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ORIGINAL | *ORIGINAL*

Water sorption and solubility of glass ionomer cements indicated for atraumatic restorative treatment considering the time and the pH of the storage solution

Sorção de água e solubilidade de cimentos de ionômero de vidro indicados para tratamento restaurador atraumático em função do tempo e do pH da solução de armazenagem

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ABSTRACT

Objective
Evaluate the water sorption and the solubility of glass ionomer cements considering the time and the pH of the storage solution.

Methods
The materials used in this survey study were the following ones: Ketac Molar Easymix, Maxxion R, Vitro Molar, Vitremer and Vitro Fil LC. Fifteen specimens of each material were fabricated and subdivided into the storage solutions (deionized water, acid artificial saliva and neutral artificial saliva), having the mass measured in 24 hours, 7, 14 and 21 days. Water sorption and solubility values (µg/mm³) were obtained and submitted to the analysis of variance (ANOVA) and the Tukey test (p <0.05).

Results
The water sorption values were statistically different for all the studied materials and solutions in each storage period, except for the Maxxion R. Considering the solubility, all the glass ionomer cements presented values that were not statistically different when evaluating the storage solutions, except for the Vitro Fill LC.

Conclusion
The water sorption and the solubility of the studied glass ionomer cements were not influenced by the various storage solutions.

Indexing terms: *Glass ionomer cements. Hydrogen ion concentration. Solubility.*

RESUMO

Objetivo
Avaliar a sorção de água e a solubilidade de cimentos de ionômero de vidro em função do tempo e do pH da solução de armazenagem.

Métodos
Os materiais utilizados foram: Ketac Molar Easymix, Maxxion R, Vitro Molar, Vitremer e Vitro Fil LC. Foram confeccionados 15 espécimes de cada material que foram subdivididos nas soluções de armazenamento (água deionizada, saliva artificial ácida e saliva artificial neutra) e tiveram suas massas mensuradas em 24 horas, 7, 14 e 21 dias. Os valores da sorção de água e solubilidade (µg/mm3) foram obtidos e submetidos à análise de variância (ANOVA) e ao teste de Tukey (p<0,05).

Resultados
Os valores de sorção de água diferiram estatisticamente para cada período de armazenamento para todos os materiais e soluções estudadas, exceto para o Maxxion R. Em relação à solubilidade, todos os cimentos de ionômero de vidro apresentaram valores que não diferiram estatisticamente comparando as soluções de armazenamento, exceto o Vitro Fill LC.

Conclusão
A sorção de água e a solubilidade dos cimentos de ionômero de vidro estudados não sofreram influência dos diferentes meios de armazenagem.

Termos de indexação: Cimentos de Ionômeros de Vidro. Concentração de íons de hidrogênio. Solubilidade.

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INTRODUCTION

Atraumatic Restorative Treatment (ART), a technique developed in Tanzania in the mid 1980's, as part of an oral care program, is of great importance, as it is a low-cost easy treatment option to assist the low-income population[1].

Glass ionomer cements (GICs) are considered the most appropriate restorative material for ART due to their biological, physical and chemical properties.[1] GICs feature recharging and fluoride release properties, adhesion to tooth tissue, coefficient of thermal expansion and biocompatibility[2-4]. High viscosity GICs were specially developed to be used with this technique. These cements were manufactured by removing calcium ions excess from glass particles, reducing the particles size and increasing the powder to liquid ratio[5]. Such materials present a longer settling time and better physic-mechanical properties when compared to previous cements[1,6], resulting in higher survival rate of the restorations[7].

The clinical success of the ARTs for restorations depends on the shelf-life of the restorative material. One of the most important properties that determines the lifetime of restorative materials is resistance to dissolution or disintegration. The water sorption properties and the solubility of the cement change the mechanical characteristics of the material by directly interfering in the half-life of the restorations[8]. Water sorption changes the mechanical properties through two effects: lamination and degradation. The solubilization of the restorative material causes loss of material mass, adversely affecting its mechanical properties, as well as causing tooth/restoration interface failure, increasing the risk of marginal microleakage and leading to restoration failure[9-10].

