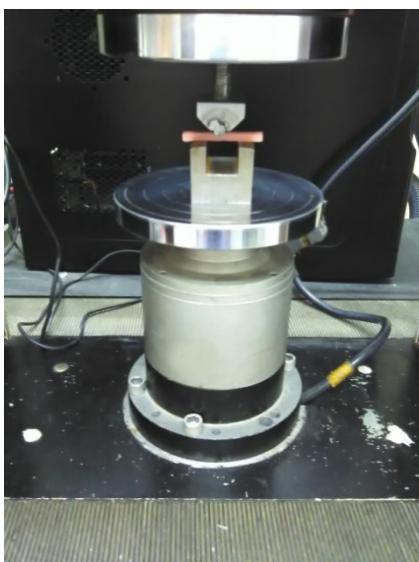


Figure 7. Universal Testing Device with acrylic sample in the appropriate position.

Parts of the samples after fracture are shown in Figures 8. and 9.



Figure 8. Triplex hot



Figure 9. Superacryl plus

The results of the maximum force were obtained which led to the break of all 20 samples, as well as the fracture values in the breakdown.

By applying the appropriate formula, the value of the flexural strength for each sample is calculated separately:

$$\sigma = \frac{F * l}{4} * \frac{6}{b * h^2}$$

F – applied force

l – support range (30 mm)

b – sample width

h – sample height

The obtained results were statistically evaluated by using Student's t-test for independent samples.

3. RESULTS

Calculation of the flexural strength (σ) yields, the results shown in Table 2. In the same table, the results of the break deflection (l), which is read directly on the device at the moment of fracture and expressed in mm, with an accuracy of 0.1 mm.

Table 3. shows the range of the flexural strength and the break deflection for the tested materials (from minimum to maximum values) as well as the standard deviation.

Table 2. The results of the flexural strength (σ) expressed in MPa and the angle of fracture (l) expressed in mm.

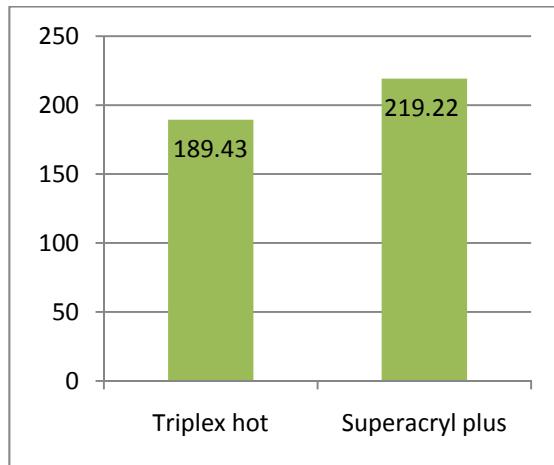
		I	II	III	IV	V	VI	VII	VII	IX	X
Triplex hot	σ	185,5	200,7	184,8	190,7	185,6	187,6	188,3	183,2	189,1	198,4
	l	1,2	1,2	1,3	1,2	1,0	1,4	1,3	1,2	1,1	1,3
Superacryl plus	σ	241,5	220,7	230,2	179,9	223,2	248,7	209,3	199,2	232,4	206,3
	l	1,4	1,2	1,2	1,1	1,2	1,5	1,0	1,3	1,3	1,4

Table 3. Minimum and maximum values of flexural strength and break deflection and standard deviation of tested materials.

	Flexural strength			Break deflection		
	Min-max	Mean	Standard deviation	Min-max	Mean	Standard deviation
Triplex hot	183,25-200,74	189,43	5,81	1,0-1,4	1,22	0,11
Superacryl plus	179,91-248,74	219,22	21,65	1,0-1,5	1,26	0,15

According to research results, Triplex hot material showed lower variability compared to Superacryl plus material. Accordingly, the first group of samples shows greater homogeneity, which

gives more precise and accurate further results of the research. The standard deviation in the break deflection does not show significant variability between the tested materials.

