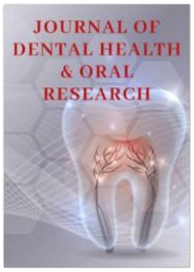

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Research Article

Radiopacity Related to Composition of Restorative Glass-Ionomer Cements with the Use of Digital Images

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Abstract

The aim of this study was to evaluate the radiopacity of fourteen restorative glass-ionomer cements and to associate the results obtained with the Energy-dispersive X-ray spectroscopy (EDX) analysis. The glass-ionomers evaluated were Maxxion R (MX), Vitro Fil (VF), Bioglass R (BG), Ionglass R (IG), IonoFil Plus (IP), GlassIonomer Type II (GI), Vitro Molar (VM), IonoStar Molar (IS), Equia Forte (EF), GC Gold Label 9 (GL9), Riva Self Cure (RV), Ketac Molar Easy Mix (KM), Fuji II (FJ2) and ChemFil Rock (CR). The materials were handled following manufacturer's instructions and six specimens were made of each material (Ø15x1 mm) (ISO 9917-1: 2007). After storage for 7 days, the radiopacity was determined for each material using a dental X-ray machine. Data were subjected to one-way ANOVA and Tukey's test (α=0.05). Superficial analysis was evaluated for the 14 materials with a Scanning Electronic Microscope equipped with an X-radiation detector. The highest radiopacity values were registered for FJ2, IS, GL9 and CR and the lowest for MX ($p<0.05$). There was significant difference in radiopacity among the materials and seven of the fourteen tested GICs could not be labeled as radiopaque materials by its manufacturers according to ISO 9917-1, however four of them are described as radiopaque in its package inserts. The strontium and zinc content appear to be associated with the higher radiopacity values found.

Keywords: Glass Ionomer Cements; Radiopacity

Introduction

The radiopacity of dental materials in general practice is a valuable diagnostic tool for direct filling, cavity liners, luting agents and adhesive systems [1]. This property is key to evaluate long-term success of restorations, enabling the detection and assessment of marginal overhangs, open gingival margins, interproximal contour and recurrent caries by the contrast difference between tooth, film and material [2-4]. It is known that those factors are directly associated with consequences beyond adaptation and aesthetics, leading to pain, anxiety, low-esteem and in worst cases, irreversible outcomes as loss of the tooth. Thus, radiographic images bring the possibility to act in advance, predicting future complications by a preventive approach. Also, neglecting those aspects adds an economic impact once the patient needs to pay for a treatment to restore or replace elements that could be avoided if well diagnosed with the use of radiographic images [5-7].

There is still lack of information in literature regarding radiopacity on restorative Glass-Ionomer Cements (GICs), despite its undeniable influence on clinical practice. Those cements have gone through many changes in the past related to their composition, in order to overcome limitations, being considered part of the future of conservative dentistry [8]. These modifications included the addition of high atomic number radiopaque components and reinforcement particles, such as strontium (Sr), barium (Ba) and zinc (Zn) [9,10]. Hence, recent restorative GICs presented satisfactory clinical durability when compared to composite resin in posterior teeth over a 6-year evaluation period [11]. On the other hand, the addition of components may also affect other significant properties, such as opacity and translucency, due to the light-refracting properties of the components. An excess of heavy metal oxides may reduce the resistance of the glass to chemical degradation, as the large particles of Ba and Sr could disrupt the aluminosilicate network [12].

As low radiopacity may hinder contrast between tooth and restoration, previous studies have concluded that a radiopacity equal or greater than dental enamel is desirable for adequate performance of restorative materials. Moreover, it has been recommended that dental restorative materials should be radiopaque [3,13]. Considering that, ISO 9917-1 stipulates a minimum radiopacity for materials to be labeled as radiopaque. Although many new brands of restorative conventional glass-ionomer cements were recently introduced on the market, there is no information on radiopacity of these materials [14]. Furthermore, previous data on radiopacity of glass-ionomer cements seem to be outdated, with no standardized methodology and most of materials tested are not available in the market today. In addition, ISO 9917-1 standard received an update in 2007 related to radiopacity tests on restorative glass ionomer cements.

The aim of the present study was to determine and compare the radiopacity of fourteen trademarks of recently introduced and of well-established restorative glass-ionomer cements, analyzing them based on the International Accepted Standards (ISO) and to investigate the chemical components and their influence in the materials radiopacity by Energy-Dispersive X-ray spectroscopy (EDX) assessment. And as a result, bring more information to understand the relationship between radiopacity and the composition of conventional glass-ionomer cements and assist professionals in choosing the best materials for clinical practice.

