OPTIMIZATION OF POLYMERIZING CONDITION OF THE COMPOSITE MATERIALS, IN ORDER TO IMPROVE THEIR PHYSICAL AND MECHANICAL PROPERTIES

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ABSTRACT: The materials used in restorative dentistry differ depending on the type of prosthetic work. They may be metallic and nonmetallic. Nonmetallic materials have a critical importance as they are the esthetic component of works. These include acrylates, composites and ceramics. A particular property of them is the hardness of the material because it directly affects the damage of antagonists tooth enamel or contact surface damage of the prosthetic work. When using porcelain or metals having hardness greater than of natural teeth, their enamel will be rubbed to contact with prosthetic works. If the hardness of the latter is far below that of natural teeth, the contact surface of the prosthetic works will rapidly deteriorate requiring their replacement at short intervals. Due to these considerations in this paper are presented a series of tests in order to increase the toughness of composite materials by changing the polymerization conditions.

KEY WORDS: dental materials, prosthesis, abrasion antagonists tooth, composite, hardness

1 INTRODUCTION

Composite materials used in dental field polymerize under laboratory condition at a temperature of 95-125 degrees Celsius and a pressure of 6 atmospheres. The main mechanical parameters for a restoration material used in prosthetic works are: modulus of elasticity, fracturing strength and hardness. The latter, hardness of the used material, is very important because it can influence the strength both the antagonist teeth, natural teeth and prosthetic work.

Regarding the hardness of the composite material, this property has a special importance as it directly affects the degradation of the antagonistic teeth enamel (encountering the dental work) or of the prosthetic contact surface of the dental works. By using porcelain or materials with hardness higher than that of the natural teeth, their enamel will be abraded when encountering the prosthetic works. If the hardness of the latter is much under that of the natural teeth, then the contact surface of the prosthetic works will be rapidly deteriorated, requiring a frequent replacing. It was found that the hardness of the ceramic masses used in dental practice is of 6.5 (Mohs scale), exceeding the hardness of the dental enamel. With this specific feature, hardness is considered one of the disadvantages of the ceramic materials which produce uneven abrades of the antagonistic teeth.

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Composite material used in dental works has a much lower hardness than the porcelain. It is desirable that the materials used in the manufacture of prosthetic work to be slightly below the enamel of natural teeth.

2 PROPERTIES OF DENTAL MATERIALS

In the literature they are given some hardness values of composite materials made of metal powders and hydroxyapatite. Using conversion tables, Shore hardness readings were converted to HRC and HV values in Table 1.

Table 1. The hardness of the composites, with 30% and 20% Hap.

r. Crt.	Composite	H SD	H RC	H V
1.	70%316L/30%HAp	71	53	560
	80%316L/20%HAp	77	73	645

The values were obtained by using the Shore method which measures the ratio of rejection of a device with diamond striking the material surface and registering the height from which it was dropped by means of directional guides. Determination of hardness in this case was done with a hardness tester ZWICK D 790 type 7206, according to ISO R 868 / 2003, on a scale from 0 to 100.

In order to optimize the properties of existing composite materials, polymerization pressure will rise from the current 6 atm. to a value of almost 10 times higher. To get such working conditions, a device would be achieved which allows to obtain values of 50 atmospheres and an adjustable temperature around 120 degrees. In order to achieve new polymerization conditions of composite materials used in dentistry, an analysis of physical and mechanical properties of materials commonly polymerized under normal conditions was performed.