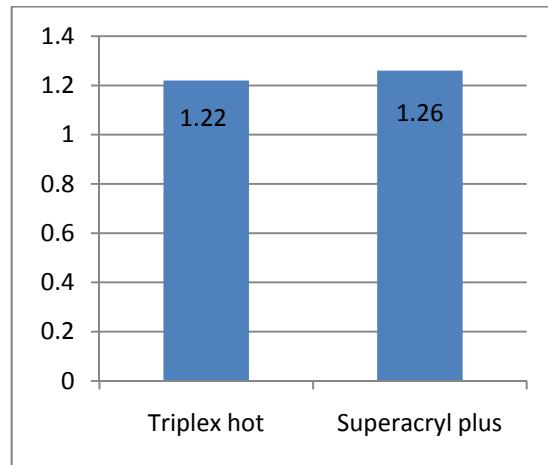
**Chart 1.** Flexural strength (MPa).

Using the Student's t-test with a significance level of $p < 0.05$, the value $t = 3.99$ was greater than 2.31, and it can be concluded that there is a statistically significant correlation according to flexural strength between two groups of tested materials. It has been proven that there is a statistically significant difference in the flexural strength between the reinforced (179.91-248.72 MPa) and the conventional heat-curing acrylic resin (183.25-200.74 MPa) (Chart 1).

For the measured break deflection, the t-test value is 2.17, which is less than 2.31 and it follows that $p > 0.05$ and states that there is no statistically significant difference (1.0-1.4 mm; 1.0-1.5 mm), and by assessing this property, none of the above materials can be given priority (Chart 2).

4. DISCUSSION

Literary data indicate that the fracture of complete denture after insertion depends primarily on the mechanical properties of the material. In this regard, increasingly modified polymers are added with the addition of new crosslinkers or rubber compounds, for example styrene-butadiene, and evaluate the effect on flexural strength. It was found that the addition of such materials yields better results of

**Chart 2.** Break deflection (mm).

flexural strength with a minimal drop of Young's module. It has been proven that reinforced PMMA shows up to 2.4 times higher flexural strength compared to standard PMMA [2,16].

The values of the tensile strength of PMMA are significantly changed by the addition of Al_2O_3 particles to the basic structure of the material. Namely, the addition in the amount of 2.5 wt% results in an increase in tensile strength by 6.36% [3].

Fracture resistance of the conventional PMMA can be significantly increased if there is an addition of aluminum/yttrium stabilizer zirconia ($\text{Al}_2\text{O}_3 / \text{YSZ}$) filler with nitrile-butadiene rubber (NBR) particles in the material structure. The optimum amount or size for improving the mechanical properties of PMMA is 10% NBR together with 5 wt% of 50% $\text{Al}_2\text{O}_3 / 50\%$ YSZ [1] and 7.5% NBR together with 2.5% $\text{Al}_2\text{O}_3 / 2.5\%$ YSZ [13].

It is believed that the best mechanical performance of PMMA material is achieved by adding 7 wt% ZrO_2 [11], while the recommended amount of added TiO_2 is 1 wt%, as with further increase in the amount of TiO_2 the tensile strength decreases [8,14].

There are no studies to confirm the clinical applicability and justification of PMMA modification with hydroxyapatite HAP ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$) in order to improve mechanical properties [11].

In our study, the conventional heat-curing PMMA (Triplex Hot) showed less resistance to fracture compared to reinforced PMMA (Superacryl plus), while the results of the break deflection were approximately equal. The material manufacturer of Superacryl Plus states that PMMA powder has been added zinc oxyde but not as much as a percentage. In the Safety Data Sheet for Triplex hot, there is no information regarding the addition of fibers or metal oxides. Also, the amount of ethylene glycol dimethacrylate is different in both tested material, Superacryl plus 5-10% and Triplex hot 3-10% which can also be one of the reasons for the superiority of the Superacryl plus material in terms of fracture resistance. However, this study did not investigate other mechanical properties of Superacryl plus material as well as biocompatibility of the same, and its absolute recommendation in terms of advantages over conventional PMMA for clinical practice cannot be performed.

Increasing the polymerization temperature reduces the content of the residual monomer. The lowest content of the residual monomer (0.07%) was determined after the hot polymerization procedure at a temperature of 100°C for a period of 12 hours, which confirms the importance of the temperature and duration of the polymerization [2]. In this study, a polymerization protocol ending at a temperature of 100°C was applied in order to minimize the percentage of residual monomers. The conversion of monomers into polymer is an important determinant of the mechanical strength of the tested material [17].