Methodology

The brands and compositions of restorative conventional glass-ionomer cements tested are described in Table 1.

Commercial Name	Manufacturer	Groups	Powder	Liquid	Powder/ Liquid Ratio	Batch Number
Bioglass R	Biodinâmica, Ibiporã, Brazil	BG	Calcium, barium and aluminum fluoride + PAA + inorganic fillers	PAA+ TA+ water	1.6:1	97515
ChemFil Rock	Dentsply Caulk, Milford, USA	CR	Calcium-aluminum-zinc-fluoro-phosphor-silicate glass + PA+ pigments + TA + water		capsule	1.51E+09
Equia Forte	GC Corporation, Tokyo, Japan	EF	Strontium fluoro-alumino-silicate + PAA + water		capsule	1611221
GC Fuji II	GC Corporation, Tokyo, Japan	FJ2	Strontium Fluoroaluminumsilicate + PAA	PAA + water	2.7:1	1601121
GC Gold Label 9	GC Corporation, Tokyo, Japan	GL9	Strontium Fluoroaluminumsilicate + PAA	PAA + other ingredients	3.6:1	1506011
GlasIonomer Type II	Shofu Inc. Kyoto, Japan	GI	Alumino fluoro silicate glass	Copolymer of acrylicacid and tricarboxylic acid + TA	2.5:1	51501
Ionglass R	Maquira Dental Products, Maringá, Brazil	IG	PAA + sodium fluorosilicate, calcium, aluminum	TA + water	1.5:1	75216

IonoFil Plus	Voco GmbH, Cuxhaven, Germany	IP	Strontium aluminium fluorosilicate glass	PAA + TA + water	4.7-5.6:1	1514030
IonoStar Molar	Voco GmbH, Cuxhaven, Germany	IS	Strontium aluminum fluorosilicate + PAA + TA + water		capsule	1618227
Ketac Molar Easy Mix	3M ESPE, Seefeld, Germany	KM	Strontium Fluorosilicate + Aluminum + lanthanum + pigments	PCA + TA + water	4.5:1	628875
Maxxion R	FGM, Joinville, Brazil	MX	Fluoroaluminum silicate + Calcium fluoride	PCA + TA + water	1.5:1	240516
Riva Self Cure	SDI, Victoria, Australia	RV	Fluorine Silicate Aluminum + PAA	PAA + TA + water	3.03:1	73072V
Vitro Fil	Nova DFL, Rio de Janeiro, Brazil	VF	Fluorine Strontium Aluminum Silicate + `PAA + Iron Oxide	PAA + TA + water	02:01	16050647
Vitro Molar	Nova DFL, Rio de Janeiro, Brazil	VM	Fluorine Barium Aluminum Silicate + PAA + Iron Oxide	PAA + TA + water	2.9:1	16030405

Table 1: Brands and composition of glass-ionomer cements tested.

The materials were handled according to manufacturers' instructions. Six disc-shaped specimens (Ø =15x01 mm) of each material were prepared according to ISO 9917-1:2007 standards using metal molds with glass plates and polyester films on both sides, all held with a clamp to ensure the correct specimen thickness [15]. The whole assembly was placed in an incubator at 37°C and after 30 minutes, the specimen was removed from mold. The thickness of each specimen was measured near its center using a screw micrometer (0,01 mm accuracy) and when necessary, the specimen was polished using 1200 grit abrasive paper until the specified thickness (1 ± 0.1 mm) was obtained. The specimens were returned to the incubator for storage in water of grade 3 (ISO 3696:1987) for 7 days.

Radiopacity Test

For the radiopacity test, the specimens (n = 5) were positioned in an occlusal sized imaging plate alongside an aluminum step wedge designed with a thickness range from 0,5 mm to 5 mm in equally spaced steps of 0,5 mm according to ISO 9917-1:2007 requirements [15]. The specimens and the aluminum step wedge were irradiated in a dental X-ray unit at 70kVp/7mA for 0.23s at a distance of 400mm. Five images of each specimen were obtained, resulting in 25 images per group and 350 imagens in total.

After irradiation, the image plates were developed in VistaScan digital and the images obtained (JPEG format) were analyzed in the photographic densitometer of Image J (National Institutes of Health) to measure their mean gray values (MGV). Each group had 5 specimens and each specimen was measured five times in order to obtain more accurate radiopacity values. These values were converted into millimeters of aluminum (mmAl), using the following equation proposed by Lachowski, et al., [13].

$$\frac{(A-B)}{(B-C)}\ x\ sample\ thickness + mmAl\ below\ material\ MGV$$

where A is the material's MGV; B is the MGV of the aluminum step wedge increment immediately below the material's MGV; and C is the MGV of the aluminum step wedge increment immediately above the material's MGV.

