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ORIGINAL ARTICLE

Comparison of Surface Roughness and Microhardness of Reinforced Glass Ionomer Cements and Microhybrid Composite

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ABSTRACT

Objectives: The purpose of this study was to compare high viscosity glass ionomer cement (HVGIC), giomer and microhybrid composite using the atomic force microscopy (AFM) and Vickers microhardness. **Methods:** Three different restorative materials Equia Forte (HVGIC), Beautifil II (giomer) and Solare X (microhybrid composite) were used in this study. A total of 30 samples were prepared, 10 of each of the restorative materials used in our study. Samples were prepared using standard cylindrical Teflon molds with a diameter of 8 mm and a height of 2 mm. The measurements of surface roughness and hardness were performed by using AFM and Vickers microhardness, respectively. The surface roughness was analyzed using the Kruskal Wallis test. One-way variance analysis (ANOVA) and LSD test was used for the surface hardness ($\alpha = 0.05$). **Results:** There was no significant difference between the groups according to surface roughness values ($p > 0.05$). A statistically significant difference was found between all groups in terms of surface hardness. **Conclusion:** Reinforced glass ionomer cements had similar and surface properties than composite resin.

Key words: atomic force microscopy, composite, giomer, glass ionomer, microhardness, surface roughness

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INTRODUCTION

Surface roughness is the main factor affecting the plaque accumulation, color change, wear and aesthetic properties of the materials. The restorative material becomes more susceptible to breakage and wear as the rough surface increases the coefficient of friction.[1] Increased roughness negatively affects dental marginal integrity and resistance to abrasion. It also leads to a clinical failure by causing the staining of the restoration, plaque accumulation and gingival irritation.[2] Quantitative techniques such as surface profile analysis (Profilometer) and qualitative techniques such as scanning electron microscopy (SEM) are used to assess the surface roughness of the restorative materials. Topographic surface analysis is the most commonly used method to evaluate surface roughness.[3] AFM, which is a scanner-tipped microscope, can provide three-dimensional detailed topographic images of surface roughness in nanometer resolution. Using the atomic interactions between the tip used for surface analysis and the surface to be analyzed, the AFM device allows the surface to be measured at a very precise level (angstrom level) in the range of 100 to 150 microns.[3] AFM, which provides detailed information about the surface quality of any dental material, emerges as a satisfactory method for evaluating the roughness of restorative materials.

The eligible surface hardness of the restorative material affects the clinical success of the restoration by increasing its resistance to abrasion and preventing it from deforming under various forces.[4] Dental restorative materials are exposed to various effects such as variable pH, temperature and chewing forces in the oral cavity. The restorative material must have high hardness values to preserve the integrity of the restoration against these effects. The most commonly

Table 1. Details of the investigated restorative materials

Materials	Manufacturers	Contents
HVGIC (Equia Forte)	GC Corporation, Tokyo, Japan	Fluoro-Aluminosilicate glass, Hybrid glass particles, polyacrylic acid powder, polyacrylic acid Polybasic carboxylic acid, Distilled water
Giomer (Beautifil II)	Shofu Inc. Kyoto, Japan	S-PRG filler, Fluoroboroaluminosilicate glass, BIS-GMA, TEGDMA, catalyst
Composite (Solare X)	GC Corporation, Tokyo, Japan	UDMA, silica nanoparticles, prepolymerized fillers containing silica nanoparticles, fluoroaluminosilicate glass fillers
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

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

used test methods in surface hardness measurements are Rockwell, Brinell, Knoop and Vickers hardness tests. The test method is selected according to the type of material. It has been reported that Vickers and Knoop hardness tests can be used to measure the hardness of all dental materials such as gold, porcelain, composite resins and cements.[5] Vickers hardness test, which is one of the hardness measurement methods, creates a trace on the material when a square-bottomed pyramid-shaped diamond tip is applied with a certain force to the surface of the material sample to be measured. The hardness values obtained from the materials are inversely proportional to the size of this tip.[5]

Various modifications have been developed by adding metal, ceramic and glass fiber particles to the second phase of glass ionomer cement (GIC), and it has been tried to improve the physical and mechanical properties and antibacterial activity of GIC.[6] High viscosity glass ionomer cement (HVGIC), one of the materials that emerged due to these modifications, is a material that has reduced sensitivity to moisture in the early curing period, increased hardness and abrasion resistance.[7] Thus, it has become usable in posterior class 2 cavities where intense chewing forces are observed.[7] Giomer, which contains pre-reacted glass ionomer (PRG) fillers and releases fluoride, forms a new class of resin-containing glass ionomers.[8] This material contains the main components of glass ionomer cements. The use of PRG fillers ensures rapid fluoride release by ion exchange in the previously reacted hydrogel phase. This effect differentiates giomer from other resin-based restorative materials that release fluoride.[9]

The present study aims to evaluate the surface roughness and hardness of the use of HVGIC and giomer as permanent restorative materials, which are shown among the reinforced glass ionomer materials, by comparing it with the composite material using the AFM method and Vickers microhardness, respectively.

