

## STUDY OF SURFACE ALTERATIONS OF COMPOSITE AND IONOMERIC MATERIALS SUBMITTED TO SIMULATION OF A HIGH CARIOGENIC CHALLENGE<sup>†</sup>

### ESTUDO DA EROSÃO SUPERFICIAL DE MATERIAIS COMPÓSITOS E IONOMÉRICOS SUBMETIDOS À SIMULAÇÃO DE UM ALTO DESAFIO CARIOGÊNICO

Alexandre Rezende VIEIRA\*  
Ivete Pomarico Ribeiro de SOUZA\*\*  
Adriana MODESTO\*\*\*

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VIEIRA, A. R.; SOUZA, I. P. R.; MODESTO, A. Study of surface alterations of composite and ionomeric materials submitted to simulation of a high cariogenic challenge. *Rev Odontol Univ São Paulo*, v. 13, n. 4, p. 321-327, out./dez. 1999.

We evaluated the surface of composite resins and glass-ionomer cements in a situation of high cariogenic challenge. Based on seventy-five standard test specimens of one glass-ionomer cement (Chelon Fil - ESPE), one resin-modified glass-ionomer (Vitremar - 3M), two polyacid-modified composite (VariGlass and Dyract - Dentsply) and one composite resin (Heliomolar - Vivadent), submitted to fourteen days of demineralization and remineralization cycling to simulate a high cariogenic challenge, the erosive aspects of the surface of the materials were assessed. All of the samples were evaluated by scanning electronic microscope and compared with another five test specimens of each material, prepared in the same way and serving as control. All of the materials studied suffered erosive action by the media, with different characteristics due to the different compositions, after being submitted to *in vitro* simulation of a high cariogenic challenge.

UNITERMS: Composite resins; Glass-ionomer cements; Erosion.

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## INTRODUCTION

Glass-ionomer cements present considerably lower resistance to abrasion than composite resins, and similar to that of silicate cements *in vitro*<sup>1</sup>. According to SETCHELL *et al.*<sup>8</sup> (1985), the mechanical forces and resistance to the combined effects of abrasion, erosion and dissolution are very important factors for a material to be considered satisfactory.

To minimize the problems relating to mechanical wear of the material, some materials were introduced to the market with important structural alterations from the original formula, so that today there is a whole spectrum ranging from “real”

glass-ionomer cements to “real” composite resins.

It was because of this that McLEAN *et al.*<sup>5</sup> (1994) suggested a new nomenclature to better identify this array of materials. Glass-ionomer cement is the material that consists of a basic glass and an acid polymer which undergo an acid-base reaction. Glass is a decomposable acid, usually made of finely crushed powder which, in contact with the watery acid released from the cement in formation, gives off Ca<sup>2+</sup>, Al<sup>3+</sup> and possibly also Sr<sup>2+</sup>, La<sup>2+</sup> or Zn<sup>2+</sup> ions, depending on its composition<sup>5</sup>. The acid polymer is typically polyacrylic acid, which can comprise polymers and copolymers of the acrylic, itaconic, maleic and phosphonic vinyl

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\* Master; \*\* Full Professor; \*\*\* Assistant Professor - Department of Pediatric Dentistry and Orthodontics - School of Dentistry - Federal University - Rio de Janeiro - Brazil.

acids. The term resin-modified glass-ionomer is related to the materials that present an acid-base reaction, like the true glass-ionomer cements, supplemented by a polymerization reaction either of the monomer (bis-GMA or 2-hydroxyethyl methacrylate), or of the lateral chains of the polyacid molecule<sup>5</sup>. The resin-modified glass-ionomers harden in the dark, but this is a slower process and the material becomes very fragile, with its resistance lower than that obtained by photopolymerization<sup>5</sup>. Finally, there is a third category of materials which present the same ingredients (decomposable-acid glass, possibly some polymeric acid), but in amounts that are insufficient to produce the acid-base reaction in the dark. These are the polyacid-modified composites.