Aiming mechanical properties of dental composite materials, we will refer to their hardness and wear resistance. The measure unit for the hardness of the composite resin surface is the Knoop index that varies by composite type. Macrofilled composites have Knoop index of 35-65 kg/mm2, while microfilled composites have Knoop index of 18-30 kg/mm2. Knoop hardness of the thermopolymerizable acrylic resin is of 20 kg/mm2, lower than dentin (that has the hardness around 65 kg/mm2) and enamel (300 kg/mm2). Referring to the wear resistance of the restoration material, it must be equal to the enamel of the restored area. Physical damage of restoration surface is determined by factors such as material abrasion and fatigue associated with intermittent stress of the material. As well, the wear of the antagonist teeth [1] enamel is important, depending on the used composite. The bigger the filling particles, the higher composite surface wear and the wear of the antagonistic teeth enamel. Composites with quartz particles give a greater wear of the antagonistic teeth enamel than barium, tin and other fillers [2].

The phenomenon of fatigue of composite materials, as a result of the mastication leads to the compromising of the restoration work by the appearance of cracks in the organic matrix. Knoop hardness test is performed by using pyramidal instrument acting on material by imprinting a footprint on this and measuring its size [3]. Knoop hardness number (KHN) represents the value of the applied force on the hardness footprint and it is calculated with the formula:

$$KHN = \frac{L}{l^2 Cp} \tag{1}$$

Where, L is applied load, l is the length of the long diagonal of the footprint, and Cp is Poisson constant.

The method to determine hardness can run with varying loads. As well, the footprint area changes with load but also with the hardness of the analyzed material. The advantage of the method is the fact that materials can be tested even if they are very hard, only by varying the load. As the smaller loads cause very small footprints, this method can be used with materials that present different values of hardness on different areas (Table 2).

Table 2. Knoop hardness (KHN) some dental materials

Material	KHN (kg/ mm^2)
Porcelain	460
Enamel	343
Dentin	68
Dental cement	40
Dental acrylate	21

One of the most important applications studied in dentistry is the action of the occlusal forces acting on both the teeth and dental restorations. Using strain gauges, holography, telemetric devices and finite element analysis were recorded forces with values of 250-3500 N. Bites of an adult decrease from molars to the incisor teeth. The forces on the first and second molar are from 400 to 800 N. Following a study conducted with a microscope, the composite samples shows that the material appearance changes depending on the degree of polymerization, the difference being given by the polymerization time.

For the PLUS VALUX material an incomplete polymerization was observed with white stains on the material surface, while for all samples of TE-SAVING material, the polymerization was uniformly made, with no stains. The surfaces of the two materials are very different, if we refer to the material homogeneity. Depending on the forces applied to composites the following conclusions were drawn:

- The obturation materials withstood to forces of:

  - 2300 N, equivalent to a strain of 117 MPa for the material Valux Plus;

  - 2500 N, equivalent to a strain of 127 MPa for TE-ECONOM material.
- *Duropont* materials withstood depending on centric or eccentric load to forces of:

  - 1600 N equivalent to a strain of 48.9 MPa for the centric compression;

  - 1000 N echivalentul to a strain of 82.77 MPa for the eccentric compression.
- Dental acrylate teeth withstood to a force of 1400 N, this is 87 MPa.

These results are highlighted by taking into account that human bite can reach a maximum value of 270 N [4].

In order to study the strength of the dental composite material, they were differently polymerized to observe different factors influencing the structure of composite material. Following analysis in which digital microscope VHX-600 was used, conclusions on the structure of polymerized material were drawn. In the pictures below you can see variations in material structure according to the polymerization times. In figure 1, there are 3D images seen on microscope of the composite TE-ECONOM after 7 minutes of polymerization.

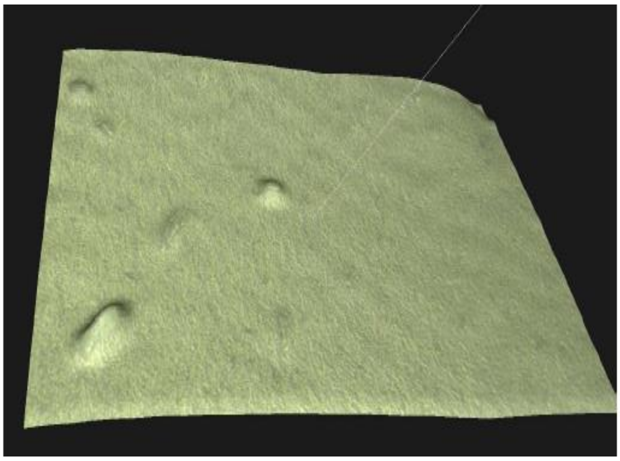

Fig. 1. 3D microscope image of the composite material TE-ECONOM after 7 minutes of polymerisation [4].