METHODS

The study protocol was approved by the Institutional Research Ethics Committee of Inönü University (Protocol no. 72867572/050/22780). Table 1 provides information about materials used. A total of 30 samples, 10 of each of three different restorative materials were prepared with a diameter of 8 mm and a height of 2 mm in Teflon molds. Excess material was provided to overflow from the edges by pressing.

Restorative materials were used according to the manufacturer's instructions. 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 molds. Giomer and composite were polymerized with a 1000mW/cm² LED light source (Woodpecker Led G, Guilin, China) for 20 seconds. After curing, the polishing procedure was done with the four-step polishing system (OptiDisc, Kerr, CA, USA) and using a low-speed handpiece rotating at 12,000 rpm (30 seconds for each step). Equia Forte Coat was applied on HVGIC samples and polymerized for 20 seconds. The samples were kept in distilled water at room temperature for one week.

AFM (NTEGRA-Solaris NT-MD, Moscow, Russia) was used for surface roughness measurement. The samples were adhered to a cylindrical carrier and placed into the device. Using NSG30 silicon tip, measurements were made in semi-contact mode, at a frequency of 240-440 kHz, by scanning areas of 20x20 µm. Two and three-dimensional images of the samples were obtained. Three measurements were made from each sample and the average was calculated.

Vickers microhardness was measured using the DuraScan 20 G5 (Emco Test, Kuchl–Salzburg,

