



Original Article / Araştırma Makalesi

**EVALUATION OF THE SHEAR BOND STRENGTH, MICROLEAKAGE AND
COMPRESSIVE STRENGTH OF REINFORCED GLASS IONOMER CEMENTS
USED AS PERMANENT RESTORATIVE MATERIALS**

**Daimi Restoratif Materyal Olarak Kullanılan Güçlendirilmiş Cam İyonomer
Simanların; Makaslama Bağlanma Dayanımları, Mikrosızıntı ve Basma Dayanımlarının
Değerlendirilmesi**

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ABSTRACT

In this study, it is aimed to compare the usage of reinforced glass ionomer materials such as high viscosity glass ionomer and giomer as a permanent restorative material with the composite resin in terms of the mechanical properties. In our study, three groups were used: Equia Forte, Beautifil II and Solare X. For the shear bond strength test; 48, and for the microleakage test; 27 extracted third molar teeth were used. A total of 30 samples were prepared for the compressive strength test. Shear and compressive strength tests were performed by using universal test devices. Standard class V cavities were prepared on the buccal and lingual surfaces of the tooth for the microleakage test. Thermal cycling (5-55°Cx10.000) was applied to the restored teeth. According to the statistical analysis, difference was found between all groups in the shear bond strength, compressive strength and microleakage tests ($p<0.05$). Solare X has showed the highest shear bond strength. Equia Forte has showed the lowest value in the microleakage test. Beautifil II has showed the highest compressive strength. Reinforced glass ionomer cements have been determined to have similar and better mechanical properties than composite resin in all the tests except the shear bond strength test.

Keywords: Compressive Strength, Giomer, Microleakage, Reinforced Glass İyonomer Cement, Shear Bond Strength

ÖZ

Bu çalışmada, yüksek viskoziteli cam iyonomer ve giomer gibi güçlendirilmiş cam iyonomer malzemelerin daimi bir restoratif materyal olarak kullanımının kompozit rezin ile mekanik özellikler açısından karşılaştırılması amaçlanmıştır. Çalışmamızda üç grup kullanıldı: Equia Forte, Beautifil II ve Solare X. Makaslama bağlanma kuvveti testi için 48 tane ve mikrosızıntı testi için 27 tane çekilmiş üçüncü azı dişi kullanıldı. Basma dayanımı testi için toplam 30 numune hazırlandı. Makaslama ve basma dayanımı testleri, evrensel test cihazları kullanılarak yapıldı. Mikrosızıntı testi için dişin bukkal ve lingual yüzeylerinde standart sınıf V kaviteler hazırlandı. Restore edilen dişlere termal siklus (5-55°C x 10.000) uygulandı. İstatistiksel analize göre; makaslama bağlanma dayanımı, basma dayanımı ve mikrosızıntı testlerinde tüm gruplar arasında fark bulundu ($p < 0.05$). Solare X, en yüksek makaslama bağlanma kuvveti göstermiştir. Equia Forte, mikrosızıntı testinde en düşük değeri göstermiştir. Beautifil II, en yüksek basma dayanımını göstermiştir. Güçlendirilmiş cam iyonomer simanların, makaslama bağ mukavemeti testi dışındaki tüm testlerde kompozit rezine benzer ve daha iyi mekanik özelliklere sahip olduğu tespit edilmiştir.

Anahtar kelimeler: Basma Dayanımı Kuvveti, Giomer, Güçlendirilmiş Cam İyonomer Simanlar, Makaslama Bağlanma Kuvveti, Mikrosızıntı

INTRODUCTION

In recent years, the use of glass ionomer cements (GIC) and composite resin materials have become widespread as a result of the increase in interest in aesthetic restorations, as well as the adoption of minimally invasive treatment techniques in dentistry (Yap, Wang, Wu, & Chung, 2004).

