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Influence of external energy sources on the dynamic setting process of glass-ionomer cements

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ABSTRACT

Objective. To evaluate the influence of external energy sources on the dynamic setting process of glass-ionomer restorative materials.

Methods. Eighteen brands of GIC were studied: Bioglass R (Biodinâmica; G1), Chemfil Rock (Dentsply; G2), Equia Forte (GC; G3), Gold Label 2 (GC; G4), Gold Label 9 (GC; G5), Glass Ionomer Cement Type II — (Shofu; G6), Ionglass R (Maquira; G7), Ion Z (FGM; G8), Ionomaster (Wilcos; G9), Ionofil Plus (Voco; G10), Ionostar Plus (Voco; G11), Ketac Molar easymix (3M ESPE; G12), Magic Glass R (Vigodent; G13), Maxxion R (FGM; G14), Riva Self Cure (SDI; G15), Vidrion R (SS White; G16), Vitro Fil R (Nova DFL; G17) and Vitro Molar (Nova DFL; G18). LED, halogen light or ultrasound (n=20 for each set) applied for 30 s was used to activate setting, and a control group of each material was allowed to set without activation. Samples were analyzed by FTIR spectroscopy using the ratio of intensities of bands at 1637 cm⁻¹ (carboxylate) and 1720 cm⁻¹ (carbonyl) as a function of time. Means and standard deviations were subjected to ANOVA and Tukey tests (p < 0.05).

Results. All three activation modes significantly reduced the time at which the carboxylate content became stable in G2, G4, G5, G6, G8, G10, G14, G16, G17 and G18. By contrast, in G1, G7, G12 and G15 no activation source had any significant effect (p > 0.05).

Significance. External activation sources, namely LED, halogen light and ultrasound, typically but not always increase the setting rate of restorative GICs.

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1. Introduction

The glass-ionomer cement (GIC) is the material of choice in several clinical situations in modern dentistry due to its favorable physical and biological properties [1]. The setting process of the glass-ionomer cement (GIC) is of the acid-base type between a powder of degradable aluminosilicate glass and an aqueous solution of polymeric acid, such as polyacrylic acid or an acrylic/maleic acid copolymer [2,3]. The aqueous phase of the liquid attacks the solid structure of the glass particles releasing calcium or strontium, aluminum and fluoride ions into the aqueous medium [4,5]. The polyacid chains become cross-linked, initially with the calcium or strontium ions, forming a solid mass [4,5]. During the first 24 h, the aluminum ions also unite with the polymer chains within the cement mixture, making it more rigid [1,6,7].

Glass-ionomer cement has the advantages of adhesion to dental substrates, fluoride release, biological compatibility and linear coefficient of thermal expansion similar to tooth structure [1,8], making the GIC a versatile material. It can be used in restorations, as cavity bases and for cementation of prosthetic and orthodontic appliances [9]. However, the prolonged time of maturation and sensitivity to moisture before completion of the setting process is a limitation of the material, as the GIC suffers syneresis or imbibition in the first 24 h after mixing [1]. Previous studies showed the presence of water at the interface with dentin, due to the permeability of GIC matrix after storage in water. This phenomenon occurs due to the fact that after the first 24 h, the material suffers an additional reaction of the curing process and it is possible for moisture to reach the GIC/dentin interface by diffusion through dentin [8] thus affecting the final strength of the material [10].

Yap et al. [11], suggested that if the curing process of the GIC were faster, it could result in the improvement of the mechanical properties. To achieve this objective, it has been suggested that the application of external sources of energy could accelerate the reaction of the initial setting process, improving the final properties of the material.

However, there still exists the need for studies that characterize the influence of external sources on modifying the chemical reactions of GICs, to result in acceleration of hardness of this material. Fourier Transform Infrared spectroscopy technique (FTIR) provides the means of identifying specific functional groups due to their characteristic absorption bands and is able to provide information on the detailed chemistry of the material, as evaluated by changes and/or emergence of new peaks [12]. Thus, the objective of this paper was to evaluate the time required to stabilize the chemical bonds in the curing process of GICs when external sources such as LED, halogen light and ultrasound waves were applied to the material after mixing. The null hypothesis is that the application of external sources to the GICs after mixing did not alter the time required to stabilize the chemical bonds in the curing process of the materials.

