RESEARCH

Effect of Thermocycling On Mechanical and Surface Properties of Three Posterior Restorative Materials

Sinem Akgül(0000-0001-5023-1454)[α], Ahmet Hazar(0000-0002-7106-0006)[β], İhsan Yıkılgan(0000-0001-5023-1454)[α], Suat Özcan(0000-0001-6948-6086)[α], Mine Betül Üçtaşlı(0000-0001-8646-4890)[α], Oya Bala(0000-0002-7106-0006)[α]

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ABSTRACT

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Background: To evaluate the mechanical and surface properties of two glass ionomer restorative systems (EQUIA Fil, Ionostar Molar) and a resin composite (Charisma Classic) after thermocycling.

Methods: Twenty disk-shaped samples were prepared from each material in teflon molds according to manufacturer's instructions. After the samples were stored in distilled water at 37°C for 24 h, microhardness and surface roughness measurements were performed from each group and repeated after 5000 and 10000 thermocycling. Scanning electron microscopy examinations were also performed. The data were analyzed by using Wilcoxon signed rank and Bonferroni corrected multiple comparison tests.

Results: EQUIA did not exhibit significant differences in its microhardness values after thermocycling ($p>0.005$). In contrast, Ionostar Molar and Charisma Classic exhibited statistically significant decreases in baseline microhardness after 5000 and 10000 thermocycling processes (both $p<0.005$). However, there were no significant differences between 5000 and 10000 thermocycling groups for Charisma Classic ($p=0.007$). Ionostar Molar exhibited no statistically significant differences between its surface roughness values before and after thermocycling groups ($p=0.067$). Similarly, there were no significant differences between baseline and 5000 thermocycling groups for EQUIA and Charisma Classic ($p>0.05$). However, a statistically significant increase was observed after 10000 thermocycles for both of these two materials ($p=0.002$ and $p<0.001$, respectively).

Conclusion: The EQUIA and Ionostar Molar exhibited mechanical features similar to those of a resin composite, and thus, represent promising materials for permanent restorations.

KEYWORDS

Glass ionomer, Scanning electron microscopy, Surface properties

ÖZ

Isıl Döngü İle Yaşlandırmanın Üç Posterior Restoratif Materyalin Mekanik Ve Yüzey Özellikleri Üzerine Etkisi

Amaç: İki cam iyonomer restoratif sistem (EQUA Fil, Ionostar Molar) ve bir kompozit rezinin ısıl döngü ile yaşlandırma sonrası mekanik ve yüzey özelliklerini değerlendirmektir.

Gereç ve Yöntemler: Her materyalden 20 adet örnek üretici firma talimatları doğrultusunda hazırlandı. Örnekler 37°C'de 24 saat bekletildikten sonra, mikrosertlik ve yüzey pürüzlülük ölçümleri yapıldı ve bu ölçümler 5000 ve 10000 ısıl döngü sonrasında tekrarlandı. Her gruptan bir örnek yüzey değerlendirmesi için taramalı elektron mikroskobisi ile incelendi. Veriler Wilcoxon signed rank testi ve Bonferroni düzeltmeli çoklu karşılaştırma testi kullanılarak analiz edildi.

Bulgular: EQUIA, ısıl döngü sonrası mikrosertlik değerlerinde anlamlı bir farklılık göstermedi($p>0.005$). Ionostar Molar ve Charisma Classic gruplarında başlangıç mikrosertlik değerlerine göre 5000 ve 10000 ısıl döngü sonrası anlamlı azalma gözlendi ($p<0.005$). Ancak 5000 ve 10000 döngü değerlendirmeleri arasında ise Charisma Classic grubunda anlamlı farklılık tespit edilmedi ($p=0.007$). Ionostar Molar yüzey pürüzlülük ölçümlerinde ısıl döngü öncesi ve sonrasında anlamlı farklılık gözlenmedi ($p=0.067$). EQUIA ve Charisma Classic gruplarında ise başlangıç ve 5000 ısıl döngü sonrası grupları arasında farklılık bulunmadı ($p>0.05$). Bununla beraber, her iki materyalde de 10000 döngü sonrası yüzey pürüzlülük değerlerinde anlamlı bir azalma tespit edildi ($p=0.002$, $p<0.001$, sırasıyla).