The identification of risk situations for the development of new caries lesions is an important step in choosing the restorative material. The capacity of releasing fluoride by ionomeric materials makes them an attractive option for the restorative treatment of the disease. However, an environment with a lot of pH variations will increase the material's erosion despite its fluoride release<sup>6</sup>.

In order to verify how these materials behave in the presence of an acidic medium, the study attempted to evaluate the surface of the samples of five restorative materials submitted to a fourteen-day process of demineralization and remineralization cycling that simulated a situation of high cariogenic challenge.

## MATERIAL AND METHOD

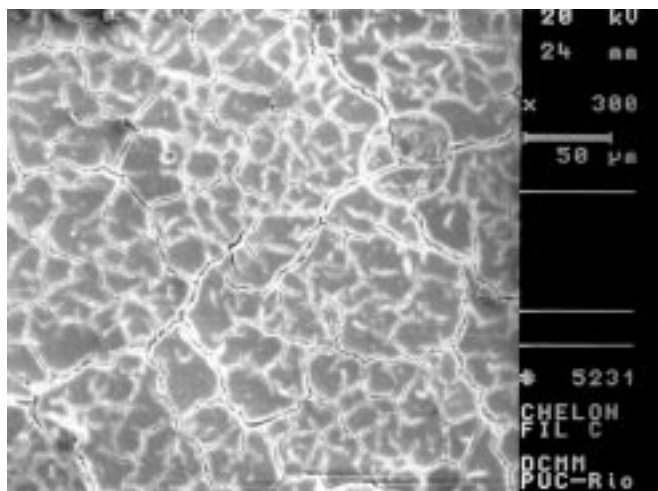
One hundred samples (twenty of each material) measuring 10.5 millimeters in diameter by 2.0 millimeters thick were obtained by means of standardized teflon matrices. The restorative materials used were proportioned and mixed according to the manufacturers' specifications and the following materials were used: Chelon Fil (ESPE) glass-ionomer cement, Vitremer™ (3M) resin-modified glass-ionomer, VariGlass™ VLC (Caulk/Dentsply) and Dyract (DeTrey/Dentsply) polyacid-modified composites and Heliomolar radiopaque (Vivadent) composite resin. The samples of Chelon Fil, Vitremer, and VariGlass were prepared using three powder/liquid measures to fulfill each matrix. Dyract and Heliomolar samples were prepared by putting small portions of each material in the matrix. After that the

photopolymerization of the materials, except for Chelon Fil, was done.

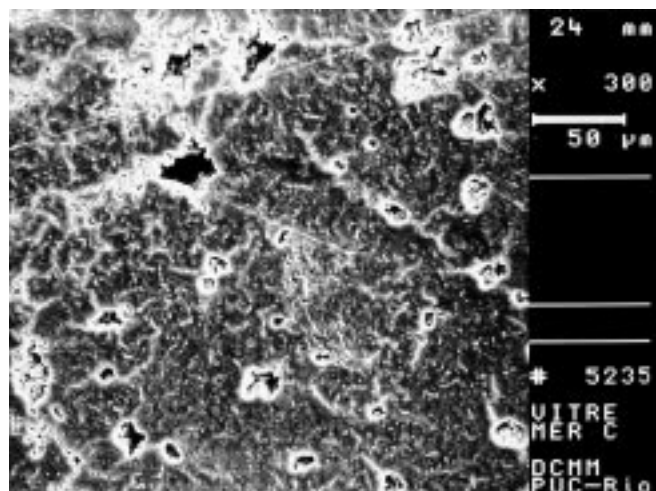
After preparation, all of the samples were protected by polyester strips and left in an oven at 37°C and 100% relative humidity for twenty-four hours to allow for maturation of the gelatinization reaction of the glass-ionomer cements and for the composites to polymerize. After this period, the polyester strips were removed from over the samples to be submitted to the cariogenic challenge. The matrices were cut with a scalpel blade for easy removal.