In figure 2, there are 3D images seen on microscope of the same composite TE-ECONOM after 9 minutes of polymerization.

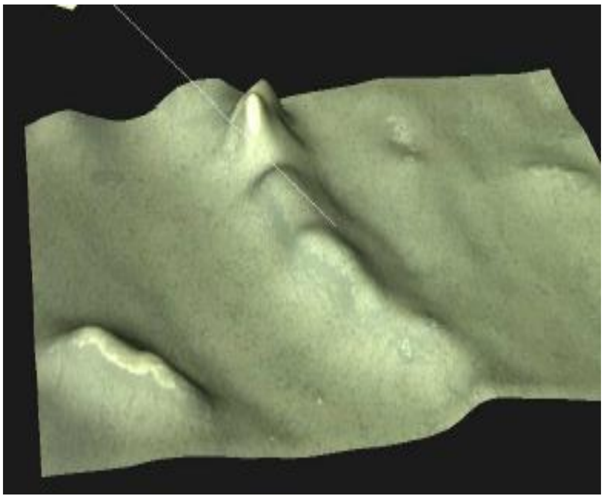

Fig. 2. 3D microscope image, of the TE-ECONOM composite material after 9 minutes of polymerization [4].

In figure 3 there are 3D images seen on microscope of the VALUX PLUS material after 6 minutes of polymerization [3].

Figure 4 presents the structure of the Concise-3M material seen at the microscope, which is a self-polymerizable composite material.

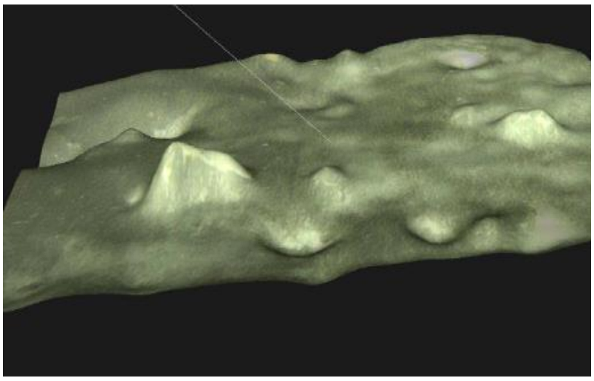

Fig. 3. 3D microscope image, of the VALUX PLUS composite material after 6 minutes of polymerization [4].

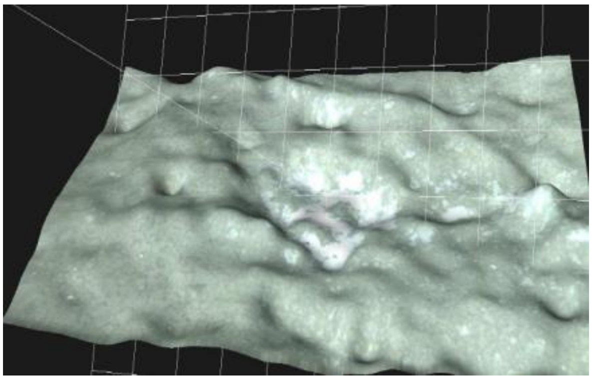

Fig. 4. 3D image of the composite material Concise-3M [4].

3 EXPERIMENTAL STUDIES

In order to obtain a composite material with a denser and with higher mechanical properties internal structure, the operating conditions will be changed and the polymerization of the same material will be experimentally achieved at a temperature ranging between 120 ° C and 180 ° C and a pressure between 6 atm and 50 atm.

