Study regarding the resistance of different materials used in dental medicine

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According to the World Health Organization, the oro-dental health is an important pylon of the quality of life. In this context, the edentation (loss of tooth) represents the pathological state peak of the oral cavity with a negative impact on the physiology of the human body. This study proposes a comparative evaluation on the resistance to tension of two highly used dental materials used in dental prosthetics such as poly (methyl methacrylate) and composite resin. The obtained result highlights a greater resistance of poly (methyl methacrylate) than composite resins, also put forward that a high temperature realizes a complete polymerization of poly (methyl methacrylate), and consequently presents higher resistance. To characterize sample TEM (Transmission Electron Microscopy) techniques was used, included conventional images, selected area electron diffraction, and high-resolution images.

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Keywords: Poly (methyl methacrylate), Composite resin, Resistance, Dental prosthetics, TEM, SAED

1. Introduction

According to several studies conducted by W.H.O. (World Health Organization), in 2012, approximately 30% of the global population presents different types of edentation [1]. Edentation is not necessarily linked to the old age; there is oro-dental disease with a multifactor and complex etiology like the periodontal disease that could lead to irreversible loss of teeth even to the young adults due to juvenile periodontal disease [2]. The W.H.O. emphases that oral health is a fundamental factor that contributes to the quality of life [3]. Therefore, the loss of teeth is a major cause that decreases the quality of life; in this direction, the public oro-dental programs included in the working plan of the European Parliament establish a line of action in the healthcare dominium in the period 2008-2013 to increase the quality of life by implementing measures to prevent the edentation and to rehabilitate the edentulous patient [4,5].

For this reasons, in the present day there are some multi and interdisciplinary studies that have as a goal the decrease of this dishabituation. Current progress registered in the field of dental prosthetics is closely linked to the technical progress, the invention of new materials that will be used in dental prosthetics, as well as the establishment of the biocompatibility level of these new materials in the human body [6].

Since the treatment of edentation with dental implants is a hardly accessible way for many patient due to the high price, the partial prosthetics or total prosthetics, remains for the time being as the most frequently used therapeutic modality; in this order of thoughts, we witness in time evolution of the materials used in dental prosthetics, starting from the simple metal alloys and ending with the new materials that have superior quality regarding the resistance and biocompatibility like resistant resins and zirconia [7].

We have selected two materials widely used in the dental prosthetics, poly (methyl methacrylate), and composite resin. The objective of this study is to test the resistance of these two materials, poly (methyl methacrylate) and composite resin, using as evaluation parameters the breaking point and tension.

2. Experimental materials

Composite resins (Amelogen Shade A3.5 and B1) and premacryl powder was embedded in epoxy resin, the sample was cut by means of Ultramicrotome Ultracut R with a diamond knife to obtain slices with 90 nm thickness. The Premacryl chemical solution was deposed directly by dripping on the copper grid covered with formvar film.

Poly(methyl methacrylate) is a synthetic resin, methyl methacrylate polymer, transparent, thermoplastic; is a material with a low mass and density of 1.17-1.20 g/cm3, has good impact resistance (more than glass and polystyrene) but still lower than the resistance of polycarbonate. For testing first group of materials, namely poly(methyl methacrylate) it was used Duracryl commercial product that has many advantages: easy handling and dimensional stability so that it can be used both in the dental office and the dental laboratory. Duracryl is composed of polymers (autopolymerizing acrylic, polymethyl methacrylate), red pigments and additives (self-curing monomer, methyl methacrylate, ethylene dimethacrylate) amines, which catalyze the reaction. The relevant physical properties of Duracryl are 0.10% -0.50% polymer humidity and a working time of 5-6 minutes at 23°C.

