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Evaluation of Glass Ionomer Restorative Materials' Surface Roughness and Microhardness In Vitro After Acidic Challenge

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Abstract

Background: This study aimed to compare the surface microhardness and roughness values of different glass ionomer-based restorative materials after acidic aging.

Methods: Three restorative glass ionomers, ChemFil Rock, Equia Forte, and Ketac Molar, were examined in this study. Glass ionomer samples with dimensions of 8 mm in diameter and 2 mm in height were produced in a Teflon mold. The samples' baseline microhardness and roughness were measured before the acidic aging process, and the measurements were taken again after the aging process. The 1-way analysis of variance test, the Bonferroni post hoc test, the Kruskal–Wallis test, and the Dunn's post hoc test were used to statistically assess the results. The significance level was set at .05.

Results: There is a statistically significant difference between the preaging material groups in terms of the mean microhardness values ($H=39.819$, $P=.000$). Accordingly, the microhardness value average of the Equia Forte group (65.57) is higher than the average of the Ketac Molar group (47.75) and the average of the ChemFil Rock group (38.31) ($P < 0.05$). After the aging procedure, the mean microhardness value of the ChemFil Rock group (36.94) was statistically lower than the mean of the Ketac Molar group (48.92) and the mean of the Equia Forte group (48.58) ($P < 0.05$). It is seen that the lowest *Ra*, *Rt*, and *Rv* values before (*Ra*: 3.05; *Rt*: 24.58; *Rv*: 10.4) and after aging (*Ra*: 2.28; *Rt*: 17.65; *Rv*: 7.52) belong to the ChemFil Rock material group ($P < 0.05$). However, no statistically significant difference was detected between the groups in terms of roughness change rates (Δ*Ra*, Δ*Rt*, Δ*Rv*) ($P > 0.05$).

Conclusion: Acidic aging adversely affected the microhardness of the materials, which is important for clinical success. The lowest microhardness value before and after acidic aging was determined in the ChemFil Rock group. No significance could be detected between the groups in terms of changes in surface roughness values.

Keywords: Glass ionomer, microhardness, acidic challenge, roughness

INTRODUCTION

Conventional glass ionomer cements are utilized in dentistry because of their biocompatibility, minimal cytotoxicity, fluoride release, and direct chemical adherence to tooth structures.[1,2]

Glass ionomer cements are indicated for class I, II, III, and IV primary tooth restorations, class III and V permanent tooth restorations, interim therapeutic restorations, and atraumatic restorative therapy.[3] However, among the shortcomings of glass ionomer cements in clinical usage are poor abrasion resistance, poor esthetics, low tensile strength, and low ultimate hardness.[4,5]

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Glass ionomer compositions are being developed to replicate the wear, durability, polishability, and esthetics of composite resins.[6] Equia Forte, a high-viscosity glass ionomer, includes a novel, higher molecular weight polyacrylic acid to improve its mechanical characteristics.[7] The ChemFil Rock restorative material is another recently discovered glass ionomer-based material.[6] Zinc is a significant component in the glass composition of ChemFil Rock.[7] According to the manufacturer, the use of zinc oxide improves the setting process and improves strength and toughness.[6] This material may have better mechanical properties because of its distinctive zinc accretion, which is projected to promote reactivity, and zinc oxide's role as a network modifier in the glass' Si–O–Si bond breaking, which increases the glass' susceptibility to acid attack.[7,8]

Studies on the mechanical and physical properties of these newly developed bioactive materials are mostly in the form of case assessments, and studies on the mechanical and physical properties of post-aging due to clinical use are few. In this study, it was aimed to compare the surface microhardness and roughness levels of 3 different glass ionomer-based restorative materials after acidic aging.

The initial null hypothesis that was investigated was that, prior to acidic aging, there would not be any variations in surface microhardness and surface roughness dependent on the type of restorative material. Second hypothesis was that the ChemFil Rock restorative material show better microhardness and roughness values after acidic aging procedure.

