

## **Microstructural analysis of restorative materials submitted to acid exposure**

### **Análise microestrutural de materiais restauradores submetidos à exposição ácida**

#### **Bruna Di Profio DAIBS**

DDS - School of Dentistry of São José dos Campos - UNESP - Univ Estadual Paulista - São José dos Campos - SP - Brazil.

#### **João Maurício Ferraz da SILVA**

PhD - Restorative Dentistry - School of Dentistry of São José dos Campos - UNESP - Univ Estadual Paulista - São José dos Campos - SP - Brazil.

#### **Daniel Maranha da ROCHA**

PhD - Restorative Dentistry - School of Dentistry of São José dos Campos - UNESP - Univ Estadual Paulista - São José dos Campos - SP - Brazil.

#### **Virgílio Vilas Boas FERNANDES JÚNIOR**

MsC - Restorative Dentistry - School of Dentistry of São José dos Campos - UNESP - Univ Estadual Paulista - São José dos Campos - SP - Brazil.

#### **José Roberto RODRIGUES**

Associate Professor - School of Dentistry of São José dos Campos - UNESP - Univ Estadual Paulista - São José dos Campos - SP - Brazil.

---

#### ABSTRACT

The study aimed to evaluate, through surface roughness and microhardness tests, the amount of damage caused by hydrochloric acid to restorative materials. Five different materials were used: direct composite resin (Z 350), indirect composite resin (Resilab Master), conventional glass-ionomer cement (GIC) (Vidrion R), resin modified GIC (Vitremer) and ceramic (Empress II). Twenty one specimens of each material were constructed and had their initial roughness and microhardness evaluated. Sixteen specimens of each material were immersed in a gastric fluid without enzymes simulating acid episodes. Other 5 specimens of each material were immersed in artificial saliva (control groups). After 7 days of immersion, the specimens had their surfaces evaluated again. Then, after another 21 days of immersion the specimens were submitted to a third mensuration. One specimen at each stage of the research was subjected to analysis in scanning electronic microscopy. The samples demonstrated changes in roughness and microhardness after the immersion of the restorative materials in the acid solution. The roughness results showed that for both glass ionomer cements, there was a significant difference between the first and last reading, with an increase in their roughness. In ceramic and direct resin materials, no significant difference among the periods was observed. Concerning to microhardness, the behavior of the materials showed a tendency towards decreasing hardness. The results showed the degradation of the restorative materials when exposed to acid episodes.

#### KEYWORDS

Restorative materials; acid erosion; roughness; microhardness.

---

## INTRODUCTION

The stress of the globalized society, the high competition, and the anxiety of the requirements related to the dictatorship of beauty created by this environment modulates in a certain way the daily routine. The demanding of stereotyped bodies often ignores the limit of health and imposes patterns which lead many people to develop psychosomatic disease as bulimia.

Bulimia nervosa occurs mainly in young women. It is a disease of emotional character in which the person misrepresents the body image, characterized by binge eating followed by purging, resulting in vomiting, excessive exercise and/or taking indiscriminately laxatives and diuretics [1]. Consequently, it leads to an almost constant homeostatic imbalance and to the triggering of several diseases associated to the immune and hormonal systems, as well as visible damage to the oral mucosa and tooth structure, such as: bilaterally swelling of the parotids, esophagus problems and peeling of oral mucosa [2]. Additionally, the fact that bulimic patients have both their salivary flow rate and buffer effect decreased makes that they be more prone to show dental erosion [3].

The dentist plays a fundamental role in the diagnosis of this type of disorders because they cause severe damages to the tooth structures and make the patients search for treatment. Therefore, the dentist is responsible for not only rehabilitating the patient undergoing tooth erosion, but also for diagnosing the type of disorder which is causing the damage and therefore referring the patient to treat the disorder [4].

Only after the adequate treatment of the disorder, the dentist is able of planning and performing the treatment of the tooth structure, that is, to rehabilitate the teeth damaged by acid erosion. High rates of wear provoke the loss of the anatomical shape due to the chemical attack of the oral environment added by the mechanical wear on the restoration, leading to the loss of interocclusal contact, migration of the antagonist tooth, alteration of the occlusal plane and masticatory cycle; also, the friction between the teeth may cause interproximal wear [4]. For the rehabilitation of erosive lesions, materials aiming to recover both the aesthetics and function of the tooth structure in a more conservative way are employed, changing the tooth shape, filling fractures, and restoring caries and erosion cavities [5].

Composite resins have been used because of their high aesthetic appeal which approximates the characteristics of color, texture, brightness,

fluorescence and translucency of the restoration to those of the natural tooth [6]. Spreafico [7] reported a clinical case of a bulimic patient rehabilitated by composite resin in which the biological, functional and aesthetic result was achieved efficiently in a short period of time.

