Original Article

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Effect of Different Polymerization Times on the Microhardness and Intrapulpal Temperature of Glass Ionomers

Farklı Polimerizasyon Sürelerinin Cam İyonomerlerin Mikrosertliği ve Pulpaiçi Isı Artışı Üzerine Etkisi

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ABSTRACT

Objective: The aim of this study was to compare the microhardness of high viscosity glass ionomer, glass carbomer (GC) and bioactive restorative material (BRM) exposed to different polymerization times, and the intrapulpal thermal changes they caused on teeth.

Methods: Sixty human molar teeth were used in this study. During Class I cavity preparation,1 mm dentine thickness was left between the pulp chamber and occlusal cavity floor.Teeth were randomly divided into six groups. Group 1: restored with high viscosity glass ionomer cement (HV-GIC), cured for 20 sec., Group 2: restored with HV-GIC, cured for 40sec., Group 3: restored with conventional glass ionomer cement, cured for 60 sec., Group 4: restored with GC and cured for 90 sec., Group 5: restored with BRMs, cured for 20 sec., Group 6: restored with BRM, cured for 40 sec. All glass ionomer cements were polymerized with a LED light curing unit except GC groups. GC groups were cured with a special thermocure lamp. As soon as the materials were placed in the cavities, temperature increase on the tooth during setting/polymerization reactions were measured with a thermocouple wire connected to a data logger. All of the specimens were polished with discs.Then, microhardness values were evaluated from three different points. Data were analyzed using one-way ANOVA, Tukey test and paired t-tests ($p<0.05$).

Results: Group 2 showed statistically significantly higher increase in temperature when compared to Group 1. Group 4 showed statistically significantly higher temperature than Group 3. There

ÖZ

Amaç: Bu çalışmanın amacı, farklı polimerizasyon sürelerine maruz bırakılan yüksek viskoziteli cam iyonomer, cam karbomer (GC) ve biyoaktif restoratif materyalinin (BRM) mikrosertliklerini ve intrapulpal termal değişiklikleri karşılaştırmaktır.

Yöntemler: Bu çalışmada 60 adet çekilmiş molar dişi kullanıldı. Sınıf 1 kavite preparasyonu sırasında pulpa odası ile oklüzal kavite tabanı arasında 1 mm dentin kalınlığı bırakıldı. Dişler rastgele altı gruba ayrıldı. Grup 1: Yüksek viskoziteli cam iyonomer siman (HV-GIC) ile restore edildi, 20 sn polimerize edildi. Grup 2: HV-GIC ile restore edildi, 40 sn polimerize edildi. Grup 3: GC ile restore edildi, 60 sn polimerize edildi, Grup 4: GC ile restore edildi ve 90 sn polimerize edildi. Grup 5: Biyoaktif restoratif materyal (BRM) ile restore edildi, 20 sn polimerize edildi, Grup 6: BRM ile restore edildi, 40 sn polimerize edildi. GC grupları hariç tüm cam iyonomer simanları LED polimerizasyon cihazı ile polimerize edildi. GC grupları özel ışık aleti ile polimerize edildi.Tüm örneklerin pulpaiçi ısı artış değerleri J tipi termometre cihazı ile ölçüldü. Daha sonra mikrosertlik değerleri üç farklı noktadan değerlendirildi. Veriler tek yönlü ANOVA, Tukey testi ve t-testleri ile analiz edildi ($p<0,05$).

Bulgular: Grup 2, Grup 1'e göre pulpaiçi ısı artışında istatistiksel olarak anlamlı fark gösterdi Grup 4, Grup 3'e göre istatistiksel olarak daha yüksek pulpaiçi sıcaklık artışı gösterdi. Gruplar karşılaştırıldığında en yüksek mikrosertlik değerleri GC gruplarında elde edildi. Grup 2, Grup 1'e göre istatistiksel anlamlı derecede

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ABSTRACT

was no significant difference between Groups 5 and 6 in terms of temperature changes. The highest microhardness values were obtained in GC groups, when the groups were compared to each other. Group 2 showed significantly higher microhardness value than Group 1. Group 6 showed significantly higher microhardness values than Group 5.

