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In Vitro Evaluation of the Stabilization Time of Chemical Bonds During Setting Reaction and Microhardness of Preheated Glass-Ionomer Cements

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Clinical Relevance

The preheating of glass-ionomer cements can influence the performance of the restoration, potentially maintaining it in the mouth for longer.

SUMMARY

Objectives: To evaluate the effect of preheating glass-ionomer cement (GIC) restorative materials on stabilization time (ST) of their metal carboxylate bonds and on microhardness.

Methods and Materials: Two conventional high-viscosity GICs, Ketac Universal (3M ESPE) and Equia Forte (GC), were evaluated. The thermographic camera was used to measure the temperature inside the glass-ionomer cement capsules before and after

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heating. The preheating of capsules was performed at 54°C for 30 seconds in a commercial device. Characterization of ST in the GICs was determined by Fourier Transform Infrared (FTIR) spectroscopy. For this, 10 samples of each material were prepared, five in the non-preheated group (control) and five with preheating. FTIR spectra were obtained 10 minutes after mixing (control group) or after heating and then every 10 minutes for 120 minutes. For the microhardness test, 20 cylindrical specimens (3 mm height x 6 mm diameter) were prepared for each material (10 preheated, 10 control). The microhardness was determined at three time intervals: 10 minutes after mixing, after the ST as detected through the FTIR part of the study, and after one week. Knoop microhardness was assessed using a diamond indenter with a 25 g load and 15 seconds dwell time.

Results: Ketac Universal showed an increase in temperatures of 15.7°C for powder and 3.6°C for liquid, while Equia Forte showed 16.4°C for powder and 8.5°C for liquid. FTIR spectra indicated that preheating reduced the ST for Equia Forte but increased it for Ketac Universal. Preheating increased the initial microhardness (T_1) of Equia Forte. With maturation over one week, it was observed that preheating significantly improved the microhardness of both materials compared with the control specimens.

Conclusion: Preheating influenced the ST and the microhardness of Ketac Universal and Equia Forte. The ST and microhardness of Ketac Universal increased after seven days, whereas Equia Forte showed a reduced ST and increased microhardness from the outset.

INTRODUCTION

Glass-ionomer cement (GIC) is a widely used material due to its properties such as biocompatibility in the mouth, fluoride release, ability to promote remineralization of dental structures,[1] chemical adhesion to tooth,[2] and bioactivity without shrinkage during the setting reaction.[3] It is a material that hardens following an acid-base reaction between fluoroaluminosilicate glass powder and an aqueous solution of polyacid.[4] According to the ISO standard (ISO 9917-1),[5] restorative GICs have setting times in the range of 1.5 to 6 minutes. The hardening reaction occurs in a short period, and despite this, it does not mean that the material has reached complete chemical stability.[2] After its initial hardening, the GIC material continues to undergo changes (maturation process) for some time.[4,6] A recent study about the dynamics of the setting process of GIC demonstrated that the time required for Ketac Molar Easy Mix (3M ESPE, Sumaré, São Paulo - Brazil) to acquire stabilization time of its chemical bonds takes up to 150 minutes.[2] The manufacturer of this material states that the setting time is only five minutes. The setting time is different from the time to reach chemical bond stability (stabilization time), as evaluated by FTIR spectroscopy.[2,7]

Contemporary GICs have improved over previous versions and have a wide range of uses.[8] Modern glass-ionomers for clinical use, the so-called high-viscosity materials, are formulated with improved particle size and particle size distribution glasses, which have more rapid set than previous types, and result in mechanically strong and durable restorations. However, glass-ionomer cements do have limitations, including their physical resistance, sensitivity to humidity, opacity, and relatively slow setting reaction.[1,4]

In an attempt to reduce these limitations, some authors have suggested applying external energy such as ultrasound, light emitting diode (LED) light, or hot metal elements after mixing the material to speed up the setting reaction and improve the mechanical properties of the set GIC.[7,9-13] Preheating has been used successfully with composite resin materials to improve the mechanical and physical properties,[14,15] but only a few studies evaluated heating before mixing the GIC.[10,16] Currently, it is not clear if there is an effect of heating before mixing GIC restorative materials. Although GICs and composite resins show different behavior due to their individual composition, heating GICs before mixing may similarly improve the mechanical and physical properties.