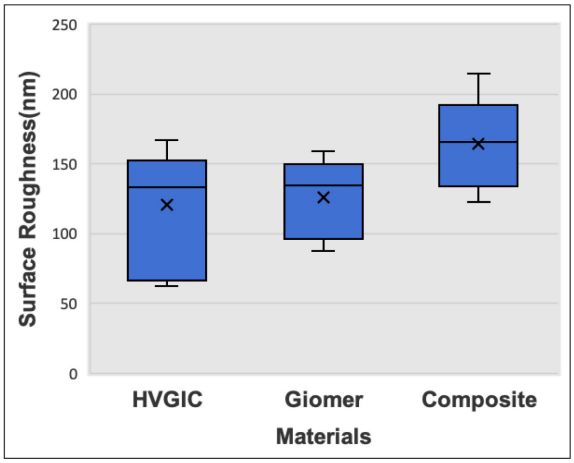


Figure 1. Box plot of restorative materials in terms of surface roughness

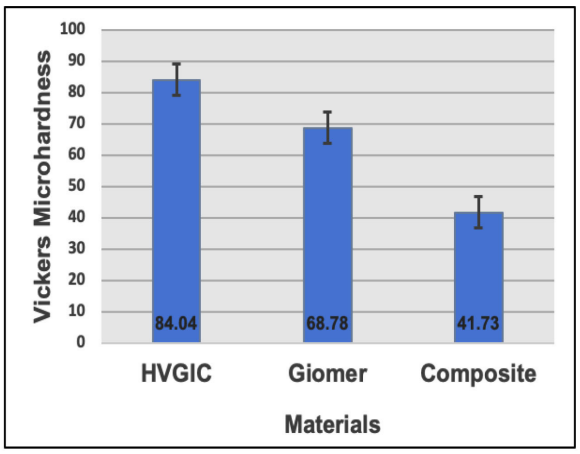


Figure 2. Vickers microhardness values of restorative materials

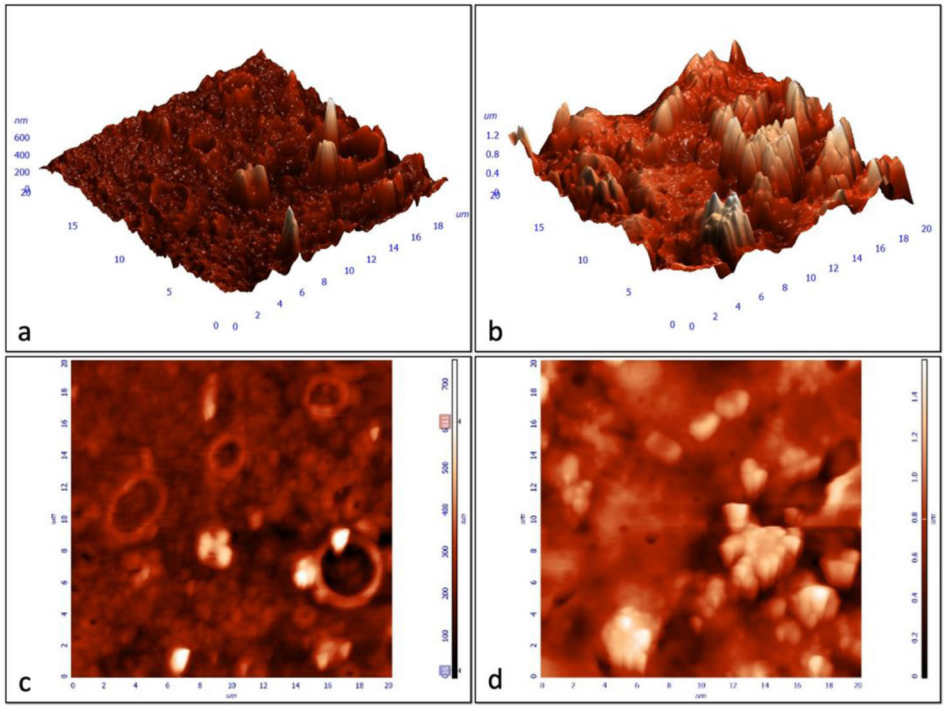


Figure 3. Two and three-dimensional images of HVGIC. a) AFM image showing the lowest roughness in the HVGIC group b) AFM image showing the highest roughness in the HVGIC group. c) and d) 2D version of the above 3D images

Austria) device. Three indentations were placed into the material and utilizing a Vickers diamond indenter while using a 50-gram load. Indentations were placed in the center of each specimen, approximately 200 µm apart from one another, with a dwelling time of 15 seconds. The Vickers hardness number (HV) of each specimen was calculated using the mean of the length of both diagonals of the three indentations.

Statistical Package for the Social Sciences (SPSS) 26 (IBM, Chicago, IL, USA) software was used to analyze the data. The mean and standard deviation of the surface roughness and microhardness were evaluated. Shapiro-Wilk tests were used to determine the distribution of the data. The Kruskal-Wallis test was conducted to evaluate the surface roughness value (S_a, nm) since the parametric test assumptions were not fulfilled. One-way ANOVA analysis was carried out to compare the microhardness among groups. Pairwise comparisons were performed using the least significant difference (LSD) with Bonferroni adjustment. The set of p-value of <0.05 with a 95% confidence interval was considered statistically significant.

RESULTS

The means, standard deviations, and variance analyses for surface roughness (Ra) values are reported in Figure 1. There was no statistically significant difference