Another material that can be used for the restoration of teeth with erosion is glass ionomer cements (GICs), which shows as their main property the fluoride releasing. When compared to composite resins, they present smaller resistance to wear, greater solubility, and slightly lower esthetic because of their opacity caused by the difference between the refraction index of their components and the tooth. Because of the biocompatibility, it decreases the caries formation as well as shows a coefficient of thermal volumetric alterations very similar to those of the tooth structure [6]. The use of composite resins and glass ionomer cements should be carefully indicated only in moderate cases in order to protect the tooth remnant against an eventual evolution of the lesions by continuing the episodes [8].

In some cases, tooth erosion damages the tooth structure at an extension that the aesthetics and function can only be obtained through the use of tooth prostheses, veneers, and resin or ceramic inlays/onlays or metal-ceramic crowns. The rehabilitation of these patients is indicated after the ceasing of the acid episodes. However, the bulimic patients often have relapses and start to provoke the vomits again, leading the oral environment, although rehabilitated, susceptible to the action of the acids. Additionally, several professionals, unfortunately, do not know yet the characteristics of the disease, therefore rehabilitating the patient without the referral to the specialist to treat bulimia.

Considering the aforementioned discussion, the aim of this study was to evaluate the damages caused by hydrochloric acid to restorative materials, simulating the continuity of the “acids exposures” in patient who already undergone the restorative/rehabilitative treatment of the acid erosion lesions.

## MATERIAL AND METHODS

This research employed an indirect resin (Resilab Master – Wilcos do Brasil Ind. E Com. Ltd.), a direct resin (Z 350 – 3M ESPE), resin-modified glass ionomer cement (Vitremmer – 3M ESPE), conventional glass ionomer cement (Vidrion R – SSWhite) and a ceramic (IPS- Empress 2 - Ivoclar-Vivadent) (Chart 1):

### Chart 1 - Experimental groups

Group IR	Resilab Master	Wilcos do Brasil Ind. E Com. Ltd.
Group DR	Z 350	3M ESPE
Group RMGIC	Vitremer	3M ESPE
Group GIC	Vidrion R	SSWhite
Group C	IPS- Empress 2	Ivoclar-Vivadent

Twenty-one specimens of each material were constructed with the aid of a metallic matrix, resulting in 6 mm diameter x 3 mm height cylinders. Considering the composite resins, they were inserted into the metallic matrix in two portions. Each portion was light-cured for 20 s, through a halogen light unit with power of 300mW/cm<sup>2</sup> (Curing Light XL 3000 – 3M Dental Products – St. Paul – USA). After the insertion and prior to the light-curing of the last portion, the composite resin was covered by a polyester strip and a glass coverslip was pushed onto it aiming to standardize the initial superficial smoothness of the specimens. To construct the conventional glass ionomer cement specimens, the material was mixed according to the manufacturer's instructions and the metallic matrix was completely filled with the aid of an insertion spatula (Duflex – SSWhite). Next, the material was covered by a polyester strip until it reached the final curing. For the resin-modified glass ionomer cement, the material was inserted similarly to the conventional GIC, covered by a polyester strip and then light-cured for 40s; to achieve the chemical curing of the material, the set matrix/material was left for 7 min.

The ceramic samples were constructed with IPS Empress 2 (Ivoclar-Vivadent – São Paulo - Brasil) manipulated according to the manufacturer's instructions.

After the construction of the samples of each material, we performed the first reading of the superficial roughness in a rugosimeter (Mitutoyo). Three readings were executed for each specimen, and then the mean value (Ra) was calculated and expressed in  $\mu\text{m}$ .

Vickers roughness was also measured through using a microhardner (FM 700, FutureTech Corp – Tokyo, Japan). Similarly, three readings were performed by using a load of 50 N for 10 s.

Additionally to the roughness and microhardness measurements, one specimen of each material was evaluated under scanning electronic microscopy (JSM – 5310 JEOL – Japan). The initial analyses

were employed to obtain an initial view of the surface. Each group comprised 21 samples: 15 were analysed in rugosimeter and microhardner at each phase of the research, 1 sample was sent to scanning electronic microscopy. Additionally, other 5 samples comprised the control group. After the initial analysis, the samples undergone one cycle of immersion into the acid substance (simulated gastric acid without enzymes) (Biofórmula – São José dos Campos - SP) for 5 min and more 23h and 55 min into artificial saliva, comprising therefore the 24h cycle. The control groups of each material composed of 5 samples were kept into artificial saliva during all the experiment. After seven days of cycle, the samples had their surfaces evaluated once more regarding to roughness, microhardness, and SEM. After this second reading, the samples were kept for more two weeks into the immersion cycles until one month of acid episodes was reached. After that period, the third reading was accomplished as previously described.