For composite resins, preheating increases the monomer's degree of conversion by reducing the viscosity of the material, increasing the microhardness and flowability,[17] increasing diametral tensile strength,[18] and improving marginal adaptation, as well as reducing microleakage.[19] Previous researchers have found that external energy applied to hand-mixed GIC after mixing potentially increases benefits, after applying thermo-light curing for 60 seconds,[13] such as increased superficial microhardness up to a depth of 4 mm and reduction of crack propagation and working time,[9,10] as well as an improvement in marginal microleakage.[11]

The aim of this study was to evaluate the influence of preheating (before mixing the material) on stabilization time of the metal carboxylate bonds and surface microhardness in capsulated restorative glass-ionomer cement materials. The null hypotheses tested

were 1) the application of extrinsic thermal energy does not modify the time needed to stabilize the metal carboxylate bonds during the setting reaction of restorative glass-ionomer cement materials, and 2) the preheating of glass-ionomer does not change the physical property of surface microhardness.

METHODS AND MATERIALS

This was an *in vitro* study to assess the effect of preheating on two restorative GIC materials on the dynamics of their setting reaction, as evaluated by infrared spectroscopy, and on the microhardness, as assessed by the Knoop hardness test. Preheating capsules was performed at 54°C for 30 seconds. For each of these analyses methods (spectroscopy and microhardness), 10 and 20 samples, respectively, of each material were made, half without preheating (control) and half with preheating the material. The GICs, batch numbers, and manufacturers of the products evaluated in this study are listed in Table 1.

Measurement of Glass-Ionomer Cement Temperature Inside the Capsules

To determine the actual or eventual temperature of the glass-ionomer cement material inside the capsules when preheated, an additional analysis was conducted. A thermographic camera (ImageIR 7300, InfraTec, Dresden, Germany), which operates in the wavelength range from 2 to 5.7 μm, was used; this was mounted upright and the sample positioned in its focal lens at 25 cm. The temperature measurements of this camera range from -40°C to 300°C, with a resolution of ±0.002°C. Measurements were performed at room temperature, which was around 22°C. The samples were placed on a waterproof paper (SDI, Victoria, Australia) suitable for GIC handling. First, three intact capsules were preheated and the internal thermographic images of both precursors, powder and liquid, were collected. After that, three new capsules were selected for the GIC preparation. They were heated for 30 seconds and immediately transferred to the mechanical agitator for mixing. Then, immediately after removing the capsule, the thermal image of each GIC was collected, using the IRBIS 3 software (Infratec). The same software calculated the average temperature variations of the samples (n=3) of each material.

Sample Preparation

Samples of each material were prepared and divided into two equal groups: one group with preheating (test group) and one group without preheating (control). For preheating the GICs, the capsule tips of the materials were slightly modified to enable them to be fitted into a heating device (Figure 1) (Calset; AdDent, Danbury, CT, USA) using a tungsten carbide bur n°1251 (American Burrs, Pedra Branca, Palhoça, Brazil) in a slow-speed handpiece. The material capsules were either preheated to 54°C for 30 seconds prior to mixing or not preheated (control). The mixing of each capsule was conducted according to each manufacturer's instructions for both groups.

For the Fourier Transform Infrared (FTIR) spectroscopy analysis, 10 samples of each material were prepared and divided into two groups: five for the non-preheated group (control) and five for the preheated group (test group). For preparation of these samples, each mixed material was placed between two polyester tapes, pressed between two plates of glass, and loaded with a constant force of 0.4 N for 30 seconds generated vertically to the specimen via the upper glass plate. After the first five minutes of setting, the samples of GIC were prepared for measurements. The GICs were ground in a mortar using an agate pestle. After that, they were mixed with potassium bromide (KBr) powder and pressed with a manual hydraulic press (Specac, Orpington, Kent, UK) under 10 SI (tons) for two minutes to obtain pellets for analysis.[7]

Following the protocol of Xie and others[20] for microhardness measurement, 20 cylindrical specimens

Table 1: *Brand Names, Batch Numbers, and Manufacturers of the Products*

Material	Chemical Composition	Batch n°	Manufacturer
Ketac Universal	Water 40%-60% Acrylic–maleic acid copolymer 30%-50% Tartaric acid 1%-10% Benzoic acid <0.2% Glass oxide >95%	3817763	3M ESPE, Seefeld, Germany
Equia Forte	Polybasic carboxylic acid 5%-10% Iron oxide (III) <0.5%	1706191	GC, Europe NV, Leuven, Belgium