Sonuç: EQUIA ve Ionostar Molar mekanik özellikleri yönünde kompozit rezine yaklaşabilecek özellikler göstermiştir bu sebeple daimi restorasyonlarda umut verici materyaller olabilirler.

ANAHTAR KELİMELER

Cam iyonomer, Tarama elektron mikroskobisi, Yüzey özellikleri

Recently, greater diversity has been developed among commercially available dental restorative materials in response to an increased number of aesthetic requests.[1] In particular, glass ionomer cements (GICs), which were identified at the end of the 1960s by Wilson and Kent, are currently widely used in clinical dentistry.[2] Conventional GICs were previously bonded to tooth structures without adhesives. The advantages of this approach include biocompatibility, low cytotoxicity, fluoride release, and good marginal adaptation.[3,4] However, they have some disadvantages such as prolonged setting time, moisture sensitivity during initial setting, dehydration, and rough surface texture. Additionally, GICs are reported as low fracture toughness and higher occlusal wear than the other restorative materials such as amalgam and composite resin.[5,6]

More recently, to overcome these shortcomings, the mechanical properties of conventional GICs have been

[α] Gazi University Faculty of Dentistry Department of Restorative Dentistry Ankara, Turkey
[β] Bülent Ecevit University Faculty of Dentistry Department of Restorative Dentistry Zonguldak, Turkey

modified with the addition of various fillers, ultrafine and highly reactive glass particules, that increase the viscosity of these cements.[7,8] These modifications also enhance mechanical properties and improve wear resistance of materials in comparison with the traditional GICs.[9] As a result, these GICs have been widely used as a permanent restorative material. A further innovation has been the development of a restorative system that consists of a highly viscous GIC combined with a nanofilled coating material called Eqiua. This resin-based coating provides a perfect seal and increases wear resistance, while also improving the aesthetic properties of this system.[1,10,11] Another product with similar features, Ionostar Molar consists of a physically reinforced GIC, a nanofilled coating material and in an easy manipulation form.[12] However, there is not yet enough evidence about the physical properties of both restorative systems.

It has been reported that reinforced GICs exhibit improved physical and mechanical properties compared with conventional GICs.[13,14] Some of the properties that have been evaluated in characterizations of GICs are comprehensive stress, microhardness, fracture toughness, creep and wear rate.[15-17]

When a restorative material exposed to the oral environment for a long time, some changes occur in its aesthetic properties such as staining, plaque accumulation, gingival irritation and discoloration.[18] Restorations are also exposed to thermal stresses during normal oral functions. Thermal stresses disrupt the structure of restorative materials and may adversely affect their mechanical properties. In many studies, the effects of thermal stresses on restorative materials have been examined.[18,19]

Scanning electron microscopy (SEM) is another effective method for evaluating the surface topography, filler amount, size, and interface of restorations. SEM is particularly recommended for evaluating the types of failures that restorations undergo, as well as surface alterations and wear.[20,21] While the surface properties of GICs have been evaluated with SEM[22,23], studies about reinforced glass ionomer restorative systems are limited.[24]

Therefore, the aim of this in vitro study was to evaluate the mechanical and surface properties of two high viscosity, resin-coated glass ionomer restorative systems, EQUIA Fil (GC, Japan) and Ionostar Molar (Voco, Germany), as well as a microhybrid posterior resin composite, Charisma Classic (Heraus Kulzer, Germany), after thermocycling process.

MATERIALS AND METHODS

Sample preparation

Two reinforced glass ionomer restorative systems and a microhybrid resin composite (included as a positive control) were evaluated in this study (Table 1).

Table 1.

