

■ Original Article

Evaluation of physical and mechanical properties of glass carbomer cement under in vitro conditions

Cam karbomer simanın fiziksel ve mekanik özelliklerinin in vitro koşullar altında değerlendirilmesi

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ABSTRACT

Aim: To evaluate the microtensile bond strength (μ TBS) and microhardness of glass carbomer cement in comparison to conventional glass ionomer cement and compomer.

Material and Methods: The bonding strength test involved 60, second deciduous molar teeth. The dentine bonding strength of the restorative materials was evaluated by a μ TBS test. Failure modes were determined by light microscopy. Plexi-glass molds of 5 × 2 mm (diameter × depth) were used for Vickers' microhardness analysis. Seventy-five samples were evaluated, considering twenty-five samples for each material. The data were statistically analyzed by the Kruskal-Wallis test, at $p \leq 0.05$.

Results: There was no significant difference between the μ TBS of the glass carbomer (2.0 MPa) and glass ionomer (1.7 MPa) ($p > 0.05$). However, the μ TBS of the compomer (9.4 MPa) was higher than the glass carbomer and glass ionomer ($p < 0.001$). No significant difference was found among the three materials, regarding adhesive, cohesive and mixed failure modes ($p > 0.05$). The compomer presented the highest microhardness value, followed by the glass ionomer and finally, the glass carbomer ($p < 0.001$).

Conclusion: The glass carbomer cement showed a lower μ TBS to the dentine than the compomer. Furthermore, the microhardness of the carbomer was lower than the compomer and glass ionomer.

Keywords: Pediatric dentistry; Glass carbomer cement; Compomers; Glass ionomer cements.

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ÖZ

Amaç: Bu çalışmanın amacı; cam karbomer simanın mikrogerilim bağlanma dayanımı ve mikrosertlik değerlerinin geleneksel cam iyonomer siman ve kompomerle karşılaştırılarak değerlendirilmesidir.

Gereç ve Yöntemler: Bağlanma dayanımı testinde 60 adet süt 2. molar dişi kullanılmıştır. Bu amaçla dentine uygulanan restoratif materyallerin dentine bağlanma kuvveti "mikrogerilim bağlanma dayanımı testi" ile değerlendirilmiştir. Kopma tipleri 20X büyütmede ışık mikroskobu altında belirlenmiştir. Mikrosertlik testi için 5 mm çapta ve 2 mm derinlikte pleksi-glass kalıplar kullanılmıştır. Her materyal için 25 örnek olacak şekilde 75 örnek değerlendirilmiştir ve materyal mikrosertlikleri 'Vicker's testi' ile incelenmiştir. Elde edilen veriler Kruskal Wallis testiyle değerlendirilmiştir. İstatistiksel anlamlılık düzeyi $p \leq 0,05$ olarak kabul edilmiştir.

Bulgular: Cam karbomer (2,0 MPa) ve cam iyonomerin (1,7 MPa) dentine bağlanma değerleri arasında istatistiksel olarak anlamlı farklılık olmadığı, kompomerin ise (9,4 MPa) istatistiksel olarak anlamlı derecede daha yüksek mikrogerilim bağlanma dayanımı gösterdiği belirlenmiştir ($p < 0,001$). Cam karbomer siman, cam iyonomer siman ve kompomer restorasyon materyalleri arasında adeziv, koheziv ve karışık kopma tipleri oranları açısından istatistiksel olarak anlamlı fark görülmemiştir ($p = 0,409$).

Restoratif materyallerin mikrosertlik değerleri arasında ise istatistiksel anlamlı fark tespit edilmiştir ($p < 0,001$). Mikrosertliği en yüksek dolgu materyali kompomer; mikrosertliği en düşük materyal ise cam karbomer olacak şekilde materyal sertlikleri kompomer > cam iyonomer > cam karbomer olarak belirlenmiştir.

Sonuç: Cam karbomer siman dentine kompomerden daha düşük mikrogerilim bağlanma dayanımı göstermiştir. Ayrıca mikrosertlik değeri kompomerden ve cam iyonomer simandan daha düşük olarak tespit edilmiştir.

Anahtar kelimeler: Çocuk diş hekimliği, Cam karbomer siman, Kompomer, Cam iyonomer siman.

