# Researches on Composite Resins Used in Dental Restorations

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*The aim of the present paper was to highlight the interaction between two composite resins available for dental restorations with artificial saliva in connection with the material properties. The study was focused on specific tribological aspects concerning wear, microhardness of composite material surfaces, friction and lubricant film. The two types of tested materials were composite resins: CERAM-X and FILTEK SUPREME. The experimental researches were based on important laboratory equipment such as microhardness device, metallographic microscope and spectrophotometer.*

*Keywords: composite resins, tribology, wear, microhardness, dental medicine*

Keeping within the limits of normality the structural integrity and stability of the materials used in restorative dentistry was a constant concern. Presence in the oral cavity of dental restorative materials in contact with saliva environment also affects both material properties and those of saliva. Dental restorative materials contain a greater or lesser amount of so-called filler elements, elements influencing the mechanical properties of the material, and especially the behavior of the oral environment. Restorative material effect on the oral environment is multiple, ranging from simple color changes to changes in the binding force between tooth and material, with initiation and maintenance of crevices or data changes caused by wear. This phenomenon is accompanied by the appearance of dimensional changes with initiation of cracks in the surface and in depth. In general, wear is dependent on the material and the interface between them, but also in the presence of a lubricant film, such as the film saliva, separating surfaces during motion and reducing friction and wear.

Previous research has shown that, in general metals are prone to an adhesive wear, corrosive, while the composites show abrasive wear by surface fatigue too [1].

In this study it is analyzed the interaction *in vitro* of composite resins used in dental crown restorations and artificial saliva, aiming both the effect of saliva on material electrochemical corrosion and superficial and deep changes occurring in the mass of material.

## Experimental part

We have studied two types of composite resins:

- CERAM – X containing filling rate of 62% by volume, including glass filler (~1 μM), nanofilling (~10 nm) and organically modified ceramic nanoparticles (2-3 nm).

- FILTEK SUPREME Plus Universal Restaurativ which contains a unique combination of nanofilling/nanomeri (discrete non-agglomerated particles and non-aggregated) ranging in size from 5-75 nm and nanobundles (weakly bound agglomerates of nanodimensional particles) embedded in an organic polymer matrix.

Processing, i.e, application, adjustment and finishing of samples was performed sequentially under producing tools and manufacturing companies instructions.

To determine the hardness it was used oughness microhardness device, Neophot metallographic microscope that allows, through microscopic analysis, to measure the hardness of different structural constituents. For microhardness measurements on penetrator (diameter D) it was used a load of about 100 gf for a certain period of time.

To determine the effect of saliva on electrochemical corrosion of materials it was used as appreciation liquid medium the artificial saliva proposed by Duffo and Quezada [2]. The composition of this saliva is presented in table 1. It was chosen this composition because it has very close corrosive properties to those of natural saliva. The *p*H value of this solution was determined with a pH-meter/mili-voltmeter RADELKIS OP-208 *p*H = 7.08.

**Table 1**

ARTIFICIAL SALIVA COMPOSITION PROPOSED BY DUFFO AND QUEZADA

| Composition (g/L) | |
|---|---|
| NaCl | 0.600 |
| KCl | 0.720 |
| $CaCl_2 \cdot 2H_2O$ | 0.220 |
| $KH_2PO_4$ | 0.680 |
| $Na_2HPO_4 \cdot 12H_2O$ | 0.856 |
| KSCN | 0.060 |
| $KHCO_3$ | 1.500 |
| citric acid | 0.030 |

To assess the surface morphology obtained after immersion in Duffo-Quezada saliva was used an APCA-2400 microscope by AFM technique (Atomic Force Microscopy).

For recording solutions spectra after electrochemical treatment of materials it was used an UV-Vis spectrophotometer - NANODROP-1000 type .

## Results and discussions

Analysis of CERAM-X material hardness shows its increased value at the sample surface of material and its decrease as we move to depth, high hardness which influence the strength in the mouth (fig. 1). This variation in hardness values is directly proportional to the degree of polymerization of the composite resin.