The oral cavity is a complex environment that may influence the properties of the restorative materials. *In vitro* studies use artificial water or saliva as a storage solution, trying to simulate the condition of the oral cavity. Therefore, it is necessary to understand the influence of the storage solution in the GIC properties along the time, to better understand the behavior of the restorative material in the oral cavity to predict its performance. Thus, this research study aims at evaluating the water sorption and the solubility of glass ionomer cements indicated for ART considering the time and the pH of the storage solution.

The null hypotheses for this study were the following ones: 1) The water sorption and the solubility of the glass ionomer cements would not be influenced by the immersion in various solutions (deionized water, acid saliva and neutral saliva); 2) There would be no difference considering the water sorption and the solubility for the glass ionomer cements after the immersion in various solutions (deionized water, acid saliva and neutral saliva); 3) There would be no difference among the various solutions (deionized water, acid saliva and neutral saliva), considering the water sorption and the solubility values of the glass ionomer cements.

Chart 1. Materials, classification, manufacturer and composition description.

Material/ Lot	Manufacturer	Particle Composition	Liquid Composition	Classification
Ketac Molar Easymix Lot: 308161	3M ESPE	Aluminum fluorosilicate glass, lanthanum and calcium, polyacrylic acid, eudragit, tartaric acid, sorbic acid, benzoic acid and pigments	Water, copolymer of acrylic acid and maleic acid, tartaric acid and benzoic acid	Conventional GIC
Maxxion R Loe:150108	FGM	Aluminum fluorosilicate glass, polycarboxylic acid, calcium fluoride and water	Polyacrylic acid	Conventional GIC
Vitremer Lot:0806000262	3M ESPE	Fluoroaluminosilicate crystals, potassium persulfate, ascorbic acid and pigments	Polyalkanoic acid, methacrylate groups, water, HEMA, camphorquinone	Resin-Modified GIC
Vitro Fil LC Lot:07101276	DFL	Strontium aluminum silicate, activators and iron oxide	2-hydroxyethyl methacrylate, aqueous solution of polyacrylic and tartaric acids, benzoyl peroxide and camphorquinone	Resin-Modified GIC
Vitro Molar Lot:08040461	DFL	Barium and aluminum silicate, dehydrated polyacrylic acid and iron oxide	Polyacrylic acid, tartaric acid and distilled water	Conventional GIC

METHODS

The materials used in this study, along with their composition and manufacturers, are shown in the Chart 1.

To prepare the specimens, the materials were handled following the manufacturers' recommendations, with temperature at 26 ± 1° C. Before handling the materials, they were weighed on an analytical balance to standardize the powder/liquid proportion. Soon after, the cements were placed in a circular mold: 10.0mm diameter and 3.0mm thickness Teflon. Resin-modified glass ionomer cements were light-cured using a Flash Lite 1401 light-curing unit, with light intensity at 400 mW/cm^2 for 40 seconds. Seventy-five specimens were prepared and distributed in 5 groups, according to the material applied. Thus, 15 specimens were prepared and subdivided according to the storage solution: deionized water, acid artificial saliva and neutral artificial saliva (n = 5).

All specimens were weighed on a precision analytical balance (AG 200 GEHAKA) and the weight recorded. Subsequently, they were placed in a heater at 37°C, 100% humidity, aiming at reaching a constant mass (m1), with a maximum weight variation of ± 0.0005g. Then, the specimens were immersed in the storage solution and weighed 24 hours, 7, 14 and 21 days after immersion (m2).

During the experiment, the specimens were stored in polystyrene plastic recipients. Each recipient received 5ml of the storage solution, which was changed, daily, in the same time. The plastic recipients, containing the specimens immersed in the storage solution, were kept in a heater at 37°C, during the entire period of the experiment.

After the storage period (21 days), the specimens were removed from the recipients, weighed on an analytical balance and maintained in a desiccator, containing dehydrated silica gel to reach a constant weight (m3). Diameters were measure in four points and a caliper rule was used to measure thickness. The data collected was used to calculate the volume (mm^3).

The water sorption was determined by the equation m2 - m3 / Volume. And for solubility, the equation m1 - m3 /V was applied.

The water sorption and the solubility values expressed in µg/mm^3, in deionized water, acidic and neutral saliva were, respectively, submitted to the analysis of variance (ANOVA) and the Tukey's test using the statistical software SPSS, version 10.0, and significance level at 5%.

