OPTOELECTRONICS AND ADVANCED MATERIALS – RAPID COMMUNICATIONS Vol. 1, No. 5, May 2007, p. 231 - 234

The characterization of some new dental composites

A. COLCERIU[*], T. BURUIANA[a], M. MOLDOVAN, C. PREJMEREAN, C. TAMAS, O. FODOR[b], L. OPREAN[c], V. POPESCU[d]

"Raluca Ripan" Chemistry Research Institute, Cluj-Napoca, Romania

[a]*"Petru Poni" Institute of Macromolecular Chemistry, Iasi, Romania*

[b]*Faculty of Medicine and Pharmacy, Oradea University, Romania*

[c] *Iuliu Hatieganu University of Medicine and Pharmacy, Cluj-Napoca, Romania*

[d] *The Technical University Cluj-Napoca, Romania*

Behind the recent studies, the researchers' attention in the field was focused upon the fillers-polymeric matrix interface instabilities, which may lead to a rapid erosion of the composites. Till now, doesn't exist clinical studies, which confirm these assumptions**.** We may specify that the filler-matrix bond is the most important for a composite with adequate properties and from this point of view is easy to justify why a filler-matrix bond unbinding could be the first failure mechanism which may be take into consideration for the composites wear and fracture. This fracture may be realized at the filler-matrix interface, filler surface, filler-silane interface or at the silane-resin interface. The purpose of this study is to evaluate the diametric tensile strength of five experimental dental composites and to acquire a SEM analysis of the intact and fractured dental composites' surface. The diametrical tensile strength was measured with the aid of a universal INSTRON type-testing machine from the LOYD Company, at 23 ^{0}C. The analysis of the intact and fractured dental composites surface was evaluated using a scanning electronic microscope, Philips XL 30 ESEM. We studied the relationships between the diametric tensile strength and the composites morphology and composition. A significant contribution in order to obtain better values for the diametric tensile strengths has SiO_2 contained in the fillers composition.

(Received April 4, 2007; accepted April 5, 2007)

Keywords: SEM, Diametric tensile strength, Polymeric composites

1. Introduction

By definition a composite is a material that consists of two or more components. Typically a dental resin composite contains an organic binder and inorganic filler incorporated into a system that would induce polymerization. Usually the filler particles are coated with a coupling agent to bond to the resin matrix [1]. Change of size and filler loading has improved the wear and fracture resistance of the early composite resins. Modern composite systems contain filler such as quartz, colloidal silica and silica glass containing barium, strontium and others. This filler increases strength and modulus of elasticity and reduces the polymerization shrinkage, the coefficient of thermal expansion, and the water sorption [2]. Also, type, size, quantity and refraction index of the fillers into composite exert an influence upon light transmission across the material and, consequently, the light attenuation and the depth of cure may be altered [3, 4].

With respect to the organic matrix, the monomers used in dental composites, have not changed significantly throughout the last 20 years [5], and still belong to the class of the so-called high molecular weight dimethacrylates. Two of them are well known under the names bisphenol glycidyl methacrylate (Bis-GMA) and urethane dimethacrylate (UDMA) [6]. The nature of the involved monomer molecules and the degree of conversion obtained in composite resin has an important effect upon mechanical properties [7], where the higher degrees of cure will improve the final properties of the material.

Behind the recent studies, the researchers' attention in the field was focused upon the fillers-polymeric matrix interface instability, which may lead to a rapid erosion of the composites. Till now, do not exist clinical studies, which confirm these assumptions**.** A Japanese research group was concerned to the increase the fillers-polymeric matrix interface stability by binding the covalent methacrylic groups to the fillers surface, increasing in this way the filler-matrix bond and also the composites hydrofobicity. The particle fillers size, and the volumetric fraction in which these fillers could be inserted into the polymeric matrix and influence the properties of the composite materials.

But, the use of fillers with submicron dimensions is nowadays becoming one of the most frequently used methods for improving the properties of dental composite materials.