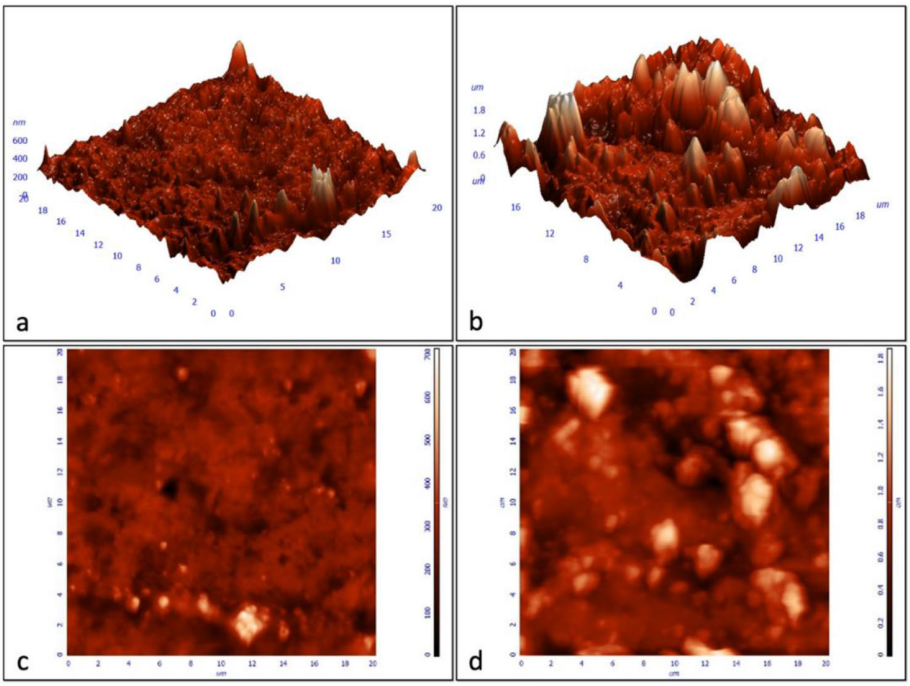


Figure 4. Two and three-dimensional images of giomer. a) AFM image showing the lowest roughness in the giomer group b) AFM image showing the highest roughness in the giomer group. c) and d) 2D version of the above 3D images

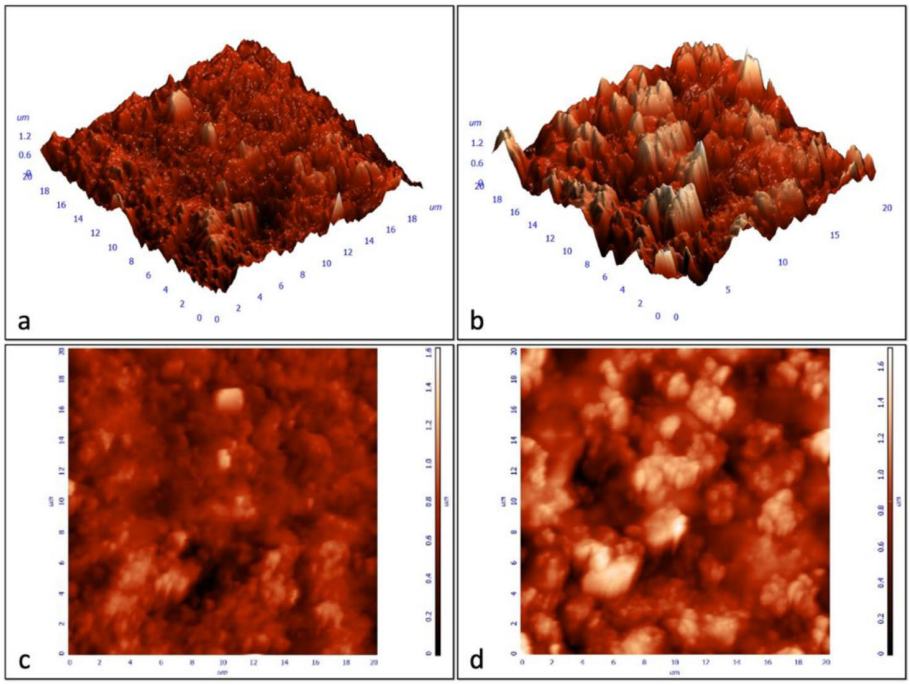


Figure 5. Two and three-dimensional images of the composite. a) AFM image showing the lowest roughness in the composite group. b) AFM image showing the highest roughness in the composite group. c) and d) 2D version of the above 3D images

between the groups concerning surface roughness ($p > 0.05$). Despite not being statistically significant, the highest mean roughness value was observed in the composite group, followed by giomer and HVGIC groups.

According to the results of the one-way ANOVA test, a statistically significant difference was observed between the groups ($p = 0.0001$). The mean and standard deviation hardness values of all groups are shown in Figure 2. The highest hardness value was monitored in Equia Forte, while the lowest was seen in Solare X (Figure 2). There was a significant difference in a pairwise comparison of all groups ($p < 0.05$).

The images presented in Figures 3, 4 and 5 are two and three-dimensional representations from the AFM scans of the restorative materials. The pictures with the

lowest and highest roughness values in each group are given in the pictures below.

When the baseline of the AFM images with the lowest roughness is evaluated, it is observed that the composite group shows a more irregular topography compared to the other groups (Figure 3-5). Low-roughness composite group samples show low height but dense peaks (Figure 5). Crater-like pits are found on the surface of samples of the HVGIC group with low roughness (Figure 3).

The irregularities on the baseline are quite prominent and severe in samples with high roughness belonging to all groups (Figures 3-5). However, in the HVGIC and giomer groups, these severe protrusions are accompanied by partially smooth flats, while in the composite group, dense and pointed protrusions are observed (Figures 3-5). 2D images also accompany 3D AFM images in terms of detecting the density of sharp peaks and the size and number of pits.