To obtain the study material, Duracryl was blended in a ratio of 3:1 (three parts powder to 1 part liquid); the resulting material was casted in four pieces in forms with approximately similar surfaces and big enough to face the testing to the breaking point. The materials were then removed from the molds and immersed in a bowl of water at 85 °C as follows: three samples were immersed for 30

minutes, and one for 60 minutes. Duracryl samples (poly (methyl methacrylate)) are obtain in the form of disc (about 5 cm diameter and 1cm thickness). Discs are trimming in blocks, suitable for ultramicrotome mounting device, using a diamond knife under water, to prevent thermic degradation of samples. Blocks for ultramicrotomy was finished at one end to form a pyramid. TEM sections was obtained using a diamond knife and mounted on 400mesh Cu grid without support.

Transmission Electron Microscopy was used to characterize morphological and structural features of materials. The sections was investigate using CM120ST microscope, follow the standard procedure for BFTEM, HRTEM and SAED. The sample annealed at 100°C and 120°C, for 5 min (p5m100c, p5m120c), 10 min (p10m100c, p10m120c), 15 min (p15m100c, p15m120c), 20 min (p20m100c, p20m120c) and 30 min (p30m100c, p30m120c) was investigate.

3. Results and discussions

3.1 Electron microscopy results

Fig. 1 shown morphological feature of composite resin slice, using a selected zone from conventional TEM image. We identified particles with characteristic dimensions that can be classified as nanoparticles, with mean size about 85 nm, and material pieces with large dimensions in range 200-1000nm.

The mean size was determined assuming a lognormal distribution of Feret diameter (Fig. 2) measured from image. Fig. 3 shows the electron diffraction, having the characteristics of an amorphous material, the two peaks situated on 0.210 nm, respectively 0.121 nm.

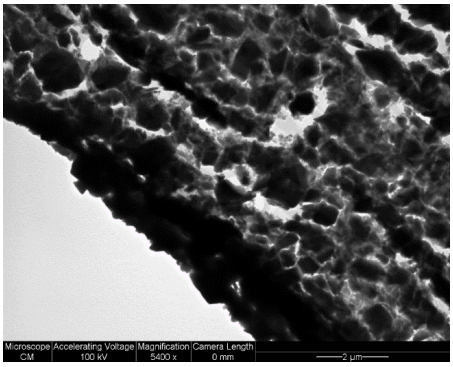


Fig.1. Selected area in composite resin slice

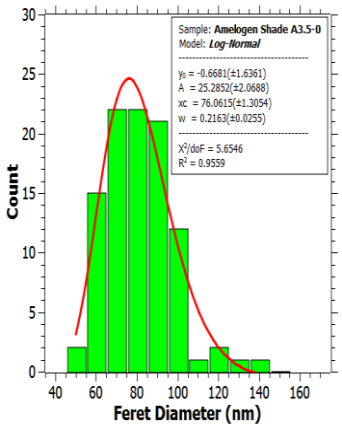
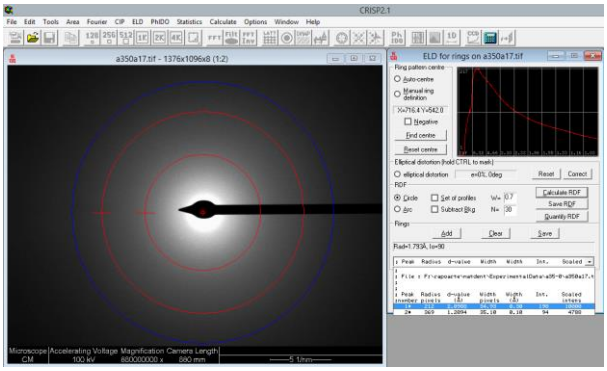
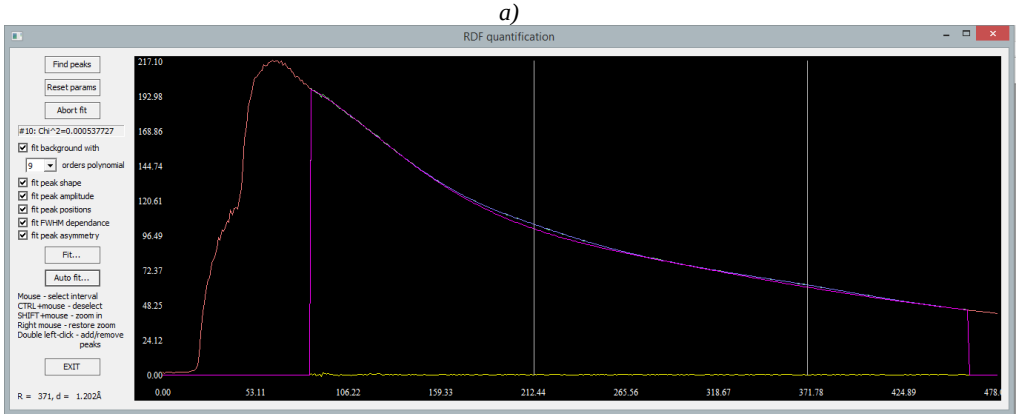