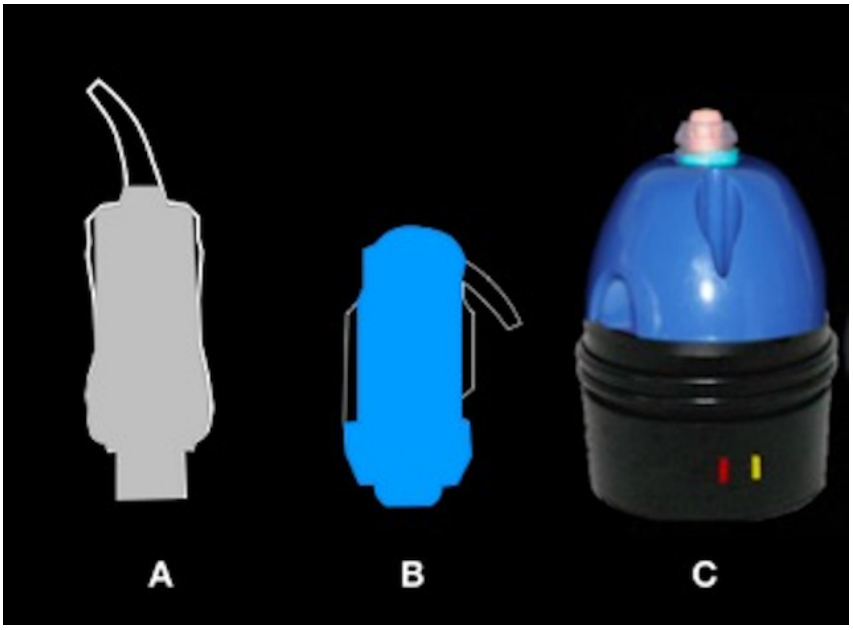

Figure 1. *(A and B) Schematic drawings showing the original capsules and the slightly modified capsules overlapped. The filled area in each drawing represents the final modified capsule (A: Equia Forte, GC Corp; B: Ketac Universal, 3M ESPE). (C) The modified capsule inside the heating device.*

of each material were prepared using a split steel mold with internal dimensions of 3 mm height and 6 mm diameter. To prepare each specimen, the mixed GIC was placed into the mold and compressed with polyester tapes on both sides of the mold with a screw clamp for 10 minutes. Control (non-preheated) specimens (n=10) of each material were mixed and fabricated at room temperature, according to the manufacturer's instructions. In the test group (preheated) specimens (n=10) of each material, the capsules were preheated to 54°C in the heating unit as in the previous section for 30 seconds prior to mixing according to the manufacturer's instructions.

Analysis of the Stabilization Time of the Chemical Bonds by Fourier Transform Infrared Spectroscopy

The characterization of the molecular stabilization time (ST) of GICs was determined with a FTIR research spectrometer (Vertex 70v, Bruker Optik GmbH, Ettlingen, Germany). To produce pellets (n=5) for this analysis, 0.002 g of GICs was diluted with 0.198 g potassium bromide (KBr), weighed with an analytical scale (GH-202, A&D Weighing, San José, CA, USA), and compressed with a manual hydraulic press (Specac, Orpington) under 10 SI (tons) for two minutes. The spectra of the KBr-sample pellets were collected by making an average of 128 scans with a 4 cm^{-1} resolution in the spectral range of 4000-400 cm^{-1}. FTIR spectra were collected 10 minutes after mixing the GIC in both preheated and non-preheated samples and again every 10 minutes for 120 minutes thereafter.

The samples were kept in the FTIR spectrometer, maintaining the vacuum condition throughout the whole 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, following the same methodology described by de Oliveira and others.[7] Analysis was performed by the variation of the ratio band intensities as a function of time. The fitting was done using an exponential decay-type function written as (Equation 1):

$$y(t) = y_0 + y_1 \, e^{(-t/\tau)}$$

with y_0 being the area of the bands after the material reached stabilization, y_1 the amplitude of the area variation, t the instant times of the measurements, and τ the characteristic decay time representing the instant t when the area of the bands decay to 1/e (approximately 37% of y_1 value). In this type of mathematical analysis, the curve shape tends to a constant value with time (approximately five times the value of τ), meaning that 99.95% of the decay variation has been reached, and

the material can be considered stabilized in terms of the changes of the chemical bond.[2] This analysis was performed to observe the dynamics of possible changes in the chemical bonds of the restorative material after preheating.