2. Materials and methods

The tests were performed in vitro using 18 brands of conventional glass-ionomer restorative cements (Table 1).

2.1. Sample preparation

The 18 groups ($n=20$, each material) were divided into control and experimental groups ($n=5$, each group) in which LED (Radii Cal, SDI, Victoria, Australia, 1200 Mw/cm², point temperature of 45 °C), halogen light (Optilux 501, Kerr Corporation, Orange California, United States, 1000 Mw/cm², point temperature of 55 °C) or ultrasound waves (DentSurg PRO, CVDentUS, São José dos Campos, Brazil) were used. The temperature reached by the halogen light and LED was measured with a digital infrared thermometer model GM 300 (Benetech, Shenzhen, China). The tip of the infrared light was positioned at 1 mm from the tip of light source turned on for 30 s. The highest temperature measured in the end of 30 s was recorded. The measurements were done in triplicate.

After being manipulated according to the individual manufacturer's instructions, each GIC was placed between two polyester tapes and pressed between two plates of glass and loaded with a constant force of 0.4 N for 30 s to standardize the thickness of the material. In the groups that received activation with one of the two light sources, the tip of the unit was positioned in contact with the surface of the material directly on the polyester tape and the photo-polymerizing device was used for 30 s. In the group in which heat and ultrasound waves were applied, after spreading the GIC in a limited area of the glass plate demarcated with an adhesive tape (10 mm²) to standardize its thickness, the tip of the ultrasonic probe was applied on the material and activated for 30 s without water cooling. In the control group, the GICs did not receive any activation. The materials were maintained between polyester tapes at ambient controlled temperature (25 °C) to protect them from desiccation and after the first 5 min of curing, the samples of GIC were prepared for measurements.

2.2. Dynamic setting analysis

2.2.1. Fourier Transform Infrared (FTIR) spectroscopy

To understand the dynamic cure of GICs, i.e. the time interval necessary for the chemical bonds to be formed and stabilized, the samples were ground in a mortar using an agate pestle, then diluted in potassium bromide (KBr) powder and pressed under 10 tons, for 2 min to obtain pellets for analysis by FTIR spectroscopy according to the methodology related by Yamakami et al. [13].

Characterization of the molecular interactions of the GIC material was determined by the Vertex 70v Fourier Transform Infrared (FTIR) spectrometer (Bruker®, Massachusetts, USA), with 4 cm⁻¹ of resolution. The spectra of the KBr/sample pellets were collected every 10 min for 120 min, making an average of 128 scans per specimen with the spectral range from 4000 to 400 cm⁻¹.

Table 1 – Brands, batch numbers and composition of conventional restorative glass-ionomer cements tested.