The same variation is observed for composite Filtek SUPREME (fig. 2). This behaviour could be attributed to

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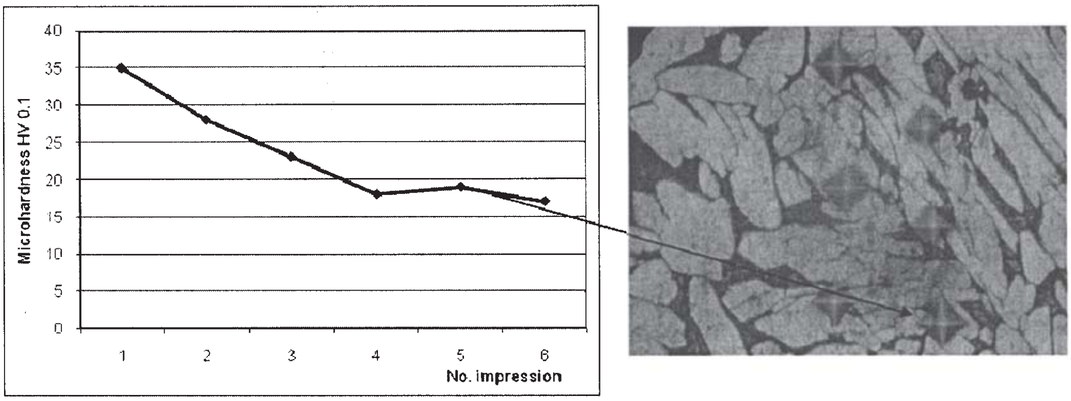


Fig. 1. Microhardness for CERAM-X with loads of 100 gf (HV 0,1)

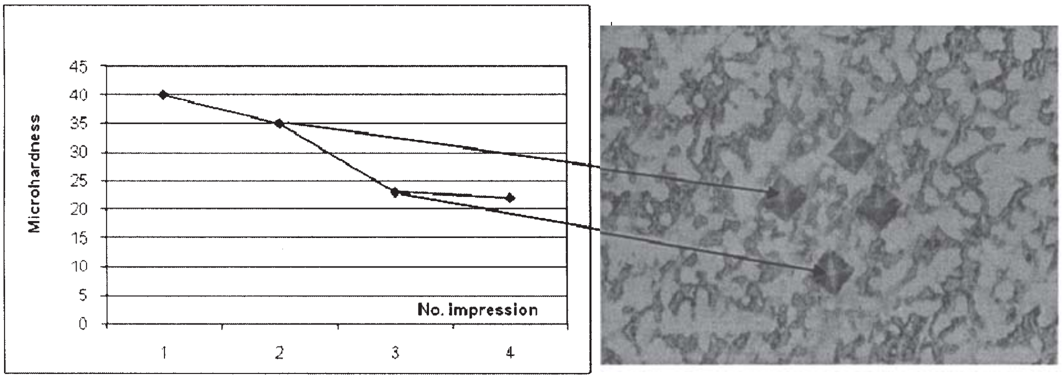


Fig. 2. Microhardness for FILTEK -SUPREM with loads of 100 gf (HV 0,1)

insufficient polymerization of the methacrylate groups from restorative material structure.

As for CERAM-X material, free radicals resulting from initiation system collide with the double links of carbon-carbon of the monomer and joins from an electron of the double bond, leaving free the other one electron. Thus, each monomer molecule becomes a free radical and initiates the formation of new connections. However, much of the initial non-activated light methacrylate groups remain nonpolymerized even after several hours. Degree of conversion of double connections at a depth of 0.2 mm is between 25-65% [3, 4].

During the photopolymerization process, the surface layer is enriched in oxygen, up 7 percent for FILTEK SUPREME XT material and low with 3 atomic percent for CERAM-X [4, 5]. This is a consequence of the formation of oxygen inhibited layer of a few micrometers on the surface, caused by the high affinity of the monomer to the oxygen with a low degree of polymerization, and thus lower mechanical properties.

Surface analysis by atomic force microscopy for FILTEK SUPREME material shows a little rough of its microstructure (average topography ranges from -38.48 nm and 32.41 nm). This shows good machinability of the material while maintaining aesthetics and smooth (fig. 3). It appears a slight congestion of reinforcement elements with large and very large sizes (clusters of nanoparticles) in well-defined directions probably by mechanical process of applying the material before photopolimeryzation. [6, 7].

For CERAM-X material is observed a topography average of -22.00 nm and 35.30 nm, much higher than in case of previous material, which is made primarily due to geometric architecture obtained in this case after applying, photopolymerazion and machanical processingf or the layer (fig. 4).