### RESULTS

Mean and standard deviation values of roughness and microhardness related to the five materials studied and the different periods of exposure to the acids were obtained.

T Student test was applied for the comparison among the experimental and control groups for each material at the different periods of exposure to acids. Generally, a tendency towards the increasing of the roughness and decreasing of microhardness was verified, although without statistically significant differences.

ANOVA was applied to verify a possible difference among the five materials studied. There was statistically significant difference in both the roughness and microhardness among the materials ( $p < 0.05$ ).

Then, Tukey test was applied (level of significance of 5%) at the different periods to identify where the difference had occurred.

By observing the graphs below (Figures 1 and 2), it can be seen that the ceramics, as time went by, was the material which had the smallest alteration in relation to the initial period. The glass ionomer cements presented the most relevant structural alterations, both for roughness and microhardness. The indirect and direct composite resins showed certain stability, however, with a structural variation greater than that of the ceramics, after the acids episodes.

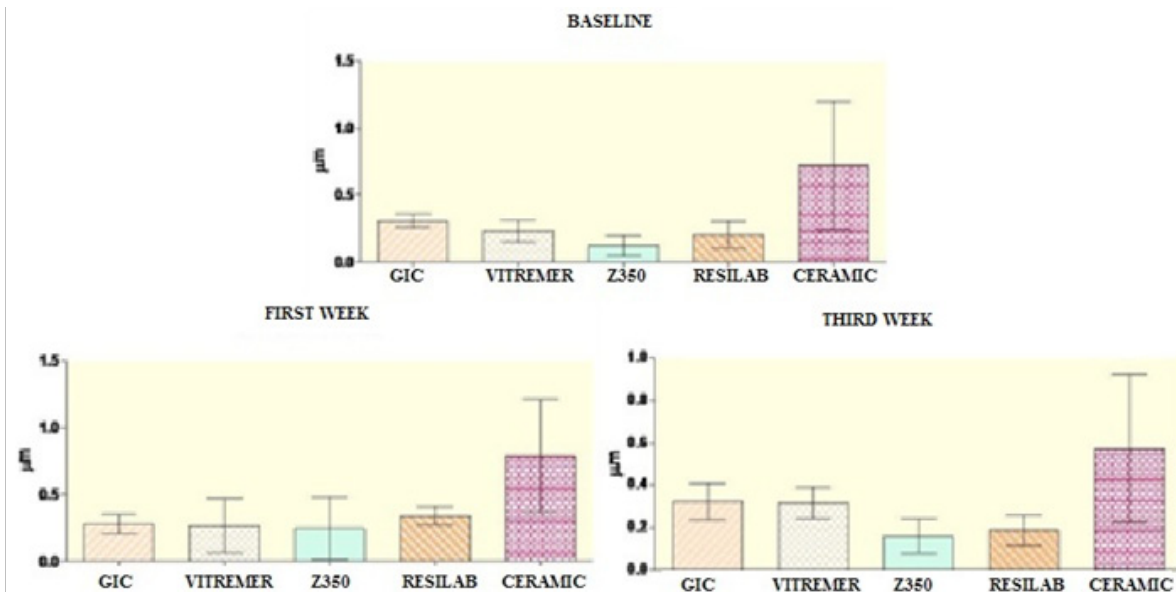


Figure 1 – Column graphs of the superficial roughness of the materials tested: initial, second and third readings. (Different letters indicate statistic difference).