Fig.2. Lognormal distribution for composite resin slice



a)



b)

Fig.3. Electron diffraction for composite resin. a) Identification of peaks in diffraction pattern, b) The position of peaks in extracted profile in Crisp2 application

In fig. 4 is presented a selected zone in a Premacryl solution on the cooper grid cover with formvar, containing spherical particles. The histogram to determine the Feret diameter based on lognormal distribution is shown in fig. 5. The approximate diameters by 60 nm and 18 nm respectively. Electron diffraction pattern (fig. 6) reveals an amorphous structure of the Premacryl solution, with two peaks around 0.210 nm, and 0.116 nm respectively

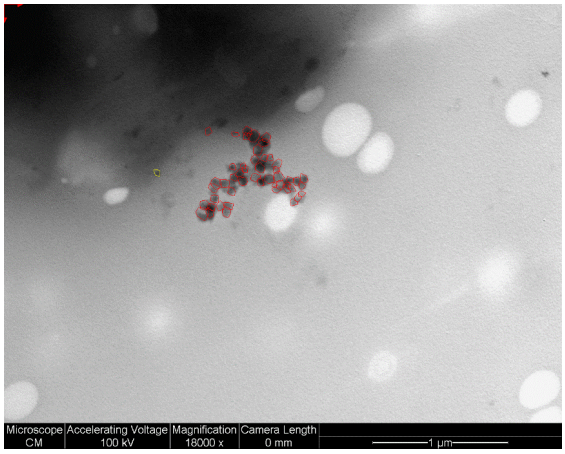


Fig.4. Selected zone to determine characteristic sizes for Premacryl solution sample

