Evaluation of Mechanical Properties by Stereo-and Scanning Electron Microscopy of Some Heat Curing Dental Resins

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Complete dentures technology uses especial heat curing acryllic resins, which are included in DIN EN ISO classification at number 1567, group 1. Polimetacrilates are fragile materials, which fracture easily and often need prosthetic pieces repairs. Evaluation of acryllic resins structural qualities allows choosing of the material with maximum mechanical resistance. Present paper intends to determine some comparative structural studies of three heat curing acryllic resins, concerning behaviour after mechanical testing . There were realized 8 samples from each heat curing resins (Meliodent, Superacryl, and Vertex), using the complete denture technology recommended by the producer. Their mechanical characteristics were determined with help of Zwick Roel equipment. Transversal and longitudinal sections of the fractured zones were evaluated by stereomicroscopy and scanning electron microscopy. Following the mechanical tests, there were obtained the following results: ultimate tensile strength 64 MPa / Meliodent, 66 MPa/ Superacryl, Vertex/ 70MPa; total elongation was 11% for Meliodent, 9% for Superacryl and 5% for Vertex; Young's elasticity modulus was 4570MPa for Meliodent, 5125 MPa for Supercryl and 6243 MPa for Vertex. By stereomicroscopy there was noticed fragmentation, number of fibres from the transversal cross section, or the aspect, shape, colour of fibre from the matrix. At the scanning electron microscopy, microscopic features may be put in evidence: the aspect of the broken matrix (which is brittle for all the samples), the manner of fracture, the nature of the interface, which is incoherent. Finally the correlation between mechanical behaviour and fracture mechanism was made for dental heat curing resins.

Keywords: heat curing acryllic resins, stereomicroscopy, scanning electron microscope

One of the most difficult prosthetic restorations is that with complete dentures, where the result is immediately "to be seen". A bad prosthesis cannot be kept on the position and is not stable from the first moment. Prosthetic restoration of edentulous patients must have a good function almost for partial mastication recovery and why not aesthetic shape and form of teeth, occlusal equilibration, homeostasis of the prosthetic field.

The introduction of heat curing methylpolymetacrylat resins in dentistry took place in 1937, and it is considered a revolution in total prosthesis technology. Today, the acrylic resins have acceptable properties, simple technology, but also disadvantages- such as dimension changes during elaboration, porosities, poor mechanical resistance. According to EN ISO 1567 there are four types of resins: heat curing resins (upper 65°C) either bicomponent, self curing (lower 65°C), thermoplastic materials in granulate form, light curing resins and microwave polymerized resins. Heat curing resins are now the most used materials for realization of partial or complete dentures. World market of bi-component heat curing resins knows a lot of product, such as Meliodent (Heraeus Kulzer), Vertex (Vertex), Superacryl (Spofa), RoyalDent (Palatinál Foggyártó KFT).

Frequently, there are non corresponding situations, when complete dentures may have cracks, producing partial or total breakdown. The problem of mechanical behaviour of acrylic resins materials is a real one, being well and repeatedly presented in literature [1-7]. The necessity of a minimum level of mechanical characteristic values guarantee may appear in order to assure the prosthesis viability on long term. The aim of present paper is the necessity of processes grounding which takes place for integration maintaining of dual components heat curing resins, and also to structural characterization of two types of resins, with differential behaviour.

Experimental part

There were analysed four acrylic resins which are ussualy used in dental practice, respectively: MELIODENT (Heraeus Kulzer, Senden, Germany), SUPERACRYL (Spofa-Dental, Markova, Czech Republic), and VERTEX (Vertex Dental, B.V., Yeist, Netherland). In accordance with dental practice, there were made by polymerization samples with the following dimensions: 2 mm thickness, 30 mm length and 5 mm width. In order to define the mechanism of fracture, all samples were loaded in the same manner. Mechanical characteristics were determined by Zwick Roel equipment with data processing using testXpert system (Zwick GmbH & Co. KG, Ulm,Germany). The loaded stress was 50kN and the rezolution 0.1μm for all experiemental samples. The experimental samples were then analysed at stereomicroscope Olympus type SZX7, equiped with image processing soft QuickphotoMicro 2.2. There were analysed both longitudinal and transversal surfaces perpendicular to fracture surface. For final structural investigation, Scaning electron microsopy was used, made on PHILIPS type microscope. SEM were taken on broken surfaces, at different magnification of the microscope, respectively x100 and x500.

