ORIGINAL RESEARCH

Chemical and Structural Characterization of Glass Ionomer Cements indicated for Atraumatic Restorative Treatment

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ABSTRACT

Glass ionomer cements (GICs) are restorative materials, which clinical use has increased significantly during the last decade. The aim of the present study was to analyze the chemical constitution and surface morphology of four glass ionomer cements: Maxxion R, VitroFill, Vidrion R and Vitremer. Twelve polyethylene tubes with an internal diameter of 3 and 3 mm in length were prepared, filled and then transferred to a chamber with 95% relative humidity and a temperature of 37°C. The surface morphology of the tested materials was examined by scanning electron microscopy (SEM) and main components were investigated by energy-dispersive X-ray microanalysis (EDX). Scanning electron microscopy revealed irregular and rough external surface. Cracking was not observed. The main constituents were found to be aluminum, silicon, calcium, sodium and fluoride. Phosphorus, sulfur and barium were only observed in Vidrion R, while chlorine were only observed in Maxxion R. Elemental mapping of the outer surface revealed high concentration of aluminum and silicon. Significant irregularities on the surface of the tested materials were observed. The chemical constitution of all GIC was similar.

Keywords: Glass ionomer cement, Chemical properties, Scanning electron microscopy, Energy-dispersive X-ray analysis.

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INTRODUCTION

Glass ionomer cements (GICs) are widely used in restorative dentistry as luting cements, as cavity liners or

bases under composites, to restore Class V cavities, small Class I cavities, deciduous teeth, long-term temporaries and core build-ups.^{24,34} In addition, GIC are the materials of choice for atraumatic restorative treatment (ART).^{14,24,37} Its popularity is related to the presence of several important properties, such as adhesion to enamel and dentin¹² fluoride release^{27,28} good biocompatibility^{7,23,36} and coefficient of thermal expansion similar to that of dentin.⁴ Despite these advantages, GIC presents limitations as restorative materials, which are related to their poor mechanical and esthetic properties.^{6,22,25,26}

Glass ionomer cements are formed by an acid-base reaction of an aqueous polymeric acid and an ion leachable acid-degradable glass.³⁴ Although the compositions of the glasses used as cement formers are complex and varied they are based on three essential constituents: silica (SiO₂), alumina (Al₂O₃) and fluoride.^{3,7} From this system appeared other more complex and with better properties by the inclusion of different filler materials, such as silver-amalgam alloy powder, stainless steel powders, carbon and alumino-silicate fibers, bioactive glass, etc.^{2,17,36}

Several comparative studies on specific properties of GIC have been published^{6-8,23} and have been shown that a number of factors present influence on compressive strength and handling properties of GIC.^{8,19,25} The glass components critically determine the mechanical properties of this GIC,⁸ thus, the relationship between the composition of the glass and these properties should be examined. However, there are limited information about the correlation of glass composition and cement properties. Griffin & Hill,¹⁸ (1999) stressed that the vast body of literature on GIC deals with the properties of commercial products of unknown composition and microstructure.

Depending on their application GIC must be classified as materials with permanent contact to living tissues.²³ This contact may produces interactions with complex biologic systems around where it occurs, resulting in a biologic response represented by the immune reaction.^{11,23} The material's constituents and surface characteristics in addition to its cytotoxicity is an indication for material's biocompatibility.^{20,38} Thus, the aim of the present study was to analyze the chemical constitution and surface

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morphology of GIC by using scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) analysis.

MATERIALS AND METHODS

Preparation of the Samples

For the experiments, four commercial available GIC were used (Table 1). The GICs were mixed according to the manufacturers' instructions and placed in standard polyethylene tubes with an internal diameter of 3 mm and a thickness of 3 mm. The tubes were placed on glass slab (75 × 25 × 1 mm), slightly overfilled with the freshly prepared materials and then transferred to a chamber with 95% relative humidity and temperature of 37°C for a period corresponding to 3 times the manufacturer's recommended setting time. Three homogeneous specimens of each material were made.

Scanning Electron Microscopy and Energy Dispersive X-Ray Microanalysis

Morphological analysis of outer surface of GIC was performed using a scanning electron microscope (JSM-6610; Jeol Ltd., Tokyo Japan) at 500× magnification, using an accelerating voltage of 10 kV and a working distance of 15 mm. The samples were sprinkled on carbon double-side tape over a metallic stub, critical point dried and sputter-coated with gold palladium (Bal-Tec AG, Balzers, Germany) at 20 mA. The morphologies of the external surface were qualitatively analyzed according criteria used by Carvalho et al (2012).