| Group | GIC | Batch no. | | Composition |
|-------|---|-----------|------------|---|
| 1 | Bioglass R (Biodinâmica, Ibioporã, Brazil) | Powder: | 974/15 | Calcium, barium and aluminum fluorosilicate, polyacrylic acid and inorganic filler |
| | | Liquid: | | Polyacrylic acid, tartaric acid and water |
| 2 | Chemfil Rock (Dentsply, Milford, USA) | Powder: | 1511000724 | Zinc-modified fluoro alumino silicate glass |
| | | Liquid: | | Polyacrylic acid and itaconic acid |
| 3 | Equia Forte (GC Corporation, Tokyo, Japan) | Powder: | 1608181 | Fluoro-alumino-silicate glass, polyacrylic acid powder, pigment |
| | | Liquid: | | Polyacrylic acid, distilled water, polybasic carboxylic acid |
| 4 | Gold Label 2 (GC Corporation, Tokyo, Japan) | Powder: | 1601161 | Fluoro-alumino-silicate glass and polyacrylic acid powder |
| | | Liquid: | 1601121 | Distilled water and polyacrylic acid |
| 5 | Gold Label Glass 9 (GC Corporation, Tokyo, Japan) | Powder: | 1506021 | Fluoro-alumino-silicate glass, polyacrylic acid powder |
| | | Liquid: | 1506011 | Polyacrylic acid, polybasic carboxylic acid |
| 6 | Glass Ionomer Cement Type II (Shofu Inc., Kyoto, Japan) | Powder: | 6144 | Fluoro-alumino-silicate glass |
| | | Liquid: | 31513 | Copolymer of acrylic acid and tricarboxylic acid, tartaric acid and others |
| 7 | Ionglass (Maquira Dental Products, Maringá, Brazil) | Powder: | 130417 | Polyacrylic acid and sodium fluorosilicate, calcium and aluminium |
| | | Liquid: | | Tartaric acid and purified water |
| 8 | Ion Z (FGM, Joinville, Brazil) | Powder: | 140116 | Fluoro-alumino-silicate glass |
| | | Liquid: | 130116 | Polycarboxylic acid and tartaric acid |
| 9 | Ionmaster (Wilcos, Petrópolis, Brazil) | Powder: | 15336 | Calcium fluoro-alumino-silicate glass powder, tartaric acid, citric acid, pigments |
| | | Liquid: | 15335 | Water, polycarboxylic acid, pigments |
| 10 | Ionofil Plus (VOCO GmbH, Cuxhaven, Germany) | Powder: | 1509454 | Fluoro-alumino-silicate glass and polyacrylic acid |
| | | Liquid: | 1506325 | Tartaric acid |
| 11 | Ionostar Plus (VOCO GmbH, Cuxhaven, Germany) | Powder: | 1607068 | Fluoro-alumino-silicate glass, polyacrylic acid, tartaric acid |
| | | Liquid: | | Polyacrylic acid solution |
| 12 | Ketac Molar Easymix (3M ESPE, Seefeld, Germany) | Powder: | 627356 | Al–Ca–La fluorosilicate glass, copolymer acid (acrylic and maleic acid) |
| | | Liquid: | 624889 | Polyalkenoic acid, tartaric acid, water |
| 13 | Magic Glass (Vigodent, Rio de Janeiro, Brazil) | Powder: | 1503044 | Strontium, aluminum, fluoride, silicate, polyacrylic acid, tartaric acid and pigments |
| | | Liquid: | 1401244 | Polyacrylic acid, water |
| 14 | Maxxion R (FGM, Joinville, Brazil) | Powder: | 21117 | Fluoro alumino silicate glass, polycarboxylic acid, calcium fluoride, water |
| | | Liquid: | 260917 | water |
| 15 | Riva (SDI, Victoria, Australia) | Powder: | 150630V | Glass powder and acrylic acid polymers |
| | | Liquid: | 15312 | Acrylic acid polymers and tartaric acid |
| 16 | Vidrión R (SS White, Rio de Janeiro, Brazil) | Powder: | 220716 | Sodium fluor silicate, calcium, aluminum, barium sulphate, polycarboxylic acid, pigments. |
| | | Liquid: | | Tartaric acid, water |
| 17 | Vitro Fil (Nova DFL, Rio de Janeiro, Brazil) | Powder: | 16030374 | Fluorine strontium aluminum silicate, dehydrated polyacrylic acid and iron oxide |
| | | Liquid: | 16030373 | Polyacrylic acid, tartaric acid and distilled water |
| 18 | Vitro Molar (Nova DFL, Rio de Janeiro, Brazil) | Powder: | 16020279 | Fluorine barium aluminum silicate, dehydrated polyacrylic acid and iron oxide |
| | | Liquid: | 16020278 | Polyacrylic acid, tartaric acid and distilled water |

2.2.2. Setting analysis

The setting process was analyzed in the spectra by the ratio of the intensities of the bands associated with the formation of the COO^- carboxylate salts of the polyacid (1637 cm^{-1}) and C=O acid group (1720 cm^{-1}) as a function of time. The ratio of the band's intensities as a function of time was adjusted by an exponential decay-type function according to Yamakami et al. [13]. The means and standard deviations of the ratios were subjected to Shapiro–Wilk normality test, ANOVA and Tukey tests at 5% significant statistic level, using R statistic software (R Foundation for Statistical Computing, Vienna, Austria).