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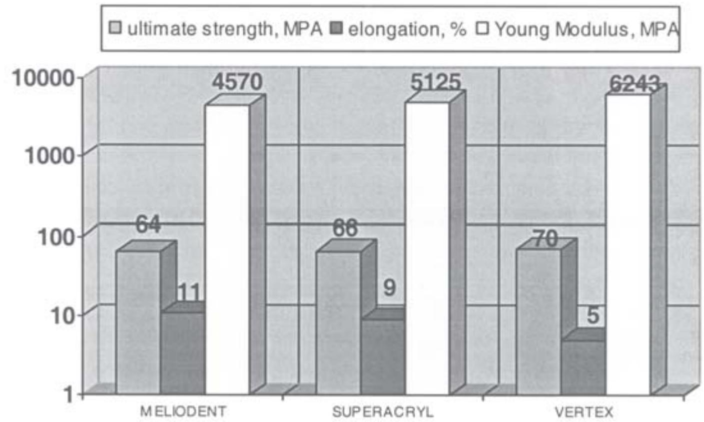


Fig. 1. Mechanical characteristics of the heat curing dental experimental resins

Results and discussions

The mechanical characteristics of the heat curing dental experimental resins are given in figure 1. So, ultimate strength is 64 MPa for MELIODENT, 66 MPa for SUPERACRYL, the highest value being for VERTEX, respectively 70MPa. Elongation was 11% for MELIODENT, 9% for SUPERACRYL and 5% for VERTEX. The Young Modulus of elasticity was 4570MPa for MELIODENT, 5125 MPa for SPERACRYL and 6243 MPa for VERTEX. As one may remark, the lowest value of the ultimate strength is for MELIODENT, than SUPERACRYL and VERTEX (with the highest values). Considering the manner of fracture we may conclude that resin VERTEX has the best mechanical behaviour among all the investigated heat curing dental resins.

Macroscopic analysis is made by stereomicroscopic analysis. The stereomicroscope that we used had two separate optical paths with two objectives and two eyepieces to provide different viewing angle to the left and right eyes. In this way it produces a three-dimensional visualization of the sample being examined. Detailed analysis under the stereomicroscope revealed interesting observations. As a general remark, the entire sample has a brittle fracture, with a quasi crystalline aspect in transversal cross section. Each resin has its own structural characteristics.

Resin type MELIODENT, given in figure 2, has in general a brittle aspect. One may remark in transversal cross section relative many chop fibers, about 3-4 red and short fibers. All the fibers cracked separately than the matrix, as a sign of a different fracture behaviour between fiber and the matrix. There is no fragmented fracture of the samples.

Resin type SUPERACRYL, given in figure 3, has a specific structural feature. The entire sample fractured in fragmented manner, resulting three parts after fracture (the so called 100% "fragmented fracture"). One may remark a small amount of short chop fibers on the transversal cross section, respectively one, or two.

Resins type VERTEX, given in figure 4, have also a fragmentary fracture, but in proportion of about 30%. In all situations, the fibers do not fracture in the same time with the matrix. These resins have the longest fibers among the investigated resins.

Complete fractography was done by scanning electron microscopy that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample surface topography. Scanning Electron Microscopy made on broken mechanical test samples are illustrated in figures 5, 6 and 7. As one may observe, all the examined heat curing dental resins have the same aspects of the broken surfaces:

- the matrix has a brittle behaviour, with a well-defined cleavage aspect,
- the fibers are not broken in any case, all of them being seen unbroken after fracture,
- a small discontinuity may be revealed at the fibers-matrix interface. This is a sign of different mechanical values between matrix and the fibers.

a. b. c. d.

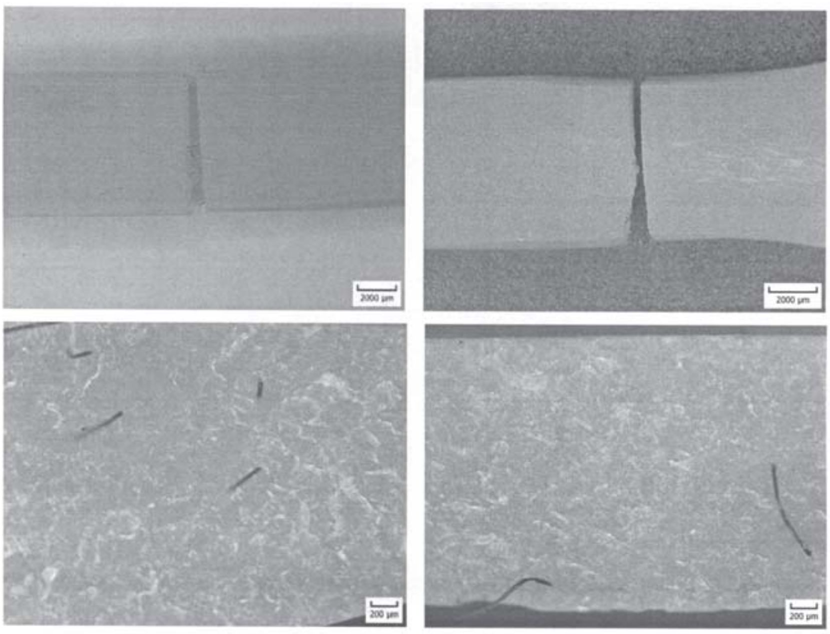


Fig. 2. Stereomicrostructural aspect of MELIODENT dental resin: longitudinal section (a,b) and transversal cross section (c,d)