Glass ionomer cements, one of the widely used restorative materials in restorative dentistry, have evolved to fulfil many functional and aesthetic requirements since their introduction by Wilson and Kent in the 1970s (Bonifácio et al., 2009). Glass ionomer cements have advantages as; being easy to use, having anticaryogenic potential thanks to their fluorine ion release and rechargeable properties, being biocompatible, being able to bind to dental tissues chemically, and having thermal expansion coefficients that are similar to dental tissues. However, in addition to these advantages, they are very sensitive to moisture during the hardening phase, and their usage as restorative material in the posterior region is limited due to low wear resistance, compression and bending strength properties (Croll & Nicholson, 2002). The existence of remineralization potential and the need to develop GIC, which has become very popular with their anticaryogenic properties, led to studies on reinforced glass ionomer cement types (Murdoch-Kinch & McLean, 2003; Peters & McLean, 2001). By adding metal, ceramics and glass fibers as a second phase particle in the powder and liquid part of the glass ionomer and developing them with different modifications, the physical and mechanical properties and antibacterial activity of GIC have been attempted to be improved (Najeeb et al., 2016; Williams, Billington, & Pearson, 1998).

Based on the manufacturer's claim Equia Forte, one of the materials released as a result of these studies, is a high viscosity glass ionomer cement (HVGIC). These cements are materials that have reduced sensitivity to moisture in the early period, increased hardness and abrasion resistance, and they can be used in the posterior areas where intense chewing forces are seen (Basting, Serra, & Rodrigues, 2002). Another modification of the glass ionomer is giomers. Giomer is available as a reinforced restorative material containing active glass ionomer particles, capable of releasing fluorine ions and curing with light. Pre-reacted glass ionomer (PRG) fillers are formed as a result of acid-base reaction of polyalkenoic acid in aqueous medium with fluoroaluminacilicate glass powders (Ikemura, Tay, Endo, & Pashley, 2008). PRG fillers have a GIC structure and are responsible for fluorine ion release (Deliperi, Bardwell, Wegley, & Congiu, 2006; Gordan, Mondragon, Watson, Garvan, & Mjör, 2007). Some composite resins contain pre-reacted fillers called prepolymerized fillers. It is thought

that the organic matrix around the prepolymerized structure has a lower hardness value compared to this structure (Yarimizu, Sakuma, Akahane, & Hirota, 2002).

Although glass ionomer cements (GIC) have many advantageous properties, they have poor mechanical properties. The development requirement of GIC has led to studies on the range of reinforced glass ionomer cements. Equia Forte Fil is a newly developed GIC with improved mechanical strength. However, data on the mechanical properties of Equia Forte Fil were not satisfactory. Comparison of GIC with existing products for impression was not sufficient as permanent restorative materials. The purpose of this study is to compare reinforced glass ionomer materials, being the HVGIC and giomer with composite material, in terms of mechanical properties which include shear bond strength, microleakage and compression strength.

H_0 hypothesis; high viscosity glass ionomer cement, giomer and micro hybrid composite are not different in terms of mechanical properties.

MATERIAL AND METHOD

The study protocol was approved by the Institutional Research Ethics Committee of Inonu University (Protocol no. 72867572/050/22780). In this study, a high viscosity glass ionomer Equia Forte (GC Co., Tokyo, Japan), a giomer Beautifil II (Shofu Inc., Kyoto, Japan), and a microhybrid composite resin Solare X (GC Co., Tokyo, Japan) were evaluated. Universal Single Bond (3M ESPE Neuss, Germany) was applied as an adhesive before the beautifil II and Solare X groups. Table 1 gives information about materials used. According to the power analysis results, when $\alpha = 0.05$, $1-\beta$ (power) = 0.80, at least 8 teeth (n) should be taken from each group.