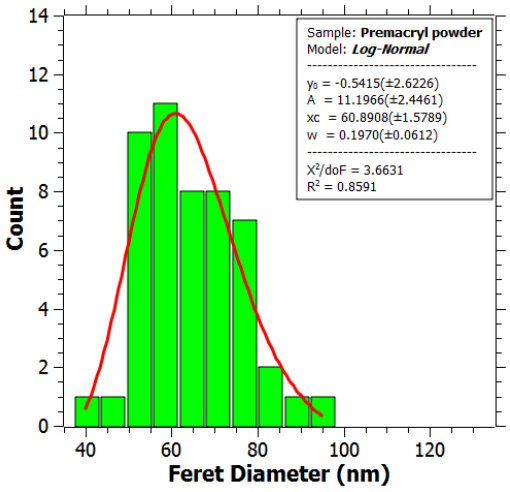
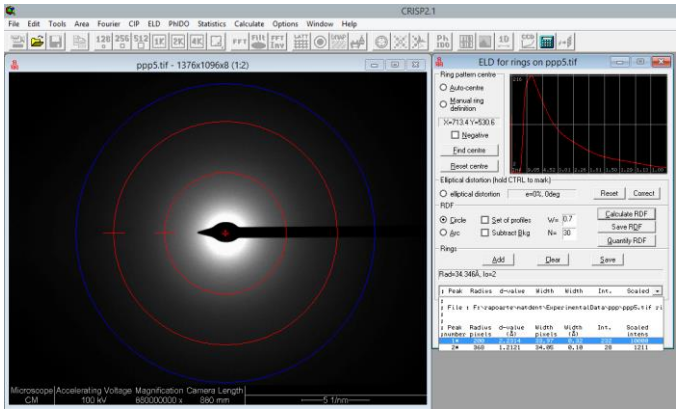
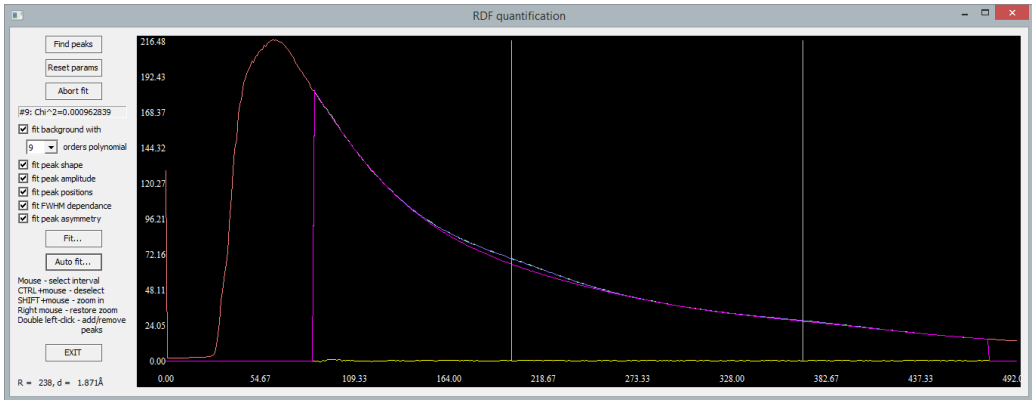


Fig.5. Lognormal distribution for Premacryl solution sample.

Duracryl samples morphologies are close to each other, an amorphous matrix, with different behavior in electron beam that depends on exposure time. In addition, we identified crystalline inclusion in amorphous matrix, but cannot be assigned to initial reactive that are used to form final material. Distribution of inclusions is not uniform, and is randomly on film as we can see in fig. 7, for sample p5m120c.



a)



b)

Fig.6. Electron diffraction Premacryl solution sample. a) Identification of peaks in diffraction pattern, b) The position of peaks in extracted profile in Crisp2 application

Comparative studies of Michelson contrast [8] ($C = \frac{I_{max}-I_{min}}{I_{max}+I_{min}} = \frac{2 \cdot SD}{mean}$), was carried out to evaluate thickness and composition of films. Fig. 8 present sample p5m100c, p15m100c, p30m100c and same sample at 120°C.

The dark regions in TEM image show are that are begin to deteriorate because of thermal heat in electron beam. The sample anneal at 120°C for 15, 20 and 30 minutes are more resistant in this case. Michelson contrast was evaluate for 256x256 pixels area. The mean value and standard deviation are presented in table 1.

Table 1. Michelson contrast

Sample	100°C			120°C		
	mean	SD	C	mean	SD	C
5 min	154	37	0.48051948	132	43	0.65151515
10 min	125	47	0.752	120	40	0.66666667
15 min	124	45	0.72580645	128	43	0.671875
20 min	135	40	0.59259259	115	40	0.69565217
30 min	130	48	0.73846154	121	47	0.7768595

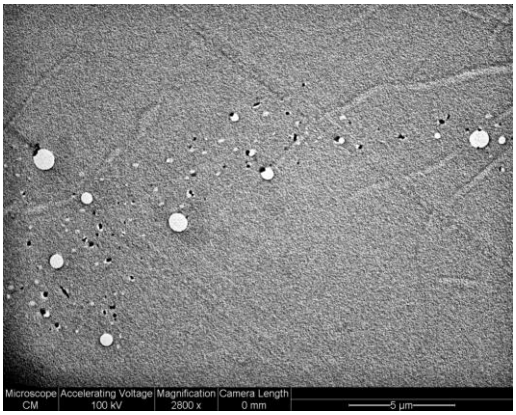


Fig. 7. Distribution of crystalline inclusions.