DISCUSSION

HVGIC indicated superior surface hardness than composite and similar results were obtained with the composite concerning roughness. Fluorine release, which is the common feature of GIC materials, provides an antibacterial effect and supports remineralization. However, since composites have lack of these positive properties and contain toxic monomers (e.g., BISGMA, HEMA and TEGDMA), their biocompatibility continues to be questioned. It contributes to the literature that HVGIC, which offers strengthened mechanical properties, is suitable for use as a permanent restorative material to take advantage of glass ionomer cements and to eliminate the negative effects of materials containing resin.

Surface roughness is one of the significant parameters used in the evaluation of dental materials and affecting the long-term success of the restorative material. Dental material roughness depends on the amount, type, shape, size and distribution of filler particles, type of resin matrix, ratio of filler and matrix combination, the flexibility of finishing and polishing tools, abrasive hardness and, of course, application methods.[10] In recent years, AFM has been used as a new technique in dental materials research. It has been reported that the AFM is the most effective method of determining surface roughness.[11] Hence, the AFM technique, which is a more up-to-date method in evaluating surface roughness, was used in our study.

The surface roughness values we determined in the restorative materials we used in our study were, in order from low to high; 120.2251 in HVGIC, 125.7490 in giomer and 163.9825 nm in the composite. However, no statistically significant difference was found between the materials. A threshold of 200 nm has been reported as the minimum value of ideal surface roughness in materials.[12] Based on this result, the materials used in our study have clinically acceptable surface roughness. In the study of Al-Angari et al.,[13] surface roughness of four different HVGICs and a nanohybrid composite resin were measured with a profilometer. Ra values of the materials were found in ChemFil R 0.79 μm, Premise C 0.68 μm Ketac M 0.62 μm, Equia F 0.14 μm, and 0.10 μm in Fuji IX, respectively. However, it has been reported that there is no statistically significant difference between them. The surface roughness of the HVGIC (Equia Forte) and composite resin (Solare X) used in our study is consistent with the results of the study of Al-Angari et al.[13]

Although the HVGIC used in our study was not statistically significant, it showed the lowest roughness value numerically. In previous studies examining the surface roughness, it has been reported that glass ionomer-containing cement materials have higher surface roughness values than resin composites.[2, 14]

In a study by Yap et al.,[15] composite resins (Z100, A110, Filtek Supreme Translucent, Filtek Supreme), ormocer (Admira), HVGIC (Fuji IX GP Fast), resin modified glass ionomer cement (RMGIC) (Fuji II LC) and compomer (F2000) compared the surface roughness values of eight different restorative materials. As a result of this study, they reported that the surface roughness values of resin-containing restorative materials were statistically significantly lower than those of glass-ionomer-containing materials.

Based on the manufacturer's claim and studies,[16,17] the nanofilled resin coating was recommended to be applied on glass ionomer restorations to form bright and smooth surfaces by filling and covering all surface irregularities. In addition, it is claimed that these materials increase the abrasion resistance of restorations thanks to their nano-content and enable them to be used successfully for longer.

In an in vitro study by Perez et al.,[18] using four different restorative materials (Filtek Supreme, Grandio, Vitremer, Meron Molar), they examined the effects of the glazed material (BisCover) applied to these materials on the surface roughness in three dimensions. They reported that the application of glaze significantly reduced the surface irregularities that occur after the finishing and polishing processes. The HVGIC and other resin-containing materials used in our study did not show a statistically significant difference concerning surface roughness. This result can be attributed to the resin structure of the glazing material (Equia Forte Coat) applied on Equia Forte after the finishing processes and to the removal of irregularities by filling the voids on the surface.

In our study, the value of the surface roughness of microhybrid composite resin was higher than the giomer while there was no statistically significant difference between them. These differences might be due to the prepolymerized fillers in the composite resin structure. It has been shown that the organic matrix around the prepolymerized structure has a lower hardness value than this structure.[19] It is thought that during the polishing process, the softer organic matrix is easier to separate from the surface and the formation of prepolymerized protrusions on the surface due to this situation increases the surface roughness.[19]

Valinoti et al.[20] evaluated the surface roughness of three different microhybrid composite resins (TPH, Concept, Opallis) and nano-filled composite resin (Supreme). The surface roughness value of one of the microhybrid composites (TPH) was found to be significantly lower while it was reported that there was no significant difference between the average surface roughness values of other microhybrid composites (Concept, Opallis). The researchers argued that although all three composites were in microhybrid structure, the different roughness values were due to differences in the particle size and material content of the materials. The results of this study support that the use of microhybrid composite resin with prepolymerized filler affects the surface roughness.