MATERIAL AND METHODS

In this study, an in vitro experimental study design was used to examine 3 restorative glass ionomers: ChemFil Rock, Equia Forte, and Ketac Molar. Table 1 shows the materials' components as well as the manufacturers. Glass ionomer specimens with dimensions of 8 mm in diameter and 2 mm in height were produced in a Teflon mold. The components were then placed in between 2 microscopic glass slides. The synthesis of the specimens was done by a single operator in accordance with the manufacturer's instructions. It was postponed until the chemical polymerization of the Ketac Molar restorative material had been completed, while ChemFil Rock and Equia Forte materials were polymerized with a light device. The hardening of the materials was stored at 37°C in a humid environment for 24 hours. The baseline microhardness and roughness of the samples were measured before the acidic aging process, and the measurements were taken again after the aging process.

Sample Size

The S_{pooled} value was initially established as 5.703 using the numbers acquired by Korkut et al.[9] This led to the value of the effect size being calculated as 2.282. The number of samples for microhardness analyses in each subgroup was calculated to be at least 6, and the number of samples for microhardness testing in this study was found to be 10 when the power of the study at the 0.05 level was assessed to be 80%.[9]

The impact size *d* value was discovered to be 2.087 after taking into account the results of the study conducted by Mirdas et al.[10] The number of samples needed in each subgroup for surface roughness was calculated to be at least 8, and the number of samples for surface roughness experiments was decided to be 8 when the power of the study at the 0.05 level was assessed to be 80%.[10]

Acidic Aging Procedure

The samples were placed in Coca-Cola (The Coca-Cola Company, Istanbul, Turkey) for 5 minutes 3 times a day for a week. In the intervals between the periods of acidic aging, all of the samples were stored in distilled water.[11]

Microhardness Measurement

The Vickers hardness test was used to measure the microhardness of the different restorative materials immediately and after aging. The Vickers hardness of the material surface was determined using a microhardness tester (HMV-G31 Series, Shimadzu Corporation, Kyoto, Japan) with pyramidal diamond indenter at 50 g load. Each sample was measured at 3 different surface points located at the beginning, middle, and end of the restorative material, with at least a 500 μm distance between each point. The Vickers hardness number (kg/mm^2) was recorded for each point.

Surface Roughness Measurements

A contact profilometer (Surtronic S-128, Taylor Hobson, Leicester, England, UK) was used to evaluate the surface roughness. In order to determine the surface roughness (*Ra*, *Rt*, and *Rv*) values, 3 different regions (in the middle and sides) of each specimen were assessed, and the mean value was calculated by the average of the results. The sampling length for each measurement of surface roughness was 1.5 mm, and the cutoff value was 0.25 mm. Prior to each new measurement session, the profilometer was calibrated.

Table 1. Composition of the Restorative Materials Used in the Study

Material	Composition	Manufacturer
EQUIA Forte Fil	Polyalkenoic acid, fluoroaluminosilicate glass, tartaric acid, and water	GC Corporation, Tokyo, Japan
ChemFil™ Rock	Calcium–aluminum–zincfluoro–phosphor–silicate glass, polycarboxylic acid, iron oxide pigments, titanium dioxide pigments, tartaric acid, and water	Dentsply DeTrey, Konstanz, Germany
Ketac Molar Easymix	Al–Ca–La fluorosilicate glass, 5% copolymer acid (acrylic and maleic acids), polyalkenoic acid, tartaric acid, and water	3M ESPE, Seefeld, Germany

Statistical Analysis

Data analysis was carried out in IBM Statistical Package for the Social Sciences Statistics 25.0 (IBM SPSS Corp.; Armonk, NY, USA)package program. The distributions of the variables were checked with the Shapiro–Wilk test, and the equality of variance between the groups was checked with the Levene test. For the variables showing normal distribution and equality of variance, multiple comparisons were made with the 1-way analysis of variance test, and pairwise comparisons were made with the Bonferroni post hoc test. For the variables that did not fit the normal distribution, multiple comparisons were made with the Kruskal–Wallis test, and pairwise comparisons were made with the Dunn's post hoc test with Bonferroni correction. The significance level was given as .05 in all analyses.