We may specify that the filler-matrix bond is the most important for a composite with adequate properties and from this point of view is easy to justify why a filler-matrix bond unbinding could be the first failure mechanism which may be taken into consideration for the composites wear and fracture.

This fracture may be realized at the filler-matrix interface, filler surface, filler-silane interface or at the silane-resin interface.

The purpose of this study is to evaluate the diametric tensile strength of five experimental dental composites and to acquire a SEM analysis of the intact and fractured dental composites' surface.

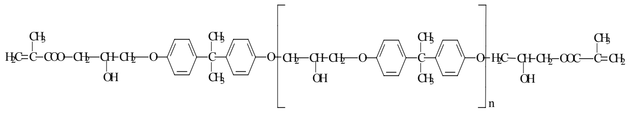
2. Experimental procedure

The experimental composites were prepared as a paste (Table 1), by dispersing the inorganic fillers in the monomers mixture.

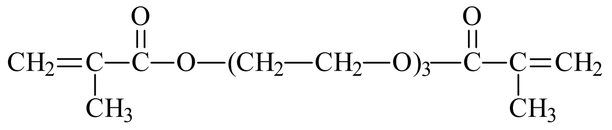
Table 1. The chemical composition of the experimental dental composites [wt%].

Cod	Organic phase	Inorganic Phase			
		G_1 Filler	**G_2** Filler	**G_3** Filler	SiO_2 colloidal
C1	18	16,4	-	49,2	16,4
C2	19	-	16,2	48,6	16,2
C3	18	20	-	42	20
C4	20	-	16	48	16
C5	20	-	-	60	20

The monomers mixture (Fig. 1) consists of polyethylene oxide di(urethane ethylmethacrylate) (synthesized in our laboratory) and dimethacrylate monomers: Bis-GMA (synthetised in our laboratory), TEGDMA (Aldrich). In each mixture dimethylaminoethylene methacrylate (Aldrich) was introduced as accelerator of polymerization and camphorquinone (Merck) as photosensitiser.

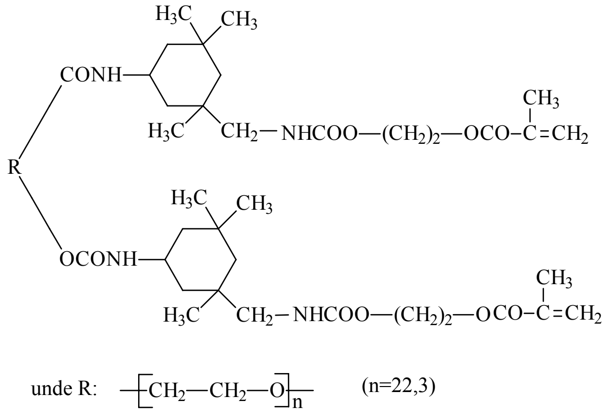


Bis-GMA monomer



$$CH_2{=}C(CH_3)-C(=O)-O-(CH_2-CH_2-O)_3-C(=O)-C(CH_3){=}CH_2$$

TEGDMA monomer



Polyethylene oxide di(urethane ethylmethacrylate) monomer

Fig. 1. Structure of the monomers used as organic phase in dental composites.

The inorganic fillers used consist of one (G_3) vitreous mass obtained through the conventional melting method and two fillers (G_1, G_2) obtained through the sol-gel method [8]. Table 2 presets the conditions for the synthesized and chemical composition for the elaborated fillers.

Table 2. The chemical composition used in inorganic fillers and the condition for the synthesis [%wt].

Oxides /Glass	**G_1**	**G_2**	**G_3**
SiO_2	72.8	-	40
Al_2O_3	-	-	10
ZrO_2	27.2	50	15
La_2O_3	-	50	-
SrO	-	-	27
AlF_3; CaF_2; NaF	-	-	8
Melting temp. [^{0}C]	1000	900	1380
Surface area (BET) [m^2/g]	75.20	83	10.0

The samples, for diametric tensile strength, were obtained in Teflon moulds (6 mm diameter and 3 mm thick), where the composites were polymerized with the aid of a 3M XL 2500 lamp for 40 s, from several directions. After the polymerization process, the samples were kept into distilled water at a temperature of 37±1 ^{o}C for 24 h. The diametric tensile strength was measured with the aid of a universal INSTRON type-testing machine from the LOYD Company, at 23 ^{o}C. The analysis of the intact and fractured dental composites surface was evaluated using a scanning electrone microscope, Philips XL 30 ESEM.

