Abstract

**Objectives**: To examine the effect of energy application on microhardness of glass-ionomer cement (GIC), and the effect of the temperature applied on pulp temperature, in vitro and in vivo.

**Methods**: Discs of EQUIA (GC Co. Japan) were examined for microhardness using Vickers indentations after heat application of 50 or 60°C for 30 or 60 seconds using light curing devices or a specific heating device, and compared to self setting GIC. The measurements were performed after 30, 60 minutes and after 5 days. Heat was applied to GIC occlusal restorations in deciduous and permanent teeth both in vitro and in vivo and the effect on pulp temperature was measured using a thermo-coupling device.

**Results**: The best microhardness results were observed using a specific heating device at 50°C for 30 or 60 seconds, and the differences to self setting material after 60 minutes were statistically significant. After 5 days the differences in microhardness were non-significant. Temperature of 60°C caused the pulp temperature to rise with more than the accepted 5.5°C and may cause irreversible damage to the pulp. Similar pulpal temperature changes were observed in vitro and in vivo in deciduous and permanent teeth.

**Significance**: The application of heat energy to GIC using a heating device improved the surface microhardness significantly during the first 60 minutes. The temperature to be used is around 50°C for 30 or 60 seconds in order not to cause irreversible damage to the pulp.

**Keywords**: Glass-Ionomer cements; Microhardness; Energy application; Pulp temperature

**Introduction**

Glass ionomer cement (GIC) is a dental restorative material used for dental fillings and luting cements. These materials are based on the reaction of silicate glass powder and polyalkenoic acid. In a paper that reports the consensus of the American Association of Pediatric Dentistry meeting in 2014 on the use of GIC in restorative dentistry it was concluded that it can be used in primary dentition for Cl I and II restorations and in permanent dentition in small, minimally invasive Cl I restorations [1]. When minimal intervention approach like ART technique was compared to traditional occlusal amalgam restoration in permanent molars, GIC showed similar success rates as amalgam after six years (72.3 and 72.6%). Restoration fracture and marginal defects were the most common causes for failure in the amalgam group compared to loss of material in the GIC group [2]. In primary molars three years follow-up showed no significant differences between amalgam and GIC in single and multiple-surface restorations [3]. GIC restorations affect mineral components and remineralization of adjacent enamel and dentin [4-5]. The main problem with GIC for restorative treatment is its inferior resistance to attrition during the first weeks compared to composite materials and amalgam.

As early as 2001, it was shown in vitro that thermal curing of GIC during setting time improved its resistance to attrition [6]. The hypothesis was that since glass-ionomer setting process is an exothermic reaction, energy application will shorten the setting time and will improve the final reaction, resulting in better resistance to dental surroundings. Thermal curing of GICs is simply an alternative to accelerate the GIC reaction for prevention of harmful effects of oral liquids and occlusal contact on the outer surface of GICs during its initial setting period. The application of energy in the form of ultrasonic vibrations or heat using LED devices, improved the GIC strength, especially during the early setting time [7], improved the shear bond strength [8], decreased micro leakage and improved marginal adaptation [9], improved biaxial flexural strength [10], improved microhardness and surface mechanical properties [11,12]. The main concern when using high temperature is that application of heat on GIC restorations may affect the temperature of the pulp so that pulp necrosis may occur. The intensity of the energy applied and the longevity of application is crucial to pulp health. An in vivo study on monkeys' teeth showed that if intra pulpal temperature is increased by 5.6°C and the heat source was allowed to rest longer than 10 seconds, 15 percent of the teeth showed pulp necrosis. If the intra pulpal temperature is increased by 11.1°C, 60% of the teeth showed pulp necrosis [13]. Based on these results, the accepted maximal pulpal temperature rise during dental treatment was determined as 5.5°C. In vitro use of LED or conventional quartz-tungsten-halogen light curing devices for polymerization of composite restorative materials caused pulpal temperature increase that was related to the time of exposure and residual dentin thickness between the restorative material and the pulp ceiling [14-16]. The lowest results were recorded for incrementally cured composite resin and when bulk filling was used the increase in pulp temperature was more than the accepted 5.5°C [17,18].

This research was designed in order to answer several questions

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regarding the application of heat energy to GIC. A. What is the best temperature and time application to promote the setting of GIC during the critical first hour and after five days? B. What is the effect of different temperatures, time of application and residual dentin thickness on pulpal temperature in premolars and deciduous molars in vitro? C. Can the results obtained for deciduous molars in vitro be compared to in vivo changes in pulpal temperature after heat application to GIC?