The conditions used in the current material polymerization appliances are: temperature of 95-120 °C and pressure of 6 atm. The container will have to withstand a pressure of over 60 atm. It will be made of stainless steel with wall of 10 mm thickness in order to avoid work chamber rusting and thus compromising the prosthetic work. It will be supplied with compressed air from a high capacity compressor, used to fill oxygen tanks or from a hand pump used to fill small size tanks. A tap attached to the container will enable supplying and discharging the working fluid. It will fill the polymerization chamber within the cylinder after the introduction of composite work and will make easier the pressure rise to the desired value, as liquids are theoretically uncompressible.

Under certain conditions water is used, but at a temperature of 100°C and normal pressure this will start to boil and turn into vapours. Glycerine has a boiling point of 290 °C but at 180 °C it is flammable. The disadvantage is that at high temperatures it turns into acrolein, which is a toxic and irritating substance. In the experiment will be used brake fluid, a glycol-based fluid, with boiling point at 245°C (if unaltered state), or DOT5, a silicone based fluid with the same properties.

The pressure used in experimental research will have successive values of 10, 20, 30, 40 and 50 atmospheres. Then the results are analyzed for the above mentioned values, regarding mechanical strength, homogeneity and other physicochemical properties of the used composite material. It particularly aims to achieve results that demonstrate increase of the polymerized material hardness and mechanical strength without negative influences on colour and elasticity.

After establishing the optimum polymerisation pressure, the experiments will be repeated, the results obtained by changing the values of the temperatures between 100 and 200°C will be analyzed. As components in the air circuit a check valve (Ss), a pressure regulator (R), a gauge of 100 atm (M), container (P), air valve (A) and thermoregulator (T) will be used. The assembly will be achieved by screwing using silicone sealing / locking and seals resistant to temperature and pressure.

Figure 5 shows the device for the polymerization of acrylates and composites made in the original form.

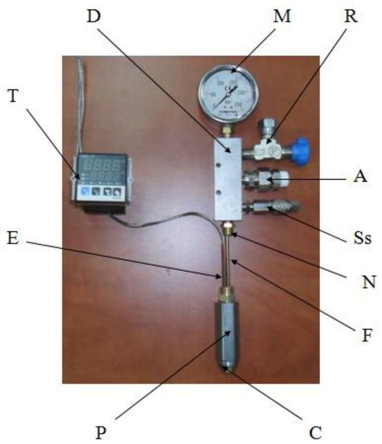

Fig. 5. Initial device.

Figure 6 shows the device comprising some changes made in order to optimize its functionality and eliminate the deficiencies resulted when testing the initial device. First of all, the replacement of the distributor D was required (Fig.5) as some cracks have appeared on it.

Fig. 6. Changed device.

In order to provide an additional control when discharging the pressurized fluid with a temperature around 120° C, a flow regulator was mounted. For the polymerization, the sample made of composite material is firstly introduced in the polymerization chamber P and the lid containing thermal probe is closed. Through tap R the liquid is introduced until the complete filling of the inner space of the device. The tap R is closed. The pressure pump is connected by means of a quick coupling as in Figure 6 and they pump to the pressure of 10, 20, 30, 40 and 50 atm displayed on gauge M. The air valve, A, located in the lower part of the pump is opened and the pressure pump is unconnected. The polymerization chamber body P is inserted inside a metal container with 150°C-resistant liquid, and the vase is placed on an induction plate.

Setting the working temperature and controlling the operation of the plate are achieved by the means of thermal probe in the polymerization chamber, which transmits the information to the thermoregulator in order to maintain the temperature around 120°C. A timer will record the polymerization time from the moment when the set temperature is reached.