Fifteen specimens of each material were submitted to a simulation of high cariogenic challenge for fourteen days<sup>3</sup>. Each test specimen was stored initially in 2.0 ml of demineralizing solution at 37° C for 6 hours and then washed briefly with deionized water and transferred to 2.0 ml of remineralizing solution at 37° C, where they stayed for 17 hours and were then transferred again to 2.0 ml of demineralizing solution, and so forth. The demineralizing solution was a buffer containing 2.0 mmol/L phosphate and 7.5 mmol/L acetate at pH 4.3. The remineralizing solution was supersaturated with calcium phosphate, *i.e.*, 1.5 mmol/L calcium, 0.9 mmol/L phosphate with potassium chloride at 150 mmol/L and 20 mmol/L cacodylate buffer at pH 7.0. After this period the fifteen samples of each material had their surfaces evaluated and compared with the other five prepared to serve as control and not submitted to the simulation of high cariogenic challenge. The control samples remained for fourteen days in an oven at 37°C and 100% relative humidity.

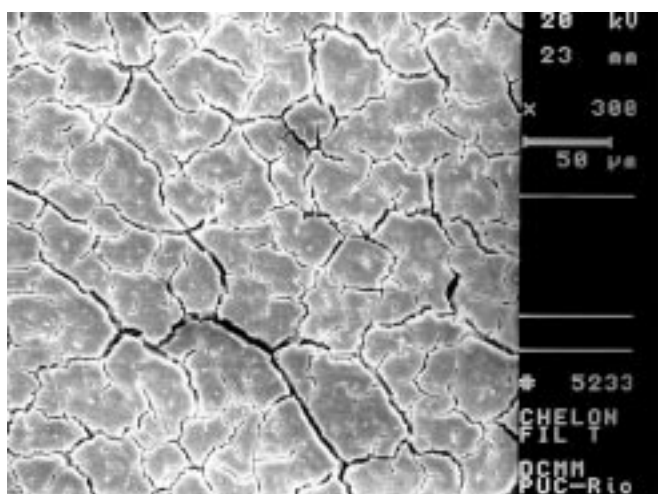
For the study of the surfaces of the samples, the materials were evaluated on the scanning electronic microscope (SEM). All the samples were attached to metallic sample-holders, given a coating of gold-palladium at 300 Å after the pH cycles. Before they were analyzed by the SEM, all samples remained in a vacuum chamber for thirty minutes in order to be dehydrated. A description was made of the aspect of the surface of the samples of the materials tested in comparison with the samples that served as control and the images were registered by electromicrographs.



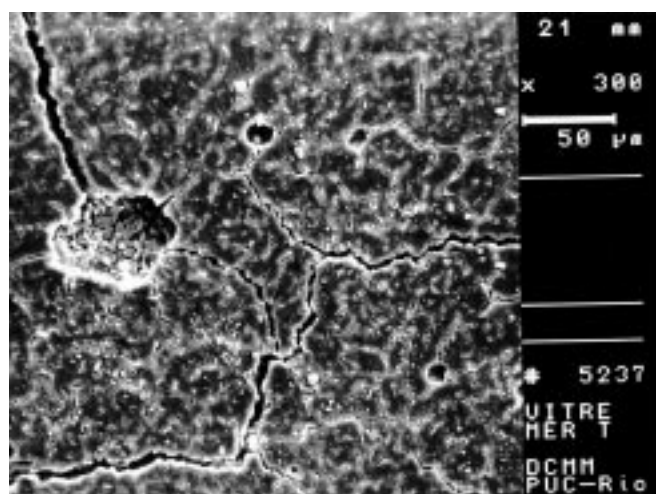
**FIGURE 1** - Photomicrograph of a control sample of Chelon Fil (300 X).