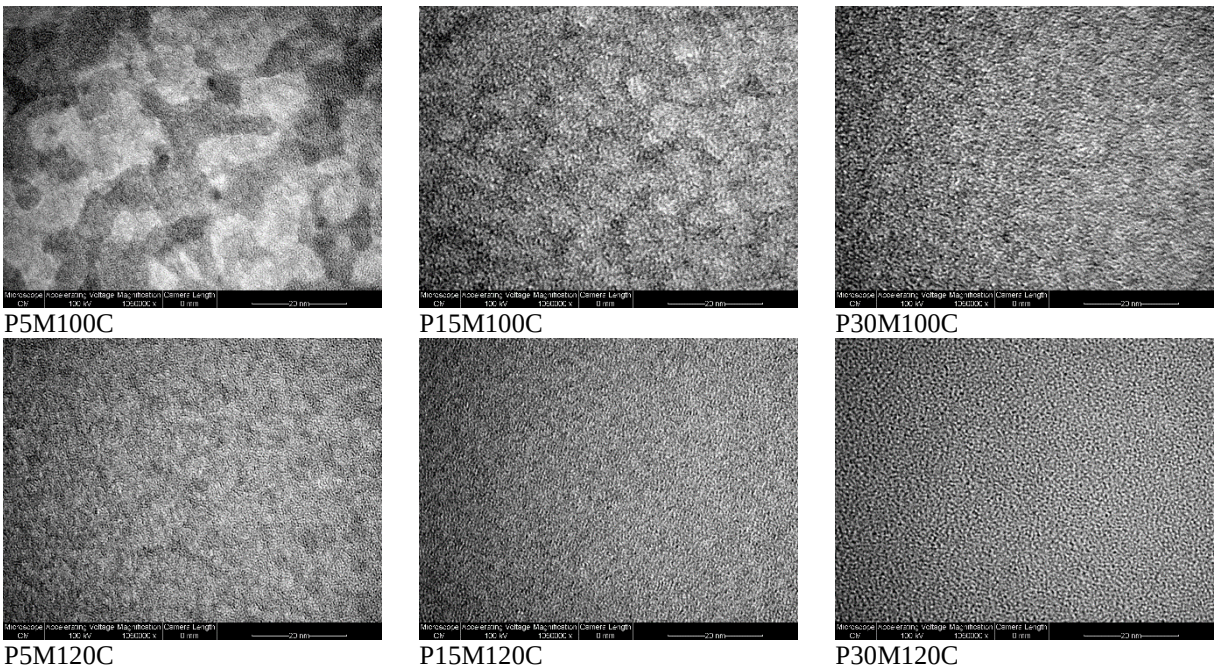


Fig. 8. BFTEM of investigated sample

We can relate the Michelson contrast to material composition, due to fact that intensity of image is direct proportional with atoms distribution (we work with electron beam so the nucleus of atoms must be consider). In fact, amorphous sample can be describe using Debye approximation [9], given by

$$I(S) = \sum_m \sum_n f_m f_n \frac{sin(Sr_{mn})}{Sr_{mn}}$$

where f_m, and f_n represent scattering factor for atoms m and n, S are given by $S = 4\pi sin\theta/\lambda$, and r_{mn}. Using Fourier representation of Debye equation, we can calculate electron distribution,

$$4\pi r^2 \rho(\boldsymbol{r}) = 4\pi r^2 \rho_0 + \frac{2r}{\pi} \int_0^\infty Si(S) sin(rS) dS$$