Conclusion: Fourty sec polymerization of the BRM positively affected the microhardness without causing an intrapulpal temperature increase. While high microhardness values were obtained in 90 sec polymerization of GC, it also caused an increase in temperature that would damage the pulp.

Keywords: Glass-ionomer, microhardness, intrapulpal, bioactive, carbomer

ÖZ

yüksek mikrosertlik değeri gösterdi. Grup 6, Grup 5'e göre anlamlı derecede yüksek mikrosertlik değeri gösterdi.

Sonuç: BRM'nin 40 sn polimerizasyonu intrapulpal sıcaklık artışına neden olmadan mikrosertliği olumlu yönde etkilemiştir. GC'nin 90 sn polimerizasyonunda yüksek mikrosertlik değerleri elde edilirken pulpaya zarar verecek derecede ısı artışına da neden olmuştur.

Anahtar Sözcükler: Cam iyonomer, mikrosertlik, pulpaiçi, biyoaktif, karbomer

Introduction

Resin-containing materials are commonly preferred in restorative dentistry due to their high mechanical and esthetic properties. However, the cytotoxic effects of the monomers that release on the pulp tissue and the applications requiring technical sensitivity, have led to the search for new materials in the field of restorative dentistry (1).

Conventional glass ionomer materials are one of the most frequently researched and developed restorative materials. Glass carbomer (GC) (GC Dental, Netherlands) is one of the new glass ionomer-based materials. GC contains nano-fluorapatite and nano-hydroxyapatite particles differently from the conventional glass ionomer cements (CGICs) (2). Containing nano-particles is believed to promote remineralization of caries-affected dentin and enamel (3). Besides, the incorporation of nano-particles provides better mechanical and chemical properties to GC when compared to CGICs (2). Actually, the clinical application procedures of GC are similar to CGICs, with the exception that heat application is recommended during the setting reaction for GC (4). Although the application of high energy polymerization unit, GC sets with an acid-base reaction chemically (3). And the use of heat is supposed to accelerate the matrix-forming reaction of GC (4). The recommended polymerization time for GC is between 60 and 90 sec (5).

High viscosity glass ionomer cements (HV-GICs) are another newly developed CGICs (Equia Fil, GC Dental Co., Tokyo, Japan). One of the main differences between HV-GICs and CGICs are the ratio of the particles and the size of the particles (6). HV-GICs have improved physical, mechanical, and esthetic properties and are less sensitive to moisture when compared with the CGICs (7,8). Equia Fil is advised to be used with a novel nanofilled coating material (Equia Coat, GC Dental Co., Tokyo, Japan) which protects the material against wearing in the oral environment (9). Coating material should be applied with heat application. Thus, the mechanical properties of Equia Fil are also improved.

Bioactive restorative material (BRM) (Pulpdent Corporation, Watertown, USA) is one of the preferred materials containing no Bisphenol A, BIS-GMA, or BPA derivates. BRM is a resin-modified glass ionomer cement (RMGIC) reinforced with rubberized resin (10). BRM showed similar flexural strength and flexural fatigue with flowable composites (10). Also, BRM demonstrated similar mechanical properties to bulk-fill resin composites (11).

Heat application is one of the operative procedures that can damage pulp tissue (2). Zach and Cohen stated that a 5.5 °C increase in the intrapulpal temperature can cause irreversible damage to the pulp (11,12). *In vitro* studies have pointed out that different light sources used during the polymerization of resin-based restorative materials may cause such an increase in the pulp temperature (11,12). In addition, thermal conduction is affected by the thickness of the remaining dentin tissue (13). It has been mentioned that remaining dentin thickness has an essential role in preserving the vitality of the pulp (14).

Studies exhibited that increasing the polymerization time may improve the mechanical properties of a material 15-18). However, there is no study evaluating the influence of extended polymerization time on the intrapulpal temperature and mechanical properties of glass ionomers materials.