Energy-Dispersive X-ray Spectroscopy

The specimens (n=1) of all groups were subjected to a superficial analysis of the elements Si, Ca, Na, Al, Sr, Ba, Zn and F (according to atomic number and composition) performed by a Scanning Electron Microscope equipped with an X-radiation detector.

Statistical Analysis

Data were statistically analyzed with SPSS for Windows v.19.0 (IBM Statistics, United States). The normal distribution and homogeneity of variances assumptions were checked for all variables using the Shapiro-Wilk test and the Levene test, respectively. As the assumptions were satisfied, data were subjected to ANOVA, followed by Tukey's test (α=0.05), for individual comparisons.

Results

The results for radiopacity are shown in Table 2 and Fig. 1. The highest radiopacity values were registered for FJ2, IS, GL9 and CR ($p>0.05$). The lowest value was recorded for MX which was different to the other groups ($p<0.05$). The other groups, KM, EF, RV, IP, IM, VM, VD, VF, BG, GM, IZ, IG and GI presented intermediate values between them. ISO 9917-1 establishes that for a material to be described as radiopaque by its manufacturer, the radiopacity must be at least equivalent to that of aluminum of the same thickness when tested in accordance with ISO 9917-1 requirements. Since ISO 9917-1 specifies specimens with a thickness of 1 mm, the minimum radiopacity should therefore be equivalent to 1 mm of aluminum (1 mmAl). Considering this reference, the GICs RV, EF, KM, CR, GL9, IS and FJ2 achieved the minimum radiopacity value determined by the ISO standard and they are correctly labeled as radiopaque in its package inserts.

Commercial Brand's Names	**Radiopacity (mm Al)**
GC Fuji II	1.72 (0.18) [a]
IonoStar Molar	1.38 (0.12) [a.b]
GC Gold Label 9	1.37 (0.34) [a.b]
ChemFil Rock	1.34 (0.13) [a.b.c]
Ketac Molar Easymix	1.25 (0.22) [b.c.d]
Equia Forte	1.15 (0.31) [b.c.d.e]
Riva Self Cure	1.05 (0.12) [b.c.d.e]
Ionofil Plus	0.96 (0.18)* [b.c.d.e.f]
Vitro Molar	0.91 (0.15)* [d.e.f.g]
Vitro Fil R	0.85 (0.22)* [d.e.f.g]
Bioglass R	0.82 (0.08)* [e.f.g]
Ionglass R	0.75 (0.19)* [e.f.g]
Glass Ionomer Type II	0.60 (0.06)* [f.g]
Maxxion R	0.52 (0.05)* [g]
* The highlighted values are below the minimum required by ISO for the manufacturer to describe the material as being radiopaque, considering the standard deviation.	

Table 2: Mean values and standard deviation (DV) of radiopacity for the fourteen brands of glass-ionomer cements.

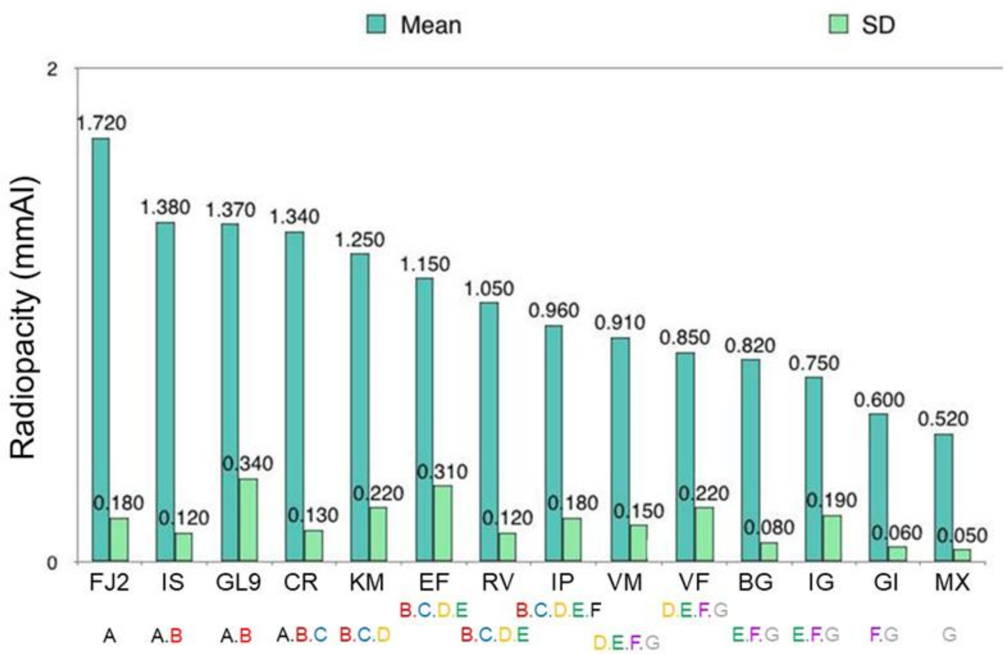

Figure 1: Mean values and standard deviation (DV) of radiopacity for the fourteen brands of glass-ionomer cements.

