

In vitro Interaction Between Two Composite Restorative Materials and Artificial Saliva

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Dental restorative materials due to their composition and structure relatively easily reacts in oral environment, subject to a chemical, electrochemical and biological degradation process. Effects vary from material discoloration and pigmentation, leading to cracks or fractures of fillings. Saliva as oral biotope is a continuous environment favouring these changes. In this saliva are dissolved chemicals coming from the external environment (food, beverages), which modifies the composition and the pH of the saliva, elements which are playing a main role inside the degradation processes, and in particular in electrochemical process. The intensity and its effects are heavily determined both by the composition, structure and quality of materials used and the composition and pH of saliva.

Keywords: composite materials, restorative dentistry, artificial saliva

In this study we analyze the interaction *in vitro* between two composite restorative materials and the artificial saliva, aiming both saliva effect on the material composition and the changes produced on some properties of saliva.

The paper continues to present data obtained by authors based on their experimental researches, part of them mentioned in previous studies [1-3].

Experimental part

There were studied Filtek Supreme XT and Filtek Z250 dental materials which were light-cured using a LED source (light emitting diode) Optilight LD MAX - Gnatus type for 60 seconds to ensure uniformity in depth material. According to the manufacturer, the composition of the two materials is the same, the difference being given by the size of the filler particles. The chemical percentage of zirconium is slightly higher in Filtek Z250 with 0.5%, indicating a higher load with oxid zirconia particles.

The liquid medium used was an artificial saliva proposed by Duffo and Quezada. It was chosen this composition because some previous studies have indicated that it is very close to the corrosive properties of natural saliva. The pH of this solution was determined by an OP-208 pH-meter/milivoltmeter RADELKIS (Budapest, Hungary): pH = 7.08.

To characterize the surface morphology of the two materials before and after electrochemical treatment it was used scanning electron microscopy technique for this purpose using VEGA II LSH-type microscope (fig. 1). In order to reduce the time needed for determining the interaction between the materials and the utilized artificial saliva, the electrode potential was kept constant at the value of 2V, the corrosion current density over 1000 times greater than the density of the instantaneous current. For all the tests of the cell volume of artificial saliva was the same, 50 mL, and the voltage between the platinum electrode and material sample $E = 2V$ and corrosion during 30 minutes. Sample material was connected to the positive terminal of the source.

For recording spectra of solutions after electrochemical treatment of materials it was used an UV-VIS spectrophotometer type NanoDrop-1000 (fig. 2).



Fig. 1. The electron microscope Vega II LSH



Fig. 2. Spectrophotometer UV-VIS

After placing the sample and performing the measurement through the display window it appears the spectrum of the used solution. In the saliva spectrum it can be distinguished two groups of absorption bands: a group in the I domain: 195-205 nm - more intense and the group in the II domain: 220-225 nm - wider and with a lower intensity.

Results and discussions

The surface of Filtek Supreme XT material presents pitting corrosion (fig. 3 - details 3c and 3d), relatively small but deep.

The changes are developed over the entire surface from dendrites limits of its microstructure. Acid attack is profound.