Introduction

In recent years, resin-based composites, resin-modified glass ionomers, polyacid-modified resin-based composites (compomers), and glass ionomers have emerged as restorative materials in the restoration of milk teeth. Glass ionomer cement (GIC) is today often preferred as a restorative material in pediatric dentistry because its thermal expansion coefficient is comparable to that of the tooth structure. Within this context, the enamel, dentin, and cement are chemically bonded; and GIC is biocompatible, has anticariogenic properties after fluoride release, and shows less sensitivity to moisture than resins [1,2]. However, GIC also has disadvantages, such as long workability for completion of hardening, difficulty in applying to the cavity, susceptibility to scratching, dehydration in early stages of hardening and finishing, poor finishing and polishing process, roughness of the final surface, as well as poor mechanical properties because of the powder-to-liquid ratio [3,4].

An excellent restorative material should be able to adhere the dentin and the enamel structure. The absence of a material

with this feature and the presence of positive and negative attributes of all existing materials, have led to the search for innovative materials. Glass carbomer cement is a new generation monomer-free restorative material, containing glass and aqueous polyacrylic acid, which is similar to conventional GIC, as well as nano-sized fluorapatite and hydroxyapatite [$\text{HAp: Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$] [5,6]. HAp has excellent biocompatibility, and both its composition and chemical composition are similar to dental structure and bone tissue [7]. For the first time in 1984, Yamamoto evidenced improved biocompatibility of conventional GIC by the addition of HAp. Compared to glass carbomer cement, which is a resin-free material, the mechanical properties of GICs are strengthened by the HAp crystals in the material [8]. Glass carbomer cement is manufactured by GCP Dental (The Netherlands) and the term "glass carbomer" has been adopted in the scientific literature, despite being a brand name and a type of glass ionomer. However, unlike conventional GIC, glass carbomer aims to provide remineralization in the oral environment, as particles in the

material help to remineralize decayed enamel and dentin [6,9]. It is claimed that the glass carbomer contains calcium fluorapatite nanocrystals that can act as a core for remineralization and initiate fluorapatite formation [9]. Based on the characteristics of this nanotechnologic approach and process, glass carbomer cement contains less matrix and more filler than GIC [10]. To fluorapatite conversion, the glass particles in the contents are smaller in size than the GIC [11]. The liquid of glass carbomer cement is polyacrylic acid. Similar to high-viscosity GICs, incorporation of the nanoparticles may also provide improved compressive strength and might wear resistance [12].

The clinical application of glass carbomer cement is similar to conventional GIC. However, in order to set the glass carbomer, dentists must use a high-energy light device in the final step [12-14]. It has been reported by specialists that through the application of heat, the compression strength of the material is increased and the clinical outcome is improved [12-15]. Furthermore, the chair time is shortened because of the accelerated curing reaction with heat application [15].

Application of a surface protector may improve the surface and insulation properties of the glass carbomer cement should be applied before the light-curing stage [12,13]. The GCP glass carbomer surface protector is a monomer-free, silicone-based material that protects the restoration from the initial curing reaction, moisture as well as saliva exposure the second phase's dehydration. It furthermore facilitates the shaping and polishing of the filler [14].

Due to the lack of published data regarding the clinical use of glass carbomer cement, laboratory tests are gaining importance in the evaluation of its physical properties. This study, compared the microtensile bond strength (μ TBS) of a glass carbomer cement, GIC and compomer.

Material and Methods

μ TBS Test

Sixty, second deciduous molar teeth with no caries, cracks or defects were used. Their use in research was approved by the Ankara University Ethics Committee (2015/12-11). The teeth were stored at room temperature in a 0.2% thymol solution for 3 months maximum. The buccal enamel surfaces were sanded with a silicon carbide abrasive (600, 800, and 1200 grit) under water cooling in a horizontal rotary sander (Gripo 2V Grinder Polisher, Metkon Instruments Ltd, Bursa, Turkey), to obtain flat dentin surfaces. The teeth were divided into three groups: (n=20): (1) compomer (Dyract Extra, Dentsply,

Konstanz, Germany) + bond (Prime&Bond NT, Dentsply Sirona, NY, USA); (2) conventional GIC (SDIRiva Self Cure, Bayswater, Australia); and (3) glass carbomer cement (Glass Carbomer Products, GCP Dental, Leiden, The Netherlands).