Microhardness Measurement: Knoop Microhardness Test

For each specimen, the microhardness test was performed at three time intervals: 10 minutes after mixing, after the stabilization time of chemical bonds as detected through the FTIR spectroscopy (Table 2), and after one week. During this period, the specimens were stored in a dry, closed environment at a room temperature of 22°C.

The microhardness was determined using a microhardness tester (FM-ARS 900, FUTURE-TECH Corp, Tokyo, Japan). The Knoop hardness test was performed using a diamond indenter with a 25 g load and 15 seconds dwell time. Three measurements were made on the surface of each of the 10 specimens for the preheated and non-preheated materials investigated.

Statistical Analysis

Data collected from FTIR spectra were submitted to Shapiro-Wilk normality, Levene homogeneity, and Student *t*-test at the 5% significance level. To analyze the influence of preheating on microhardness by the time (initial, ST, and seven days), the repeated measures factorial one-way analysis of variance (ANOVA) and *post hoc* comparison of means (Tukey test) were performed to determine any significance over time (p=0.05). To analyze the influence of preheating on GIC, the *t*-test was applied. The statistical analysis was carried out with SigmaPlot 12.0 (Systat Software Inc, San Jose, California, USA) software.

RESULTS

Figure 2 shows the thermal images obtained with the thermographic camera. On the left side and in the middle are the images for room or ambient temperature in the capsule and after preheating at 54°C for 30 seconds, respectively, for both powder and liquid precursors positioned inside the capsules. The images for the capsule after preheating and mixing are on the right side. Ketac Universal (Figure 2A) showed an increase in temperature from the ambient temperature in the capsule of 15.7°C for powder and 3.6°C for liquid, after 30s of preheating, while Equia Forte (Figure 2B) showed increases of 16.4°C for powder and 8.5°C for liquid. On the right, after preheating and mixing, the temperature rise in the two GICs in relation to room temperature was 7.3°C for Ketac Universal and 5.7°C for Equia Forte.

FTIR Spectroscopy

The FTIR results (Figure 3) showed that preheating the capsules of Ketac Universal increased the time of stabilization of chemical bonds and reduced the ST for Equia Forte (Table 2).

Microhardness Measurement: Knoop Microhardness Test

Based on the stabilization time of chemical bonds, the microhardness test was performed on the materials with and without preheating, at three different time intervals: 10 minutes after mixing (t_1), after the

Table 2: Mean Stabilization Times (in Minutes with Standard Deviations) of Chemical Bonds Obtained with Fourier Transform Infrared (FTIR) Spectroscopy

Groups	Time in Minutes			
	Room Temperature (22°C)		Preheated	
	τ	Stabilization Time (ST) (5×τ)[a]	τ	Stabilization Time (ST) (5×τ)[a]
Ketac Universal	26.26±5.49	131.30±27.46 A	35.55±6.02	177.75±30.08 B
Equia Forte	29.61±2.23	148.08±11.14 A	23.79±3.14	118.99±15.72 B

Abbreviations: ST, stabilization time.
[a]Means followed by the same letter comparing the columns (materials preheated and non-preheated) are not statistically different (p>0.05). n = 5 specimens/group. τ = the time characteristic of the chemical reaction, and 5x this time was considered the time of stabilization, 99.95% of the reaction occurred in that period.