Table 1. Details of the Investigated Restorative Materials

Material	Manufacturer	Contents	The batch (LOT) numbers
HVGIC (Equia Forte)	GC Corporation, Tokyo, Japan	fluoro-aluminosilicate glass, hybrid glass particles, polyacrylic acid powder, polyacrylic acid, polybasic carboxylic acid, distilled water	1803121
Giomer (Beautifil II)	Shofu Inc. Kyoto, Japan	S-PRG filler, fluoroboroaluminosilicate glass, BIS-GMA, TEGDMA, Ccatalyst	061758
Composite (Solare X)	GC Corporation, Tokyo, Japan	UDMA, silica nanoparticles, prepolymerized fillers containing silica nanoparticles, fluoroaluminosilicate glass fillers	1808231
Equia Forte Coat	GC Corporation, Tokyo, Japan	25-50% methyl methacryl, 10-15% silicon dioxide, 0.09% camphoroquinone, 30-40% urethane methacrylate, 1-5% phosphoric ester monomer	1803121

Single Bond Universal	3M ESPE, Neuss, Germany	MDP phosphate monomer, methacrylate resins, HEMA, silane methacrylate, polialkenik acid copolymer, ethanol, water, initiators	620318
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Abbreviations: S-PRG, Surface pre-reacted glass-ionomer; Bis-GMA, bisphenol A glycidyl dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate. Data are provided by manufacturers.

Shear Bond Strength Testing

48 human 3rd molar teeth without caries were disinfected in 10% formalin solution. Teeth were randomly divided into 3 groups for the study (n=16), samples were prepared by cutting the occlusal surfaces of the teeth with diamond separators. This was followed by manual polishing of the dentinal surface with wet 600 grit silicone carbide paper. Teflon molds of 2 mm length and 2 mm height were placed on the flat surfaces where dentine was exposed. Restorative materials were used according to the manufacturer's instructions. Before the giomer and composite, universal bond were applied to the dentin surface for 20 sec, air dried for 10 sec and light cured for 10 seconds. The HVGIC capsule was activated just before mixing and was placed in the amalgamator immediately. The restorative material mixing time was 10 seconds and the setting time was 2 minutes 30 seconds after placing into the cavity. Afterwards, equia coat was applied and light cured for 20 sec.

Group 1:HVGIC (Equia Forte)

Group 2:Giomer (Beautifil II)

Group 3: Composite (Solare X)

The prepared samples were kept in distilled water at 37° C for 24 hours. Using a shear bond test universal tester (MTS Criterion 42, MTS Systems Corp, USA), a force was applied to the material-dentine regions of the samples with a blade-shaped tip at a head speed of 0.5 mm/ min. Fracture type analysis was undertaken by examining the surfaces where the fracture occurred with a x10 magnification stereomicroscope (DZ1100, Euromex, Netherlands).

Microleakage Testing

Twenty-seven non-carious human 3rd molar teeth were used for microleakage testing. Class V cavities of 4x2x2 mm were prepared on the buccal and lingual surfaces of the teeth, occlusal wall in enamel and gingival wall in dentin (n=9). The sizes of the preparations were measured with the help of a periodontal probe. Restorative material applications were placed in the cavities as mentioned above and the restoration surfaces were polished with medium, fine and superfine disks (Sof-Lex, 3M ESPE, St. Paul, MN, USA), respectively.

All teeth thermal cycles were placed and 5° - 55° C for 10,000 cycles were applied (20 sec applications, 10 sec wait). The root ends of the teeth removed from the thermal cycle were covered with wax and stained with two layers of nail polish, leaving 1 mm around the restoration. The samples were put in 0.5% basic fuchsin. The samples, which were kept in basic fux for 24 hours, were washed with water and embedded vertically in acrylic blocks.

Acrylic embedded in the teeth cutting device (Microcut 152, Metkon, Istanbul, Turkey) with the aid of a water-cooled diamond wheel teeth bucco-lingual was cut away. Sections were examined under a stereomicroscope at x40 magnification and microleakage scoring for both the gingival and occlusal wall was scored by two researchers using the same scale (R. M. Araujo, de Paula Eduardo, Duarte Junior, M. A. M. Araujo, & de Castro Monteiro Loffredo, 2001). The final score was created by reaching consensus among researchers.