RESULTS

The results achieved in this research study showed that the Maxxion R glass ionomer cement (FGM) presented statistically higher water sorption values when compared to other cements in all the studied conditions, except for neutral saliva in the 24-hour period, in which the Maxxion R did not statistically differ from the Vitro Molar. On the other hand, the Ketac Molar EasyMix glass ionomer cement presented the lowest water sorption and solubility values for the great majority of the studied solutions and storage periods (Table 1, 2, 3 and 4). Regarding the storage period, the water sorption values of the Maxxion R glass ionomer cement did not statistically differ in the periods of 24 hours, 7, 14 and 21 days, when evaluating all the studied solutions. For the additional materials, such values presented statistically significant differences in each one of the storage periods (Table 1, 2 and 3). In relation to the solubility, every glass ionomer cement presented values that did not present statistically differences when evaluating the storage solutions, except the Vitro Fill LC that presented statistically lower solubility values in neutral saliva when compared to other solutions (table 4).

Table 1. Water sorption of the materials (µg / mm^3) in acid saliva in the different periods.

	Period			
Material	24 hours	7 days	14 days	21 days
Ketac Molar Easymix	63.50 aA	83.32 aB	90.39 aB	94.06 aB
Vitro Fill LC	77.08 abA	77,60 aA	83.67 aB	82.09 aAB
Vitremer	88.37 bcA	101.66 bAB	113.38 bB	114.41 bB
Vitro Molar	105.73 cA	102.70 bA	109.74 bA	111.09 bA
Maxxion R	156.59 dA	153.30 cA	146.72 cA	148.02 cA

Note: Mean values followed by the same lowercase letter, in the columns, and the same capital letter, in the rows, do not present statistically significant differences by the Tukey test at 5% probability.

Table 2. Water sorption of the materials (µg / mm^3) in neutral saliva in the different periods.

	Period			
Material	24 hours	7 days	14 days	21 days
Ketac Molar Easymix	71.71 aA	89.56 abB	103,62 abBC	105.86 bC
Vitro Fill LC	73.78 aA	111.89 cB	136.59 cdBC	141.19 cdC
Vitremer	77.76 abA	79.67 aA	85.33 aA	79.69 aA
Vitro Molar	108.01 bcA	106.65 bcA	122.41 bcA	128.24 cA
Maxxion R	126.45 cA	145.54 dA	148.78 dA	151.71 dA

Note: Mean values followed by the same lowercase letter, in the columns, and the same capital letter, in the rows, do not present statistically significant differences by the Tukey test at 5% probability.

Table 3. Water sorption of the materials (μg / mm³) in deionized water in the different periods.

	Period			
Material	24 hours	7 days	14 days	21 days
Ketac Molar Easymix	80.56 aA	81.60 aA	83.80 aA	81.87 aA
Vitro Fill LC	88.89 aA	100.81 bB	105.88 bA	106.09 bA
Vitremer	90.93 aA	114.58 bcB	123.83 cB	123.57 cB
Vitro Molar	95.64 aA	122.69 cB	131.46 cC	128.09 cC
Maxxion R	158.04 bA	143.76 dA	145.80 dA	140.80 dA

Note: Mean values followed by the same lowercase letter, in the columns, and the same capital letter, in the rows, do not present statistically significant differences by the Tukey test at 5% probability.

Table 4. Solubility (μg/mm³) of each material according to the storage solution.

	Solution		
Material	Acid Saliva	Neutral Saliva	Deionized Water
Ketac Molar Easymix	37.9670 aA	37.9929 aA	36.7565 aA
Vitro Fill LC	67.2524 abA	52.6918 abB	67.5708 bA
Vitremer	68.3019 abA	69.3109 abcA	72.5587 bA
Vitro Molar	90.9312 bA	80.0308 bcA	65.9717 bA
Maxxion R	106.4730 bA	95.4704 cA	115.2089 cA

Note: Mean values followed by the same lowercase letter, in the columns, and the same capital letter, in the rows, do not present statistically significant differences by the Tukey test at 5% probability.

DISCUSSION

The chemical composition of the material plays an important role in its physicochemical properties. Considering sorption and solubility, the differences in the composition of the GICs determined the behavior of the materials which were evaluated in this research study, when stored in various solutions (neutral saliva, acid saliva and distilled water). The particle size exposed to water, the presence of pores, the conversion level, the polymerization initiation system (chemical or physical) and the chain density of the materials play an important role in this process[12-13].