RESULTS

The materials' surface microhardness values both before and after acidic challenge are shown in Table 2. There is a statistically significant difference between the pre-aging material groups in terms of the mean microhardness values (H=39.819, P = .000). Accordingly, the microhardness value average of the Equia Forte group (65.57) is higher than the average of the Ketac Molar group (47.75) and the average of the ChemFil Rock group (38.31) (P < .05). Likewise, the mean microhardness value of the Ketac Molar group was statistically higher than the mean of the ChemFil Rock group (P < .05). A statistically significant difference was found between the mean microhardness values of the material groups after aging (H=20.281, P = .000). Accordingly, the mean microhardness value of the ChemFil Rock group (36.94) was statistically lower than the mean of the Ketac Molar group (48.92) and the mean of the Equia Forte group (48.58) (P < .05). There was no significant difference between the Equia Forte and Ketac Molar groups (P > .05).

The averages of the differences between the microhardness values of the material groups before and after acidic aging are shown in Table 3. It is seen that there is a higher decrease in microhardness values as a result of acidic aging of Equia Forte material (Figure 1).

The data on the surface roughness values of the materials before and after aging are shown in Table 4. It is seen that the lowest *Ra*, *Rt*, and *Rv* values before and after aging belong to the ChemFil Rock material group (P < .05). The highest *Ra*, *Rt*, and *Rv* values were determined in the Ketac Molar group. When the differences between the roughness values before and after aging were evaluated, no statistically significant difference was found between the materials in terms of *Ra*, *Rt*, and *Rv* measurements (P > .05) (Figures 2, 3, and 4) (Table 5).

DISCUSSION

Long-term clinical performance depends greatly on the mechanical qualities of restorative materials.[12] In terms of the material's resistance to chewing pressures, surface hardness is a crucial factor in the choosing of restorations.[13] When choosing a material for clinical usage, surface roughness is one of the selection factors.[14] Bacterial adherence is substantially impacted by the roughness of the restorative dental materials employed.[14] Depending on the surface change, plaque accumulation, discoloration, gingival problems, and caries may occur in restorations.[15-17] In addition, dietary intake of acidic beverages causes high tooth wear, deterioration of restorative materials, restoration failure, and problems

Table 2. Comparison of Microhardness Values of Materials

Microhardness	Material Group	Mean	Mean Rank	SD	H^a	P	Post Hoc^b
Before acidic challenge	Equia Forte	63.77	65.57	12.65	39.819	.000*	Equia Forte-Ketac Molar Equia Forte-ChemFil Rock Ketac Molar-ChemFil Rock
	Ketac Molar	52.33	47.75	12.37			
	ChemFil Rock	38.31	23.18	10.66			
After acidic challenge	Equia Forte	48.58	54.62	10.05	20.281	.000*	Equia Forte-ChemFil Rock Ketac Molar-ChemFil Rock
	Ketac Molar	48.92	54.15	13.77			
	ChemFil Rock	36.94	27.73	7.85			

[a]Kruskal–Wallis test.
[b]Dunn's test with Bonferroni correction.
*Significant P-value at .05 level.

Table 3. Comparison of Difference in Microhardness (ΔVickers Hardness Number) Values of Materials

Microhardness	Material Group	N	Mean	SD	F^a	P	Post Hoc^b
ΔVHN	Equia Forte[1]	30	−15.20	15.68	6.672	.002*	1-2 1-3
	Ketac Molar[2]	30	−3.42	17.32			
	ChemFil Rock[3]	30	−1.37	14.32			

[a]One-way analysis of variance test.
[b]Bonferroni post hoc test.
*Significant P-value at .05 level.

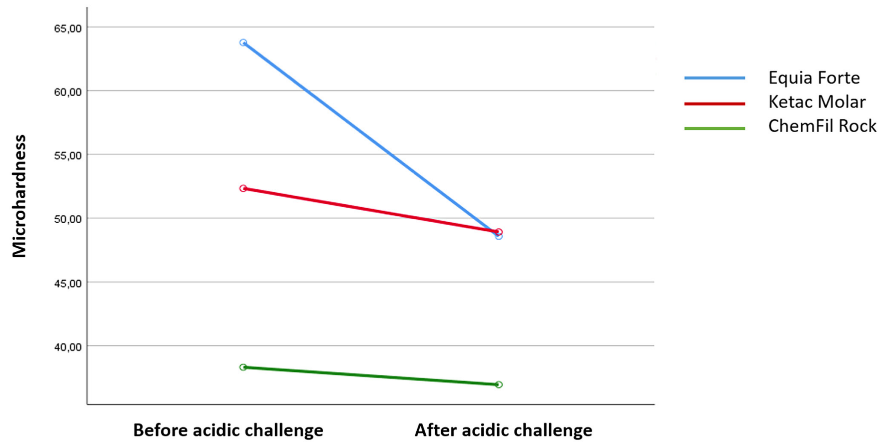

Figure 1. Change of microhardness values.