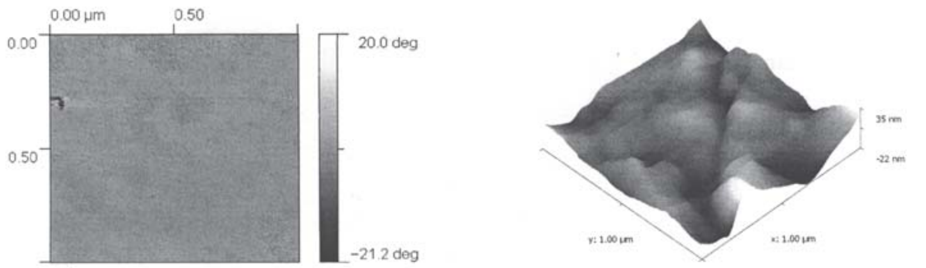


Fig. 3. Aspects for worn surface for FILTEK SUPREM

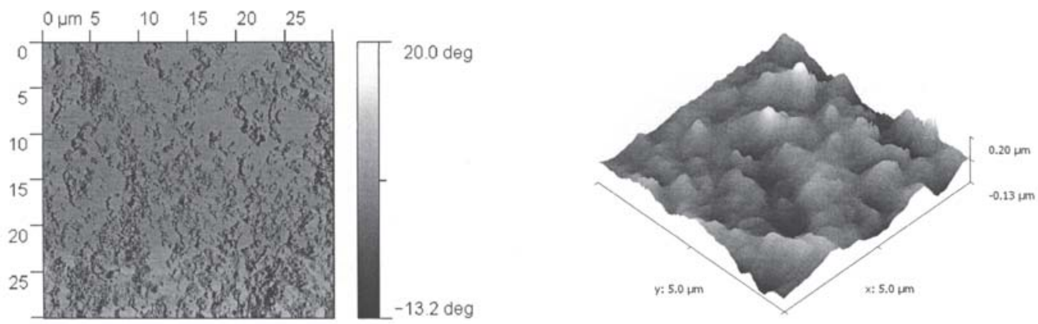


Fig. 4. Aspects for worn surface for CERAM-X

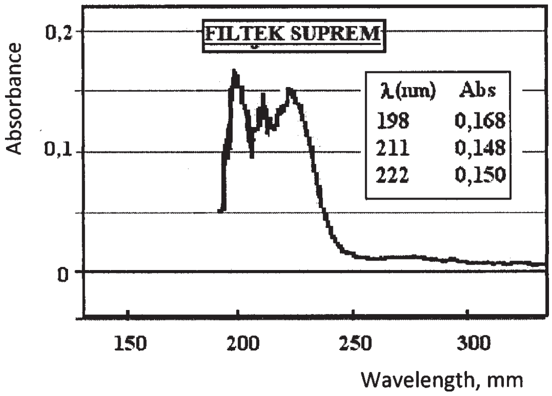


Fig. 5. UV spectrum of saliva Duffo-Quezada after electrochemical treatment of FILTEK SUPREM material

Below are presented both the UV spectra of artificial saliva in which were treated electrochemically the studied materials and the characteristic absorption bands. (fig. 5, 6)

In the case of artificial saliva in which it was treated FILTEK SUPREME material movement the bands group from domain I is reduced to a single band, wider and less intense, and the bands from the domain II remain as intense but grouped. Instead a distinct band appears at a wavelength of 211 nm, band attributable to the silicon past in solution.

For the artificial saliva in which it was treated CERAMI-X material obvious attenuation of absorption band from wavelength of 195 nm, accompanied by the amplification band from 205 nm - domain I and from 226 nm in domain II. A series of overlapping bands appear as a “shoulder” in the 210-216 nm domain attributable to aluminum silicates past in solution.

As shown, the restorative material behaviour depends on the resin matrix, silane coupling agent integrity, the type, quantity and size of the filler particles. Wear process is complex, even if the material is not under the direct action of important restorative forces, but rather chemical or thermal variations.

Hydrolytic degradation of barium and strontium in particular can cause pressure at the interface resin-filler material with the appearance of microcracks. Un-reacted methacrylate groups degrade quickly and can be “extracted” from resin, amplifying the effect of degradation components. All this results in lower hardness material [8]. The cracks appearing on the surface amplifies the effects of abrasion, leading to increased porosity. To this we can add the fact that chemical attack can cause failure of silane agent, leading to the destruction of the relationship created between the filler-matrix material [3, 8].