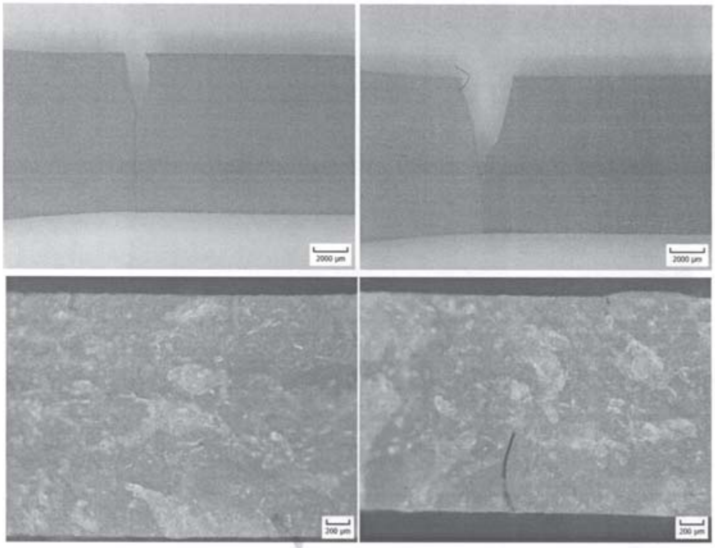


Fig. 3. Stereomicrostructural aspect of SUPERACRYL dental resin: longitudinal section (a,b) and transversal cross section (c, d)

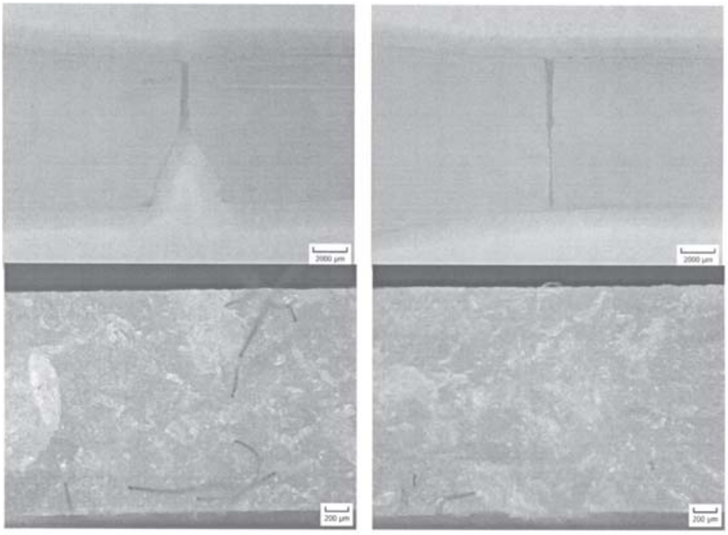


Fig. 4. Stereomicrostructural aspect of VERTEX dental resin: longitudinal section (a,b) and transversal cross section (c,d)

