Evaluation of mechanical properties of some composite biomaterials

A. SOANCA[*], A. ROMAN, M. MOLDOVAN[a], C. I. BONDOR, M. ROMINU[b]
"Iuliu Hatieganu" University of Medicine and Pharmacy, 8th Victor Babes, 400023, Cluj-Napoca, Romania
[a]*Institute for Research in Chemistry "Raluca Ripan", Babes-Bolyai University, 30th Fantanele, 400294 Cluj-Napoca, Romania*
[b]*"Victor Babes" University of Medicine and Pharmacy, 2nd Eftimie Murgu Place, 300041 Timisoara, Romania*

The composite materials used for dental restorations must have adequate mechanical properties in order to resist to the high masticatory forces to which they are subjected in the oral environment. The knowledge of these properties is important for understanding the clinical behavior of composite materials and for the success of the restorative treatment. The present study investigates the mechanical properties like flexural, compressive and diametral tensile strengths of a Romanian composite resin in comparison with two registered trademark dental biomaterials. The Romanian composite has a mechanical behavior similar with the control materials, being suitable for the oral environment conditions.

(Received November 24, 2011; accepted February 20, 2012)

Keywords: Biomaterials**,** Composite resins, Mechanical properties, Filler

1. Introduction

Composite resins became the most frequently used biomaterials for dental restorations due to the continuously growing aesthetic demands of patients. These materials are the main topic in many research studies as they continue to develop constantly, with the improvement of their properties in order to fulfill the clinical requirements for restorations [1-4].

In oral environment, composite restorations are subjected to high masticatory forces, which can affect the general properties of materials. These forces act both on the teeth and restorations producing changes in material dimension and quality, inducing deformation, and finally, restoration failure [5]. For this reason, the knowledge of mechanical properties is important for selecting the adequate restoring composite material [6,7].

Composite resins must have superior mechanical properties so that the dental restoration could resist to fracture and degradation in time. Fracture, flexural or compressive strengths are properties which influence the success of composite restorations [4,8]. When a force is applied on the material, a reaction of the same intensity occurs in opposite direction. Depending on the characteristics of the applied force, different tensions appear leading to structural and dimensional changes. The ratio between the force and the consequent deformation determines the mechanical behaviour of the material. However any material presents a certain degree of elasticity, a resistance to deformation; when these limits are exceeded, a permanent deformation occurs, followed by fracture [6].

In real clinical situations, there are many forces i.e. compression, traction, flexure, shear that tend to deform the restorative materials.

Dental composite biomaterials are complex mixtures of an organic matrix and inorganic filler. The mechanical properties are influenced by their composition and structural characteristics, especially the quantity and size of the inorganic particles [9].

The success of composite resin restorations depends on the mechanical properties of composites and on their behavior under the stress of the masticatory forces from the oral environment. The purpose of this study is to evaluate the following mechanical properties: flexural, compressive and diametral tensile strengths of a Romanian (experimental) composite resin [Barodent] in comparison with the properties of other materials, already successfully used on the dental market. This study continues our previous research studies on composite biomaterials that focused on the microstructural characterization as well as on the evaluation of the marginal adaptation and adhesion of these materials to dental tissues [10-13]. The goal is to provide a complete characterization of these materials, in order to start a clinical study.

2. Experimental

The materials under investigation are:

- a Romanian composite, Barodent, manufactured at the Institute for Research in Chemistry "Raluca Ripan" (ICCRR), Cluj-Napoca, Romania
- and two control composites BelleGlass® (Kerr Corporation,Orange, USA) and Gradia® (GC, Tokyo, Japan), used for comparison.

The composite materials were used following the manufacturer's instructions. All mechanical tests were performed according to the international standards in the field, preparing 10 specimens for each investigated material (n=10).

For the *flexural strength* testing, rectangular bar shaped specimens were prepared according to ISO standard 4049:2000 [14] with the following characteristics: 25± 2.0 mm length, 2 ± 0.1 mm width, and 2 ± 0.1 mm height.

For the *compressive strength* testing, cylindrical shaped specimens were prepared with the following characteristics: 4 mm diameter and 8 mm height, according to ADA standard, specification no. 27/1993 [15].