3. Results

The values in minutes of the minimum time required for stabilization of the chemical reactions of the formation of

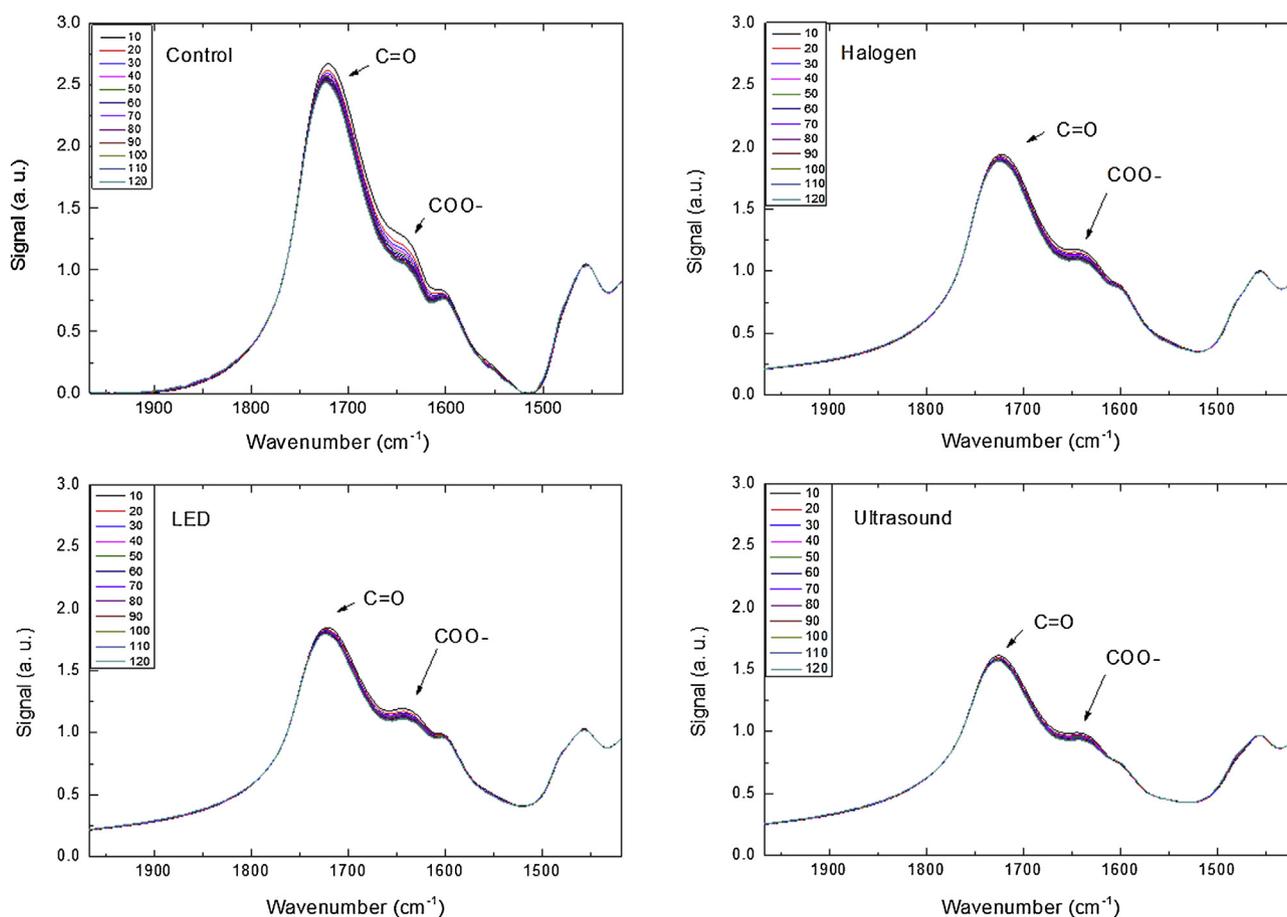
$\text{COO}^-/\text{C=O}$ bands are represented by means and standard deviations in Table 2.