Therefore, this study aimed to compare the microhardness of HV-GICs, GC, and BRM polymerized at different times and evaluate the intrapulpal thermal changes during increased polymerization times.

The null hypothesis of the study are;

1. The microhardnesses of HV-GICs, GC, and BRM do not differ depending on the different polymerization times.

2. Intrapulpal thermal changes do not differ depending on the increased polymerization time applied on hV-GICs, GC, and BRM.

Methods

Tooth Selection and Preparation

Sixty extracted, caries-free human molars were stored in 0.5% Chloramine T solution until the test started. Class I cavities (2 mm widht, 2 mm depth, 3 mm length) were prepared with diamond burs (G&Z Instruments, Austria). 1 mm dentin thickness that was measured with a digital micrometer was left between the pulp chamber and occlusal cavity floor. After cavity preparations, the roots of each tooth were removed. Then, all teeth were randomly divided into six subgroups (n=10):

Group 1: Equia Fil + Light emitting-diode (LED) curing light (VALO Cordless, Ultradent, South Jordan, Utah) 20 sec.

Group 2: Equia Fil + LED curing light 40 sec.

Group 3: GC + GC CarboLED thermocure lamp (Carboled, GC Dental Netherlands) 60 sec.

Group 4: GC + GC CarboLED thermocure lamp 90 sec.

Group 5: BRM + LED curing light 20 sec.

Group 6: BRM + LED curing light 40 sec.

Materials used in this study are provided in Table 1.

Bezmialem Vakıf University Non-invasive Research Ethics Committee (number: E-54022451-050.01.04-7929/date: 20.03.2021).

Restoration Procedures

Group 1: Capsulated HV-GICs, Equia Fil, was mixed for 10 sec. The mixture was applied to the cavity in bulk immediately. After 2 min 30 sec, the Equia Coat was applied and cured for 20 sec.

Group 2: Capsulated HV-GICs, Equia Fil, wasmixed for 10 sec. The mixture was applied to the cavity in bulk immediately. After that, the Equia Coat was applied and cured for 40 sec.

Group 3: GC capsule was mixed for 15 sec with its mixer (GC Dental, Netherlands). GC material was placed in the cavity in a single increment. After the cavity was filled, the surface cover with silicone was applied to the cavity and condensed with finger pressure. Finally, it was polymerized with a CarboLED light device set at a power of 1,400 mW/cm^2 for 60 sec.

Group 4: GC capsule was mixed for 15 sec with its mixer. GC material was placed in the cavity in a single stage. After the cavity was filled, the surface cover with silicone was applied to the cavity and condensed with finger pressure. Finally, it was polymerized with a CarboLED light device set at a power of 1,400 mW/cm^2 for 90 sec.

Group 5: Cavities were selectively etched with 37.5% phosphoric acid (Ultradent, South Jordan, USA) for 15 sec, rinsed with water, and dried. Later, BRM was placed into the cavity using a syringe, according to the manufacturer's instruction with the bulk technique. Finally, the samples were polymerized with LED light curing unit (Ultradent, South Jordan, Utah, USA) for 20 sec.

Group 6: Cavities were selectively etched with 37.5% phosphoric acid for 15 sec, rinsed with water, and dried. Later, BRM was placed into the cavity using a syringe, according to the manufacturer's instruction with the bulk technique. Finally, the samples were polymerized with LED light curing unit for 40 sec.

The Experimental Design and Measurement of Intrapulpal Temperature

The pulpal microcirculation model was demonstrated in Figure 1, designed by Savas et al. (19).

A thermal gel (Hutixi, HTGY 260, China) was injected into the pulp chamber to facilitate heat transfer from the roof of the pulp chamber to the thermocouple. As soon as the materials were placed in the cavities, temperature increases in the tooth during setting/polymerization reactions were measured with a j type-thermocouple (Fluke 54 II, Washington, USA) connected to a data logger. For all specimens, initial and highest temperature values were recorded. In addition, differences between initial and highest temperatures were determined (Δt) (19).