In the first group, a double bond (Prime & Bond NT) was applied to the dentin surfaces. All samples were treated with a GCP CarboLED CL-02 lamp (1850 mW cm²) (GCP Dental) for 10 s, to standardize the dentin surfaces in the compomer group. Subsequently, the compomer, conventional GIC, and glass carbomer cement was applied with a thickness of 2 mm, by using transparent plastic molds of 5 mm length and diameter. The compomer was polymerized for 20 s and the glass carbomer cement for 90 s, with the high-energy LED device. The specimens were stored in artificial saliva (mmol/L): CaCl₂·2H₂O (0.7), MgCl₂·6H₂O (0.2), KH₂PO₄ (4.0), KCL (30), HEPES solution (20), NaN₃ (3.0) at 37°C for 24 h, as recommended by the manufacturer (GCP Glass Carbomer). For the μ TBS test, the samples were cut to obtain approximately 1.0×1.0 mm sticks and mounted in a universal testing machine (Micro Tensile Tester, Bisco Inc., Schaumburg, IL, USA) at a crosshead speed of 1 mm/min, creating fracture at the interface of the restorative materials and dentin. The rupture force was recorded (N), and then the units of the measured μ TBS values were converted to megapascal (MPa) (N/surface area = MPa). The failure modes were evaluated, with a digital stereomicroscope (Leica MZ12, Meyer Instruments, Houston, TX, USA) at 20× magnification and classified as adhesive, cohesive and mixed.

Vicker's Microhardness Test

Twenty-five disk-shaped specimens of each material were prepared using a split plexi-glass mold (5 × 2 mm). The plexi-glass molds were placed on transparent bands on glass (SNA, Universal Strips, Germany). The glass carbomer samples were light-cured with the LED device for 90 s, and the compomer samples were light-cured for 20 s. The specimens were stored in artificial saliva. The microhardness test was conducted on the top surface of the samples, using Vicker's microhardness tester (Zwick/Roell ZHV 10, Germany), with 200 g load and 17 s dwell time. The hardness was measured at three different points on a single surface of the restorations, and the average value was calculated afterwards. The formed traces were also examined by light microscopy at 200× magnification.

Statistical Analysis

Data was statistically analyzed by the Kruskal-Wallis test at $p \leq 0.05$. During this process, a Bonferroni correction was made to control the Type I error in all possible multiple comparisons.



Results

μTBS

While the μTBS values of the test groups (Table 1) revealed no significant difference between the glass carbomer and glass ionomer ($p>0.05$), the compomer presented the highest μTBS ($p<0.001$). There was no statistically significant difference found among the glass carbomer cement. As well as, no significant difference was found among the three materials, regarding adhesive, cohesive and mixed failure modes ($p>0.05$).

Table 1: Microtensile bond strength of Compomer, Glass Ionomer and Glass Carbomer restorative materials.

Group	Microtensile bond strength (MPa)
Compomer *	9.4 (14.12) *
Glass ionomer	1.7 (1.05)
Glass carbomer	2.0 (1.19)

† Kruskal Wallis test, a: The difference between the compomer group and the glass ionomer group was statistically significant ($p<0,001$), b: The difference between the compomer group and glass carbomer group was statistically significant ($p<0,001$).

Vicker's Microhardness

Significant differences were found in the Vicker's surface microhardness values of the three types of material ($p<0.001$) (Table 2). The compomer displayed the highest microhardness value, followed by the glass ionomer and, finally, the glass carbomer.

Table 2: Microhardness values of Compomer, Glass Ionomer and Glass Carbomer restorative materials.

Group	Microhardness (VHN)
Compomer *	118.7 (8.67) *
Glass ionomer *	92.3 (9.83) *
Glass carbomer	66.0 (4.50)

†Kruskal Wallis test, a: The difference between the compomer group and the glass ionomer group was statistically significant ($p<0,001$), b: The difference between the compomer group and the glass carbomer group was statistically significant ($p<0,001$), c: The difference between the glass carbomer group and the glass ionomer group was statistically significant ($p<0,001$).

Discussion

GICs and compomers are frequently used as restorative materials in deciduous teeth[16,17]. The GIC has various advantages, such as chemical bonding to the dentine and fluoride release. These physical and chemical properties make the glass ionomer the ideal restorative material in pediatric dentistry[18]. Consequently, the compomer and GIC have been preferred as control materials for understanding the physical and mechanical properties of glass carbomer cement.

Stress occurs between the tooth surface and the restorative material, due to the effects of temperature changes in the mouth throughout the day. This stress affects the interconnection of the material and the tooth surface. Therefore, when evaluating the suitability of a material presented in clinical dental practice, the bonding strength to dental enamel becomes very important. When the μTBS values of the restorative materials were examined, it was found that the compomer had the highest bond strength, while the glass carbomer and the GIC had similar bond strengths. There are no current publications that have tested the μTBS of glass carbomer cement. However, previous studies have evaluated the bond strength of the glass carbomer cement to dentine, using various methods.