**FIGURE 3** - Photomicrograph of a control sample of Vitremer (300 X).



**FIGURE 2** - Photomicrograph of a test sample of Chelon Fil (300 X).



**FIGURE 4** - Photomicrograph of a test sample of Vitremer (300 X).

## RESULTS

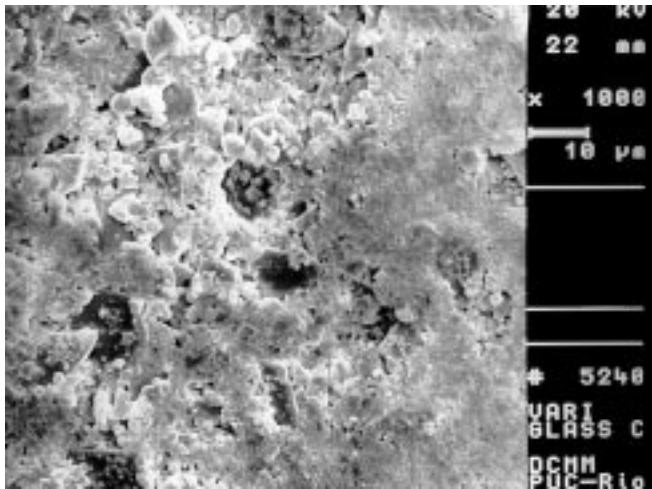
The Figures presented exemplify the pattern of surface verified in all of the samples, both those that served as control and those submitted to the demineralization and remineralization cycles.

The pattern observed in the samples used as control for the Chelon Fil material was that of a cracked surface (Figure 1). After undergoing the demineralization and remineralization cycles, the samples still presented a cracked surface pattern (Figure 2). When we compare Figures 1 and 2, we

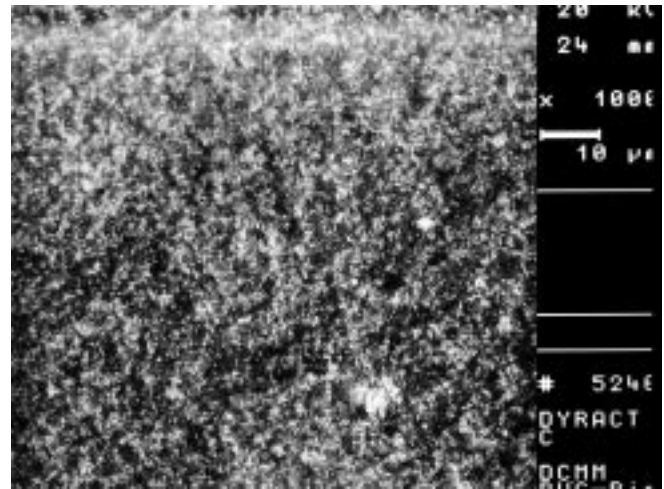
note that the cracks seen in the sample that underwent the cycles are more marked, more evident, and the small cracks and surface flaws seen between the larger cracks are more evident in the sample submitted to the cycles.

The pattern observed in the Vitremer samples that served as control of the material was that of a surface with a great deal of porosities (Figure 3), and some particles were seen protruding from the surface. In the samples submitted to demineralization and remineralization cycles, the presence of cracks could be observed apparently joining to-