For the *diametral tensile strength* testing, cylindrical shaped specimens were prepared with the following dimensions: 6 mm diameter and 3 mm height, according to ADA standard, specification 27/1977 [16].

The composite materials were placed inside the special designed moulds, and then covered with transparent foil and light-cured using a halogen unit Optilux 501®/ Kerr Corporation. The specimens were pull out from the mould and light-cured from the opposite direction. All samples were post-cured, then finished and polished with abrasive paper disks (Optidics®/Kerr Corp).

Specimens were kept in distilled water, at 37°C for 24 hours. After drying, all specimens were subjected to mechanical testing for the determination of flexural strength (FS), compressive strength (CS) and diametral tensile strength (DTS). The tests were performed in a universal testing machine (Lloyd LR5K Plus/ Lloyd Instruments Ltd, Fareham, England; piston speed =0.75 mm/min.), according to the standard procedure. All data were visualized and analyzed with Nexygen PC software. The maximum force for the fracturing point was registered and FS, CS, DTS values (expressed in MPa) were calculated using well-known formulas [14-16].

All mechanical data were *statistically analyzed*. The normal distribution of data was performed with Shapiro-Wilk test. The results for flexural strength test were statistically analyzed using ANOVA and PostHoc Bonferonni tests, while the results for compressive strength and diametral tensile strength didn't have a normal distribution and they were processed with Mann-Whitney test [17]. The significance level was 0.05. For the statistical analysis, SPSS13.0 and Statistica 7.0 software were used.

3. Results and discussion

The goal of the study was to determine the mechanical properties i.e. flexural, compressive and diametral tensile strengths of some materials designed for dental restorations. The materials under investigation contain Bis-GMA (bisphenol A glycidyl methacrylate) or UDMA (urethane dimethacrylate) resins as organic matrix and various types of hybrid materials as inorganic fillers (Table 1).

Table 1. The composite materials used in the study and their main characteristics.

Composite material	/Organic matrix	Inorganic filler	Polymerization protocol
Barodent/ ICCRR	*Bisphenol A-glycydil-methacrylate (Bis-GMA) * Triethyleneglycol dimethacrylate (TEGDMA) *Urethane dimethacrylate (UDMA)	Hybrid filler (65 wt.%) ∗ Barium based glass (50%); ∗ Colloidal silica (20%) ; ∗ Quartz (30%).	∗ Light-curing (20 sec.) ∗ Post-curing (T=135°C; p=60 psi; N2 atmosphere)
BelleGlass/ Kerr	*Bisphenol A-glycydil-methacrylate (Bis-GMA)	Hybrid filler (87 wt%) ∗ Large prepolymerized particles; ∗ Structural particles 0.4μ; ∗ Silica nanoparticles	∗ Light-curing (20 sec.) ∗ Post-curing (T=135°C; p=60 psi; N2 atmosphere)
Gradia/GC	*Urethane dimethacrylate(UDMA)	Hybrid filler (75 wt%) ∗ Silica micro-filler mixed with UDMA resin, heat-polymerized and grounded in small particles (10-50 μm); ∗ Silanised ceramic fine particles (<2 μm)	∗ Light-curing (20 sec.) ∗ Post-curing (in the special light-curing unit; 3 min.)

The materials Barodent, BelleGlass and Gradia were used for specific specimen manufacture; the respective specimens were submitted to the mechanical testing and the results were statistically processed.

Flexural strength

The flexural strength (FS) indicates the material resistance to bend before fracturing [5]. The FS-testing implies subjecting rectangular bar shaped specimens to a "three-points" bending force producing tensile stresses on the inferior surface and compressive stresses on the superior surface (where the force is applied), followed by the specimen fracture [4,14].

The ANOVA test showed significant differences between the three investigated materials; the level of significance *p* was equal to 0.0003 (i.e. < 0.05). The PostHoc Bonferonni test was used for data correction. Mean value (mean) and standard deviation (SD), as well as the level of significance (*p*) were calculated and the results are shown in Table 2.

Table 2. Mean values and standard deviations for FS (n=10).