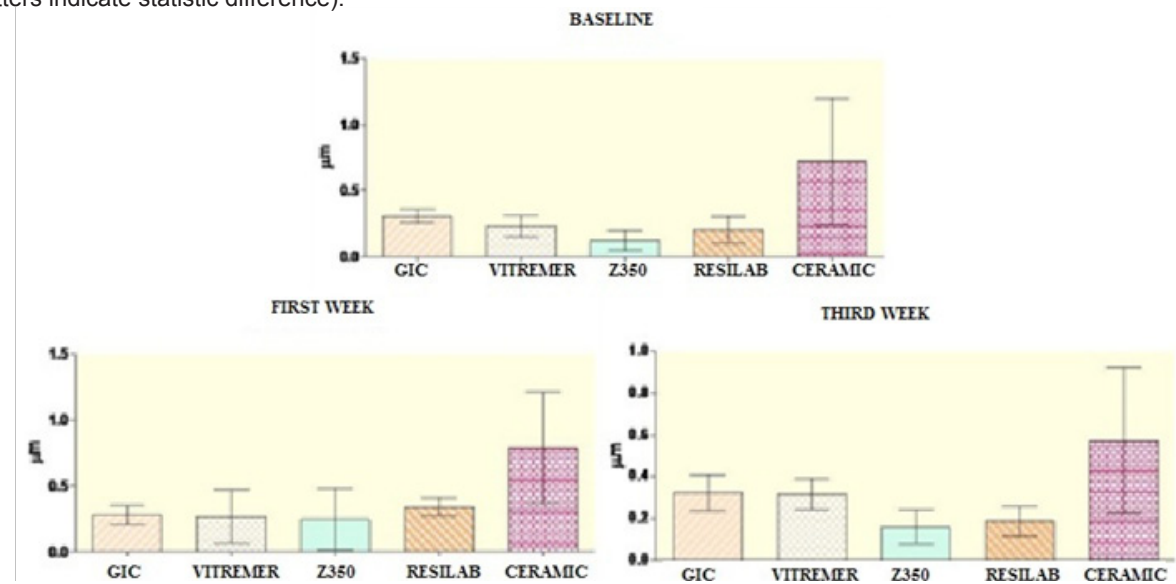


Figure 2 - Column graphs of the microhardness of the materials tested: initial, second and third readings. (Different letters indicate statistic difference).

For illustration, SEM images were performed for all materials tested at the different phases of evaluation of the samples (initial, 7 days and 30 days), at x 10,000 magnification, where the microstructure of the materials can be seen with more details (Figures from 3 to 7).

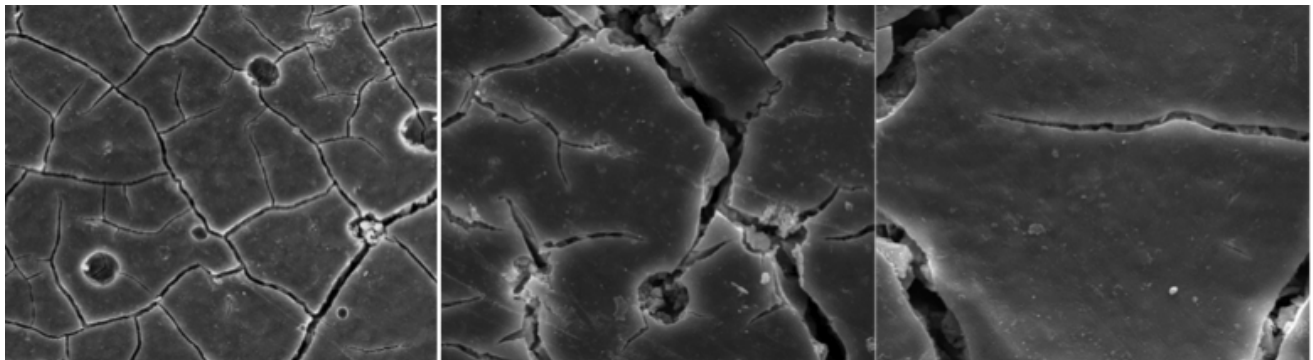


Figure 3 – Photomicrography - Glass Ionomer Cement (Vidron): a) initial reading; b) second reading; c) third reading.

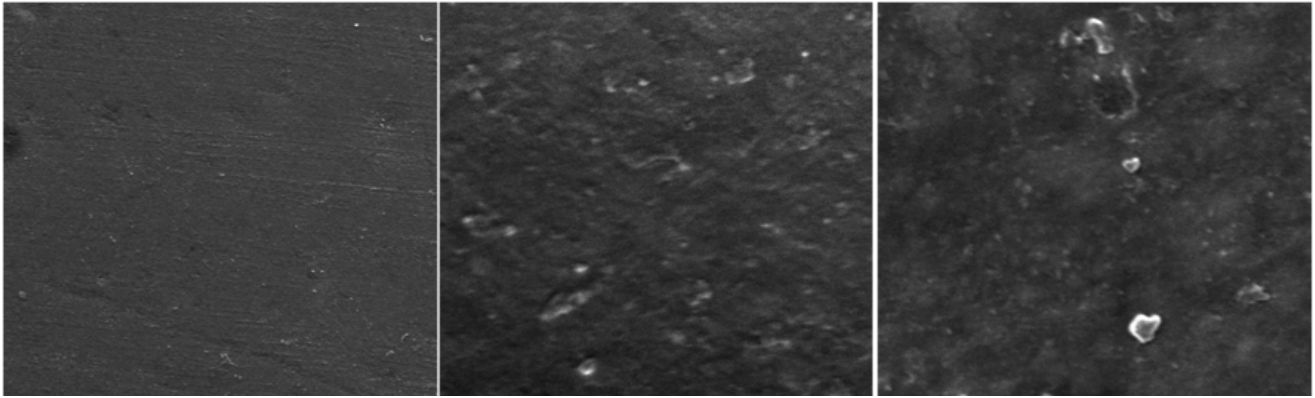


Figure 4 – Photomicrography - Direct Resin (Z350): a) initial reading; b) second reading; c) third reading.