The type and composition of the tested materials

Material	Manufacturer	Type	Composition
EQUIA	GC, Tokyo, Japan	Highly viscous GIC	Powder: Strontium fluoroalumino-silicate glass
			Liquid: Aqueous polyacrylic acid, polybasic carboxylic acid, water
EQUIA Coat	GC, Tokyo, Japan	Low-viscosity nanofilled surface coating resin	Methyl methacrylate, colloidal silica, camphoquinone, urethane methacrylate, phosphoric ester monomer
Ionostar Molar	Voco, Cuxhaven, Germany	Highly viscous GIC	Powder: Fluoroalumino-silicate glass,
			Liquid: Polyacrylic acid,
Final Varnish LC	Voco, Cuxhaven, Germany	Low-viscosity nanofilled surface coating resin	Bis-GMA, Diurethane dimethacrylate, HEDMA, Catalyst
Charisma Classic	Heraus Kulzer, Germany	Microhybrid resin composite	Bis-GMA, TEGDMA, Ba-Al-F glass, SiO_2

To prepare samples for analysis, restorative materials were placed in teflon molds with a diameter of 5 mm and a depth of 2 mm and then were prepared according to each manufacturer's instructions. Initially, each mold was mounted on top of a mylar strip and a glass plate. The mylar strip was positioned on the mold and another glass plate was placed on top of the filled mold. A slight pressure was applied to obtain a standard thickness and surface. For the resin composite group, the samples were cured for 20 s with a LED light curing unit (G Light, GC, Japan) with 1000 mW/cm intensity. For the glass ionomer samples, after the self-polymerization process was completed, the mylar strips were discarded and surface coating agents were applied and light cured for 20 s. The samples were then stored in distilled water at 37 ^{0}C for 24 h. Twenty samples were prepared from each material.

Microhardness measurements

Ten samples from each of the material groups were subjected to microhardness tests. A Vicker's hardness number (VHN) (kg/mm^2) was determined for each sample prior to thermocycling by using a microhardness tester (Shimadzu HMV-2, Japan). Three indentations were made on the top of each surface with application of a 50 g load for a 15 s dwell time and an average microhardness value was

determined for each sample. The samples were subsequently immersed in a water bath and thermocycled 5000 times between 5 ^{0}C and 55 ^{0}C with a dwelling time of 15 s in each bath (MTE 101 Thermocycling Machine, Esetron, Turkey). The measurements were performed again. Thermocycling was then repeated an additional 5000 times and microhardness measurements were repeated as described above.

Surface roughness measurements

Ten samples from each material group were evaluated. Briefly, a profilometer was applied to three different points on the top of each surface (Surfest SJ-301 Mitutoyo Japan) and an average surface roughness value was determined for each sample. The measurements were repeated after the first and subsequent 5000 thermocycling processes.

SEM evaluation

One sample from each group was prepared for examination by SEM. Briefly, after the samples were dehydrated, they were gold-sputtered and examined at 1000X magnification. SEM evaluations were performed after both thermocycling processes.

Statistical analysis

Mean±standard deviation (SD) values were estimated. The Wilcoxon signed rank and Bonferroni corrected multiple comparison tests were used to analyze alterations in the surface properties of the prepared samples after thermocycling.

RESULTS

Microhardness evaluation

Mean VHN±SD values are reported for the tested restorative materials before and after the 5000 and 10000 thermocycling processes at Table 2.

Table 2.

After 24 h and after thermocycling microhardness values (mean±standard deviation) of the tested materials

Material	After 24 h of preparation	After 5000 thermocycling	After 10000 thermocycling
EQUIA	26.09± 1.37^{a}	25.13±1.30^{a}	24.52±1.90^{a}
Ionostar Molar	22.19±1.10^{b}	20.33±0.83^{c}	19.04±1.20^{d}
Charisma Classic	65.71±2.06^{e}	63.19±1.57^{f}	61.51±1.83^{f}

In each row, values with different superscript letters indicate significant differences (p<0.005) whereas same superscript letters indicate no significance differences (p>0.005).

According to the Wilcoxon signed rank test, the EQUIA material did not exhibit a significant difference after either thermocycling process (p>0.005). In contrast, the Ionostar Molar and Charisma Classic materials exhibited statistically significant decreases in their VHN values after both the 5000 and 10000 thermocycling processes compared with the VHN value at baseline (each p<0.005). For the microhybrid resin composite group, there were no significant differences between the 5000 and 10000 thermocycling samples (p=0.007).

Surface roughness evaluation

Mean surface roughness±SD values for the tested restorative materials before and after the thermocycling processes are reported at Table 3.

Table 3.