In a study that compared the mechanical properties of materials with different polymerization methods, it has been shown that the polymerization method influences the mechanical performance of PMMA in terms of better light and thermal polymerization acrylates compared to autopolymerizing [8].

Data on the influence of polymerization methods on acrylic materials on mechanical properties are different, so *Memon et al.* found that microwave polymerizing acrylates as well as a new group of materials based on polyurethane compared to conventional PMMA show no advantage when compared flexural strength [2,18,19].

A review of the literature has shown that the optimal period of storage of samples of polymerized acrylate prior to testing of mechanical properties for 15 days, which was used in this study [20,21].

The results of this study showed that after the break, each sample of both tested materials was broken into two parts. A study by other authors shows that the addition of PMMA glass fibers during the examination of the mechanical properties of the mate-

rial happens that the fragments after the applied force remain linked by the central reinforcement, or that there is no complete separation of the fragments [22].

5. CONCLUSION

By strengthening the polymer of acrylate denture base materials, improved mechanical properties are achieved, above all, greater fracture resistance, and it should be aimed at improving these technologies.

6. REFERENCES

- [1] H. Alhareb, Z. Akil, Ahmad, *Mechanical Properties of PMMA Denture Base Reinforced by Nitrile Rubber Particles with Al₂O₃/YSZ Fillers*, Procedia Manufacturing, Vol. 2 (2015) 301–306.
- [2] V. Bubalo, V. Jerolimov, I. Baucic, M. Andreis, Z. Veksili, *Akrilatni polimeri u dentalnoj protetici: Priprava i mehanicka svojstva*, Polimeri, Vol. 33 (2013) 22627.
- [3] M. Vojdani, R. Bagheri, A. Khaledi, *Effects of aluminum oxide addition on the flexural strength, surface hardness, and roughness of heat-polymerized acrylic resin*, Journal of Dental Sciences, Vol. 7 (2012) 238–244.
- [4] B. Petronijevic, D. Markovic, I. Sarcev, A. Andelkovic, M. Jeremic Knezevic, *Fracture resistance of restored maxillary premolars*, Contemporary Materials, Vol. III-2 (2012) 219–225.
- [5] M. Potran, *Ispitivanje biokompatibilnosti i mehanickih karakteristika polimera za bazu zubne proteze*, doktorska disertacija, Autorski reprint; 2015.
- [6] A. E. Prombonas, D. S. Vlissidis, *Comparison of the midline stress fields in maxillary and mandibular complete dentures: A pilot study*, Journal of prosthetic dentistry, Vol. 95 (2006) 63–70.
- [7] J. A. Filho, L. E. Butignon, R. Pereira, M. Lucas, F. Mollo Junior, *Flexural strength of acrylic resin repairs processed by different methods: water bath, microwave energy and chemical polymerization*, Journal of Applied Oral Science, Vol. 19 (2011) 249–253.
- [8] I. L. Ali, N. Junus, M. I. Abu Hassan, *Hardness, flexural strength, and flexural modulus comparisons of three differently cured denture base systems*, Journal of Prosthodontics, Vol. 17 (2008) 545–549.
- [9] S. Shirkavand, E. Maslehfard, *Effect of TiO₂ nanoparticles on tensile strength of dental acrylic resin*, Journal of Dental Research, Dental Clinics, Dental Prospects, Vol. 8 (2014) 197–203.