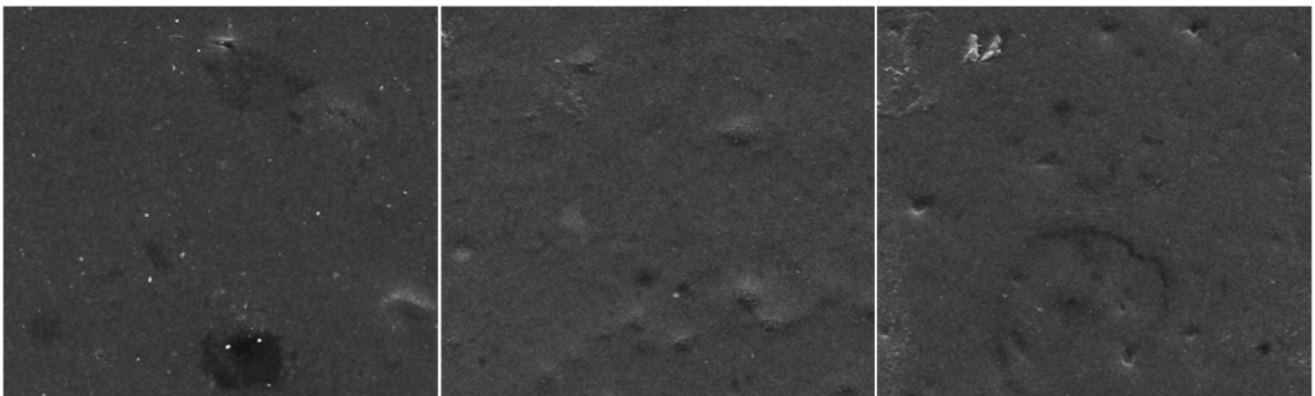


Figure 5 – Photomicrography - Indirect Resin (Resilab): a) initial reading; b) second reading; c) third reading.

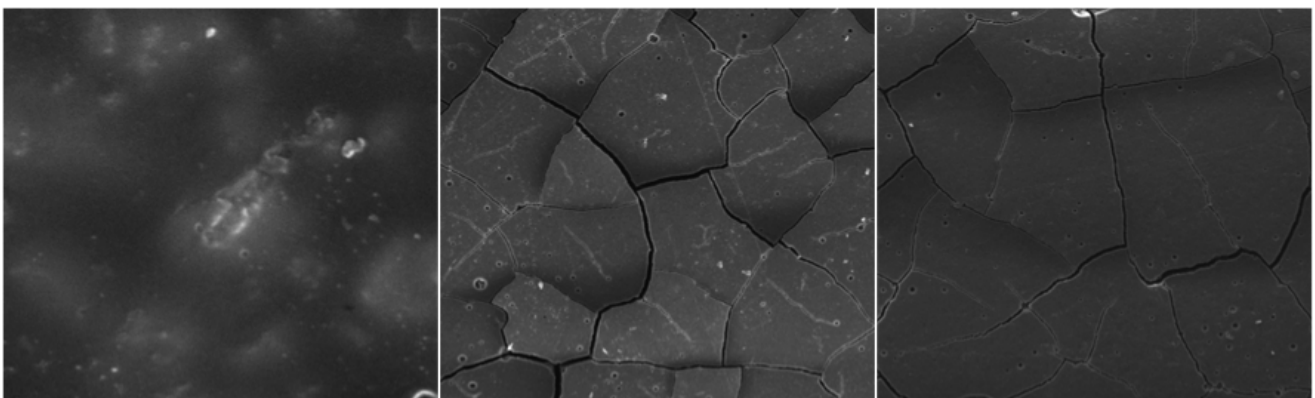


Figure 6 – Photomicrography - Resin-modified Glass Ionomer Cement (Vitremer): a) initial reading; b) second reading; c) third reading.