3. Results and discussion

Traction resistance (ADA *Sp.27*) was measured using the diametric tensile strength (DTS) test. The values (in MPa) were calculated using the formula:

$$R_T = 2xF/\pi xDxT$$

Were: **F** represents the force registered at the sample breaking moment (in N),

D represents the diameter of the sample (in mm),

T represents the sample thickness (in mm).

The obtained values for the elaborated dental composites are between 50-42 MPa, the highest values being registered for C2 composite (Fig. 2).

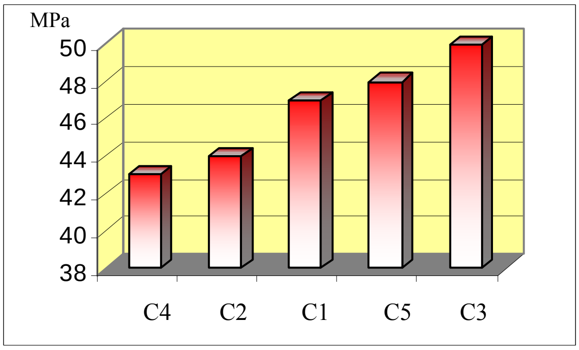


Fig. 2. Diametric tensile strength values for the elaborated dental composites.

One can be noticed that the hybride composites (C1, C5, C3) based on mixture that contains Zr (G3), nanofiller (G1) and colloidal silica presents values for the diametric tensile strenght of 47-50 MPa, values close to those corresponding by the International Standards (ADA) in the case of these restorative dental materials.

The composites C2 and C4 have smaller diametric traction strength of about 42MPa.

The sample C3 that contains fillers obtained by conventional melting method (G3), by sol-gel method (G1) and colloidal silica have the higher values of diametric tensile strength. That can be explained by the high content of SiO_2.

The use of fillers based on ZrO_2 and La_2O_3 leads to of a composite with the smallest diametric tensile strength values, which confirm once again the important role of the SiO_2 upon the mechanical properties of the dental composites.

Scanning electron microscope (SEM) analysis appears to be an efficient and acceptable method of examining features such as surface topography, filler size and distribution, interface adhesion and porosity.

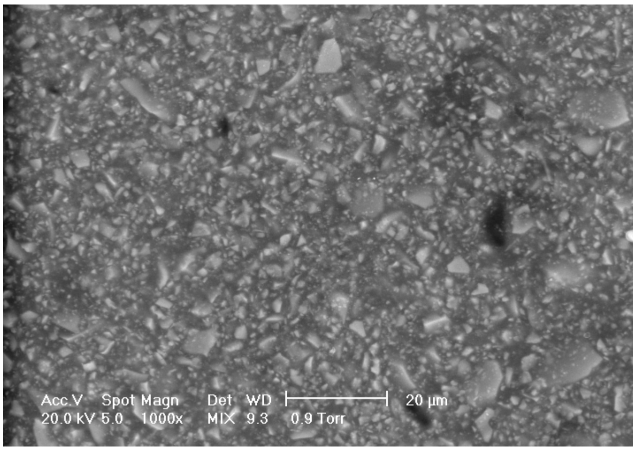


Fig. 3. SEM analysis of the C2 intact dental composites surface.