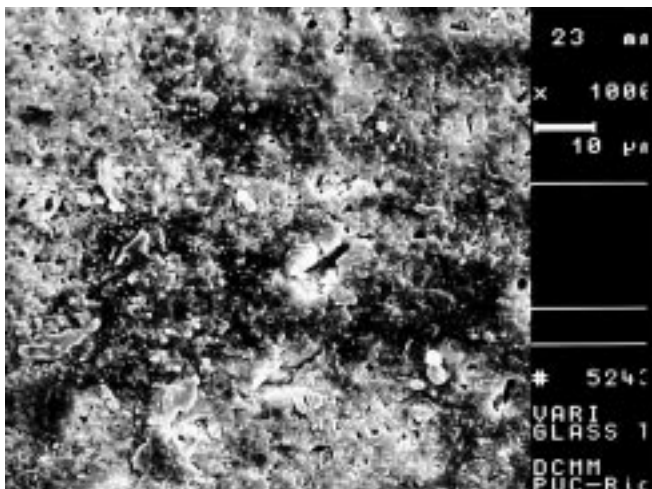




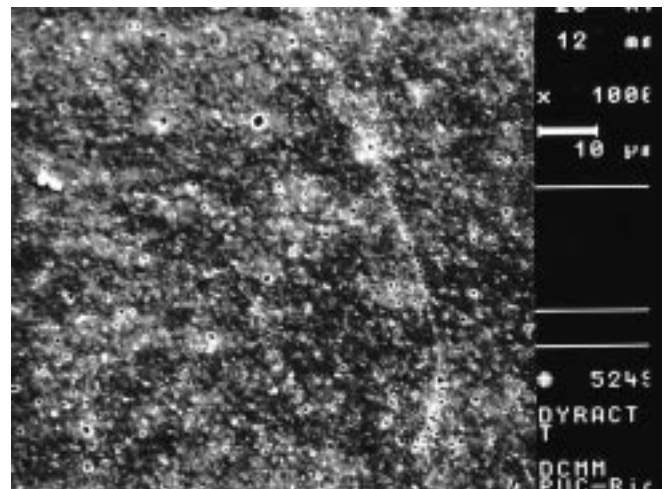
**FIGURE 5** - Photomicrograph of a control sample of VariGlass (1,000 X).



**FIGURE 7** - Photomicrograph of a control sample of Dyract (1,000 X).



**FIGURE 6** - Photomicrograph of a test sample of VariGlass (1,000 X).



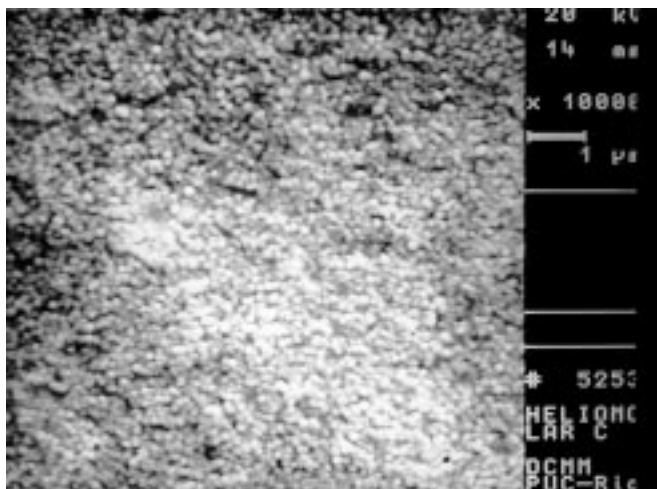
**FIGURE 8** - Photomicrograph of a test sample of Dyract (1,000 X).

gether the larger porosities (Figure 4). No particles were seen to stand out from the matrix, but many cracks were seen between the larger fissures, giving the impression that the surface had become less irregular.

The pattern verified in the VariGlass samples used as control of the material was that of a surface with few porosities, and particles were observed apparently protruding from the matrix (Figure 5). These particles presented a variety of sizes, measuring as much as about thirteen micrometers, and prominent edges. In the samples exposed to the demineralization and remineralization cy-

cles, the presence of few porosities was also observed, yet the impression was that the surface became less irregular (Figure 6). The particles did not have the same dimension as seen in Figure 8, appearing to be rounder in shape, even in the deeper parts of the porosities.

The surface pattern of the Dyract samples that served as control denoted regularity, and no marked flaws were seen (Figure 7). The samples submitted to demineralization and remineralization cycles showed a similar pattern to that of the control samples, however a greater amount of porosities was verified (Figure 8).