Table 1. Materials used in this study

Material	Manufacturer	Chemical composition
GCP (Glass Carbomer Cement) GCP Gloss	GCP Dental, Netherlands	Floraluminosilicate glass, apatite, polyacid Modified polysiloxanes
Equia Fil (High Viscosity Glass Ionomer) Equia Coat	GC, Tokyo, Japan	Floraluminosilicate glass, carboxylic acid, polyacrylic acid, water Methyl methacrylate, colloidal silica,camphorquinone, urethane methacrylate, phosphoric ester monomer
Activa Bioactive (Bioglass-reinforced Glass Ionomer Cement)	Pulpdent, USA	Mix of methacrylates and diurethane with modified polyacrylic acid; reactive glass Filler; inorganic filler, rubberized resin, Water
GCP CarboLED Valo Cordless (standart mode)	GCP Dental, Netherlands Ultradent, South Jordan, Utah, USA	1,400 mW/cm^2 power out-put 1,000 mW/cm^2 wavelenght 480 nm

Measurement of Microhardness Values

The specimens were polished with discs (Sof-Lex, 3M ESPE, USA) from coarse to fine. Then, microhardness values were evaluated from three different points by applying a load of 200 g for 10 sec on top surfaces using a micro Vickers hardness test machine (Shimadzu, Japan).

Statistical Analysis

The sample size was calculated at the significance level of 0.05 and power of 0.90 using G*Power v3.1 (Heinrich Heine, Universitat Dusseldorf, Dusseldorf, Germany). Statistical analysis of the data was performed by one-way ANOVA, Tukey, and paired t-tests. A p value of <0.05 was considered to be statistically significant.

Results

Group 2 (±4.49) showed a significantly higher increase in pulpal temperature than Group 1 (±3.29) (p=0.018). For Group 4, temperature increases over 5.5 degrees were observed. However, the highest temperature increase was calculated in Group 4 (±6.72) when polymerized for 90 sec. Group 4 showed a significantly higher increase in pulpal temperature than Group 3 (±5.49) (p=0.040). There were no significant differences between Group 5 (±3.95) and Group 6 (±4.48) (p>0.05). Intrapulpal thermal changes were shown in Figure 2.

The highest microhardness value was observed in Group 4 (±48.67). Group 2 (±37.09) showed a significantly higher microhardness value than Group 1 (±32.83) (p=0.045). There were no significant differences between Group 3 and Group 4 regarding microhardness values (p>0.05). Group 6 (±28.94) showed a significantly higher microhardness value than Group 5 (±26.92) (p=0.020). Microhardness values were shown in Figure 3.

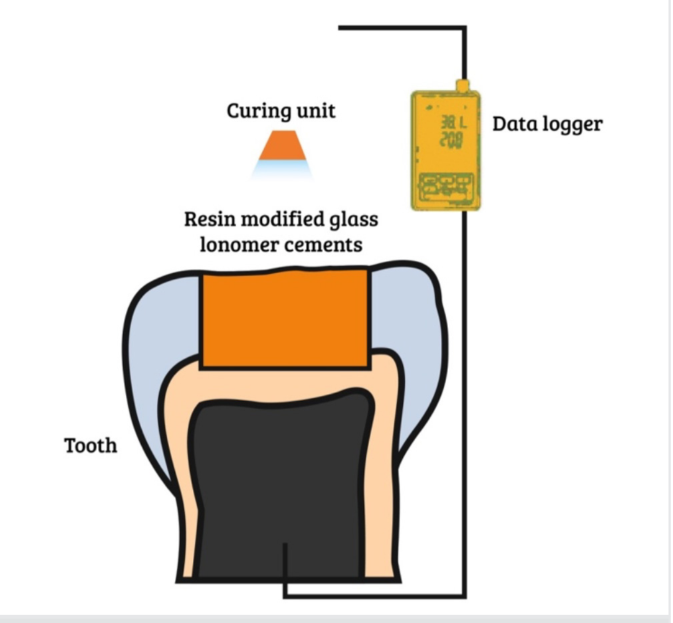

Figure 1. Diagram of measurement of intrapulpal thermal changes

Discussion

The purpose of this study was to compare the microhardness of high viscosity glass ionomer, GC, and BRM polymerized at different polymerization times and evaluated the intrapulpal thermal changes during increased polymerization times. Pulpal temperatures were found significantly different between Groups 1 (±3.29) and 2 (±4.49) and also between Groups 3 (±5.49) and 4 (±6.72). Additionally, microhardness values were found significantly different between Groups 1 and 2, and Groups 5 and 6. The null hypotheses were partially rejected.