In the above-mentioned studies, surface roughness values of the materials were obtained with a profilometer that provides two-dimensional measurements. In this study, surface roughness values were calculated on the three-dimensional measurements in nm by using AFM. The absence of three-dimensional measurement values in nm prevents a correct comparison. Therefore, further studies with the AFM method are needed.

The mechanical properties of the materials used in clinical dentistry directly affect the success of the restorations.[21] Another the most important mechanical property of materials is surface hardness.[13] Restorative materials are exposed to many effects in the mouth. Thus, they must have high hardness values to successfully resist these effects. Low surface hardness increases the wear of the material.[13]

Moshaverinia et al.[22] compared three different glass ionomer cements (Equia Forte, Fuji IX GP and ChemFil Rock) in terms of mechanical properties. Equia Forte, which shows the highest surface hardness value among glass ionomers, was reported to have a wide range of clinical applications as a restorative dental material in dental practice.

Vijayan et al.[23] found that giomer showed a higher hardness value than composite resin, but one study reported the opposite. [24] In a study using three microhybrid composites, they obtained the lowest hardness value in composites containing prepolymerized fillers.[25] The low value of hardness of the microhybrid composite seen in our study may be attributed to containing prepolymerized fillers of the material.

Al-Angari et al.[13] reported that nanohybrid composite resin shows significantly lower hardness values than Equia Fil. In another study, the hardness of Equia Fil was found to be lower than bulk-fill composites.[26] In this study, Equia Forte was used, which is a new and improved form of Equia Fil. Equia Forte showed higher hardness than microhybrid composite. This result may be due to the addition of glass hybrid fillers to the structure of Equia Forte.

Faraji et al.[27] found that the samples which were applied to a resin coat with a nanohybrid filler were harder than an un-filled resin coat, while Bagheri et al.[28] stated that a resin coat with a nanohybrid filler reduces the hardness. In our study, Equia Forte's highest hardness value is due to the positive effects of glass hybrid filler structure on hardness. In addition to strengthening the surface of the glass content, its protective impact from the external liquid environment may allow complete maturation of the glass ionomer cement reaction with delayed water exposure.

Restorative materials should be able to the best mimic enamel and dentin, which are natural tooth tissues. Enamel, the hardest tissue in the human body, protects the tooth from mechanical damage through a hierarchical arrangement of enamel crystallites.[29] In one study, the hardness of human enamel was measured between 320 and 380 kg/mm2.[29] In another study measuring the hardness of enamel and dentin, the microhardness values for enamel ranged from 260 to 279 VHN, while dentin ranged from 46 to 53 VHN.[30] Cuy et al.[31] measured enamel hardness between 2.7 and 6.4 GPa. They attributed the different variations of this enamel hardness value seen in the studies to the degree of mineralization of the enamel, the placement of the enamel rods, and the increased porosity near the enamel-dentin junction. A study comparing the hardness of glass ionomer and composite materials with enamel and dentin found the highest hardness value in enamel, and the hardness of the materials showed similar values with dentin.[30] Yazkan and Ermiş[32] found enamel hardness of 330 VHN and roughness of 0.02 µm by using a profilometer. Another study reported that the surface structure of natural enamel has a roughness in the range of 0.59 to 0.66 µm.[33] The specified surface roughness limit (Ra) for the adhesion of dental biofilm is 0.2 µm, and it has been stated that exceeding this value causes bacterial accumulation.[33] However, this value was determined only for restorative materials, not for the enamel surface. The enamel surface is highly complex with different irregularities that allow bacterial colonization.[34] Retzius grooves, pits, minor

imperfections and mineral deposits may contribute to the presence of enamel's natural roughness.[35] In this study, while the materials did not show as successful hardness as enamel, they were close to dentin. The materials provided clinically acceptable values and smoother surfaces than enamel.

CONCLUSION

The findings obtained in the present study suggest that Equia forte and giomer were more successful than the composite concerning surface hardness. The surface roughness of the Equia Forte and giomer is similar to composite resin and conforms to established standards for permanent restorative materials.

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CONFLICT OF INTEREST

The authors declare no conflict of interest for the authorship and/or publication of this article.

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