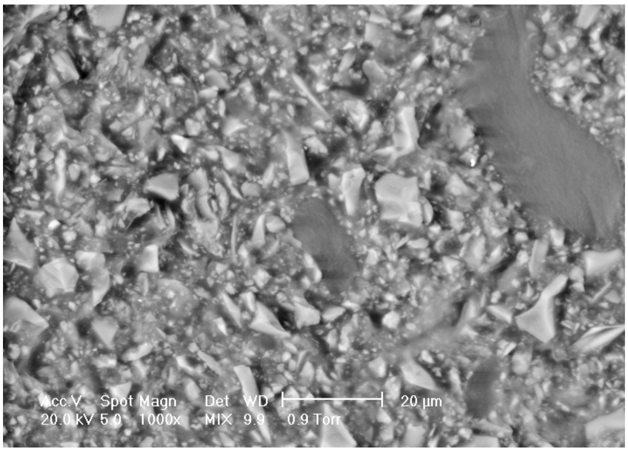


Fig. 4. SEM analysis of the C2 fractured dental composites surface.

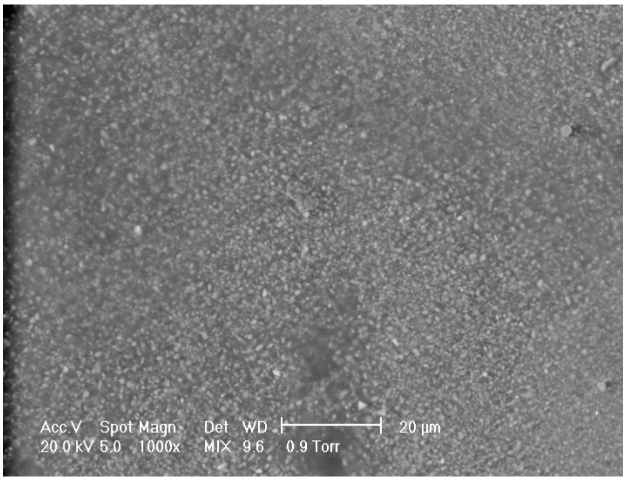


Fig. 5. SEM analysis of the C3 intact dental composites surface.

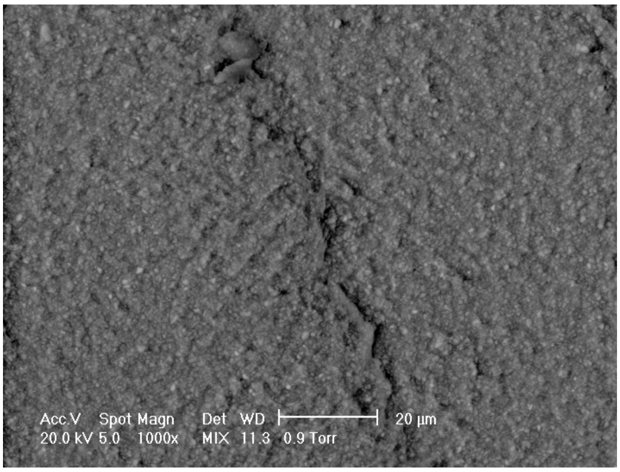


Fig. 6. SEM analysis of the C3 fractured dental composites surface.

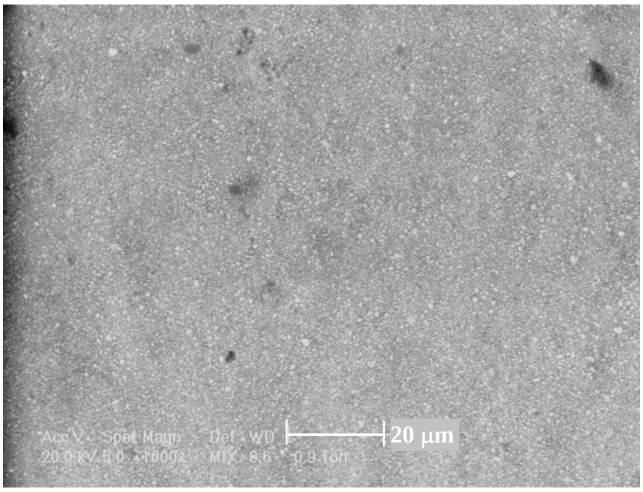


Fig. 7. SEM analysis of the C5 intact dental composites surface.