Score 0: Paint penetration is none

Score 1: Dye penetration up to 1/3 of cavity wall

Score 2: Dye penetration up to 2/3 of cavity wall

Score 3: Axial wall including paint is not penetrated

Score 4: Dye penetration in the axial wall

Compressive Strength Test

Compression-resistant samples of Teflon molds with a height of 6 mm and a diameter of 4 mm were used according to ISO 9917-1 standard (ISO, 2007). A total of 30 samples, 3 for each of the restorative materials, were prepared as mentioned above and in line with user instructions (n=10). Samples were kept in distilled water at 37° C for 1 week. Using a universal mechanical tester (Shimadzu Ag-Xd 50kN, Shimadzu Corp, Printed, Japan), the test was performed at a head speed of 1 mm / min. The compressive strength values in N / mm² were recorded by the device.

Statistical analysis

SPSS for Windows Version 22.0 software was used for the statistical evaluation of our research data. The data related to the quantitative variables were tested with the Shapirro-Wilk normality test. The Kruskal Wallis test and the Conover tests were used as the binary comparison test since the data did not show normal distribution in the shear bond strength and

microleakage assessment. One-way analysis of variance (ANOVA) and LSD (least significant difference method) as paired comparison test were used for compressive strength assessment.

RESULTS

The means, standard deviations, and variance analyses shear bond strength (MPa) values are reported in figure 1, respectively. While the highest binding value was seen in resin composite, the lowest binding value was seen in Equia Forte. Statistically, differences were observed between groups ($p=0.0001$). A significant difference was found in the pairwise comparison of the groups ($p=0.0001$). Percentage distribution of fracture types analysis is shown in Table 2. In Equia Forte, cohesive type, Giomer and Resin composite materials were mixed most frequently. There was no significant difference in terms of fracture types between groups ($p> 0.05$).

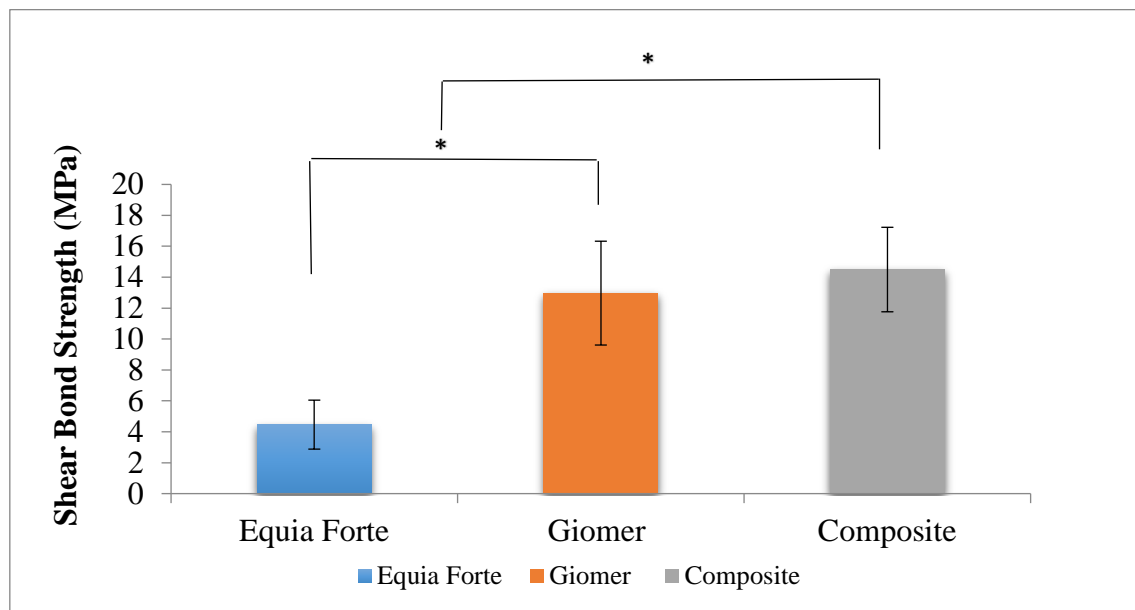


Figure 1. Average Shear Bond Strength Values of Materials. * Indicates the Difference in Pairwise Comparison $p<0.05$.