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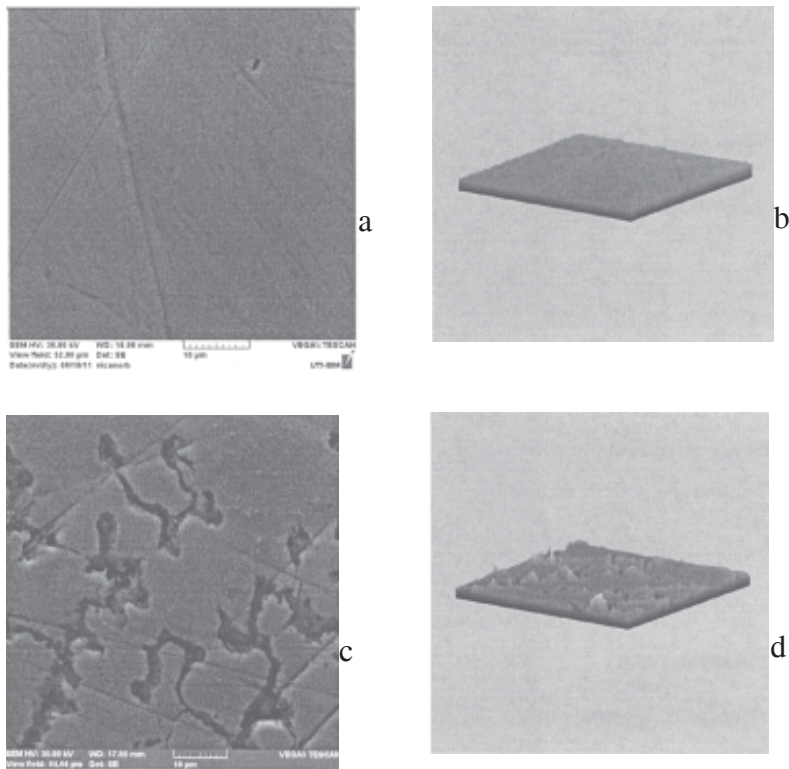


Fig. 3. Filtek Supreme microstructure of the surface before corrosion (a and b) and after corrosion (c and d)

Filtek Z250 material behavior is very similar to the material Filtek Supreme, but corrosion points are much higher and deeper, although the electrode potential and time of treatment were the same (fig. 4).

On the material surface it was formed a complex deposit in which there can be distinguished some cracks and microcrystalline deposits.

Some literature studies [4, 5] showed the formation of an oxygen-inhibited layer (OIL) to the surface of the methacrylate resin, light-cured in the presence of oxygen. This is the result of high affinity of oxygen free radicals, which is greater than the attraction to carbon-carbon double bonds and therefore slows down the formation of the polymer. In this way the OIL can be defined as an inhibited layer of the monomer which is presented on the methacrylate resin surface. The thickness of this layer has

been experimentally found within very wide limits, from $4\mu\text{m}$ to $84\mu\text{m}$ and is dependent on the viscosity of the resin, filler loading, temperature, [4]. Clinically, the presence of oxygen inhibited layer is important because the layers application of the composite restoration required by deep cavities and curing thickness, the existence of interfaces between them decreases the restoration mechanical properties [8, 9].

Below there are presented the UV spectra of artificial saliva in which there were electrochemically treated the two studied materials and characteristic absorption bands. Thus, for Filtek Supreme material the lanes group of the I domain is reduced to a single lane, wider and less intense, and bands of the II domain remain as intense but grouped. However a distinct band appears at a wavelength of 211 nm ; this bandwidth can be assigned to the structure of