where $\rho(\boldsymbol{r})$ are densities at distance $\boldsymbol{r}$ from origin point, ρ_0 mean density, and $i(S) = \frac{I}{N} f^2 - 1$, can be evaluated experimentally. In table 1, we observe that standard deviation has a mean value close to 43, so we can conclude same behavior for all sample. HRTEM image on some crystalline inclusions are show in fig. 9, for sample p5m100c (with fringes associated with 0.129 nm crystalline planes) and for sample p30m120c (with fringes associated with 0.376 nm and 0.188nm crystalline planes).

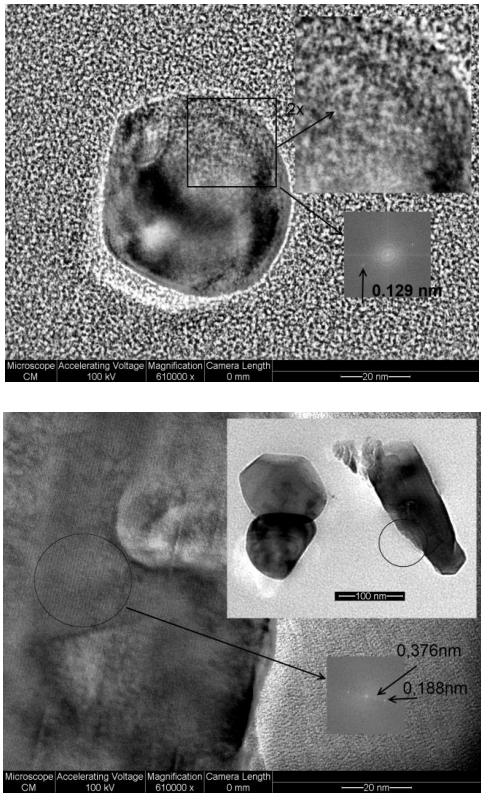


Fig. 9. HRTEM on sample p5m100c and p30m120c.

In conclusions, Duracryl samples exhibit amorphous structures with concentration that depends on temperature and time of thermal treatment. In addition, all sample have crystalline inclusion with linear dimensions around 50 nm.

3.2 Mechanical testing

The Composite dental resins are synthetic resins highly used in dentistry as restorative materials and adhesives and have the following qualities: insoluble, aesthetic, low tendency to dehydration, easy to handle and relatively inexpensive. Dental composite is a resin based on a matrix (bisphenol A-glycidyl methacrylate) or a urethane dimethacrylate and a photo initiator such as silicon dioxide.

To test the second group of materials we used commercial product Aelita APB, light-cured composite hybrid resin. From APB Aelita product two samples were poured with approximately equal surfaces and large enough to face the testing to the breaking point; subsequently the two samples were light cured.

Since in this study we aimed to compare these two materials in terms of resistance, the all six samples made from these two materials had similar surfaces. In fig. 1 we present the first 4 samples poly (methyl methacrylate).

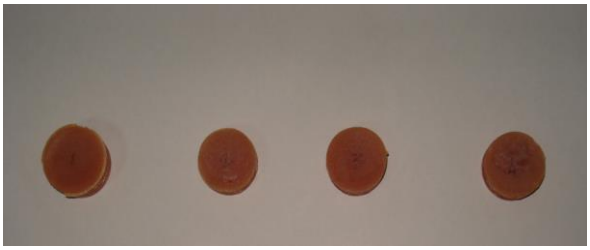


Fig.10. The Final form of Duracryl samples

To characterize these six samples was provided to the Universal Testing Machine, which is intended for testing tensile strength, compressive strength and bending strength of different materials. The machine is equipped with a low- inertia-bar dynamometer with a recorder for stress strain diagrams and a load stabilizer. Beyond static tests, the machine also permits to carry out dynamic tests within the tensile and compressive pulsating load range. An infinitely variable driving unit is used to operate the pulsator on each desired load cycle frequency within the frequency range; the maximum load that can be applied is 100 Ton. The stroke volume of the pulsator is 0 - 240 cc and the amplitude of vibration is 0 - 5 mm (Fig. 11).