As is shown by [7], filler particle size can influence the hardness and wear resistance of these composite resins in artificial saliva. Thus, the loss of material is determined by the release in solution of small particles, while larger particles are more difficult to drive [9-11]. For this reason, increasing the amount of inorganic filler increases the surface cracks, with the advent microcracks formed before main crack extension and reducing the concentration of forces around the crack tip.

## Conclusions

Based on the obtained results we can say that both materials have nearly identical behaviour in terms of material properties, especially hardness of the material.

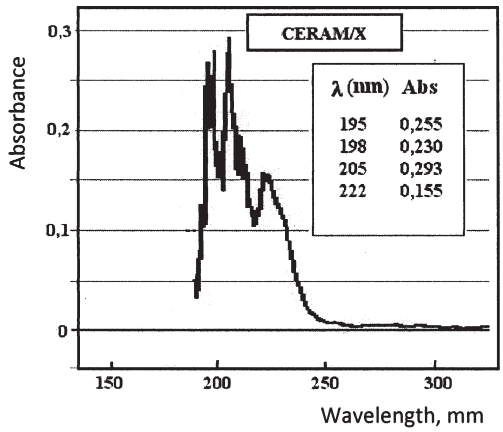


Fig. 6. UV spectrum of saliva Duffo-Quezada after electrochemical treatment of CERAM-X material

But introduced in Duffo-Quezada saliva is found an absorption bands change. Atomic force microscopy of the composite FILTEK SUPREME XT showed a low surface roughness, which reflects good machinability of the material and the clinical appearance of finished surface is smooth, glossy and aesthetically default. On the contrary, the composite CERAMIC-X has increased roughness due to large reinforcement elements, partially embedded in the polymeric matrix. This leads to good machinability of the material, but with compromised esthetics and stability in the saliva.

## References

1. KIYOSHI KAKUTA, AMORNRAT WONGLAMSAM, SHIN-ICHI GOTO, HIDEO OGURA, Surface textures of composite resins after combined wear test simulating both occlusal wear and brushing wear, Dental Materials Journal, 31, nr. 1, 2012, p. 61

2. DUFFÓ, G., S., QUEZADA C., E., Development of an Artificial Saliva Solution for Studying the Corrosion Behavior of Dental Alloys. Corrosion, 2004, 60, nr. 6, 2004, p. 594

3. KIREMITCI, A., ALPASLAN, T., GURGAN, S., Six-year clinical evaluation of packable composite restorations, Oper Dent., 34, nr. 1, 2009, p. 11

4. KON, M., KAKUTA, K., OGURA, H., Effect of occlusal and brushing forces on wear of composite resins, Dent Mater J, 25, 2006, p. 183

5. DUARTE, S., JR., BOTTA, A., C., PHARK, J., H., SADAN, A.. Selected mechanical and physical properties and clinical applicaton of a new low-shrinkage composite restoration, Quintessence Int., 40, nr. 8, 2009, p. 631

6. BAGIS, Y., H., BALTACIOGLU I., H., KAHYAOGULLARI, S., Comparing microleakage and the layering methods of silorane-based resin composite in wide Class II MOD cavities, Oper Dent., 34, nr. 5: 2009, p. 578

7. KAKABOURA, A., FRAGOULI, M., RAHIOTIS, C., SILIKAS, N., Evaluation of surface characteristics of dental composites using profilometry, scanning electron, atomic force microscopy and gloss-meter, J Mater Sci Mater Med., 18, 2007, p. 155

8. SHEILA REGINA MAIA BRAGA, TARSILA YUMI TACHIBANA, NARCISO GARONE-NETTO, MARIA ÂNGELA PITA SOBRAL, Abrasion resistance of different resin composites, J Health Sci Inst., 29 nr. 2, 2010, p. 85

9. DCHIMELLO, D., T., DIBB, R., G., P., CORONA, S., A., M., LARA, E., H.G., Assessing Wear and Surface Roughness of Different Composite Resins After Toothbrushing, Materials Research, 4, nr. 4, 2001, p. 285

10. ATTAR, N., Effect of Finishing and Polishing Procedures on the Surface Roughness of Composite Resin Materials, Journal of Contemporary Dental Practice, 8, nr. 1, 2007, p. 27

11. WONGLAMSAM, A., KAKUTA, K., OGURA, H., Effects of occlusal and brushing cycles on wear of composite resins in combined wear test, Dent Mater J, 27, 2008, p. 243

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