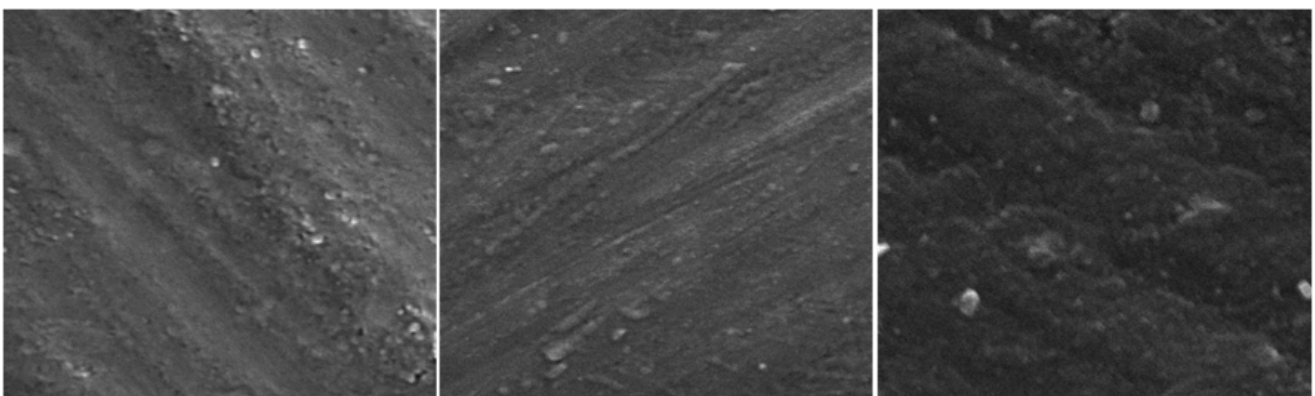


Figure 7 - Photomicrography Ceramic (Empress II): a) initial reading b) second reading; c) third reading.

The images obtained through scanning electronic microscopy allowed to visualize in the conventional GIC group that the acid episodes caused cracks in the microstructure of this material, which increased in number and size with the following episodes. Similar images were obtained for RMGIC group, however, this material showed a smaller amount of cracks, which were only more evident after one month of immersion (Figures 4 and 5).

The direct and indirect resins showed similar results regarding to degradation, which was proved by the images showing, as time went by, a degradation of the polymeric matrix with consequent increasing of roughness and decreasing of microhardness (Figures 6 and 7).

The images of the ceramic showed that this material, although suffering a degradation as the immersion time went by, underwent a smaller degradation than the other materials. It can be seen in the images the maintenance of the surface during the first two readings, and the presence of bubbles and grooves on the surface of the material, suggesting a slight degradation.

## DISCUSSION

The technology of new materials is in constantly evolution, which is very evident in Dentistry, where we observe the appearance of aesthetic restorative materials, such as the composite resins, the ceramics, among others. This technology is helping us to solve problems within a society with very aesthetic requirements.

However, it is worth highlighting that similarly to the tooth structure, mostly the aesthetic restorative materials are submitted to the constant contact with liquids of low pH, which is the case of the hydrochloric acid coming from the stomach juice [6,9,11-13]. The degradation varies according to the concentration, exposure time, and flow of the acid fluids [14].

By analyzing the results obtained in this research, it was observed the increasing of the superficial roughness, that is, the reduction in the smoothness of the materials studied. Sadaguiani et al. [15] found numbers that emphasize the findings of our study, by studying the erosion of compomers and Vitremer after the exposure of these materials to the acidity of alcoholic mouthrinses. They reported and expressive increasing in the roughness of Vitremer in the first four weeks of acid challenge, endorsing the findings of the authors who expressed this increasing in wear during the initial periods of exposure. The alcoholic acidic mouthrinses demonstrated alteration in the

roughness of all samples tested [16]. In this present study, Vitremer and Vidrion demonstrated results considered homogenous within the different groups analyzed. RMGIC (Vitremer) showed statistically significant alterations in comparison with the other materials and control group, mainly at the third week of cycling and readings in rugosimeter. Vidrion, although presenting a result similar to Vitremer did not show statistically different alterations in comparison with other materials and control group. The erosion in GIC materials is explained by the leaching phenomenon, in which the acid consumes the material's particles and the chemically linked molecules are lost instead of reconstituting the ideal initial linkages; this results in their releasing in the medium, exposing the new surface of the material which successively is submitted to the process again, suggesting that the addition of the attrition factor to the erosion phenomenon potentializes the increasing of the superficial roughness [15].

Some authors demonstrated alterations in roughness after the immersion of restorative materials in fluids, even in distilled water. Notwithstanding, these alterations are more evident when submitted to low pH media, such those of acid drugs. Acid conditions cause the degradation of the restorative materials, therefore reducing their physical and chemical properties and additionally creating sites for bacterial colonization and increasing the risks of oral diseases, which is agreement with our findings [17-21]. In this study, the samples were also assessed by SEM and it was found that the resins showed superficial alterations because of the degradation of the polymeric matrix, once several empty spaces and cracks were observed in the specimens [22]. The direct and indirect resins showed similar behaviors (increasing in roughness and decreasing in the microhardness), but the indirect resin presented significant results. The reduction in microhardness after acid immersion is because of the hydrolysis of the ester moiety of the monomer (dimethyl methacrylate), that is, Bis GMA, Bis EMA, UDMA and TEGMA [23]