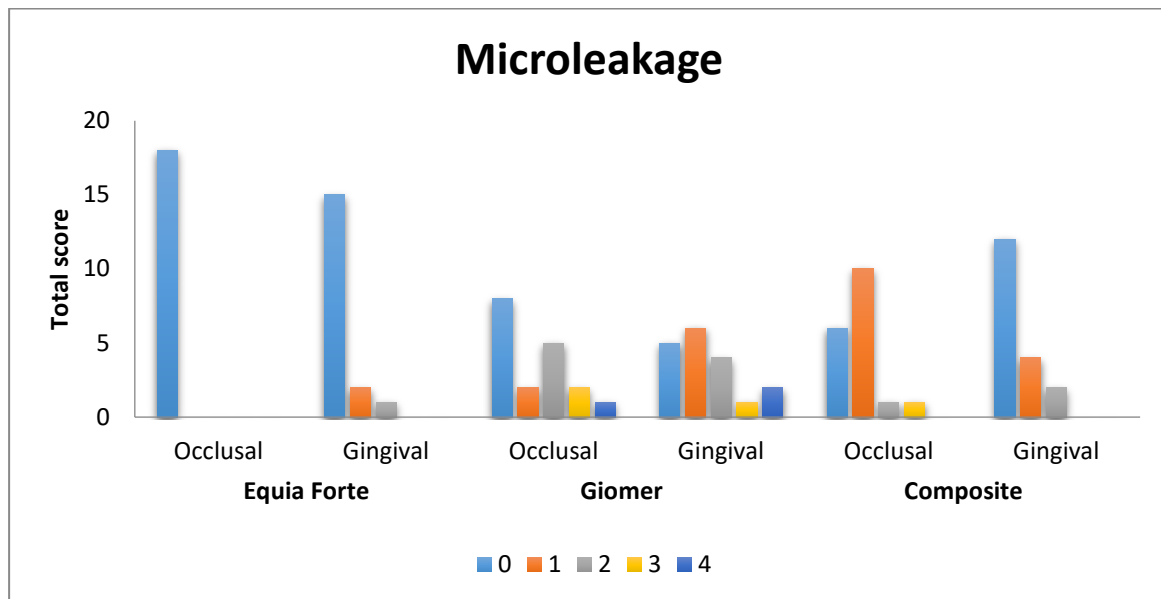
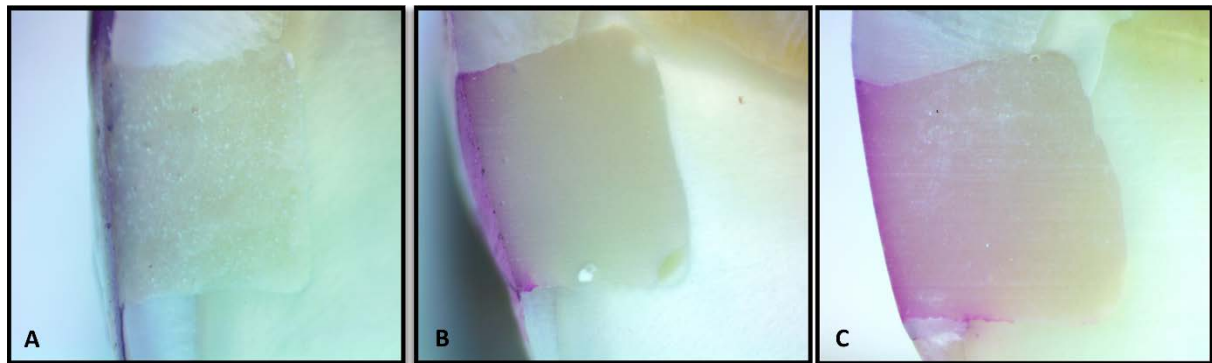
Table 2. Distribution of Fracture Failures by Materials

	Adhesive	Cohesive	Mixed
Equia forte	2 (12.5 %)	8 (50%)	6 (37.5 %)
Giomer	2 (12.5 %)	6 (37.5)	8 (50%)
Resin composite	2 (12.5 %)	5 (31.2 %)	9 (56.3 %)

The microleakage score values of the materials on the occlusal and gingival cavity walls are shown in Table 3 and figure 2. Microleakage stereomicroscope images of the materials are shown in figure 3.