Composite materials		***Mean±SD***		**p**
Batch # 1	*Batch* # 2	*Batch* # 1	*Batch* # 2	
Barodent	BelleGlass	63.16±7.58	84.58±13.64	0.0002
Barodent	Gradia	63.16±7.58	61.68±7.09	1.0000
BelleGlass	Gradia	84.58±13.64	61.68±7.09	0.0001

The mean value for flexural strength for BelleGlass composite (84.58 MPa) was significantly higher in comparison with Barodent (63.16 MPa) and Gradia (61.68 MPa), the differences between them being statistically significant ($p<0.05$). After data correction, statistical significant differences were obtained between BelleGlass and Barodent (p= 0.0002), and respectively, between BelleGlass and Gradia (p= 0.0001). No significant differences were noted between Barodent and Gradia (p=1.0000)

The flexural strength illustrates the material's resistance to compressive and tensile forces that act simultaneously on it [18]. The FS-data could be explained by the differences existing between the materials' composition as well as by their protocol of polymerization.

All composites have a post-curing step that assures a high rate of monomer conversion and a superior bonding between the organic matrix and inorganic filler. Barodent has the same post-curing protocol with BelleGlass, performed under pressure, in nitrogen atmosphere, while Gradia implies an additional light-curing process. The differences between Barodent and BelleGlass (p= 0.0002) which have the same post-curing protocol could be justified by the different amount of inorganic filler, that is 87 % in weight for BelleGlass and 65% in weight for Barodent. There are many studies that underline this hypothesis [9,19,20]. Kim et al. (2002) underlined that composite material with higher amount of inorganic filler, exhibits higher resistance to flexure and hardness [21]. The post-curing protocols of BelleGlass and Gradia could explain their significant differences between them (p=0.0001) [18].

Compressive strength

The compressive strength (CS) measures the force a material can support, at a single impact before breaking and gives information about the materials resistance to deformation [4][6]. The CS-testing consists in applying two axial forces from opposite direction on a cylindrical shaped specimen [12].[22]

The mean values and standard deviations for CS data were calculated with the Mann-Whitney test, the level of significance being 0.05 (Table 3). Barodent, Gradia and BelleGlass composites had the following mean values: 214.93, 226.11 and 241.64 MPa, respectively. There were no statistical differences between the three materials regarding compressive strength ($p>0.05$).

Table 3. Mean values and standard deviations for CS (n=10).

Composite materials		***Mean±SD***		**p**
Batch # 1	*Batch* # 2	*Batch* # 1	*Batch* # 2	
Barodent	BelleGlass	214.93±20.45	241.64±42.61	0.08
Barodent	Gradia	214.93±20.45	226.11±21.25	0.25
BelleGlass	Gradia	241.64±42.61	226.11±21.25	0.48

According to the literature data, the mechanical and physical properties of materials are influenced by the type, size and shape of the inorganic particles [23]. The CS values obtained for the materials under investigation could be explained by the amount and characteristics of the inorganic filler. For Barodent the filler is formed by a mixture of barium based glass, colloidal silica, quartz (65wt%), for BelleGlass the filler consists in a mixture of large pre-polymerized particles and nanoparticles (87 wt%), while for Gradia, it is formed from ceramic microparticles (75 wt%). The mean values were higher for the materials with a bigger amount of filler.

These results are in agreement with those presented in other studies that also show high CS values for composites with high quantity of filler and also for those with ceramic particles in their composition [23].

Diametral tensile strength

The compressive strength can be measured indirectly by the *diametral tensile strength (DTS)* consisting in applying a force, perpendicular to the longitudinal axis of a cylindrical shaped specimen [4].

The mean values and standard deviations were analyzed with Mann-Whitney test (Table 4). Gradia composite had the higher mean value (43.21±5.71), followed by Barodent (42.25±3.81) and BelleGlass (37.23±5.94) composites.

Table 4. Mean values and standard deviations for DTS (n=10).

Composite materials		***Mean±SD***		**p**
Batch # 1	*Batch # 2*	*Batch # 1*	*Batch # 2*	
Barodent	BelleGlass	42.25±3.81	37.23±5.94	0.04
Barodent	Gradia	42.25±3.81	43.21±5.71	0.85
BelleGlass	Gradia	37.23±5.94	43.21±5.71	0.06

Statistical significant differences were observed only between Barodent and BelleGlass composites (p=0.04). Even though BelleGlass (37.23 MPa) has lower values for DTS in comparison with Gradia (43.21MPa), there were no statistical differences between the two materials (p=0.06).