The results of the EDX analyses of the material with higher radiopacity are presented in Fig. 2. Strontium concentrations were highest for the materials FJ2, IS and GL9 and are 13.49, 12.23 and 13.35 weight percent respectively. The element zinc was found in many brands as GI, VM, EF, IP, but the higher value was observed for CR (11.26%), which also presented a lower amount of strontium (6.26%). Likewise, BG, VM and VD also presented some amount of barium (more than 5%). The Fig. 2 demonstrates EDX analysis and the correspondent elements. In all cases, data are expressed in terms of normalized mass percentage of oxides of the elements with atomic number higher than 10, with special attention to the elements zinc, barium and strontium.

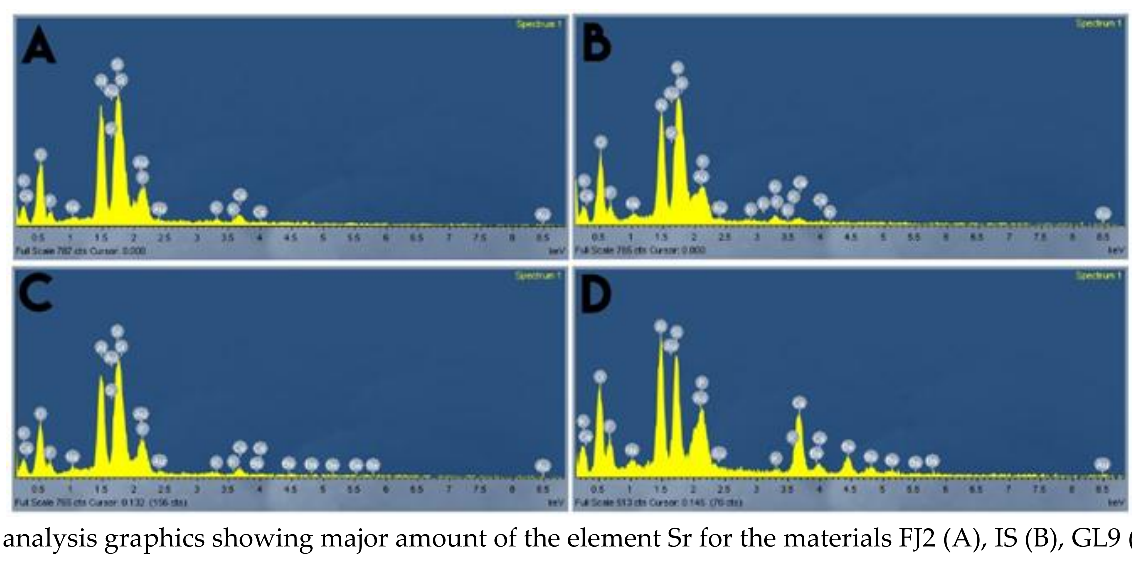

Figure 2: EDX analysis graphics showing major amount of the element Sr for the materials FJ2 (A), IS (B), GL9 (C) and CR (D).

Discussion

In the present study, radiopacity showed variable results among restorative glass ionomer cements. Unfortunately, it is difficult to compare our results with literature data, since many studies evaluate radiopacity results among different types of restorative materials [1,17,18]. There is not a recognized ideal value for radiopacity, although many authors discuss it. A tendency to strive for the highest achievable radiopacity was seen but the possible "Match effect" would be a concern, with enhancement of the contrast between a light and a dark area and consequent inadequate diagnosis of marginal areas [3,19]. Thus, the radiopacity considered acceptable for restorative materials is when it slightly exceeds the enamel radiopacity [3,13,17,18,20]. As seen in Fig. 1, no material presented radiopacity to give rise to the “Match effect” levels.