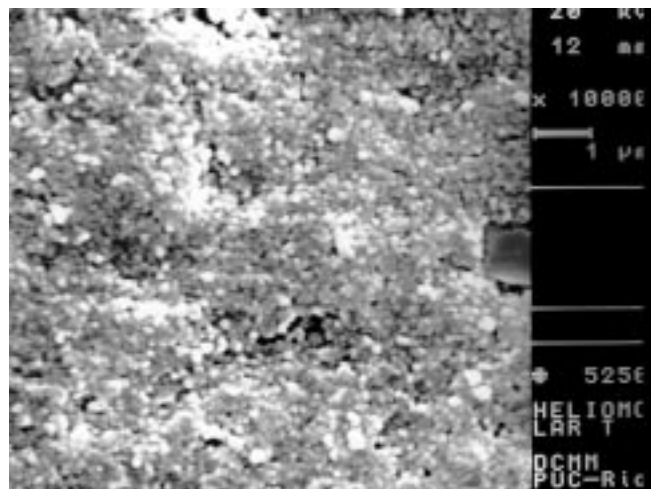


**FIGURE 9** - Photomicrograph of a control sample of Heliomolar (10,000X).

The standard pattern of the samples that served as control of the Heliomolar presented quite a regular, smooth surface. At greater magnification it was possible to perceive that numerous small particles seemed to protrude from the matrix of the material (Figure 9). The presence of some porosities was observed. The surface pattern of the samples that underwent the demineralization and remineralization cycles also proved to be quite regular. Likewise, it was possible to note the presence of some porosities and small particles that seemed to protrude from the material (Figure 10). On comparing the two types of samples, it can be seen that in the samples submitted to cycling there no longer exists the smooth surface pattern shown by the sample that served as control. It can be noted that the porosities of the samples submitted to the cycles are more visible and marked, and their surface presents a more irregular pattern.

## DISCUSSION

The samples to be evaluated by scanning electronic microscope are submitted to adverse conditions inside the equipment, such as the high vacuum of the column, the high temperature resulting from heating of the tungsten filament to generate the current of primary electrons, and bombardment by the bundle of secondary electrons to produce an image. This may influence the evaluation of the sample, making small defects on the surface of the specimen to be evaluated more



**FIGURE 10** - Photomicrograph of a test sample of Heliomolar (10,000X).

pronounced. According to VIEIRA *et al.*<sup>9</sup> (1996), teeth or dental materials studied by this technique could carry with them defects originating in the very process of high drying and the original characteristics of the sample could be lost. This aspect is liable to be minimized when the evaluation is carried out on a more extensive surface and on more than one sample. Previous experiments<sup>6,9</sup> showed that using five samples for the purpose of control would be enough to describe the effects of the demineralization and remineralization cycles.

Glass-ionomer cement is an anionic polymer that possess groups of carboxylates connected by covalent links that are stable when attacked by acids. Only the crossed links are ionic, and some of them may break and cause the matrix of the material to decompose<sup>10</sup>. This is the mechanism probably related to the erosion presented by Chelon Fil and perhaps, in part, by Vitremer, since the latter possesses such a quantity of original components of glass-ionomer cement to allow the existence of an acid-base reaction while the material is hardening.

As far as erosion of composite resins is concerned, KUSY; LEINFELDER<sup>4</sup> (1977) suggested that a composite surface in contact with a hostile medium could present the loss of inorganic particles through cohesive flaws in the matrix. With the acid aggression, the weakest portion of the material — silane — would be degraded and this would lead to loss of charge particles and the appearance of depressions, which are of a progressive nature.



FAN; POWERS<sup>2</sup> (1980) added that this degradation of the surface is accompanied by an increase in imperfections and fractures and by reduced cohesion of the material. This mechanism might explain the degradation presented by Heliomolar, Dyract, VariGlass and Vitremer, because all of these materials are, at least, partially constituted of composites.

POWERS; FAN<sup>7</sup> (1980) demonstrated that in *in vitro* conditions of accelerated aging, the matrix of the composite resins suffers erosion and exposes the charge particles. Different materials presented different patterns of substance loss. This can be observed in the present study, where different characteristics of surface erosion of the materials tested were verified.