Wan Bakar et al. [13] through using different types of acids (hydrochloric, citric and orthophosphoric) conducted an acid challenge in aesthetic restoration (GIC, RMGIC, CR and porcelain) and observed that the greatest effects regarding to erosion went from citric and hydrochloric acids, even in short cycles of only two hours on GIC and RMGIC. These results are similar to the roughness and microhardness findings of this present study. The research of this authors revealed that the marginal degradation was very high in these materials and the surrounding tooth structure

suffered greater damage in relation to both the enamel far from them and the CR and the porcelain. The level of wear on the CR and the ceramic was smaller than that of GIC and RMGIC. It is known that the results found in vivo are different from those found in vitro, because of the number of variables, such as acquired pellicle, salivary flow and composition, constitution and thickness of the bacterial plaque, acid diet, and endogenous gastric disturbances. The greatest susceptibility to degradation and dissolution of glass ionomer cements validates the use of the technique, so-called sandwich technique, where the GIC is protected from the oral cavity by some resin material.

This present study demonstrated reduced microhardness levels for all aesthetic restorative materials analyzed after the acid challenge, at different periods. Other studies reached the same results, such that of Attin et al. [24] who aimed to observe the dissolution of GIC, RMGIC, and CR after exposure to acid beverages as soft drinks, orange and apple juices, on dentin and enamel, concluding that mainly GIC and RMGIC presented a greater reduction in microhardness than composite resin, corroborating our study. Other authors [9] also demonstrated the damage caused by acid soft drinks proving the reduction in hardness of the samples submitted to acid challenges. When RMGIC was immersed in neutral medium, 0.9% NaCl for one month, 6 months and one year, there was an increasing in hardness by the adsorption of the liquids. On the other hand, when it was submitted to Coke®, during the first month of immersion, there was a slight increasing in microhardness followed by a marked reduction after

one year. GIC did not undergo dissolution during the period of one year immersed in Coke®, however, it presented total dissolution when submitted to immersion in fruit juice. Conventional CIC presented results similar to those of RMGIC except that the latter did not undergo very significant alterations in fruit juice immersion. Composite resin did not dissolve in fruit juice. During periods very short, for example 10 days, the authors did not obtained statistically significant differences when comparing to distilled water, Coke®, orange and apple juice. This factor explains some results of this present study, which were different from what was expected. As it is evidenced, there is not a standard protocol for some studies, for example, acid challenges for tooth erosion.

We verified that there is a tendency towards degradation of the restorative materials when submitted to acid exposure. Such fact increases the responsibility of the dentist by both indicating the material to be used in rehabilitation and knowing the exact moment to treat the patient undergoing acid episodes.

Considering the materials analyzed, all experienced a certain degree of degradation after the acid episodes, although in different levels. By analyzing the results, it can be concluded that the ceramics are the material most indicated for the rehabilitation of patients undergoing acid episodes, followed by the composite resins and glass ionomer cements. Notwithstanding, we emphasize that all materials present their indication and regardless of which material will be employed, the most important is to stop the acid episodes to enable that the materials reach their maximum actuation capacity within oral rehabilitation.

---

## RESUMO

O estudo teve como objetivo avaliar por meio dos testes de rugosidade superficial e microdureza os danos causados pelo ácido clorídrico em materiais restauradores. Utilizaram-se 5 materiais: resina composta direta (Z 350), resina composta indireta (Resilab Master), cimento de ionômero de vidro (CIV) convencional (Vidrión R), CIV modificado por resina (Vitremer) e uma cerâmica (Empress II). Vinte e um corpos de prova (cdp) de cada material foram confeccionados e tiveram suas superfícies iniciais avaliadas em um rugosímetro e um microdurômetro. Em seguida 16 cdp de cada material foram imersos em um suco gástrico sem enzimas simulando episódios ácidos. Outros 5 cdp de cada material serviram como grupo controle e ficaram imersos em saliva artificial. Após 7 dias de imersão os cdp tiveram suas superfícies novamente avaliadas. Após a segunda leitura, foi realizada imersão por mais 21 dias, completando um mês de experimento, e os cdp foram submetidos à terceira leitura de suas superfícies. Além disso, 1 cdp em cada fase da pesquisa foi submetido a uma análise em microscopia eletrônica de varredura (MEV) apenas como efeito ilustrativo. Amostras demonstraram alterações de rugosidade e microdureza diante da imersão de materiais restauradores em ácido. Os resultados de rugosidade mostraram que tanto para o ionômero convencional quanto para o modificado por resina houve uma diferença significante entre a primeira e a última leitura, havendo um aumento na rugosidade. Já para a cerâmica e a resina direta, não houve alteração significativa entre os períodos. O comportamento dos materiais mostrou uma tendência de diminuição da microdureza. Conclui-se que existe uma deterioração dos materiais restauradores frente a episódios ácidos.

## PALAVRAS-CHAVE

Materiais restauradores; erosão ácida; rugosidade; microdureza.