Koenraads et al. (2009) compared the compressive strengths of glass carbomer cement, composite and reinforced GIC and reported higher values for the composite than GIC and glass carbomer cement, and found no significant difference between the GIC and glass carbomer cement[19]. Lucas et al. (2003) assessed the shear bond strength of HAp-reinforced GIC to dentine in comparison with conventional GIC and reported results consistent with this study. The bonding was measured from 15 min to 56 days, and no difference was found between the two materials[11]. The bonding strength of the GIC to dentine is thought to be higher under invivo than invitro conditions because the vital tooth contains moisture and it supports dentin-GIC bonding[20].

In glass ionomer-based materials no acid or bonding application is used. Therefore, after the application of the material to the flat dentin surface as a part of the μTBS test deterioration of the dentin and GIC connection occurred, in addition to the loss of the sample. Hence, this may explain why no previous study has evaluated the bonding of glass carbomer cement to dentine with the GIC, by using the μTBS test. Although there was no statistically significant difference between the μTBS values of the glass carbomer cement and GIC, more GIC samples were lost than the glass carbomer cement group during the testing stages, which implies that the glass carbomer cement shows better bonding to dentin than GIC. Similarly, Glavina et al. (2009) found that the shear bond value of the glass carbomer was significantly superior to the conventional glass ionomer, and therefore, the glass carbomer could be used safely in the clinic[21].

One of the most crucial physical properties that contribute to the clinical success of restorative materials is surface

microhardness and the mechanical properties of restorative materials. Surface microhardness enhances resistance to scratching and abrasion and also affects clinical success by preventing the material from ready deformation against various forces[22]. In this study, the compomer had the highest microhardness value, followed by the GIC and glass carbomer, respectively. These results concur with Menne-Happ and Ilie (2013), whom investigated the influence of heat and surface protective application on the mechanical properties of glass carbomer cement compared to the resin-modified GIC. Furthermore, in their study, the hardness of the glass carbomer cement ranged from 62.3–67.4 VHN, which is comparable to that found in the current work[14]. Although Yap et al. (2002) showed that the addition of HAp increased the hardness of GIC, this conflicts with the results of this study[23]. Although the glass carbomer cement has been reported to increase the pressure and wear sresistance of nano-sized filler particles [24], in the present study, the glass carbomer cement had a lower microhardness value than the control materials.

The differences in hardness values can be attributed to the physical characteristics, chemical composition, and filler content properties of each material[25]. Chung and Greener (1990) observed that high surface hardness values were measured in materials with a high filler content[26]. In result of this study, compomer showed considerably high surface hardness values compared to the other tested restoration materials. This result can be explained in relation to the filler content of the material. Although the filler content of the glass carbomer cement is higher than the GIC, the hardness value was lower, due to other chemical properties of the material or to the smaller-sized glass particles present in the contents relative to the GIC.

During the microhardness measurement of our study, crack lines were detected in the glass carbomer cement samples when examined under the light microscope (figure1,2). Likewise, Chen et al. (2010), Çehreli et al. (2013), and Menne-Happ and Ilie (2013) also reported that in the glass carbomer group, catastrophic internal and surface crack lines, resembling ice cracks, were evident in specimens[12,14,27]. Besides micro-leakage along the cavity walls and the pulpal floor, Çehreli et al. (2013) evidenced dye penetration within the crack lines, suggesting the severity of the loss of integrity[12]. In our study, it was thought that these broken lines were caused by the low hardness of the material.

Also, the only in-vivo study about the glass carbomer cement was recently published. After 12 months follow up, glass

carbomer cement showed lower survival rates compared to two different high-viscosity glass ionomer cement [28]. This result is an important indicator for the success of glass carbomer cement.

Conclusion

Within the limitations of this study, the following conclusions were drawn;

1. Microtensile bond strength values of compomer was higher than glass carbomer and glass ionomer.
2. The microhardness and μ TBS values of the glass carbomer were lower than both, the compomer and glass ionomer, highlighting two major disadvantages of this material.
3. Also use of a high-energy CarboLED device for an extended period limits the clinical utilization of the glass carbomer, by increasing the chair time.

In order to ensure a routine use process, of the glass carbomer in pediatric dentistry clinics, other mechanical properties should further be tested in future in-vivo and in-vitro studies.

Declaration of conflict of interest

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