After 24 h and after thermocycling surface roughness values (mean±standard deviations) of the tested materials

Material	After 24 h of preparation	After 5000 thermocycling	After 10000 thermocycling
EQUIA	0.15±0.07^{a}	0.17±0.06^{a,b}	0.19±0.08^{b}
Ionostar Molar	0.15±0.04^{c}	0.21±0.06^{c}	0.21±0.06^{c}
Charisma Classic	0.10±0.05^{d}	0.17±0.05^{d,e}	0.19±0.07^{e}

**In each row, values with different superscript letters indicate significant differences (p<0.005) whereas same superscript letters indicate no significance differences (p>0.005).*

According to the Bonferroni correction multiple comparison test, The Ionostar Molar material exhibited no statistically significant differences in surface roughness values before and after the thermocycling processes (p>0.067). Similarly, there were no significant differences between the surface roughness values at baseline and after 5000 cycles for the EQUIA and Charisma Classic materials (p=071 and p>0.029, respectively). However, a statistically significant surface alteration was observed between baseline and after the 10000 cycle thermocycling process for both materials (p=0.002 and p<0.001, respectively).

SEM evaluation

Representative SEM photomicrographs of all of the tested materials are shown in Figure 1. Topographically, there were no apparent differences in the surfaces of the Charisma Classic resin after thermocycling. In contrast, there were large cracks and ruptures in the surface of the EQUIA samples after thermocycling, while only partial surface alterations such as little and superficial degradations were observed for the Ionostar Molar samples.

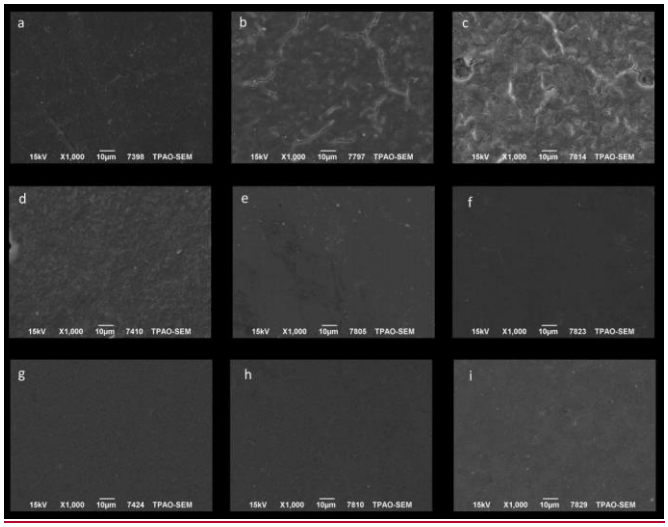

Figure 1

SEM microphotographs of tested materials. (a) EQUIA before thermocycling, (b) EQUIA after 5000 thermocycling, (c) EQUIA after 10000 thermocycling, (d) Ionostar Molar before thermocycling, (e) Ionostar Molar after 5000 thermocycling, (f) Ionostar Molar after 10000 thermocycling, (g) Charisma Classic before thermocycling, (h) Charisma Clasic after 5000 thermocycling, (i) Charisma Classic after 10000 thermocycling

DISCUSSION

GICs are widely used as permanent restorative materials due to their physical and mechanical properties. By the end of the 1990s, highly viscous GICs had been developed which were characterized by an easy application method and high mechanical properties.[25] In addition, nanofilled resin coatings were developed to enhance the longevity and wear resistance of GICs.[11] The aim of this in vitro study was to evaluate the microhardness and surface properties of two more recently developed highly viscous glass ionomer restorative systems, EQUIA and Ionostar Molar, and to compare these properties with those of a microhybrid-filled resin composite, Charisma Classic.