---

## REFERENCES

1. Kell PK, Haedt A. Evidence-based psychosocial treatments for eating problems and eating disorders. *J Clin Child Adolesc Psychol.* 2008;37(1):39-61.
2. Traebert J, Moreira EAM. Behavioral eating disorders and their effects on oral health in adolescence. *Pesqui Odontolo Bras.* 2001;15(4):359-63.
3. Filipi K, Halackova Z, Filipi V. Oral health status, salivary factors and microbial analysis in patients with active gastroesophageal reflux disease. *Int Dent J.* 2011;61(4):231-7.
4. Garone Filho W, Abreu e Silva V. Lesões não cariosas: o novo desafio da Odontologia. São Paulo: Santos; 2008.
5. Dugmore CR, Rock WP. A multifactorial analysis of factors associated with dental erosion. *Br Dent J.* 2004;196(5):283-6.
6. Anusavice, KJ. *Materiais dentários.* 10.ed. São Paulo: Guanabara Koogan; 2002.
7. Spreafico RC. Composite resin rehabilitation of eroded dentition in a bulimic patient: a case report. *Eur J Esthet Dent.* 2010;5(1):28-48.
8. Dietschi D, Argente A. A comprehensive and conservative approach for the restoration of abrasion and erosion. Part I: concepts and clinical rationale for early intervention using adhesive techniques. *Eur J Esthet Dent.* 2011;6(1):20-33.
9. Van Rooke NB, DDS, MSD. Gastroesophageal reflux disease, tooth erosion, and prosthodontic rehabilitation: a clinical report. *J Prosthodont* 2003;12(4):255-9.
10. Aliping-Mckenzie M, Linden RWA, Nilcholson JW. The effect of Coca-Cola and fruit juices on the surface hardness of glass-ionomers and 'compomers'. *J Oral Rehabil.* 2004;31(11):1046-52.
11. Czarnecka B, Nilcholson JW. Ion release by resin-modified glass-ionomer cements into water and lactic acid solutions. *J Dent.* 2006;34(8):539-43.
12. Wens BM, Kitchens M. The erosive potential of soft drinks on enamel surface substrate: an in vitro scanning electron microscopy investigation. *J Contemp Dent Pract.* 2007;8(7):11-20.
13. Wan Bakar WZ, McIntyre J. Susceptibility of selected tooth-colored dental materials to damage by common erosive acids. *Aust Dent J.* 2008;53(3):226-34.
14. Shellis RP, Finke M, Eisenburger M, Parker DM, Addy M. Relationship between enamel erosion and liquid flow rate. *Eur J Oral Sci.* 2005;113(3):232-38.
15. Sadaguiani L, Wilson MA, Wilson NHF. Effect of selected mouthwashes on the surface roughness of resin modified glass-ionomer restorative materials. *Dent Mater.* 2007;23(3):325-34.
16. El Badrawy WA, McComb D, Wood RE. Effect of home use fluoride gels on glass-ionomer and composite restorations. *Dent Mater.* 1993;9(1):63-7.
17. Gao F, Matsuya S, Ohta M, Zhang J. Erosion process of light-cured and conventional glass-ionomer cements in citrate buffer solutions. *Dent Mater J.* 1997;16(2):170-9.
18. Jaeggi T, Gruninger A, Lussi A. Dental erosion. *Monogr Sci.* 2006;20:200-14.
19. Nicholson JW, Millar BJ, Czarnecka H, Limanoska-Shaw H. Storage of polyacid-modified resin composites ("compomers") in lactic acid solution. *Dent Mater.* 1999;15(6):413-6.
20. Sidhu SK, Sherriff M, Watson TF. In vivo changes in roughness of resin-modified glass ionomer materials. *Dent Mater.* 1997;13(3):208-13.
21. Silva RC, Zuannon ACC. Surface roughness of glass ionomer cements indicated for atraumatic restorative treatment. *Braz Dent J.* 2006;17(2):106-9.
22. Valinotti AC, Neves BG, Silva EM, Maia LC. Surface degradation of composite resin by acid medicines and pH-cycling. *J Appl Oral Sci.* 2008;16(4):257-65.
23. Wonghantee S, Patanapiradej V, Maneenut C, Tantbijon D. Effect of acidic food and drinks on surface hardness of enamel, dentine, and tooth-colored filling materials. *J Dent* 2006;34(3):214-20.
24. Attin T, Meyer K, Hellwig E, Buchalla W, Lennon AM. Effect of mineral supplements to citric acid on enamel erosion. *Arch Oral Biol.* 2003;48(11): 753-9.

Received: 10/06/2011

Accepted: 12/09/2011

Corresponding Author:

Bruna Di Profio Daibs

Rua Tutóia 587, apto 11

Vila Mariana - CEP 04007-003

São Paulo – SP – Brasil

bdiprofio@gmail.com