EDX was performed with detection-analysis-system NSS Spectral Analysis System 2.3 (Thermo Fischer

Scientific, San Jose, CA, USA) to determine the constituent elements of the tested materials. One EDX spectrum was collected from the central region of each specimen under the following conditions: 25 kV accelerating voltage, 110 µA beam current, 10⁻⁶ Torr pressure (high-vacuum), 130 × 130 µm area of analysis at 1000× magnification, 100s acquisition time and 30-35% detector dead time. The elemental analysis (weight% and atomic%) of samples was performed in nonstandard analysis mode, applying PROZA (Phi-Rho-Z) correction method. The elemental maps were archived by NETCOUNTS method, with high resolution, using the same detection-analysis-system (NSS Spectral Analyses System 2.3).

RESULTS

The results obtained from SEM analysis are shown in (Figs 1A to D). It was noted that all GIC had an irregular external surface. Cracking was not observed.

A quantitative result of the main components of the tested materials is presented in Table 2. Similar chemical elements were found in all materials and there was a small variation between them. Essentially, the materials were composed of elements namely aluminum (Al), silicon (Si), calcium (Ca), sodium (Na) and fluoride (F). Phosphorus (P), sulfur (S) and barium (Ba) were only observed in Vidrion R, while chlorine (Cl) were only observed in Maxxion R. EDX wide spectrums are presented in (Figs 2A to D). Elemental mapping revealed the elements distributed throughout the outer surface (Figs 3A to D). Aluminum and silicon were strongly detected by such mapping.

Table 1: Composition of the materials and their manufactures

Material	Type	Composition (MSDS data)		Manufacturer	Lot
		Powder	Liquid		
Maxxion R®	Conventional	Glass of fluoro aluminium silicate Polycarboxylic acid Calcium fluoride	Tartaric acid Water	FGM, Joinville, PR, Brazil	020310
VitroFil®	Conventional	Strontium aluminum silicate Dehydrated polyacrylic acid Iron oxide	Polyacrylic acid Distilled water Tartaric acid	DFL, Rio de Janeiro, RJ, Brazil	0207597
Vidrion R®	Conventional	Sodium-calcium-alumino-fluoro silicate Barium sulfate Polyacrylic acid Pigments	Tartaric acid Water	SS White, Rio de Janeiro, RJ Brazil	0050611
Vitremer®	Resin-modified	Fluoroaluminosilicate glass Potassium persulfate Ascorbic acid Pigments	Aqueous solution of polycarboxylic acid modified Photoinitiators Water 2-hydroxyethyl methacrylate (HEMA)	3M ESPE, St. Paul, MN, USA	1104000163

DISCUSSION

GIC are restorative materials, which clinical use has increased significantly during the last decade. GIC may be classified according to its chemical composition into conventional, metal-reinforced, high viscosity and resin-modified.^{2,36} In the present study, the chemical constitution and outer surface of four different GIC, that were chosen based on their different formulations (three conventional cure GIC and one resin-modified GIC) and popularity, were analyzed by using EDX and SEM analysis.

EDX is a reliable, precise and reproducible method to quantify the major constituents or compounds present in a material or mixture.^{9,13,31,37} The identification of the major constituents of the material is important as it will lead to understanding of its physical, biological, chemical and mechanical properties.^{21,30} However, this method presents limitations regarding the detection of elements of low molecular weight. The proportion of ionizing events that result in emission of X-rays decreases as the atomic number of the element becomes smaller. Thus, quantification of organic compounds, which have carbon, hydrogen and oxygen, cannot be performed accurately.³⁹

All the tested cements had a similar chemical constitution as verified by EDX, and although the values varied, the GIC were composed primarily of Al, Si, Ca, Na and F (Table 2). Yap et al⁴⁰ (2003) characterized the glass

powder of 'fast-set' high viscous GIC (Fuji IX GP Fast and Fuji IX GP) with regards to particle size, size distribution and composition. The EDX analysis revealed that the three main elements present in Fuji IX GP Fast were oxygen (66.75%), silicon (13.18%) and aluminium (12.82%). Fuji IX GP also presented the same three main elements in the following composition: oxygen (64.42%), silicon (16.77%) and aluminium (16.72%). Zanata et al (2011) evaluated the chemical composition of high viscosity GIC (Fuji IX, GC Dental Co., Tokyo, Japan) after 10 years of clinical service. The cavities were restored according to the ART approach and were chemically analyzed with SEM/EDX. The main chemical components, as well as their average value (weight%), were: F (5.1), Al (16.9), Si (15.8), P (2.7), K (1.1), Ca (3.7) and Sr (11.1).