Table 3. Microleakage of the Different Groups

Group	Occlusal					Gingival				
	0	1	2	3	4	0	1	2	3	4
Equia Forte	18	0	0	0	0	15	2	1	0	0
Giomer	8	2	5	2	1	5	6	4	1	2
Composite	6	10	1	1	0	12	4	2	0	0

**Figure 2.** Graph of Total Microleakage Score on Occlusal and Gingival Wall of the Materials**Figure 3.** Stereomicroscope Images According to Microleakage Scores A: Occlusal Score=0 Gingival Score=0 (Equia Forte), B: Occlusal Score = 1 Gingival Score=1 (Composite), C: Occlusal Score = 2 Gingival Score=2 (Giomer)

No leakage was observed in the glass ionomer group at the occlusal edges (enamel) ($p=0.001$). Microleakage was observed mostly in the giomer group, and no statistically significant difference was found between the composite group and the giomer group ($p > 0.05$). While the giomer group shows the most microleakage at the gingival margins (dentine), in a statistically significant ($p=0.002$) manner, other groups were found similar ($p > 0.05$). The mean and standard deviation microleakage values of all groups are shown in Table 4. While the

occlusal edges showed less leakage than Equia Forte, Giomer and Resin composite, there was no significant difference between Giomer and Resin composite. On the gingival margins, there was no difference between Equia Forte and Resin composite, but the most leakage was seen in Giomer.

Table 4. Representation of Microleakage Distributions in Occlusal and Gingival Materials

Group		Occlusal	Gingival
Equia Forte	Mean	.0000 ^a	.2222 ^c
	Std.Deviation	.00000	.54832
Giomer	Mean	1.2222 ^b	1.3889 ^d
	Std.Deviation	1.30859	1.28973
Resin composite	Mean	.8333 ^b	.4444 ^c
	Std.Deviation	.78591	.70479

Within the column, different upper case superscript letters show mean values with a statistically significant difference ($p < 0.05$). Within the column, the same lower case superscript letters show mean values with no statistically significant difference ($p > 0.05$).

Compressive strength test results are shown in figure 4. While the highest average compressive strength value was observed in Giomer (256.902 MPa), the average compressive strength value of Resin composite and Equia Forte was 195.39 MPa and 87.269 MPa, respectively. A statistical difference was observed between the groups according to the ANOVA test ($p = 0.0001$). There was a difference in the binary comparison of all groups ($p < 0.05$).

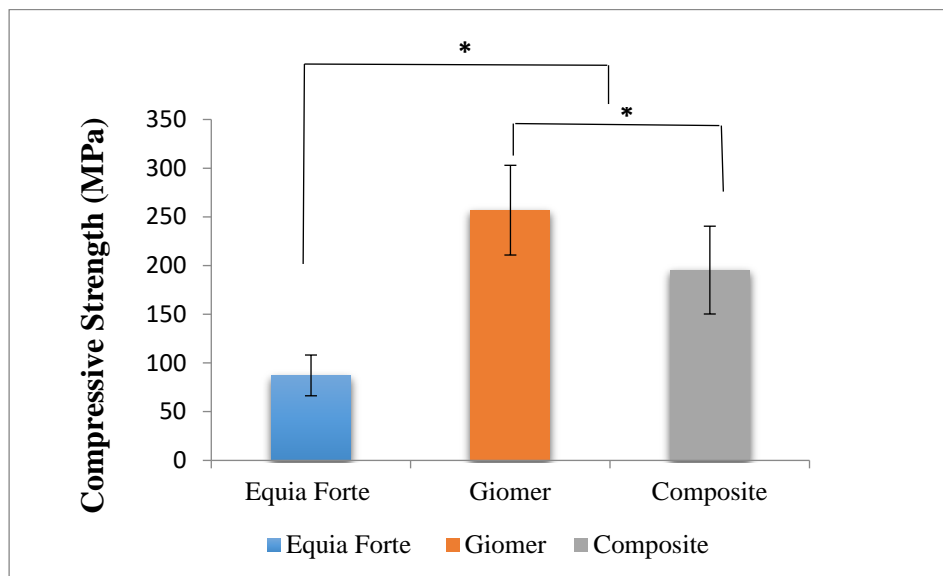


Figure 4. Average Compressive Strength Values of Materials (MPa). * Indicates the Difference in Pairwise Comparison $p < 0.05$.