The DTS testing indicates the resistance of the dental restorations to lateral forces generated by tooth functionality [24]. Barodent composite has an intermediate DTS value between the two well-known composites. Although BelleGlass has lower values than Gradia, no significant differences are observed between them (p=0.06) because of the standard deviation which is higher for BelleGlass, that is 5.94 in comparison with 5.71 for Gradia. These results could be explained by the differences between the material's compositions [4].

The DTS testing can show different mean values for apparently similar biomaterials. This could be explained by the differences in organic matrix type [4].

The organic matrix of the majority of composite materials is based on Bis-GMA resin which is an aromatic dimethacrylate ester with a high rigidity and viscosity. In order to improve the fluidity of the Bis-GMA resin and to favour the filler dispersion, the resin is usually diluted with other polymers with lower molecular weight, like TEGDMA; unfortunately they increase the polymerization contraction with consequent negative effects on the material properties. It has been stated that replacing Bis-GMA with TEGDMA increases the DTS and decreases the FS [4]. This could explain the results for Barodent that has an organic matrix containing BIS-GMA, TEGDMA and UDMA. UDMA monomer has a similar molecular weight like Bis-GMA, but it is less viscous, so it assures a better polymerization [25]. The replacement of Bis-GMA with UDMA improves the mechanical properties, like flexural strength and diametral tensile strength [26]. This could explain our results, relative to the materials under investigation. One can add that BelleGlass has an organic matrix based on Bis-GMA, but also contains small amounts of UDMA. This could explain that, the FS and DTS results for BelleGlass are comparable with those for Gradia composite.

The comparative mechanical testing results of the investigated materials Barodent, BelleGlass and Gradia are depicted in Fig. 1.

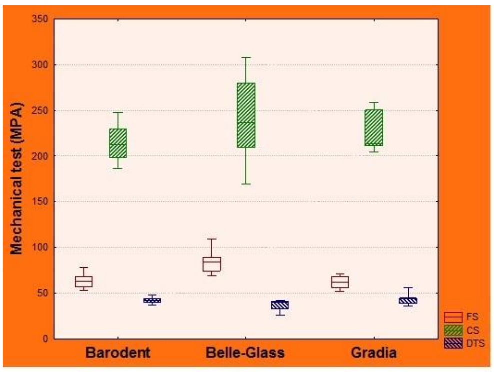

Fig. 1. Comparative analysis of mechanical tests results (median, 25%-75% percentiles, minimum, maximum).

The mechanical properties of composites are criteria on which the choice of the restorative materials and technique is based. The flexural, compressive, diametral tensile strengths are important qualities for a durable restoration to occlusal forces in the wet oral environment.

5. Conclusions

The present study investigated the following mechanical properties: flexural, compressive and diametral tensile strengths of a Romanian composite resin (Barodent) in comparison with two registered trademark dental materials (Gradia and BelleGlass).

The statistical analysis of experimental data shows that Barodent composite has lower values for flexural strength in comparison with BelleGlass, but similar with Gradia. Regarding the compressive and diametral tensile strength, the results were similar with those of the materials used for control.

The mechanical properties of Barodent composite material are determined by its composition, that is the type of organic matrix (Bis-GMA based resin), as well as by the quantity (65 wt%) and type of inorganic filler (mixture of barium glass, colloidal silica and quartz).

The mechanical properties of the experimental composite material Barodent similar with the control materials suggest that the material can resist to the functional masticatory forces, being suitable for the oral environment conditions. This fact remains to be validated by clinical studies.

Aknowledgements

This work was supported by CNCSIS-UEFISCSU, project number PN II-RU, PD-538/2010.

References

[1] A. Soanca, M. Rominu, M. Moldovan, C. I. Bondor, C. Nicola, A. Roman, Digest Journal of Nanomaterials and Biostructures, **6**(1), 383 (2011).
[2] C. Alb, M. Moldovan, C. Prejmerean, M. Trif, C. Sarosi, D. Dudea, V. Simon, Optoelectron. Adv.