- [10] K. Narva, L. Lassila, P. Valittu, *The static strength and modulus of fiber reinforced denture base polymer*, Dental Materials, Vol. 21 (2005) 421–428.
- [11] Y. Pan, F. Liu, D. Jiang, H. Yu, M. Zhu, *Novel acrylic resin denture base with enhanced mechanics properties by the incorporation of PMMA modified hydroxyapatite*, Progress in natural science: Materials International, Vol. 22 (2013) 89–93.
- [12] M. Ahmed, M. Ebratim, *Effect of zirconium oxide nano-filters addition on the flexural strength, fracture toughness, and hardness of heat-polymerized acrylic resin*, World Journal of Nano Science and Engineering, Vol. 4 (2014) 50–57.
- [13] A. O. Alhareb, H. M Akil, Z. A. Ahmad, *Impact strength, fracture toughness and hardness improvement of PMMA denture base through addition of nitrile rubber/ceramic fillers*, The Saudi Journal for Dental research, Vol. 8–1/2 (2017) 26–34.
- [14] A. Sodagar, S. Bahador, A. Khalil, S., M. Kassaei, *The effect of TiO₂ and SiO₂ nanoparticles on flexural strength of poly(methyl methacrylate) acrylic resins*, Journal of Prosthodontic Research, Vol. 57 (2013) 15–19.
- [15] H. Lee, C. Lee, K. Asaoka, *Correlation in the mechanical properties of acrylic denture base resins*, Dental Materials Journal, Vol. 31 (2012) 157–164.
- [16] F. Foat et al., *Impact and flexural strength, and fracture morphology of acrylic resins with impact* modifiers, The Open Dentistry Journal, Vol. 3 (2009) 137–143.
- [17] A. Maletin, D. Markovic, I. Nesovic, B. Petronijevic, T. Veljovic, I. Ristic, *Degree of polymerization of dual cure resin cement measured by FTIR*, Contemporary Materials, Vol. IX-1 (2018) 16–21.
- [18] M. Memon, N Yunus, A. Razak, *Some mechanical properties of a highly cross-linked, microwave-polymerized, injection-molded denture base polymer*, International Journal of Prosthodontics, Vol. 14–3 (2001) 214–218.
- [19] A. Loncar, D. Vojvodic, F. Matejicek, V. Jerolimov, *Savojna cvrstoca materijala za izradu proteznih baza*, Acta stomatologica Croatica, Vol. 40–2 (2006) 151–161.
- [20] G .Uzun, N. Hersek, *Comparison of the Fracture Resistance of Six Denture Base Acrylic Resins*, Journal of Biomaterials Applications, Vol. 17–1 (2002) 19–28.
- [21] A. Cilingir, H. Bilhan, O. Geckili, T. Sulun, E. Bozdag, E. Sunbuloglu, *In vitro comparison of two different materials for the repair of urethan dimethacrylate denture bases*, The Journal of Advanced Prosthodontics, Vol. 5–4 (2013) 396–401.
- [22] I. Mikulic, *Ojacanje dentalnih polimera staklenim vlaknima*, završni rad, Zagreb, Sveučilište u Zagrebu, Fakultet strojarstva i brodogradnje, 2015.

СОДРЖИНОВА

ИСПИТИВАЊЕ ОТПОРНОСТИ НА ЛОМ ТОПЛО-ПОЛИМЕРИЗУЈУЋЕГ АКРИЛАТА ЗА ИЗРАДУ БАЗЕ ТОТАЛНЕ ЗУБНЕ ПРОТЕЗЕ

Сажетак: Дуги низ година материјал избора за израду базе тоталне зубне протезе представља полиметил-метакрилат из разлога бројних добрих и пожељних својстава, као што су: једноставност рада, могућност репаратуре, задовољавајућа естетика и приступачна цена. С обзиром на недовољну чврстоћу и отпорност на лом, постоји тежња за побољшањем механичких својстава материјала мењањем његовог основног састава. Циљ истраживања био је одређивање отпорности на лом топло-полимеризујућег акрилата за израду базе тоталне зубне протезе.

Материјал и методе: За потребе истраживања припремљено је укупно 20 узорака два топло-полимеризујућа акрилата, стандардног и акрилата ојачаног мрежастом структуром. Након чувања у физиолошком раствору у трајању од 15 дана, мерења отпорности на лом извршена су на Универзалној кидалици. Подаци су статистички обрађени применом Студентовог т-теста за независне узорке.

Резултати: Мерењем савојне чврстоће и угиба при лому доказано је да постоји статистички значајна разлика савојне чврстоће између ојачаног (179,91–248,72 MPa) и стандардног топло-полимеризујућег акрилата (183,25–200,74 MPa). Измерени угиб при лому показао је приближно исте вредности код оба материјала (1,0–1,4 mm; 1,0–1,5 mm).

Закључак: Ојачавањем полимера постижу се побољшана механичка својства акрилатних материјала за израду базе тоталне зубне протезе, пре свега већа отпорност на лом, те треба тежити усавршавању ових технологија.

Кључне ријечи: топло-полимеризујући акрилат, отпорност на лом, угиб при лому, тотална зубна протеза.

СВЕЧА