The three activation modes tested had a significant influence on the reaction of ten GIC brands tested in G2, G4, G5, G6, G8, G10, G14, G16, G17 and G18. It can be observed that application of ultrasound to GICs from G3, G5, G17 and G18 reduced the stabilization time by at least 10 min. In the case of G3 and G17, the time for stabilization of chemical reactions with ultrasound decreased by more than half the time needed compared to the control group. The FTIR spectra of the dynamic setting process of the GIC in G3 submitted to different external energy sources as a function of time are shown in Fig. 1.

For G1, G7, G12 and G15, no differences were observed in reaction stabilization times after treatment with external activation sources. For G11 and G13, only the use of LED as external activation had statistical significance compared to the control

Table 2 – Means and SD values of time (in minutes) needed to stabilize the chemical bonds during the setting reaction of some conventional glass-ionomer cements available in the market.

| Group | Control | | Halogen | | LED | | Ultrasound | |
|-------|----------|---|----------|----|-----------|----|------------|----|
| 1 | 102 ± 12 | a | 125 ± 7 | b | 111 ± 9 | ab | 93 ± 12 | ab |
| 2 | 165 ± 24 | a | 87 ± 5 | b | 91 ± 8 | b | 85 ± 9 | b |
| 3 | 186 ± 11 | a | 149 ± 20 | a | 154 ± 7 | a | 61 ± 32 | b |
| 4 | 242 ± 20 | a | 136 ± 24 | b | 156 ± 5 | b | 130 ± 4 | b |
| 5 | 205 ± 19 | a | 127 ± 10 | b | 151 ± 23 | b | 125 ± 14 | b |
| 6 | 206 ± 20 | a | 155 ± 22 | b | 146 ± 10 | b | 121 ± 13 | b |
| 7 | 129 ± 10 | a | 178 ± 4 | b | 131 ± 9.6 | a | 119 ± 10 | a |
| 8 | 149 ± 22 | a | 70 ± 8 | b | 99 ± 8 | b | 99 ± 16 | b |
| 9 | 126 ± 24 | a | 61 ± 8 | b | 74 ± 7 | bc | 100 ± 10 | ac |
| 10 | 199 ± 14 | a | 106 ± 11 | bc | 87 ± 5 | b | 117 ± 10 | c |
| 11 | 146 ± 6 | a | 148 ± 16 | a | 95 ± 9 | b | 139 ± 5 | a |
| 12 | 118 ± 15 | a | 120 ± 10 | a | 140 ± 9 | a | 118 ± 19 | a |
| 13 | 109 ± 18 | a | 85 ± 10 | ab | 77 ± 12 | b | 109 ± 7 | a |
| 14 | 152 ± 11 | a | 99 ± 11 | c | 123 ± 5 | b | 120 ± 5 | b |
| 15 | 142 ± 8 | a | 136 ± 16 | a | 112.7 ± 2 | a | 150 ± 27 | a |
| 16 | 162 ± 5 | a | 140 ± 15 | ac | 114 ± 9 | b | 133 ± 11 | bc |
| 17 | 200 ± 14 | a | 120 ± 17 | c | 114 ± 13 | c | 70 ± 11 | b |
| 18 | 164 ± 6 | a | 111 ± 4 | c | 100 ± 14 | b | 111 ± 5 | bc |

ANOVA and post hoc Tukey. Different letters indicate statistically significant difference ($p < 0.05$).**Fig. 1 – FTIR spectra in the region between 1900 and 1500 cm^{-1} of the Equia Forte[®] (G3) subjected to different external energy sources as a function of time. The sample was measured up to 120 min, at intervals of 10 min. The spectra shows the dynamic setting process of glass-ionomer cement in G3 with the acid group (C=O) and carboxylate salt of the polyacid group (COO⁻) identified.**

group. The time of stabilization for G9 was influenced significantly by LED and halogen light.