Chelon Fil, a glass-ionomer cement, presented a surface pattern of cracks and fissures that became intensified after fourteen days of demineralization and remineralization cycles. It was observed that the cracks and fissures became more marked and evident (Figures 1 and 2). Vitremer, a resin-modified glass-ionomer which accordingly contains a composite component in its formulation, also presented cracks after the period of cycling. Nonetheless, a large amount of small cracks was observed and the larger ones were presumably joining the porosities present after the material hardened. The particles that seemed to protrude from the matrix of the material, which is a characteristic of composite resins, were no longer visible after the cycles (Figures 3 and 4). The surface pattern presented by Chelon Fil after hardening should favor the highest rate of mechanical wear suffered by glass-ionomer cement in comparison with composites. The erosive action should only accelerate this process. Vitremer, by adding composite to its formulation, should present greater resistance to wear, and it may be suggested that VariGlass and Dyract, which possess an even greater proportion of composite, since they do not possess original constituents of glass-ionomer cement in sufficient quantities to produce an acid-base reaction, should theoretically present an even greater resistance to mechanical wear than Vitremer.

Upon evaluation of its surface after hardening, VariGlass, which is a liquid-powder system, demonstrated that its particles possess a variety of

sizes, reaching over thirteen micrometers (Figure 5). Some porosities are also observed, and after the cycles one sees that the surface was altered, for the particles present less prominent extremities (Figure 6). Unlike VariGlass, Dyract is a material that is presented as a mass ready for use, just like conventional composite systems such as Heliomolar. Its particles presented sizes far smaller than those of VariGlass (Figure 7). In Dyract, just as in Heliomolar, the porosities present in the control samples are intensified in the samples submitted to the demineralization and remineralization cycles (Figure 8). This study would not enable us to predict whether there is greater intensity of mechanical wear in VariGlass than in Dyract and in Heliomolar *in vivo*. The surface pattern of Dyract and Heliomolar after the period of cyclings presented less changes than that of VariGlass, and perhaps this was an indicator that VariGlass is less resistant to wear.

The materials under study, despite having different compositions, suffered the action of the medium and presented surface changes due to the environment of high cariogenic challenge. MODESTO *et al.*<sup>6</sup> (1996) claimed that the clinical indication of a material similar to those studied in this paper would depend on the need to use a more resistant material or one less susceptible to wear, but with cariostatic action.

Confronted with these arguments, a material with greater resistance and with a long-term cariostatic effect would seem to be the ideal. The polyacid-modified composites, which possess ionomeric glass particles but in quantities insufficient to produce an acid-base reaction, are promising materials in this sense. From now on, studies should be directed towards evaluating *in vitro* and *in vivo* the mechanical resistance of these materials and their demineralization-inhibiting potential by verifying their longevity and capacity to release and recharge fluoride.

## CONCLUSIONS

All of the materials studied underwent erosive action presenting different characteristics, due to their varied compositions, after being submitted for fourteen days to demineralization and remineralization cycles to simulate a high cariogenic challenge.

VIEIRA, A. R.; SOUZA, I. P. R.; MODESTO, A. Estudo da erosão superficial de materiais compósitos e ionoméricos submetidos à simulação de um alto desafio cariogênico. **Rev Odontol Univ São Paulo**, v. 13, n. 4, p. 321-327, out./dez. 1999.

O objetivo deste trabalho foi avaliar a superfície de compósitos e cimentos de ionômero de vidro, frente a uma situação de alto desafio cariogênico. A partir de setenta e cinco corpos-de-prova padronizados de um cimento de ionômero de vidro (Chelon Fil - ESPE), um ionômero de vidro resina-modificado (Vitremmer - 3M), dois compósitos poliácido-modificados (VariGlass e Dyract - Dentsply) e um compósito (Heliomolar - Vivadent), e após serem submetidos a quatorze dias a ciclagens de desmineralização e remineralização, para simular um alto desafio cariogênico, foram avaliados os aspectos erosivos da superfície dos materiais. Todos os corpos-de-prova foram avaliados ao microscópio eletrônico de varredura e comparados com cinco outros corpos-de-prova de cada material, confeccionados da mesma forma e que serviram como controle. Todos os materiais estudados sofreram ação erosiva dos meios, com características distintas, devido às suas diferentes composições, após serem submetidos à simulação *in vitro* de um alto desafio cariogênico.

UNITERMOS: Resinas compostas; Cimentos de ionômeros de vidro; Erosão.

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