The ability of GIC to release fluoride has been a significant factor in their increasing use in dentistry.³³ Previous studies have documented the caries-protective effect resulting from their long-term release of F.^{3,16,24} The incorporation of fluoride to the GIC, besides caries-protective effect, improves its strength and handling properties.^{10,19} Several factors can influence the fluoride release of GIC, such as composition, sample dimension, handling technique, pH, application area and powder-liquid ratio.³³ Ideally, fluorine should be constantly released in small quantities in order to provide high and consistent levels.⁴¹ In the current study, F was observed

Figs 1A to D: SEM photomicrographs of the outer surface features of the tested materials (500×): (A) Maxxion R, (B) VitroFil, (C) Vidrion R and (D) Vitremer

in all GIC, with a concentration ranging from 8.14 wt% to 17.29 wt%. Zanata et al (2011), who also used EDX analysis, observed lower F concentrations (3.0 wt% to 5.1 wt%), however, this author evaluated the chemical constitution of restoration with 10 years of clinical use. Although some cements, as Vidrion R[®], presented lower amounts of F (Table 2), Ngo et al (1997) demonstrated that even small amounts of F can provide protection against demineralization. EDX revealed F in the composition of the VitroFil[®] (12.16 wt%), but the manufacturer does not cite any compound that has this element.

Gjorgievska et al (2012) determined the extent to which ions released from fluoride-containing dental restoratives migrated through the enamel and dentine of extracted teeth. A cervical (Class V) cavity was prepared in each tooth, then filled with one of: a conventional glass-ionomer, a resin-modified glass-ionomer, a polyacid-modified composite resin ('compomer') or a fluoride-releasing resin composite. After 1 and 18 months, the specimens were prepared and examined under SEM/EDX. The conventional glass-ionomer showed the highest level of ion migration whereas the fluoridated composite resin showed little if any ion migration. For Zanata et al. (2011) the lower amounts of F ions released by resin-modified GIC is due the absence of the phase of initial F release, usually observed in conventional GIC. In the present study, the highest concentration of F was found in the resin-modified GIC (Vitremer[®]; 17.29 wt%).

Aluminum is a substantial constituent of GIC and is responsible for the stability of the set cement.³⁴ Andersson & Dahl (1994) studied the release of aluminum from GIC during early water contact and observed increased release of aluminum from the auto-cured materials (GC Fuji I and II). For the authors the considerable release of aluminum from GIC during early water exposure may explain the reported lack of mineralization of predentin in the pulp beneath GIC. Griffin and Hill (1999) presented a study about the properties of GIC with varying the aluminum to silicon ratio and observed that the aluminum to

silicon ratio of the glass had no clear influence on the compressive strength of the hardened cement pastes. For the authors the maximum compressive strength would have been expected for cements made with glasses that had Al-Si ratios close to one.

Barium (Ba) is an element of high molecular weight and is associated with esthetics being considered an optic modifier. This element was only observed in Vidrion R[®] in the concentration of 4.24 wt%. Witten et al⁴² (2012) demonstrated that aluminum, barium and sulfur are considered cytotoxic or genotoxic depending on the concentration in which they are used. Souza et al (2006) evaluated the effects of resin-modified GIC (Rely X Luting Cement, Vitremer and Vitrebond) applied on culture of cells or implanted into subcutaneous tissue of rats. The materials significantly influenced cell respiratory activity and promoted a moderate to intense inflammatory reaction. For the authors, GIC may cause noticeable inflammatory response when in direct contact to connective tissue and the toxic effects of this kind of soluble material depend on the amount of components released in the aqueous environment.

The Ca⁺² ions form the calcium polycarboxylate, which reduces the mobility of the chains and increases viscosity leaving the cement with a rubber aspect. This ion was observed in all conventional GIC tested. Wang et al³² (2007) investigated the effects of environmental calcium/phosphate and pH on the hardness and elastic modulus of two glass-ionomer cements and suggested that the high calcium and phosphate rates may have positive effects on the surface hardness of GIC. But due to the clinical requirement for radio-opaque materials, calcium is frequently replaced by radio-opacifiers, such as strontium, lanthanum and barium.⁸

The importance of P in controlling ion release from the glass surface has been identified in a number of studies.^{3,19} Griffin and Hill (2000) evaluated the influence of the glass phosphorus content on the handling properties of GIC and observed that increasing phosphorus content

Table 2: Main components of glass ionomer cements analyzed with energy-dispersive X-ray microanalysis