Fig.11. Universal Testing Machine used for testing tensile strength, compressive strength and bending strength of different materials.

The values obtained by testing the samples 1-6, on an increased pressure, are shown in table 2.

As we have shown in table 2, poly(methyl methacrylate) samples 2,3,4 that were immersed in water for 30 minutes had an average resistance strength of 51,69 MPa; the composite resin samples 5,6 had an average resistance strength of 27 MPa. This composite resin had an average resistance strength 1.91 times smaller than the poly (methyl methacrylate). From the group samples of poly (methyl methacrylate), the sample 4 registered the smallest resistance with a value of 39.29 MPa, obtained value due to the incorporation of air in the process of blending the material.

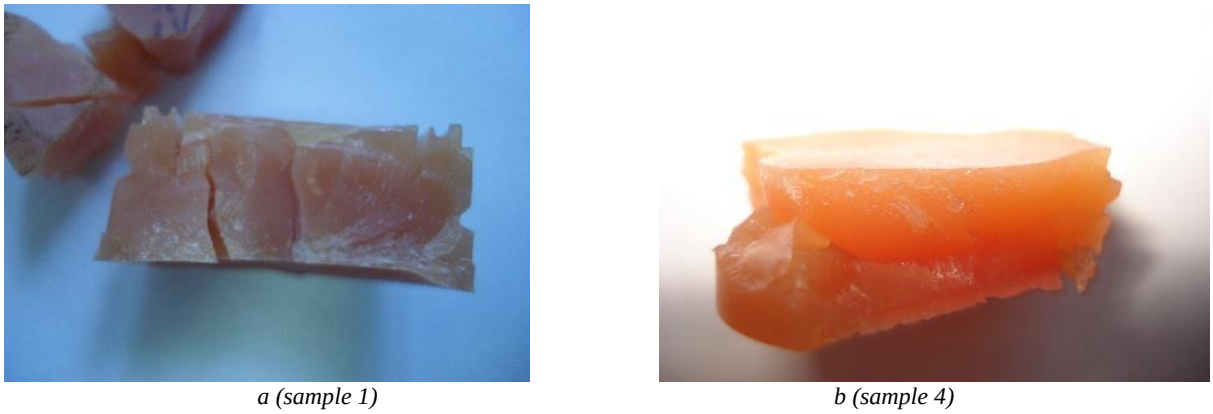
Table.2. The breaking point and the resistance of our studied materials

Sample	Material	Surface[mm²]	Breakout force[N]	Tension[MPa]
1	Poly(methyl methacrylate)-Duracryl	352.9789	54936	155.6353
2		356.3168	19620	55.063336
3		274.6378	16677	60.72362
4		274.6378	10791	39.29175
5	Composite resins-Aelite APB	363.0396	9319.5	25.67075
6		380.1215	10791	28.38829

Sample 1 of poly(methyl methacrylate) had the highest resistance (155.63 MPa),resistance that can be explained due to the fact that this sample had a doubled time (60 minutes) in immersion in water at 85°C.

To determine the influence of time of immersion in water on the material to be analyzed, sample 1 was immersed in water for 60 minutes at 85^{o}C. With these new parameters, an increased breaking point of this material up to 155,63MPa, was found. Visually we can observe that an incomplete polymerization leads to a reduced resistance. Sample 1 was destroyed under the action of the compression force, with no material displacements, in opposition to sample 4, which has material displacement due to the lack of cohesion.

In fig. 12 are presented the sample 1(a) with a high resistance and sample 4(b) with a low resistance.