Materials and Methods

- Effect of different temperature and time application on GIC microhardness- Discs of reinforced glass-ionomer (EQUIA, GC Co. Japan, LOT 140513A, Exp 2016-05, Color A2) with a diameter of 1.5 cm and 2 mm thickness were examined for microhardness using Vickers indents with Duramin 5 device (Struers Co., Denmark). Six groups of five discs in each group were analyzed: 1. Self setting, 2. Heat application using LED curing device (D-light DUO, RF, Switzerland, mean temperature 52°C) for 60 seconds, 3. Heat application using heating device (Model Wax, Carlo di-Giorgi, Italy, art.738/00) with mean temperature of 50°C for 30 seconds, 4. Heating device, 50°C for 60 seconds, 5. Heating device, 60°C for 30 seconds, 6. Heating device, 60°C for 60 seconds. On each disc 5 indentations were performed (load 981.2 mN and press time 10 seconds) after 30 minutes (groups 1, 2 and 5) and 60 minutes (all 6 groups). Groups 1 and 2 were also examined after 5 days of storage in water. Two discs from groups 1, 2 and 3 were analyzed under SEM at x1000 enlargement to understand the effect of energy on GIC setting (Figure 1).

- In vitro examination of changes in pulpal temperature during heat application on occlusal EQUIA restorations- Intact 10 deciduous second molars and 20 premolars extracted for orthodontic reason were prepared. On the occlusal surface a Class I cavity was performed using 330 carbide bur under copious amount of water and air spray. The pulp chamber was exposed from the apical direction. The residual dentin thickness was measured using a metal thickness measuring device (Leon dental, Germany), so that 2 mm or 1 mm of residual dentin remained. A thermo-coupling device (SmartMeter, Novus) was placed in close proximity to the pulp ceiling. The apical opening was sealed using IRM. The cavity was filled with EQUIA and heat was applied using two LED light curing devices, with a mean starting temperature of 50°C (Ledex, Dentmate) and 58°C (Secura Light, Silmet) for 60 seconds. The maximal increase in pulpal temperature was recorded (Figure 2).

- In vivo examination of changes in pulpal temperature during heat application to occlusal EQUIA restorations. Informed consent was obtained from the patient parents. 4 intact first deciduous molars, from a 8 years old boy, programmed for extraction due to orthodontic reason, were used. The comprehensive dental treatment, including the extractions, was performed under general anesthesia due to behavioral problems. The teeth were isolated using rubber dam. The pulp of each tooth was exposed from the Buccal surface and the thermo-coupling device was inserted in close proximity to the pulp ceiling. An occlusal cavity was performed using a 330 carbide bur to a depth of 1.5 mm. The cavity was filled with...
EQUIA and heat was applied using the heating device (50°C on the right upper and lower molars and 60°C on the left side upper and lower molars) for 60 seconds. The changes in pulp temperature were measured after 30 and 60 seconds (Figure 3).

Statistical analyses using SPSS students t-test between groups of microhardness results were performed with α=0.05.

**Results**

Microhardness of EQUIA after 30 and 60 min of self-setting vs energy application. (Table 1) summarizes the mean Vickers microhardness (MV) observed regarding the temperature application and time length after 30 or 60 minutes. After 30 minutes the effect of energy application using Light curing device or heating device increased the GIC surface microhardness by 30% and the differences were statistically significant (P<0.05). After 60 minutes the best results were observed for the use of heating device with 50°C for 30 seconds and it increased the GIC surface microhardness by 56%. There were significant differences between three groups: a. the self setting, b. the light curing device and the heating device using 60°C and c. the heating device using 50°C. After 5 days the difference between self setting and energy application using light curing device were only 8% with no statistically significance. The SEM analyses of the surfaces showed small inorganic particles and a high number of cracks in the self setting GIC, an increase in size and number of inorganic particles and reduced number of cracks in light cured energy application to GIC and no cracks with all the surface filled with large inorganic particles in GIC with energy application with the heating device at 50°C for 30 seconds.

(Table 2) shows *in vitro* the temperature increase in the pulp chamber after heat application from 2 light curing units (50°C or 58°C) on GIC surface in upper first premolars and second deciduous molars, with residual dentin of 1 or 2 mm. When using 50°C the increase of pulp temperature in both permanent and deciduous teeth was bellow the accepted raise in pulpal temperature. When using 58°C, in premolars with residual dentin of 1 mm the increase of pulp temperature was above the accepted level, while in deciduous molars even with 2 mm residual dentin the increase was above 5.5°C.

*In vivo* changes in pulp temperature after heat application to GIC surface using 50°C and 60°C for 60 seconds. (Table 3) shows the temperature at the beginning and after 30 and 60 seconds of heat application using the heating device. The basic temperature in upper first deciduous molars was 3°C less when compared to lower teeth and higher on the left side compared to right side. The main increase in pulp temperature occurs during the first 30 seconds. When using only 50°C the increase of pulp temperature was always bellow 5.5°C. When using 60°C for 60 seconds the increase was above 5.5°C.