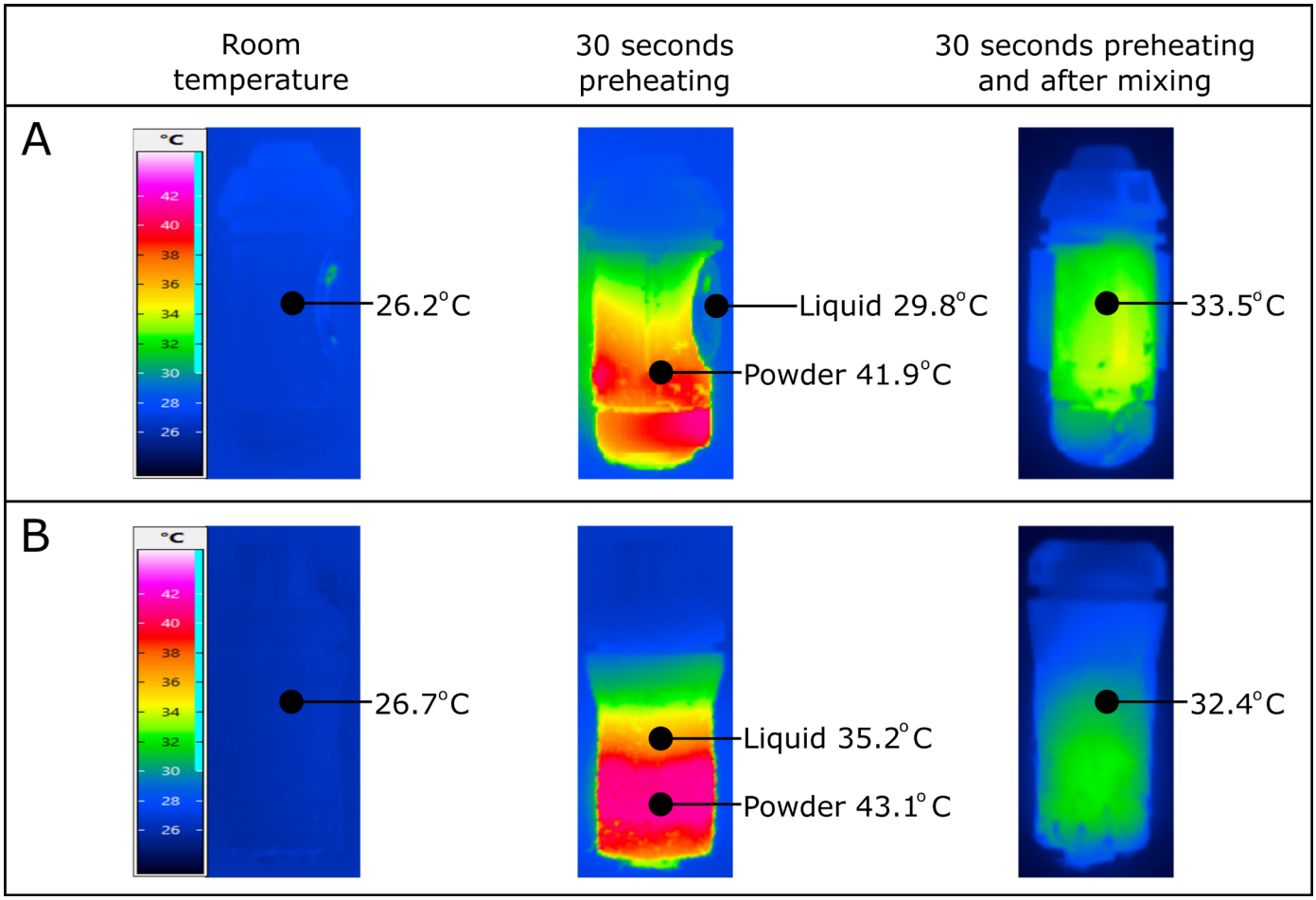

Figure 2. *Thermographic images of the precursors, powder, and liquid at room temperature (left) and 30 seconds after preheating at 54°C (middle). Images on the right are the heated and mixed GICs, Ketac Universal (A) and Equia Forte (B).*

stabilization time of chemical bonds (t_s), and after seven days (t_7). The means and standard deviation values of the microhardness test are shown in Tables 3 and 4.

Analyzing the microhardness values over time (Table 3), for Equia Forte non-preheated, there was a significant increase in microhardness between t_1 to t_s, and t_1 to t_7, but not between t_s to t_7. For each GIC, preheating showed a significant difference in microhardness between all of the time intervals.

It was observed that preheating increases significantly the microhardness of Equia Forte at t_1 ($p<0.05$; Table 4). With maturation after one week, it was observed that the microhardness increased significantly comparing non-preheated with preheated Ketac Universal ($p<0.05$; Table 4) and Equia Forte ($p<0.05$; Table 4).

DISCUSSION

The null hypotheses tested were rejected because the application of heat before mixing modified the time of stabilization of the chemical bonds and the surface microhardness of the GICs tested. With the spectroscopy analysis, a significant increase in the stabilization time of the chemical bonds for Ketac Universal and a significant reduction in the ST of Equia Forte were observed. It was also observed that preheating significantly increased the microhardness of the two materials. Menezes-Silva and others[21] reported that the longer the stabilization time of the chemical bonds the greater the mechanical properties of the material. In the present study, it was observed that preheating Ketac Universal increased the ST by approximately 40 minutes, with a significant increase in surface microhardness over time.