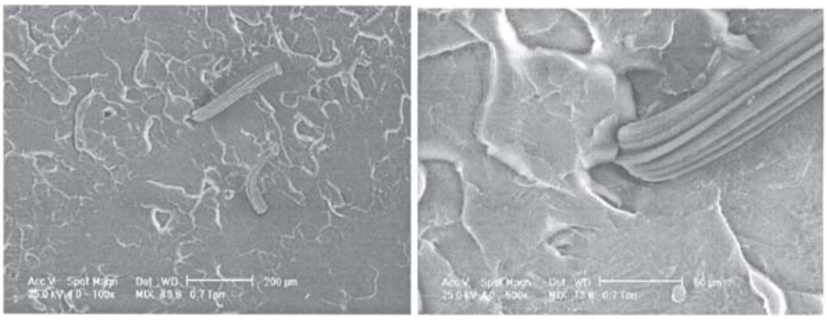


Fig. 5. SEM images for MELIODENT dental resin: a- x100, b- detail of a, x500

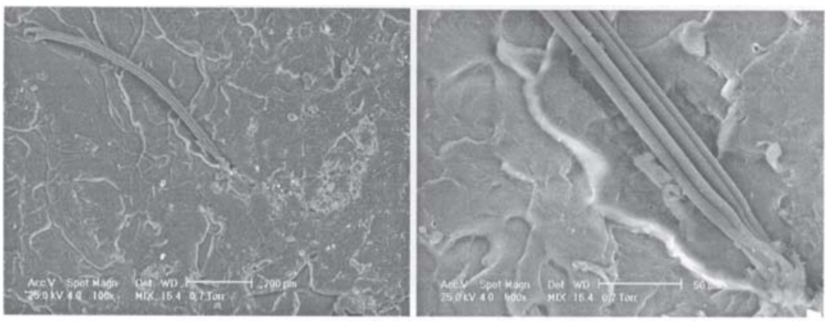
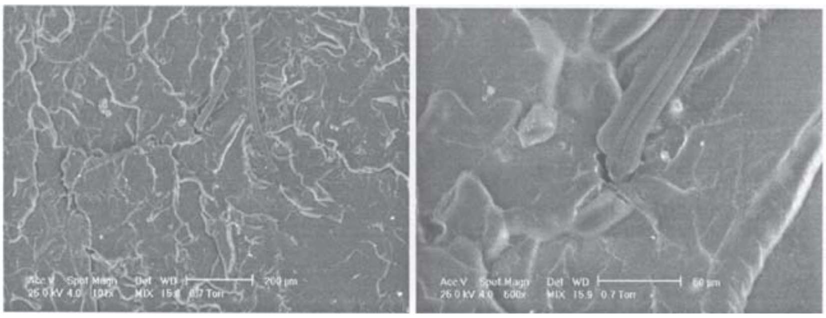


Fig. 6. SEM images for SUPERACRYL dental resin: a- x100, b- detail of a, x500

Results and discussions

In these investigations the fracture behavior of heat curing dental resins was studied through two different methods, based on microscopic analysis. The heat curing dental resins used in the experiments are typically the most common fiber-reinforced composite materials. These types of composite contain short fiber reinforced materials laid in a random fashion. Among the shape of the fiber generally used, in heat curing resins these come in the form of chip and random mate. From studies [6-8], it is clear that defects of compactity in the acrylic resins base like voids, porosities, scratches, notches are predominant factors in the fracture of the experimental samples and so of the future dentures. These defects may be considered as stress



a b

Fig. 7. SEM images for VERTEX dental resin: a- x100, b- detail of a, x500

concentrations sites which lead to crack nucleation, growth and propagation. Also, reinforcement with fibers enhances the mechanical properties of the material, such as ultimate strength, yielding strength, elongation, Young's modulus of elasticity. By using modern techniques, which reduce voids and porosities releasing residual stress, proper experimental samples were obtained for experiments. All our samples were made from common heat curing resins, such as MELIODENT, SUPERACRYL, VERTEX.

Structural investigations made with two different methods and techniques reveal some conclusions, which are in accordance one to another. So, whether at stereomicroscope some macroscopic features may be observed, such as fragmentation, number of fiber from the transversal cross section, or the aspect, shape, color of fiber from the matrix, at the scanning electron microscope microscopic features may be put in evidence: the aspect of the broken matrix (which is brittle for all the samples), the manner of fracture, the nature of the interface, which is incoherent.

Conclusions

Comparative analysis of fracture behaviour for four heat curing dental acrylic resins, respectively type MELIODENT, SUPERACRYL, and VERTEX, may reveal the following aspects.

All the heat curing dental acrylic resins present fragmented fracture, but in different proportion: 100% for MELIODENT and SUPERACRYL, and 30% for VERTEX.

Mechanical characteristic values of samples were in the range of 60- 70 MPa for ultimate strength, in the range of 5 – 10 % for elongation and in the range of 4500 -6300 for Young Modulus of elasticity. So, ultimate strength is 64 MPa for Meliodent, 66 MPa for SUPERACRYL, the highest value being for VERTEX, respectively 70MPa. Elongation was 11% for MELIODENT, 9% for SUPERACRYL and 5% for VERTEX. The Young Modulus of elasticity was 4570MPa for MELIODENT, 5125 MPa for SUPERCRYL and 6243 MPa for VERTEX.

Different behaviour of dental acrylic heat curing resins is due to significant differences between mechanical characteristics of reinforced fibers and matrix. In general, red small chop fibers may or may not fracture in the same time with the matrix. Considering our heat dental curing resins, MELIODENT, VERTEX and SUPERACRYL, all the fibers from all experimental samples do not fracture in the same moment with matrix. This is a sign of a brittle behavior of the resins

The best mechanical behaviour of the investigated heat curing dental acrylic resin is for VERTEX, with the highest amount of reinforced fibers among the other experimental resins.

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