DISCUSSION

HVGIC have been found to have mechanical properties as good as composite. HVGIC, which is currently a superior material over composite with fluorine release, provides evidence to the literature for the clinical use of patients with high caries incidence. This study suggests the usage of HVGIC in cavities with low mechanical load, as a result of compressive strength and bond strength tests.

The degree of connection between dental hard tissues is a very important criterion in evaluating the suitability of a restorative material offered for clinical use. Shear bond strength is a frequently used test in evaluating the binding properties of restorative materials (Chen, Huang, Kao, & Ding, 2006).

According to the result of an in vitro study by Manuja et al., while nanocomposite showed better bonding value than giomer and GIC, GIC showed the lowest bonding value (Manuja, Pandit, Srivastava, Gugnani, & Nagpal, 2011). In another study by Almuammar et al., while the highest bond strength values were seen in composite resin, glass ionomer cement showed the lowest values (Almuammar, Schulman, & Salama, 2001). The result of this study is consistent with other conducted studies. In the studies of Carvalho et al., it was stated that the studies testing the bonding strength of GIC to dentin found values varying between 1.32 - 4.10 MPa, and these values showed some improvement in HVGIC and were higher values in the range of 4.9 - 7.6 MPa (Carvalho, van Amerongen, de Gee, Bönecker, & Sampaio, 2011). In this study, Equia Forte, a glass hybrid cement with high viscosity, showed similar bonding values (4.350 MPa) with these studies.

In the study of Küçükyılmaz et al., the microtensil binding value of Equia Fil, which was pretreated with polyacrylic acid and dentine, was found to be 12 MPa. In addition, it has been reported that cohesive failures are predominantly seen and this indicates the stress that occurs within the material rather than bonding with dentine (Küçükyılmaz, Savaş, Kavruk, Yaşa, & Botsalı, 2017). The high value obtained for Equia in this study may have been caused by the use of the microtensile test method that gives high binding values and the application of polyacrylic acid. Similar to the study conducted in this study, cohesive type failure was observed more frequently, which indicates that Equia Forte could not accurately measure the bond strength to dentin.

One of the most important conditions for the success of the restoration is the prevention of microleakage by properly connecting the restorative material to the cavity walls. The inability of the restorative materials to achieve complete marginal sealing causes micro-cracks

formation, in which ion, liquid and bacteria leakage occurs, and leads secondary caries, tenderness and pulpal infections (Al-Dahan, Al-Attar, & Al-Rubaei, 2012). It is reported that approximately 30% of the cause of restoration renewal is due to microleakage (Hakimeh et al., 2000). For this reason, one of the largest factors that determines the success of the restorative material is microleakage.

In studies performed, it was reported that there was no significant difference between Equia and composite resin in terms of microleakage, but they showed the least microleakage among the other materials (Gopinath, 2017; Peker et al., 2017; Yıkılğan, Akgül, Özcan, Bala, & Ömürlü, 2016). Similar to these studies, in our study, there was no difference in dentin between Equia Forte and Solare X. In the enamel, Solare X showed more leakage than Equia Forte. The reason for this can be thought to be that the one-step self-etch adhesive system we used in our study was used without acid etching on the enamel edge.

In the study of Walia et al., giomer showed more leakage than composite resin and GIC (Walia et al., 2016). The results of this study are consistent with our study. However, in another study, no significant difference was observed in terms of microleakage on the enamel and dentin margins of the giomer and composite groups used with their own brand bond (Pasricha, 2011). In that study, it has been reported that FL Bond II used with giomer is a giomer-specific bond that strengthens the bonding interface. In our study, the failure of the giomer in terms of microleakage than the composite may be due to the fact that the FL Bond II recommended to be used together was not used.