The differences between the two materials tested can be attributed to the complexity of the setting reaction. It was initially previously shown that the setting of glass-ionomer cements involved not only neutralization of the polyacid component but also reaction of inorganic species arising from the ion-depleted glass particles.[22] Subsequent studies suggested that the key components were phosphate species.[23] Preheating the cement

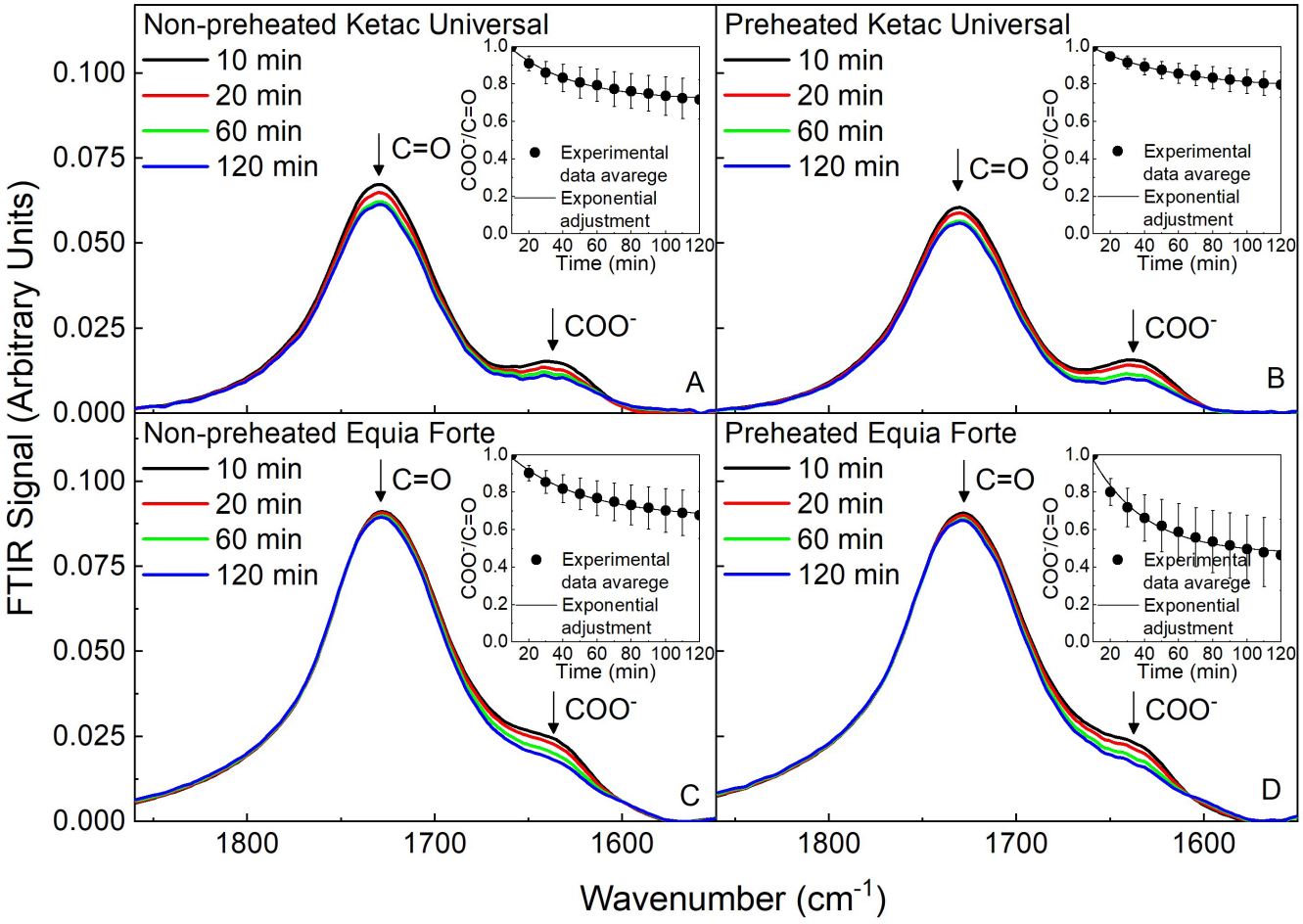

Figure 3. *Fourier Transform Infrared spectra: (A) non-preheated Ketac Universal (control), (B) preheated Ketac Universal, (C) non-preheated Equia Forte (control), and (D) preheated Equia Forte. The inserts show the changes of the COO-/C=O ratio for the samples (n=5) as a function of time adjusted with the exponential function (Equation 1).*