Intrapulpal thermal changes can be affected by several factors such as polymerization procedures, cavity preparation procedures, remaining dentin thickness, and type of restorative materials (18). It was reported that increasing the polymerization time can damage the vitality of the pulp tissue (19). It was also reported that increased polymerization time changes the microhardness of the restorative materials (16).

Calorimeter, thermocouple, infrared camera, and differential thermal analysis are techniques to evaluate intapulpal thermal changes (20). However, when the studies were examined, thermocouple device was generally used for the measurements of the intrapulpal thermal changes due to their reliable and sensitive outcomes in temperature changes (21,22).

The tooth pulp is an extensive vascularized tissue (23). Due to this structural property of pulp, intrapulpal temperature increase can be absorbed when the dental tissue is exposed to thermal stimulus (23). Studies reported a high intra-pulpal temperature increase when the pulpal microcirculation model was not used

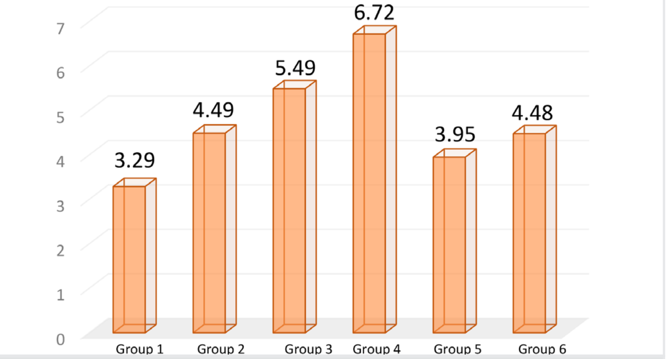

Figure 2. Intrapulpal thermal changes in groups

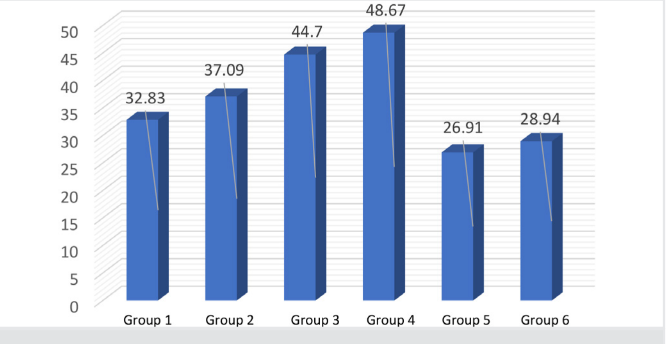

Figure 3. Microhardness values in groups

(24-27). If we had used the microcirculation model, perhaps the intrapulpal thermal changes would have been different or lower than these results.

The thermal changes in the pulp tissue vary according to the thickness of the dentin in the pulp chamber, cavity preparation technique, the type of restorative material, and the light unit used (18, 28-30). Also, the intensity of the light source and polymerization time can affect the temperature changes in the pulp chamber (31,33). In this study, an LED curing unit was used, 1,170 mW/cm^2, 385-515 nm at different times for Equia Fil and BioActiva. In addition, Carboled with 1,400 mW/cm^2 for the polymerization of GC fillings was used. The highest pulp temperature increase was obtained in the 90 sec polymerization in Carboled used group. This may be due to the high output power of the light device and the longer activation time. The higher temperature increase in the Equia Fil group in which the LED was applied for 40 sec compared to the 20 sec may also be due to the prolonged polymerization time. In a study by Altan et al. (30) the temperature increase of Equia Fil and GC was compared and they found the lowest temperature increase in Equia Fil Group and the result of that study was similar to the present study.