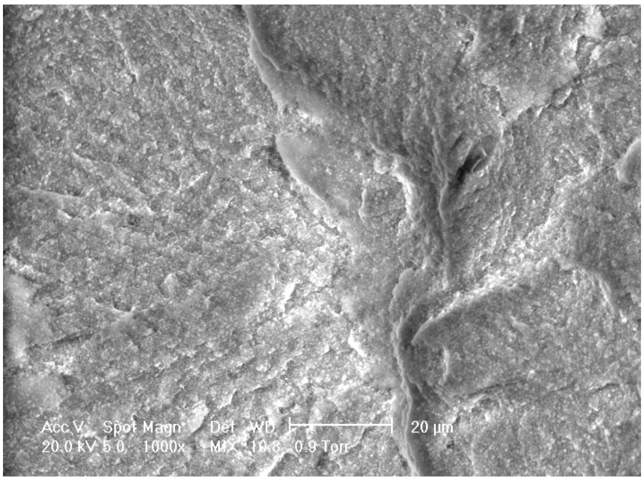


Fig. 8. SEM analysis of the C5 fractured dental composites surface.

Almost all the obtained images present a homogeneous structure, with uniform filler distribution in the organic matrix. The exception is the C2 composite (Fig. 4), at whose surface larger particles of filler can be seen, with a non-even distribution on the surface, from the dimensional point of view, with irregular shapes and sharp edges.

Also, good resin cohesion for the fractured samples can be observed, which means a good adhesion at the organic matrix/inorganic filler interface. A fractured surface with an even distribution of the inorganic filler can be observed (Fig. 6, 8), with uniform size of filler particles.

4. Conclusion

In this work, we studied dental composite materials obtained using urethane aliphatic dimethacrylate monomers (polyethylene oxide di(urethane ethylmethacrylate)), Bis-GMA and TEGDMA as organic phase, with zirconium oxide containing glass, filler based on ZrO_2-La_2O_3, filler based on SiO_2-ZrO_2 and colloidal silica as inorganic fillers. We studied the relationships between the diametric tensile strength and the composite morphology and composition. A significant contribution in order to obtain better values for the diametric tensile strengths has SiO_2 contained in the fillers composition.

The micrographs of the specimens illustrated the influence of the composition of inorganic filler on the morphology of obtained composites. The C3 composites reported a homogeny structure of composites surface and an even distribution of the inorganic filler on the fractured surface. That may be explained by a good incorporation of inorganic fillers in polyethylene oxide di(urethane ethylmethacrylate)/ Bis-GMA/TEGDMA monomers.

Acknowledgement

This work was partially supported by ATNANTECH Program under Project 188(401).

References

[1] D. Fortin, M. A. Vargas, JADA, **131**, 26 (2000).
[2] I. L. Dogon, Int J Technol Assess Health Care, **6**, 77 (1990).
[3] F. A. Rueggeberg, W. F. Caughman, J. W. Curtis Jr, H. C. Davis, Am. J. Dent. **6**, 91 (1993).
[4] S. L. Correr, A. A. de Lima, S. Consani, M. A. C. Sinhoreti, J. C. Knowles, Braz. Dent. J., **11**, 11 (2000).
[5] A. C. Obici, M. A. Coelho Sinhoreti, L. Correr Sobrinho, M. F. de Goes, S. Consani. Braz. Dent. J. **15**, 199 (2004).
[6] R. Hickel, W. Dasch, R. Janda, M. Tyas, K. Anusavice, International Dental Journal **48**, 16 (1998).
[7] L. G. Lovell, J. E.Elliott, J. W.Stansbury, C. N. Bowman, Dent. Mater. **17**, 504 (2001).
[8] K. K. Rao, M. Vithal, GY. S. K. Swamy, K. R. Kumar Materials Letter **54**, 208 (2002).

---

[*] Corresponding author: aura15ro@yahoo.com