components is likely to affect both possible setting reactions with two possible consequences. Depending on the relative speeds, both reactions may speed up and result in increased rates of formation of both the ionically crosslinked polyacid chains and the inorganic network. Alternatively, if the inorganic network forms too quickly, this will reduce the mobility of polymer chains and could reduce the rate at which the acid functional groups react to form carboxylate salts. It is this latter reaction that was observed using FTIR and on which the calculation of stabilization time was based. Consequently, it is possible that either an increase or decrease in stabilization time may occur with heat; also, we would not expect these changes to be correlated with variations in hardness determined mechanically.

Although preheating has different effects on the stabilization time of the chemical bonds within GICs, the heating is believed to increase the rate of diffusion of ions, accelerating the overall setting reaction, with consequent reduction in working time and reaction time.[19] Therefore, GIC heating would be beneficial for improving its initial properties, when the material is more susceptible. It is suggested that the application of heat to GIC, especially in the hand-mixed versions, evaporates part of the water in the cement matrix and accelerates the chemical reaction of the material.[24] However, it is important to point out that the hardening time (setting time) of the material presented by the manufacturer is different from the time to reach chemical stabilization.[21] There are certainly advantages of a shorter working time (i.e., reduction of contamination with saliva exposure and increase of the initial resistance of the material).[25] Although it has been observed that GIC loses mass when it is heated in a dry environment after mixing, at the same time, there is heat absorption that generates expansion of the material.[26] Nevertheless, heating up to 50°C promotes minimal dimensional changes in the material, keeping in the volume in balance.[27] With the application of external energy and consequent temperature rise in the

Table 3: *Means and Standard Deviations of the Surface Microhardness (Kg/mm²) Values of the Groups Studied at Three Different Time Intervals*

Group	Initial (at t_1)[a]	At stabilization time (t_s)[a]	At 7 days (t_7)[a]
Ketac Universal	36.0±10.4 A	64.3± 7.2 B	74.3±7.8 C
Preheated Ketac Universal	39.9±7.2 A	69.0±7.6 B	87.6±9.6 C
Equia Forte	32.1±6.7 A	61.1±6.3 B	68.5±7.2 B
Preheated Equia Forte	37.8± 3.9 A	58.6±9.1 B	77.0±7.2 C

[a]*Means followed by the same letter (i.e., time: initial, after stabilization, and after seven days of each material) are not statistically different (*p*<0.05). n=10 specimens/group.*

Table 4: *Means and Standard Deviations of the Surface Microhardness (Kg/mm²) Values of Materials Preheated or Not Preheated at Three Different Time Intervals*

Group	Initial (at t_1)	*p*-Value	At stabilization time (t_s)	*p*-Value	At 7 days (t_7)	*p*- Value
Ketac Universal	36.0±10.4	0.464	64.3±7.2	0.171	74.3±7.8	0.003[a]
Preheated Ketac Universal	39.9±7.2		69.0±7.6		87.6±9.6	
Equia Forte	32.1±6.7	0.04[a]	61.1±6.3	0.504	68.5±7.2	0.017[a]
Preheated Equia Forte	37.8±3.9		58.6±9.1		77.0±7.2	

[a]*Statistical significance (*p*>0.05).*

GIC after mixing, weak water bonds are the first to be lost, promoting mass loss and small structural changes, which can be reversed after contact with water.[27]

The analysis of spectra data showed a difference in the intensity of the peaks with no displacement (Figure 3). This suggests that the preheating promotes a modification in the time of the chemical reaction of the cement, but it was not possible to observe the presence of damage or changes to its molecular structure. GIC formulations consist of glass particles with complex structure and several components.[28] Any change in the proportion of these components as well as the polyacid concentration and the size and shape of the glass particles may influence the final reaction result.[20] de Oliveira and others[7] suggest that the variation in the behavior of GIC after heating can be attributed to the different compositions of the material, as well as porosity, hydrophilicity, and thermal properties.