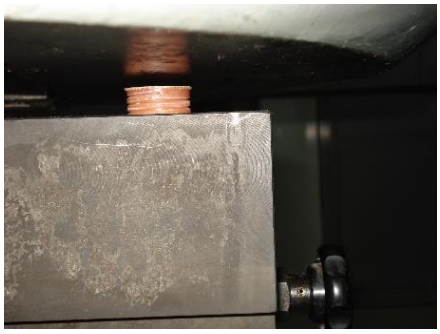
a (sample 1) *b (sample 4)*

Fig.12. Two samples tested with the Universal Testing Machine 100T.

In fig. 13 are presented two samples (a. polymethyl methacrylate, b.composite resins) that were tested with the Universal Testing Machine 100T and the measurement of the breaking point.

The average value of resistance for the samples from the polymethyl methacrylate-Duracryl group 2, 3, 4 (51.69) is closed to the one that is presented by the producer (65MPa).

For the composite resins, Aelite APB the producer does not specify the resistance of this material; there are some studies regarding the strength and resistance of this material, but made by other methods. Thus, C. Bortun et all[10] find higher values of resistance, but the testing was done by elongating the materials.



a)

b)

Fig.13. Determination of the breaking point: a.polymethyl methacrylate, b.composite resins

Regarding the material resistance, there is a low research literature, which is inhomogeneous due to the different method that was used to determine resistance. In the terms of polymethyl methacrylate-Duracryl resistance, Noraniah Kassim et all [11] realized similar studies that use the same experimental method and found an average value of resistance higher than our study; similar values were found by Thomas R. Meng et all[12].

The highest resistance value of polymethyl methacrylate-Duracryl was determined by Fernanda de Carvalho Panzeri Pires-de-Souzaa[13] that used the same method as our study, the only difference was the testing machine, Universal Test Device EMIC-MEM 2000 and the average value of resistance was found at 100,1MPa.

These differences recorded between our study and the ones found in the literature can be explained by the fact that during the testing all the researchers used a vacuum chamber. The absence of air during the blending of material leaded to a higher resistance to compression force and the final expression of this fact is the higher values obtained during the experiment.

We appreciate that the real value of resistance of polymethyl methacrylate-Duracryl is the one shown in our study because in the current practice of dental medicine the blending of the material is done in ambient atmosphere.

The resistance of Duracryl conditions the quality of dental prosthetics and, we consider that for the future practical solutions must be found in which a higher resistance of this material will be obtained. In this order of thoughts, our experiment comes somewhat to meet the solving of this problem because for the sample 1 polymethyl methacrylate-Duracryl we found a far more higher resistance in opposition with the 2,3,4 samples of the same material by extending the time of immersion in water.

The higher resistance of this sample can be explained due to a complete polymerization at a higher temperature than the one presented by the producer. For the improvement of polymethyl methacrylate-Duracryl processing steps, is necessary to determine the optimum temperature for a maximum resistance.

4. Conclusions

Amelogen sample has inhomogeneous morphologies, with large amorphous pieces, and nanoparticles at 85 nm. Electron diffraction reveals two peaks situated on 0.210 nm, respectively 0.121 nm. The approximate diameters of Premacryl are 60 nm and 18 nm respectively Electron diffraction pattern reveals an amorphous structure of the Premacryl solution, with two peaks around 0.210 nm, and 0.116 nm respectively. Duracryl samples exhibit amorphous structures with concentration that depends on temperature and time of thermal treatment. In addition, all sample have crystalline inclusion with linear dimensions around 50 nm.

Differences recorded, for average value of resistance, between our study and the ones found in the literature can be explained by the fact that during the testing all the researchers used a vacuum chamber.

Our experiments demonstrate that polymethyl methacrylate-Duracryl has a higher value of resistance than dental composite resins Aelite APB. Our study demonstrate the necessity to extend this research regarding how the temperature variations influences the polymerization and thus establishing the correlation between temperature and polymerization and the resistance to different forces generated by the jaw muscles.

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