The studies showed that the remaining dentin thickness was effective in causing pulp damage by intrapulpal thermal changes (33,34). Aguiar et al. (34) observed an intrapulpal temperature of 5.6 °C for 1 mm remaining dentin, 5.3 °C for 2 mm remaining dentin, and 2.4 °C for 3 mm remaining dentin. Botsali et al. (18) reported that the intrapulpal temperature increase in 1 mm remaining dentin was more than that in 2 mm dentin thickness. Botsali et al. (18) found that both the 1 mm and 2 mm remaining dentin thicknesses for the GC Group showed the highest intrapulpal temperature increase when compared to two different resin-modified GIC cements (34,35). In this study, the highest intrapulpal temperature increase was observed in GC groups at 1 mm dentin thickness.

Surface microhardness is one of the methods used to evaluate the physical strength of materials (36). Brinelll (37) are commonly used in measuring the microhardness value of restorative materials. Vickers test method was used in this study due to the availability of equipment and suitability for all materials and surfaces (38,39). In addition, surface hardness is related to the content and size of the restorative material (14,15).

Heat application is recommended to improve the mechanical properties of GIC (40,41).

When a glass-ionomer based material was heated to a high temperature, the evaporation of the liquid may result in an increase in the powder to liquid ratio, which in turn strengthens the cement (42). This study measured the microhardness by applying heat for different periods to all restorative materials. For all of the restorative materials, surface microhardness was higher in groups exposed to long polymerization time. Therefore, it can be concluded that the prolonged polymerization time may increase the microhardness and improve the mechanical properties of the materials positively.

It is known that the mechanical properties have become better as the particle size of the restorative materials decrease (43). GCs have developed with the application of nanoparticle technology to create an enamel-like structure (44,45). It is known that enamel is the hardest and stiffest tissue in the human body (46). In addition, fluoroapatite and hydroxyapatite are added to the nanoparticle structure to strengthen their mechanical and physical properties. In this study, the highest hardness value was found in the GC group which might be due to its nanoparticle-containing structure which created an enamel-like structure.

Surface coating application is recommended in glass ionomer cements to prevent early moisture contamination and improve surface properties (9,47). According to the manufacturer's instructions, a nanofill resin surface coat was applied to the Equia Fil Group and (48) silicon-based surface coat material was applied to GC Groups (42). Therefore, the higher surface microhardness of Equia Fil and GC compared to BRM Groups could be due to the application of surface coating materials. Besides, higher microhardness values obtained in GC than in Equia Fil Groups may be due to the different content of surface coating materials.

Although BRM is known as a type of RMGIC, it differs from RMGIC with some structural features. BRM has reactive ionomer glass fillers and rubberized resin component (49). Due to the different content of BRM from other glass ionomer cements, the increase in intrapulpal temperature may not be adversely affected.

The lack of intraoral conditions and the pulpal circulation model were the limitations of this in vitro study.

Conclusion

1. Polymerization of the bioactive material for a long time positively affected the microhardness of the material without causing an increase in pulp temperature while negatively affecting the other glass ionomer-based materials causing an increase in pulp temperature.

Increasing the polymerization time of bioactive material can be recommended.

No temperature increase that would cause pathological damage to the pulp was observed in other groups except for the group of GCs polymerized with light for 90 sec.

During application of GC in clinical situations, clinicians should avoid curing for prolonged time.

In the use of GC remaining dentin thickness is recommended to be more than 1 mm to protect pulp from damage.

Ethics

Ethics Committee Approval: Bezmialem Vakıf University Non-invasive Research Ethics Committee (number: E-54022451-050.01.04-7929/date: 20.03.2021).

Informed Consent: *In vitro* study.

Peer-review: Externally peer reviewed.

Authorship Contributions

Surgical and Medical Practices: Z.B.K., Design: Z.B.K., E.E.D., N.D., Data Collection or Processing: Z.B.K., Analysis or Interpretation: N.D., M.K., Literature Search: Z.B.K., E.E.D., M.K., Writing: Z.B.K., N.D., M.K.

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