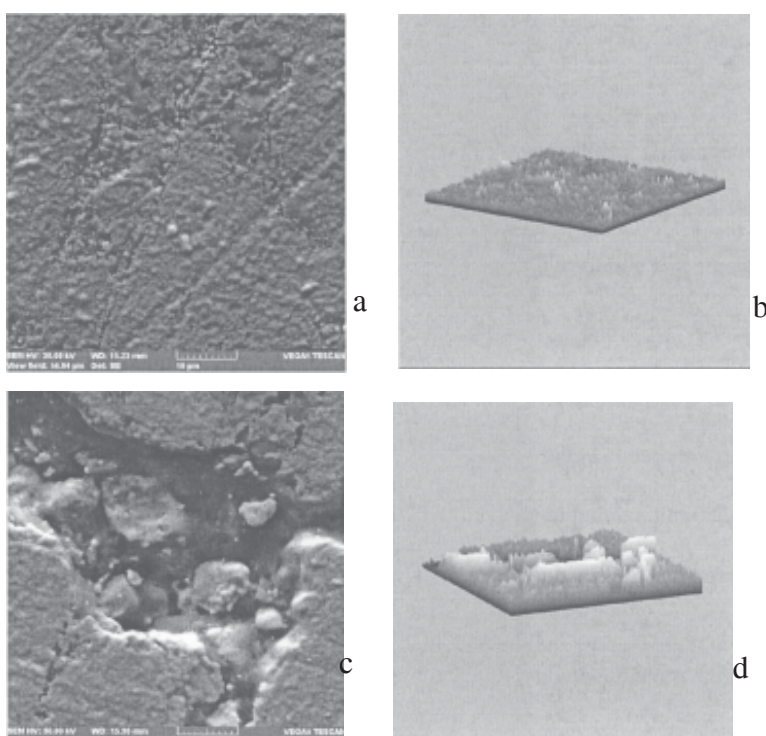


Fig. 4. Filtek 250 microstructure of the surface before corrosion (a and b) and after corrosion (c and d)

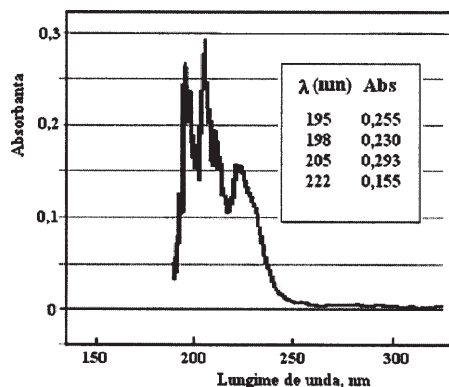


Fig. 5. UV-Quezada Duffo saliva spectrum after electrochemical treatment of Filtek Supreme material

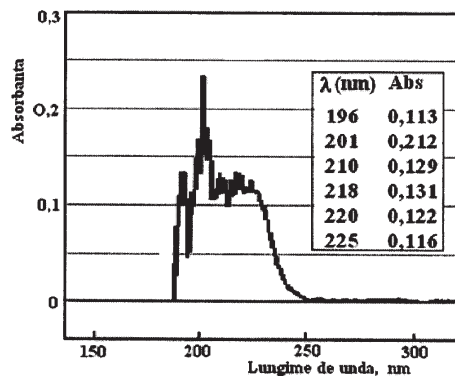


Fig. 6. UV-Quezada Duffo saliva spectrum after electrochemical treatment of Filtek Z250 material

zirconium material that went into solution. It is amplified to the 205 nm band of the I domain and the 226 nm band of the II domain. A series of overlapping bands appear as a "shoulder" in the 210-216 nm domain (fig. 5).

For Filtek Z250 material, the changes notified for the first material are more obvious (more distinct); the wavelength band of 195 nm is more attenuated and the wavelength band of 205 nm becomes the most intense; the bands of 210-218 nm are not presenting the shoulder and there are distinct and approximately of the same intensity. The 210 nm band is attributed to the zirconium passed in solution, and the band of 215-218 nm range is due to the silicon ions in solution (fig. 6).

The electrochemical treatment restorative materials in solution alter the composition of the solution, so the dissolution of ions in the material and by changing some of the components of the solution. Depending on the material used and its degree of corrosion may occur changes in the absorption bands, or by changing the position or the changes in the intensity. Unfortunately, the complexity of the solution compositions, materials and corrosion products and the lack of precise methods of analysis, it can only be a qualitative analysis of the changes produced on the solution.

During the curing process, the top layer of restorative material enriched in oxygen, up 7 normalized atomic percent for Filtek Supreme XT and more pronounced with 3 normalized atomic percent normalized for Filtek Z250. This is a consequence of the formation of a few micrometers oxygen inhibited layer on the surface determined by high affinity of the monomer to oxygen with a low degree of polymerization, and therefore lower mechanical properties [6, 7].

Conclusions

Concerning Filtek Z250 hybrid composite must be mentioned the presence of a strong tendency to agglomerate nanoscale filler particles in clusters with sizes up to 2 μm with the existence of free zones in the polymer

matrix without observable particles. For Filtek Supreme XT nanocomposite it is a more uniform spatial distribution of particles without agglomeration, with maximum dimensions of approximately 1 μm.

Due to the composite material corrosion there were actually lost percentages of silicon and zirconium and less of the composite material matrix of the, UV spectrum of ions which appear in the saliva analysis.

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