Most restorative materials must withstand forces during manufacturing or mastication. The mechanical properties are important to understand and predict the behaviour of the material under loading. Because a single mechanical property cannot give a true measure of quality, understanding of the principles involved in a variety of mechanical properties is essential in order to achieve maximum quality.

Forces, stresses, strains, strength, hardness, friction and wear properties may help identify material properties. One of the most important applications in dentistry is the study of occlusal forces applied to teeth and dental restorations. Equally important for the study of the forces applied to natural teeth is the measuring of forces and tensions in restorations, such as insertions, fixed links to partial prostheses and whole prostheses fixing.

For comparison, the average forces of biting at the permanent teeth are of 665 N for molars and 220 N for incisor teeth. In general, biting force for women is 90 N lower than that of men. Studies like this indicate that the force of mastication on the first molar for the patients with fixed prostheses is about 40% of the force exerted by the patients with natural dentition.

The distribution of forces between the first premolars, second premolars and first premolar at a complete dentition was established as approximately 15%, 30% and 55% of normal force. When a force acts on a body tends to produce a deformation and generates a resistance to this external force. Internal reaction is equal as intensity and opposite as direction to the exterior applied force. This reaction is called tension and is analyzed as the ratio between the forces applied per area unit.

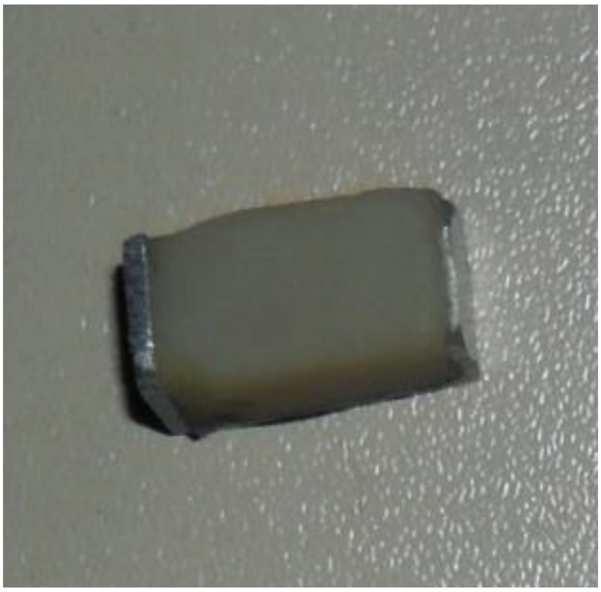

Fig. 7. The sample polymerized.

Technically, the tension is the internal resistance of the body in terms of force per area units. Since the internal resistance of the applied force is difficult to measure, the most convenient procedure is to measure the external force applied to the cross section, which can be described as tension, usually denoted S or σ. The unit of measure for tension is MPa, where 1 MPa = 106 Pa, and 1Pa = 1 N/m2 = 1 MN/mm2. After 20 minutes of polymerization of the composite material, cycle of work will be interrupted. The sample polymerized at 50 atm is shown in figure 7. The used polymerization material is the composite type DUROPONT, the most requested by dentists, with stability over time, aesthetic quality and superior physical properties (Fig. 8).

Fig. 8. Barothermopolymerizable composite material (dentin).

Hardness tests are carried out on composite materials barothermopolymerizable [5], [6] at pressures of up to 50 atm. Samples were tested in order to establish hardness on a hardness microtester FM 700 (Fig. 9). The marks imprinted on material give information required to establish the material hardness.

Fig. 9. Testing the composite DUROPONT to establish hardness on a hardness microtester FM 700.

4 CONCLUSION

Experiments performed and presented in this paper show the fact when polymerization composite material at a pressure of 10 atm, hardness rise by 4%, while polymerization at a pressure of 50 atm, hardness rise by 18%, comparing to the original polymerizing at a pressure of 6 atm of the composite material with a hardness of 65 kg/mm2. It is taken into account the testing of the behaviour of the composite material regarding the hardness increasing by polymerization under pressure around 100 atmospheres.

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