The first few hours after handling and inserting the material into the cavity are the most critical. The material is more vulnerable to moisture, which influences the susceptibility to fracture and initial wear.[2] Clinically, a protective agent is used to avoid the problems of syneresis and imbibition. This aspect was not investigated in this work because the protective material could interfere in the hardening process of the GIC and also may present additional FTIR bands that could overlap with those from the GIC. Furthermore, for the FTIR test, protection was not possible because the reading was performed during the hardening of the material. For the microhardness test, the protection was also not performed because it was intended to maintain the same condition in both tests. To prevent dehydration during the microhardness test, the samples were maintained dry in a small, closed environment controlling the dehydration of the material, with only the water present in the liquid allowing the material to harden after seven days. Maintaining the water balance of the cement is important. The specimens were maintained for 10 minutes inside the mold, without contact with oxygen. In cement, the water undergoes some kind of interaction with other chemical species present and is strongly linked. There seem to be several ways in which water is attached to ionomer cements over time.[4] One is the hydration of the cations released by the glass; they are all present in the anhydrous state, but they are capable of strongly "coordinating" the water and will form highly hydrated ions under

appropriate conditions.[29] It seems like this occurs inside the cement and that the hydrated ions formed are stable and capable of retaining their water molecules, even under desiccation conditions.[4]

Clinically, the faster the material hardens, the better its initial mechanical properties will be and the less susceptible it will be to the early masticatory load.[30] However, recently, it has been demonstrated that the slower the ST of GIC, the more the chemical bonds will be formed, consequently improving the final mechanical properties of the material.[21] Therefore, with the reduction of working time and setting of GIC, the restorations are expected to have increased durability, and for patients with difficult handling, GICs may be the material of choice. Some authors also found an increase in the microhardness of GICs after applying external energy in the form of heat after mixing and attributed this to the composition of the material.[9,12,31] O'Brien and others[10] observed that the preheating of capsules prior to mixing had a greater influence on the microhardness depth of the GIC compared with the application of heat with ultrasound and LED light after mixing. Unlike composite resins, the viscosity of GICs apparently increased with preheating, which could impair the adaptation of the material to the cavity and encourage the inclusion of air voids.

To date there is little work[10,16] on preheated GICs, making it difficult to compare the data. Most of the studies involved warming the materials after mixing. In the present study, the time required for preheating was established from a pilot study conducted by the authors. It was observed that 30 seconds was sufficient to promote changes in the stabilization time of the chemical bonds of the material and allow a sufficient working time. With time greater than 30 seconds, the material became very viscous and was difficult to express from the capsules.

Analyzing the results of the thermographic camera, it was observed that the material inside the capsule did not reach the temperature recommended by the heating device. This may have occurred because the capsule prevented the transmission of heat. The temperature of 54°C was determined from the literature,[14] and it was observed that there is significant improvement of the superficial initial hardness when GIC is heated to a maximum of 60°C. Previous research on external energy applied with LED light showed that the light temperature reaches a maximum of 60°C after 60 seconds of activation. As GIC has low thermal conductivity, heating is considered a safe procedure.[32] Therefore, training and working rapidly are necessary so that the material does not lose heat before the restorative procedure is carried out. Also, care is necessary to avoid bubbles and the formation of gaps. The literature shows that composite resin loses 50% of temperature approximately two minutes after preheating.[33] This may also occur with GICs.

Commercial GIC capsules are not uniform in construction and contain a volume of air in the powder region of the capsule. Furthermore, the location of the GIC liquid is different in each product. The Equia Forte liquid is in a more central region of the capsule, whereas the Ketac Universal capsule has the liquid in a small compartment more at the capsule periphery. Thus, there are unequal internal heating patterns between the two capsulated materials, and this was demonstrated with the thermographic camera data. The thermographic camera was found to be a useful method for measuring the temperature changes in this kind of research.

Preheating is an easy method that could be used in a dental office. The devices used for preheating composites could also be used with GIC capsules with slight modification. This may prompt manufacturers to modify the design of their GIC capsules to fit into standard preheating devices or, alternatively, to produce devices specifically for their capsulated materials.

A huge benefit of using GIC as a restorative material is its ability to release fluoride. If preheating will improve the mechanical properties and the ability to release fluoride, the preheating technique could be adopted to improve the material properties. Future research should be conducted to further clarify the benefits of preheating GIC.

CONCLUSIONS

Preheating influences the stabilization time of the chemical bonds and increases the microhardness of the GICs tested. For the microhardness test, the heating influenced the maturation of the cements in the stabilization time and in seven days. These results demonstrate that preheating is a promising technique.

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Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in

any product, service, and/or company that is presented in this article.

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