4. Discussion

The null hypothesis was rejected once the application of external sources to the GICs after mixing altered the time required to stabilize the chemical bonds in the curing process of the materials.

To date, there have been few studies that characterize the setting reaction of GICs [13], and to our knowledge this is the first to demonstrate the time required for stabilization of chemical bonds after application of external activation sources.

In the present study, setting rate was evaluated from the ratio of infrared bands due to carboxylate (1637 cm^{-1}) and carbonyl (1720 cm^{-1}) respectively. In this ratio, the carboxylate band is the one forming from the original carboxylic acid groups present on the polymer, and the carbonyl group, which does not change on setting, acts as an internal reference band [13]. Using this approach, the results indicate that the use of external activation can significantly reduce the time required to stabilize the carboxylate bonds within the material. In some cases, such as G3 and G17, ultrasound application reduced the reaction time by more than half when compared to the control group. Acceleration of chemical reactions has the advantage of shorter clinical working time, reduced risk of erosion by initial exposure to saliva, and increased initial resistance of the material [14]. Clinically this is important to establish recommendations to the patient after the procedure, in order to avoid mechanical stresses for a shorter period of time and to prevent premature loss of the restoration [13]. In addition, it has implications for the clinical protocol, for example, the minimum time necessary before removal of the matrix used for insertion of the material into cavities as well as for removal of excess material.

The GICs have unique properties such as favorable adhesion to dental structures, fluoride release, as well as the coefficient of linear thermal expansion and modulus of elasticity being similar to the tooth [1]. However, they have deficiencies such as brittleness, which leads to relatively poor wear characteristics [15,16]. To overcome these limitations, resin-modified glass-ionomers have been developed, which are similar to conventional GICs in terms of good adhesion to enamel and dentin, with the additional advantage of immediate hardening by application of an external light source; this provides greater initial mechanical resistance and less sensitivity to moisture [7]. However, there are some disadvantages inherent in resin use, such as polymerization contraction, aqueous expansion, and lower long-term mechanical properties compared to conventional GICs [7]. The presence of monomer within them also compromises their biocompatibility [17].

With the objective of improving the properties of GICs, several studies on the application of external sources to accelerate the setting reaction have previously been carried out. One such study, testing the effect of conventional manipulation, heating and ultrasonic excitation, demonstrated that heating of the material decreased microleakage and improved marginal

adaptation of the restoration [18]. Another study showed an improvement in the mechanical properties of the material after ultrasonic excitation and heating [7]. Dehurtevent et al. [9], showed more significant results with these external energy sources in accelerating the setting reaction and increasing the material strength.

Although it is not yet clear in the literature how ultrasound specifically influences the mechanism of GIC setting, it is known that ultrasound generates tiny cavities in reaction mixtures, and that these cavities grow rapidly then collapse, generating large amounts of heat in very small volumes [19]. This has the effect of significantly increasing reaction rates in two-phase mixtures, such as those used in GICs. There is also the possibility that ultrasound breaks up aggregates of glass particles, increasing the available surface area, and also contributing to the increase in the setting rate [20]. The main advantage of ultrasound is that the equipment is commonly present in dental offices and in addition to reducing working time, it helps in the flow of restorative material into the prepared dental cavity. For the majority of GICs used in the present study, ultrasound significantly reduced the $\text{COO}^-/\text{C}=\text{O}$ bond stabilization time compared to conventional time, demonstrating its clinical application for restorations with this material.

In addition, excitation with ultrasound in GICs improves adhesion to the dental surface and the fluoride release [21,22]. The external application of ultrasound does not modify the chemical composition of GICs and, according to the present work, it can stabilize the reactions of the formation of the material more quickly; this overcomes the disadvantages associated with the resin-modified GICs [7] and also increases the compressive strength and surface hardness of the material [20,23].