In general, the setting process for GICs is based on an acid-base reaction that occurs between a polyacid liquid and glass powder over several weeks.[26] The reaction begins immediately upon mixing and precipitation of the cement occurs within the first 3–6 min after mixing. Consequently, moisture contamination of these restorative materials is an important consideration for the clinical conditions of this process.[5] Gemalmaz et al. observed that early moisture contamination decreased the mechanical properties of GICs and affected surface alterations in the restorations.[27] The application of resin coating for GICs is a widespread protective approach. Resin-based coating materials prevent early moisture contamination that improves the mechanical and surface properties of GICs which play an important role in the clinical survival of the restorations. Several in vitro studies have demonstrated the positive effect of these coating materials on the mechanical characteristics of GICs.[28-30]

Thermocycling is an aging procedure that imitates rapid thermal changes in order to evaluate hydrolytic and thermal alterations that occur in materials.[31] To mimic the oral environment after a period of 6 months or 12 months, 5000 and 10000 thermocycles, respectively, have been applied[32] and were also applied in the present study. The application of this aging process to evaluate the surface properties of resin-based restorative materials was also previously demonstrated.[33] In the current study, the mean microhardness and surface roughness values of the resin composite tested significantly decreased after thermocycling. A similar result was achieved by Tuncer et al. with the application of 10000 thermocycles to other resin composites.[34] Meanwhile, the Ionostar Molar had significantly lower microhardness values after thermocycling, while the EQUIA exhibited no significant difference. When the EQUIA material was compared with zinc-reinforced GICs in a previous study, higher microhardness values were observed.[35] In another study, application of the EQUIA material with a surface coating resulted in higher microhardness values after aging compared with other restorative materials.[36] Given that EQUIA is reinforced with strontium, this property may explain these results.

In the present study, resin-based coating agents were applied to the surfaces of both of the GICs that were examined according to the manufacturer's directions. In previous studies, it was observed that resin-based coating agents that were applied to GICs surfaces enhanced the mechanical properties of the materials.[37,38] The resin components of the surface coating agents used in the present study differed from those previously used, and this may be the reason for the difference in microhardness values between the GIC groups in this study and those of other studies. It is known that the application of Final Varnish LC to the Ionostar Molar material is predisposed to water absorption due to the presence of Bis-GMA in the former[39] and absorption of water can lead to weakening of polymer structures and deterioration at the interface between the matrix and filler.[40]

When defining "surface quality", properties such as roughness, color, gloss, and morphology have been evaluated.[41] Surface roughness is a clinically important factor due to its retention potential of dental plaque and its increased risk of secondary caries.[42] Furthermore, previous studies have reported that the surface roughness of GICs is affected by filler size, shape, amount, distribution of particles in the matrix, and liquid content.[43,44]

In the current study, the Ionostar Molar samples exhibited no significant surface alterations after thermocycling, while the EQUIA samples exhibited significant surface alterations after 10000 thermocycles. The coating agent in the former was Final Varnish with Bis-GMA and was G-Coat Plus with methyl methacrylate

methacrylate (MMA) in the latter. When Zang et al. evaluated water solubility of monomers in relation to the degree of conversion for resin materials, the mobility and water solubility of Bis-GMA were found to be reduced compared with MMA due to particle size.[45] Furthermore, high water solubility of a component in a material can adversely affect the surface properties of restorations.[46] These findings are compatible with the present observations where coating of EQUIA with an MMA-containing coating agent resulted in significantly greater surface alterations after thermocycling. Correspondingly, in SEM photomicrographs, large cracks and ruptures were observed at the surface of the EQUIA samples after 10000 thermocycles, and this is attributed to the colloidal silica component of the coating agent that broke away from the surface over time. Moreover, it is possible that the remaining filler particles could have influenced the observed surface alterations of EQUIA as well. EQUIA' surface roughness values weren't compatible with SEM images, but this may be related to the examining only one sample from each group and evaluated a limited and small area in SEM imaging. Additionally, in Ionostar Molar's SEM images, there was only small surface degradations were observed in accordance with the surface roughness values.

CONCLUSION

Within the limitations of this *in vitro* study,

1. The surface properties of the GICs were found to be lower than those of the resin composite.
2. Application of thermocycling as an in vitro aging procedure may have influenced the mechanical properties of the GIC restorations.
3. The SEM images obtained showed significant alterations had occurred at the surface of the EQUIA samples after thermocycling.

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Corresponding Author:

Sinem AKGUL
Gazi University Faculty of Dentistry
Department Restorative Dentistry,
Ankara, Turkey
Phone : +90 312 203 43 99
E-mail : sinemakipek@gmail.com