In this study, Equia Forte was found to be more successful in terms of microleakage in both enamel and dentin. This result is thought due to polymerization shrinkage of resin-containing materials, giomer and composite. In some studies, the low microleakage values in GIC have been similarly justified.

Compressive strength is an important parameter in evaluating the mechanical properties of materials. It is used as a measure of the resistance of a material to chewing forces (Bonifácio et al., 2009). Most of the chewing forces are compressive. For this reason, it has been investigated whether the compression forces occurring during the chewing process create a breakage problem in the restorations. According to the results obtained, it was stated that the minimum resistance force of the posterior teeth against chewing forces is 125 MPa in permanent teeth and 100 MPa in milk teeth (Williams & Billington, 1989).

In this study, giomer has shown the highest compression strength value compared to composite resin and reinforced glass ionomers. In the study of Walia et al. comparing giomer

and other reinforced glass ionomer materials, the giomer group showed the highest pressure resistance. (Walia et al., 2016). These results are consistent with our study. The fact that giomer shows higher values than composite resin is thought to be due to the fact that PRG fillers in the giomer increase the durability of this hybrid structure, while the prepolymerized fillers in the composite resin structure weaken the mechanical properties (Quader, Alam, Bashar, Gafur, & Al Mansur, 2012).

In studies conducted with GIC and HVGIC, it has been reported that all glass ionomers show values above 125 MPa, which is an acceptable value for chewing pressure in permanent teeth (Bonifácio et al., 2009; Pereira et al., 2002). In a study comparing reinforced glass ionomer cements which are Equia Forte Fil, Fuji IX GP and ChemFil Rock in terms of compressive strength, Equai Forte showed a higher value than the acceptable chewing pressure value (Moshaverinia et al., 2019). Similarly, in the study conducted with Equia Forte, compression strength was reported as 358 Mpa, thereby above the acceptable value for chewing pressure in permanent teeth (Molina, Cabral, Mazzola, Lascano, & Frencken, 2013). Contrary to the studies reporting the high pressure strength values of Equia Fil, there is also a study reporting the value of 32 MPa which is below the acceptable chewing pressure (Gjorgievska et al., 2015). As a result of our study, a value lower than the chewing pressure of 87 MPa was found for Equia Forte.

When the studies on the compression strength of Equia were examined, contradictory results were observed. Therefore, more studies are needed on the mechanical properties of Equia Forte in order to recommend its' use as a permanent restorative material in the posterior region.

Considering the results of this study, materials containing glass ionomer have shown similar or superior mechanical properties compared to composite resin. One of the most important features of glass ionomer-containing materials is the fluoride release. Studies have reported that HVGICs have high fluoride release (Dionysopoulos, Koliniotou-Koumpia, Helvatzoglou-Antoniades, & Kotsanos, 2013; Mousavinasab & Meyers, 2009). Therefore, especially Equia Forte can be recommended as a permanent restorative material in patients at high risk of caries.

There are various discussions on biocompatibility of resin-containing materials as they contain toxic monomers such as HEMA, TEGDMA, UDMA and BisGMA (Reichl et al., 2006). In order to take advantage of glass ionomer cements and to eliminate the negative effects of resin-containing materials, our suggestion is that; Equia Forte, which offers strengthened

mechanical properties that are different from the traditional glass ionomer structure might among the permanent restorative materials. However, the limitation of this in vitro study is the inability to mimic biological changes as chewing forces and chemical attacks with acids and enzymes, which impair the durability of restoration in the oral cavity. Therefore, more research is needed in respect of the physical, mechanical, biological and clinical features of Equia Forte.

CONCLUSIONS

Beautiful II portrayed good properties by showing acceptable values in terms of all mechanical properties. In addition, the use of giomer may be preferred to composite resin, as it has anticaryogenic properties such as fluorine release and recharge. However, it should be kept in mind that the composite resin we used in our study has poor mechanical properties due to its prepolymerized filler content, and that a general judgment cannot be made about the superiority of giomer over composite resin materials from the results of this study.

When we evaluate all of the mechanical tests we have undertaken, we think that Equia Forte can be preferred as a permanent restorative material that can be used successfully in posterior areas that do not take in stress.

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