Element	Maxxion R [®]		VitroFill [®]		Vidrion R [®]		Vitremer [®]	
	wt%	at. %	wt%	at. %	wt%	at. %	wt%	at. %
Al	28.24	28.26	31.23	32.07	28.46	23.08	28.22	29.26
Ba	—	—	—	—	4.24	17.51	—	—
Ca	9.70	14.47	1.94	2.96	13.12	15.81	—	—
Cl	3.87	5.09	—	—	—	—	—	—
F	13.82	9.74	16.81	12.16	8.14	4.65	17.29	12.62
Na	19.70	16.80	3.39	2.97	5.20	3.59	3.52	3.11
P	—	—	—	—	3.89	3.62	—	—
S	—	—	—	—	4.51	4.34	—	—
Si	24.67	25.70	46.62	49.84	32.44	27.39	50.97	55.01

Figs 2A to D: Representative EDX spectrum of the tested materials: (A) Maxxon R, (B) VitroFil, (C) Vidriion R and (D) Vitremer

increases the working time of the cement paste but does not necessarily result in an increase in the cement strength. Phosphorus was only identified on Vidriion R cement.

The EDX analysis revealed a discrepancy between the elements found and the main components described by the manufacturers. Vitremer® and Vitrofil® showed large amounts of fluoride, a result consistent with the main component of Vitremer®, described by the manufacturer as $\text{Al}_2(\text{SiF}_6)_3$. However, the same does not occur with the main component Vitrofil®, whose prescription is $\text{SrAl}_2\text{Si}_2\text{O}_8$. The same discrepancy was observed in the others tested materials.

For checking material properties and interactions with biological systems (biocompatibility and cytotoxicity analysis), it is necessary to understand the materials' surface.⁹ SEM has been shown to be a powerful research tool for investigating the particle size or granulation present on materials' surface.^{1,13,36} Elemental mapping permits to reveal the elements distributed throughout the materials' outer surface, which may maintain direct contact with the tissue.

The requirements of an ideal restorative material include a smooth surface texture.²⁹ GIC have some clinical limitations, such as prolonged setting reaction time, moisture sensitivity, dehydration and rough surface texture.^{6,22,25,26} High surface roughness is likely to increase significantly bacterial adhesion, dental plaque maturation, and acidity, which act on material surfaces, thus increasing caries risk.² Bala et al (2012) evaluated surface roughness and hardness of six different GIC and determined whether there is any correlation between GIC's surface roughness and hardness. All the tested GIC showed lower surface roughness values after the polishing procedure. The authors suggested that the differences in the composition of GIC may affect their surface roughness and hardness. In addition, other factors may contribute to the material's surface aspect, such as: shape, distribution and number of particles, interfacial bonding between particles, interfacial bonding between the particles and matrix, storage media of GIC specimens, GIC's liquid component, powder: liquid ratio, etc.^{15,40}

SEM analysis of material surfaces revealed that all GIC showed uniform distribution of the elements, with

Figs 3A to D: EDX elemental map of aluminum (green) and silicon (red) distribution throughout the outer surface of tested materials: (A) Maxxion R, (B) VitroFil, (C) Vidrion R and (D) Vitremer

irregular aspects. Increased number of irregularities was observed in Vitremer[®], the only resin-modified GIC analyzed. In the present study, no attempt was made to measure the surface roughness and to classify the particle size of the tested materials. The materials' surfaces were qualitatively analyzed and classified according to Carvalho et al (2012). No cracks in the surface morphology of the studied materials were observed. Previous studies^{2,5,17,36} had observed several cracks in the surfaces of cure GIC-based materials. Cracks facilitate the phenomena of materials' surface degradation so as to allow the bio-fluid to penetrate through surface and discoloration.¹⁷ Furthermore, materials presenting denser surface textures with less and smaller voids showed higher hardness values.^{17,35} Xie et al (2000) suggested that factors such as the integrity of the interface between the glass particles and the polymer matrix, the particle size, and the number and size of voids have important roles in determining the mechanical properties. However, cracks can be produced by dehydration during specimen preparation for SEM analysis.^{5,12}

The results of the present survey lead to a more comprehensible understanding of the interactions that

occur between GIC and tooth tissues. This extended comprehension should help investigators to design new products, with well-defined characteristics, for a wider variety of applications in restorative dentistry.

CONCLUSION

The GIC analyzed by SEM images and EDX showed irregular surfaces but with elements uniformly distributed. It was observed discrepancy between the main elements found and the components described by the manufacturers.

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