Mater.–Rapid Commun. **3**(4), 350 (2009).
[3] A. Colceriu, T. Buruiana, M. Moldovan, C. Prejmerean, C. Tamas, O. Fodor, L. Oprean, V. Popescu, Optoelectron. Adv. Mater. – Rapid Commun. **1**(5), 231 (2007).
[4] A. Della Bona, P. Benetti, M. Borba, D. Cecchetti, Braz Oral Res, **22**(1), 84 (2008).
[5] H. F. Albers, Materials Science in Tooth-Colored Restoratives: Principles and Techniques, 9th ed., Bc Decker Inc, London, UK (2002).
[6] L. Wang, P. H. P. D'Alpino, L. G. Lopes, J. C. Pereira. J Appl Oral Sci, **11**(3), 162 (2003).
[7] A. Popovici (Soanca), D. Borzea, A. Pop, A. Roman, O. Pastrav, M. Moldovan, C. Prejemerean, Eng of Biomaterials, **47-53**(VII), 15 (2005).
[8] J. Manhart, H. Chen, G. Hamm, R. Hickel, Oper Dent, **29**(5), 481 (2004).
[9] S. A. Rodrigues Junior, C. H. Zanchi, R. V. Carvalho, F. F. Demarco, Braz Oral Res, **21**(1), 16 (2007).
[10] A. Popovici (Soanca), O. Fodor, M. Moldovan, A. Roman, D. Borzea, Optoelectron. Adv Mater.-Rapid Commun, **2**(12), 891(2008).
[11] A. Popovici (Soanca), M. Trif, M. Moldovan, O. Fodor, C. Nicola, D. Borzea, Optoelectron Adv Mater.–Rapid Commun, **3**(6), 616 (2009).
[12] A. Popovici (Soanca), A. Roman, H. Tassery, R. Seceleanu, Revista Română de Medicină Dentară, **XI**(4), 57, (2008).
[13] A. Popovici (Soanca), C. Nicola, M. Moldovan, C. I. Bondor, C. Badet, A. Roman, G. Baciut, M. Baciut, S. Bran, J. Optoelectron. Adv. Mater., **11**(4), 490 (2009).
[14] International Standard Organisation, Dentistry–polymer-based filling, restorative and luting materials, ISO 4049, 3rd ed (2000).
[15] American National Standard. American Dental Association Specification no. 27 for Resin-based filling materials, (1993).
[16] New American Dental Association, Council on Dental Materials and Devices. Specification No. 27 for Direct filling resins, J Am Dent Assoc, **94**, 191 (1977).
[17] A. Achima, Cadariu, Metodologia cercetării Stiintifice medicale, Edit. Univ. Iuliu Hasieganu, Cluj-Napoca, Romania (1999).
[18] M. E. Klymus, R. S. A. Shinkai, E. G. Mota, H. M. S. Oshima, A. M. Spohr, L. H. Burnett Jr., Baltic Dental and Maxillofacial Journal, **9**, 56 (2007).
[19] W. L. Kraig, S. Vandewalle, Dent Mater, **26**(4), 337 (2010).
[20] A. F. S. Borges, G. M. Correr, M. A. Coelho Sinhoreti, S. Consani, L. Correr Sobrinho, R. M. P. Rontani, J Dent, **34**, 478 (2006).
[21] K. H. Kim, J. L. Ong, O. Okuno, J Prosthet Dent, **87**, 642 (2002).
[22] E. Bresciani, T. J. E. Barata, T. C. Facundes, A. Adachi, M. M. Terrin, M. F. L. Navarro, J Appl Oral Sci, **12**(4), 344 (2004).
[23] A. Da Fonte Porto Carreiro, C. A. Dos Santos Cruz, C. E. Vergani, J Oral Rehab, **31,** 1085 (2004).
[24] I. S. Medeiros, M. N. Gomes, A. D. Loguercio, L. E. R. Filho, J Oral Sci, **49**(1), 61 (2007).
[25] D. S. M. Casselli, C. C. Worschech, L. A. M. S. Paulillo, C. T. S. Dias, Braz Oral Res, **20**(3), 214 (2006).
[26] M. C. C. G. Tolosa, L. A. M. S. Paulillo, M. Giannini, A. J. S. Santos, C. T. S. Dias, Braz Oral Res, **19**(2), 123 (2005).

*Corresponding author: andrapopovici@yahoo.com