The conventional glass ionomer cement Maxxion R (FGM) showed the highest water sorption and solubility values in all the solutions, statistically differing from the evaluated materials. Thus, the first and the second null hypotheses were rejected. This occurred due to the composition of the cement that presented calcium fluoride. On the other hand, the glass ionomer cements from the Ketac Molar Easymix (conventional) and the Vitro Fill LC (resin) trademarks were less sensitive to water sorption, which can be explained by the large number of carboxylic acid groups in the liquid of such cements. Both present tartaric acid in their composition, a dicarboxylic acid with two carboxyl radicals. Therefore, a large number of crosslinking is established between the polymer chains, reducing the empty spaces and, thus, the water inflow into the material[14].

The Vitro Molar (DFL) also presents tartaric acid in its composition, however, it did not present satisfactory behavior for water sorption and solubility throughout the storage period. This is probably due to the composition of the conventional cements which presents barium silicate glass and aluminum. The glass ionomer cements prepared with these glass types exhibit lower hydrolytic stability when compared to aluminum silicate glass[14].

The water sorption rate in the polymer chain is mainly controlled by two factors: the polarity of the resin, which is given by the concentration of hydrogen bond forming niches with water; and the topography of the polymer chain, related to the cohesive energy density of the polymer network[15]. In this study, the Vitro Fill LC showed lower sorption values with statistically significant difference when compared to the Vitremer after the storage periods of 7, 14 and 21 days. The Vitro Fill LC presents aluminum strontium glass and inorganic fillers in its composition and, in the liquid, aqueous solution of polycarboxylic acid and HEMA. The Vitremer presents aluminosilicate glass powder and, in the liquid, the aqueous solution of polycarboxylic acid, with methacrylate and HEMA. Probably, the initial sorption behavior of the Vitro Fill LC, in 24 hours of immersion in water, is due to the presence of tartaric acid in its composition. However, although the water diffusion sorption mechanisms were smaller, very probable due to the earlier presence of a dense polymer matrix, the water stability did not increase. This fact can be verified by the result of increasing solubility for this material in the period evaluated, probably due to the hydrolytic degradation on its surface. The presence of strontium in the Vitro Fill LC composition may be the explanation, as strontium reacts rapidly with water[14].

Per the results achieved in this research study, there was no influence of the storage solution in the water sorption and the solubility of the studied glass ionomer cements, except for the Ketac Molar Easymix and the Vitremer, which absorbed more water when stored in deionized water. Thus, the third null hypothesis was rejected. This is probably due to some chemical changes in such materials, since both materials are from the same manufacturer (3M/ESPE). This result corroborates the findings of additional studies[16]. In theory, polymers immersed in distilled water should absorb more water than those in artificial saliva due to osmotic pressure[17].

In this research sttudy, no relationship was found considering the type of cements, conventional or resin-modified cements, when evaluating the water sorption and the solubility properties, corroborating the results of additional studies[18]. However, previous studies showed lower water sorption rate for resin-modified GICs when compared to conventional GICs[19-20]. In the literature, the great variety of results is due to methodological and composition differences. Therefore, playing an important role in the properties of the materials, justifying the conduction of research studies to evaluate the properties of the materials available in the market, aiming at assisting the practitioners to correctly choose the materials to be used in clinical situations.

The water sorption and the solubility properties should be considered when choosing the restorative material to be used in restorations, especially when using the Atraumatic Restorative Treatment technique, as they are directly related to the lifetime of treatments.

CONCLUSION

Based on the results achieved in the experiment and within the limitations of an *in vitro* study, it can be concluded that: a) water sorption and solubility of the studied glass ionomer cements were not influenced by the various storage solutions; b) the highest water sorption rate, for all the materials, occurred in the first twenty-four hours after immersion, with a stabilization tendency for some materials in 7 and 14 days.

Collaborators

RBW LIMA, contributed to the design, acquisition of data, analysis and interpretation of data, and drafting the article. JFG FARIAS, contributed in the acquisition of data, analysis and interpretation of data and drafting the article. AKM ANDRADE, contributed with the design of the article, interpretation of data and drafting the article. FDSCM SILVA, contributed to conception and design of the article, acquisition of data and drafting the paper. RM DUARTE, contributed to conception and design, acquisition of data, and analysis and interpretation of data. Additionally, this author was important in drafting the article and revising it critically for important intellectual content.

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