It is known that 1 mm of dentin corresponds to approximately the equivalent radiopacity of 1 mm of Al used in the step wedge [21]. The International Organization for Standardization (ISO) for water-based cements determines that a material should contain at least 1 mmAl in order for manufacturers to label it as radiopaque. According to Bouschlicher, et al., even when the radiopacity value complies with ISO standards, it may not be enough to detect small defects and the restoration limits [22]. The density of dentin should be the least to assure that the material would not be mistaken for carious dentin radiographically [13]. The results found shows that seven out of the fourteen materials tested have less than 1mmAl, which means that the manufacturer of those brands should not label those materials as radiopaque, however IP, VM, VF and BG are labeled as radiopaque materials in its package inserts. That information is concerning, once glass-ionomer cements are widely used as restorative materials, especially in the primary dentition [23] and for temporary restorations, where conservative removal requires radiographic visualization.

Lack of information on composition by manufacturers led to the need of surface analysis through Energy-dispersive X-ray spectroscopy (EDX), in order to explain the results found. There is only one study in the literature reporting results of radiopacity associated with surface analysis by X-ray energy dispersive, although it was carried out with composite resins, instead of glass-ionomer cements [24]. Earlier glass-ionomer cements were radiolucent, limiting their use as restorative materials. Thus, the need for more radiopaque cements led to the addition of other components. Molecular structure influences the material radiodensity, i.e. the relative inability of radiation to pass through a particular material [18]. As seen in Table 1, the powder composition of conventional glass-ionomer cements is basically aluminosilicate glass. Atomic numbers of aluminum, silicon and calcium are 13, 14 and 20 respectively and are considerably low. Accordingly, incorporation of aluminosilicate glass alone leads to radiolucent glass-ionomer cements [13,14,25]. Studies shows that incorporation of elements with high atomic number is associated with high radiopacity and the presence of the elements zinc, strontium and barium (atomic numbers 30, 38 and 56 respectively) is attributed to that improvement [24,26]. From those, barium and strontium are the most used today (both in group II on the Periodic Table). This inclusion is usually done with fillers as zinc oxide, strontium oxide and barium sulphate to the glass particles [10].

The materials GL9, FJ2 and IS, according to EDX analysis, presented the highest content of strontium (more than 10% weight) and also presented the highest radiopacity values among all materials tested. It corroborates other literature findings, which conclude that radiopacity increases linearly with the strontium content [10]. CR was the unique material presenting a high quantity of zinc in the EDX analysis (more than 10%) and is among the few materials with adequate radiopacity values. Thus, the most viable explanation for the high value of radiopacity for CR is the high amount of zinc found in the analysis along with the content of strontium, superior to other materials. Some materials, such as IP and VF, presented some amount of Sr on the surface (less than 10%), but low values of radiopacity. This suggests that just the presence of Sr alone is not enough to increase radiopacity; there must be a minimal amount necessary to produce satisfactory radiopacity results. The presence of zinc may influence this property, but the authors suggest that it is associated to the simultaneous presence of strontium. Despite the high atomic number, all Ba-containing cements tested were radiolucent, which suggests that addition of barium (BG, VM) probably had no influence on radiodensity or was not high enough to have an influence on this property.

Radiopacity should not be enhanced at the expense of other properties or characteristics [24]. Many fillers added to the glass particles produce “opaque” cements or have the tendency to increase erosion rate whereas incorporation of considerable amounts of heavy metal oxides may reduce the resistance to chemical degradation by the presence of large ions disrupting the network matrix [24,27]. The consequences are weakened materials, increased wear rates with time and notably lower success rate of restorations. From a biological aspect, studies have shown that elution of Sr was not cytotoxic for human dental pulp cells also considering the relatively small amount that is present in restorative materials composition and that is released from that [28-30]. In fact, strontium demonstrated to have a positive response accelerating bone ingrowth around implants in rats [31]. There are still few data on cytotoxicity for barium and zinc in glass ionomer cements, although studies concluded that both lead to cell necrosis only in high concentrations [32,33]. Besides, glasses with high Sr concentration (equivalent to 40% SrO) are associated with poor wear resistance [24]. Regarding translucency, one study showed a reduction of 7% or more in optical opacity for all five Sr containing cements after 24hrs [10].

The present study used standardized tests to reduce the risk of bias, yet *in-vitro* studies do not reproduce oral environment perfectly. Thus, further research should concentrate on clinical outcomes to validate and provide reliability for the results found. More studies on chemical analysis and cytotoxicity of metal oxides should be performed to better understand and support the indication of Sr-containing cements over other cements.

Conclusion

It was concluded that from 14 brands tested 7 of them did not achieve the minimum radiopacity value to the manufacturers label the product as a radiopaque material, but 4 of those GICs are labeled as radiopaque in its package inserts.

Conflict of Interest

The authors declare no conflict of interest.

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None.

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