**Discussion**

The setting reaction, the acid-base reaction between polyacrylic acid and the fluoro-alumino-silicate particles is known as a key for determining the final properties of GICs and is characterized by the release of protons from the carboxyl groups of the polyacrylic acid. This is followed by a crosslinking step (maturation), in which chains of calcium polyacrylate and aluminum polyacrylate are formed over a period of approximately 3 month to 1 year [19]. Heat-curing of the exothermic GIC setting reaction changes the molecular kinetic energy promoting a more stable zone of ionic exchange for better interaction with tooth substrate, increases the particle size of GIC due to particle agglomeration, coalescence and growth at high temperature and promotes higher powder/liquid ratio due to removal of loosely bound water in the GIC. The use of ultrasound alone during setting or together with heating the GIC capsule improved surface microhardness and creep by an order of magnitude, particularly within the first 24 hours [6-7,11,20] and the differences vanish after the first month. The biaxial flexural strength of EQUIA improved after 30 or 60 seconds of heat application using a LED device by 7.5 and 15% [10]. After 1 week, 1 or 3 months, the microhardness of self-setting GIC is similar to the effect of heat application by LED curing unit [21]. Our study showed that the surface microhardness of EQUIA improved after energy application during setting by as much as 56% after 60 minutes, and only by 8% after 5 days. The best results were observed using a heating device with 50°C for 30 or 60 seconds. *In vivo* analysis of the surface showed no cracks and large inorganic particles when a heating device was used. The difference between the LED light curing device and the heating device is that the heating device keeps the temperature constant through the curing period.

while the light curing devices has circles of heat and cools every 10 or 20 seconds. When using LED light curing devices for composite materials there is a significant increase in pulp temperature, above the acceptable limit, when bulk application is used in comparison to layering method [17]. When heat was applied to the GIC capsule and ultrasonic devise was used during setting, the temperature inside the material reached 49°C, a temperature that may be iatrogenic to the pulp [20]. We measured the effect of heat application to GIC during setting on the pulp temperature both *in vitro* and *in vivo* and the results showed that 60°C can be harmful to the pulp by raising the pulpal temperature by more than 5.5°C. *In vivo* results showed that the basic temperature
Conclusions
• The application of heat to GIC during setting improved significantly the surface microhardness during the critical 60 minutes when the cement has to be protected from additional water in order to prevent the dissolution of metal cations. The microhardness values are almost similar after 5 days.
• The use of heating device of 50°C for 30 or 60 seconds gave the best results in comparison to LED curing device or to temperature of 60°C. The microstructure of the GIC surface showed larger inorganic particles and lack of cracks when the heating device was applied.
• The use of 60°C on GIC restoration increased the pulpal temperature above the accepted 5.5°C, in both permanent and deciduous teeth in vitro. Similar trend was observed in deciduous first molars and premolars in vivo.

References

Table 1: Vickers microhardness (VM) of GIC surface after energy application.

<table>
<thead>
<tr>
<th>Tooth</th>
<th>Upper Pm1 (10 teeth)</th>
<th>Upper PM1 (10 teeth)</th>
<th>Lower dm2 (5 teeth)</th>
<th>Lower dm2 (5 teeth)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Residual dentin</td>
<td>Mean (SD)</td>
<td>Mean (SD)</td>
<td>Mean (SD)</td>
<td>Mean (SD)</td>
</tr>
<tr>
<td></td>
<td>1 mm</td>
<td>2 mm</td>
<td>1 mm</td>
<td>2 mm</td>
</tr>
<tr>
<td>Basic Temperature</td>
<td>26.9</td>
<td>27.2</td>
<td>29.8</td>
<td>30.8</td>
</tr>
<tr>
<td>Temperature applied by heating device</td>
<td>50°C</td>
<td>60°C</td>
<td>50°C</td>
<td>60°C</td>
</tr>
<tr>
<td>Pulp temperature increase after 30 seconds</td>
<td>3.5°C</td>
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<tr>
<td>Pulp temperature increase after 60 seconds</td>
<td>4.2°C</td>
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<td>4.6°C</td>
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Table 2: The increase in pulp temperature after heat application to GIC surface- in vitro.

<table>
<thead>
<tr>
<th>Tooth</th>
<th>URdm1</th>
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Table 3. In vivo effect of heat application to GIC on pulpal temperature of the pulp is lower than the temperature of the human body core, 36.2 to 37.5°C [20,22], and we also found that there are differences between upper and lower and between right and left sides in deciduous first molars, probably due to differences in blood supply.

GIC content has a preventive effect against white spot lesions around restorations [23] and showed distinct effects in the remineralization of proximal artificial caries lesion in situ by affecting the superficial lesion [24]. Controlled clinical trials showed that the success rate of GIC versus amalgam for tooth restorations was similar although the laboratory results showed better results for amalgam [25], implicating that the GIC can be used safely for restorations in deciduous and permanent dentitions.

Note: HD- Heating device Groups with similar letter are not different statistically.

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Note: LC-Light curing device

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