However, standardization for the time of application of the external source is not yet established in the literature and may vary from one commercial product to another. In the present study, the application time for all external activation was standardized at 30 s. Talal et al. [10], demonstrated that ultrasonic excitation for 15 s did not affect or increase the reaction for Ketac Molar (3M ESPE, Seefeld, Germany). On the other hand, both cements tested, Fuji IX (GC Corporation, Tokyo, Japan) and Ketac Molar, showed higher rates in the COO^- formation relative to the $\text{C}=\text{O}$ band after the application of the ultrasound for at least 35 s [10]. In the present study, the results for ultrasound were not statistically different from the conventional method for G1, G7, G12 and G13, possibly due to the time of application of the external source may not have been sufficient to increase the reactions of these materials.

Besides ultrasound application, heating of GICs has been proposed with the objective of accelerating the setting and improving the quality of the material. The mechanism of effect of the heating process is based on the fact that high temperatures can increase the powder/liquid ratio by evaporating the liquid, thus improving the mechanical properties of the GIC [24]. Thermal transfer leads to an increase in ionic mobility at the initial phase of the material reaction, reducing viscosity and accelerating the setting reaction [25]. The variations in the behavior of the GICs after treatment with the external activation sources can be attributed to differences in

the composition of the materials, as well as to the differences in porosity, hydrophilicity and thermal properties.

The influence of heating the GICs with LED for 120 s and ultrasonic application for 55 s was tested by Dionysopoulos et al. [14] for the GIC materials EquiaFil (GC Corporation, Tokyo, Japan), FX-II (Shofu, Kyoto, Japan) and AHfil (Advanced Health Ltd, Tonbridge, England). The results revealed an increase in surface hardness, indicating that LED and ultrasound treatment may be useful for restorations with GIC to reach the initial mechanical resistance more quickly, thus decreasing the procedure time.

Gorseta et al. [18], tested the use of a standard curing light with halogen lamp for 40 s to heat the material in class V cavities. They observed that the heating of GIC during the setting process reduced microleakage, improved marginal adaptation and adhesion in enamel, suggesting that this procedure is suitable for clinical practice [18]. On the other hand, Kleverlaan et al. [7], evaluated the mechanical properties of different GICs submitted to external activation sources such as ultrasound for 45 s and heating with an external source at 70 °C for 5 min. They observed a clear relation between the increase of the temperature of the sample and the resistance to material compression, concluding that activation may increase the survival rate of GIC restorations.

The use of external sources may suggest heating of the material leading to concerns regarding effects on the pulp. However, Gavic et al. [26] tested heat transfer properties of modern restorative glass-ionomers and measured the temperature changes at three different depths (2, 3 and 4 mm) after cure times of 20, 40 and 60 s using three different light emitting diode cure lamps designed for clinical use. The study demonstrated that because the thermal conductivity of glass-ionomers is low, temperature rises at 4 mm depths were much lower than at 2 mm. At no time did the temperature rise sufficiently to cause concern about potential damage to the pulp [14]. Thus, application of the external sources used in this study are safe procedures to be included in clinical protocols, as the temperature in GICs does not increase sufficiently to cause pulp problems.

Another point to be considered is that GIC tends to set faster than pure GIC when in contact with dentin, due the increased presence of minerals available for the reaction, which consumes the carboxyl groups of the cement more rapidly. This was described previously in the literature [13] and suggests that all the GICs tested in the present study could present even faster chemical bond stabilization time in contact with dentin when external energy sources are applied.

Studies such as this, testing greater times of ultrasonic activation, LED and halogen light, are necessary to identify the minimum time required to accelerate the setting reaction of materials where no significant differences were found in relation to the control group.

5. Conclusion

The use of activation sources may reduce the time required for the stabilization of chemical bonds during the setting reaction of some conventional restorative glass ionomer cements available on the market, which may be clinically advantageous in

allowing the material to harden in a shorter period of time. New studies evaluating the influence of the use of external sources of activation on the mechanical properties of GICs could prove the clinical relevance of the use of this protocol.

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