Table 4. Comparison of Roughness Values of Materials

Roughness	Material Group	Mean	Mean Rank	SD	H^a	P	Post Hoc[b]
Baseline Ra	Equia Forte[1]	3.66	34.38	2.42	4.293	.117	–
	Ketac Molar[2]	5.05	43.54	4.05			
	ChemFil Rock[3]	3.05	31.58	1.21			
Final Ra	Equia Forte[1]	2.60	34.67	1.10	6.869	.032*	2-3
	Ketac Molar[2]	4.04	45.17	2.72			
	ChemFil Rock[3]	2.28	29.67	0.90			
Baseline Rt	Equia Forte[1]	29.75	35.21	15.21	8.731	.013*	2-3
	Ketac Molar[2]	41.44	46.00	25.50			
	ChemFil Rock[3]	24.58	28.29	11.00			
Final Rt	Equia Forte[1]	23.21	36.58	12.58	9.427	.009*	2-3
	Ketac Molar[2]	32.77	45.73	24.05			
	ChemFil Rock[3]	17.65	27.19	7.21			
Baseline Rv	Equia Forte[1]	13.77	34.79	7.71	14.699	.001*	2-3
	Ketac Molar[2]	19.17	48.83	9.97			
	ChemFil Rock[3]	10.40	25.88	3.83			
Final Rv	Equia Forte[1]	10.67	38.35	5.27	15.492	.000*	1-3 2-3
	Ketac Molar[2]	15.31	47.33	10.59			
	ChemFil Rock[3]	7.52	23.81	2.94			

[a]Kruskal–Wallis test.
[b]Dunn's test with Bonferroni correction.
*Significant P-value at .05 level.

in long-term clinical use.[18] Studies have shown that acidic beverages negatively affect the microhardness and microroughness of composite and glass ionomer-based restorative materials.[19,20]

This study aimed to measure the microhardness and roughness values of glass ionomer-based restorative materials before and after acidic aging, and Coca-Cola was used for the acidic aging protocol. In previous studies, Coca-Cola was often preferred for acidic erosion due to its low pH value for acidic aging and the high corrosive effect of phosphoric acid.[21-23]

According to the results of the study, the surface microhardness value of ChemFil Rock restorative material before and after acidic aging was found to be statistically significantly lower than other materials, while the surface roughness was found to be lower. Therefore, while the first hypothesis of the study was rejected, the second hypothesis was partially accepted in terms of roughness.

Studies on ChemFil Rock restorative material, which is a glass ionomer fortified with zinc, are mostly in the form of evaluation of its mechanical properties, and there are very few studies in the literature about the effects of aging of the

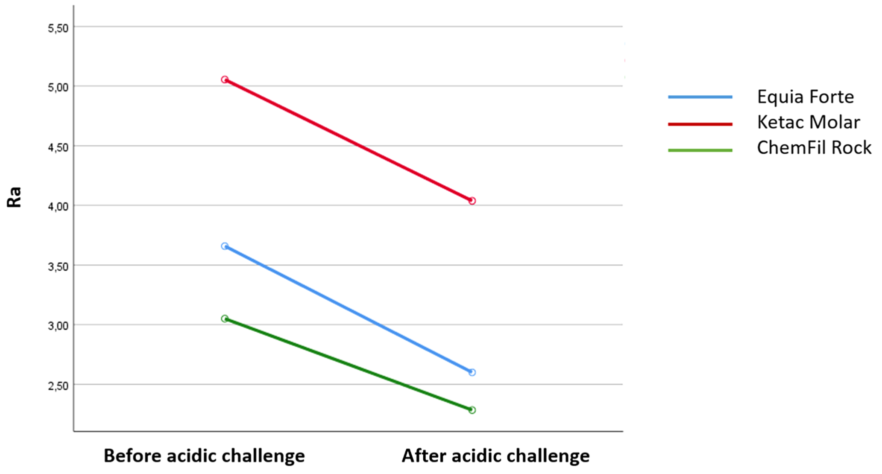

Figure 2. Change of Ra values.

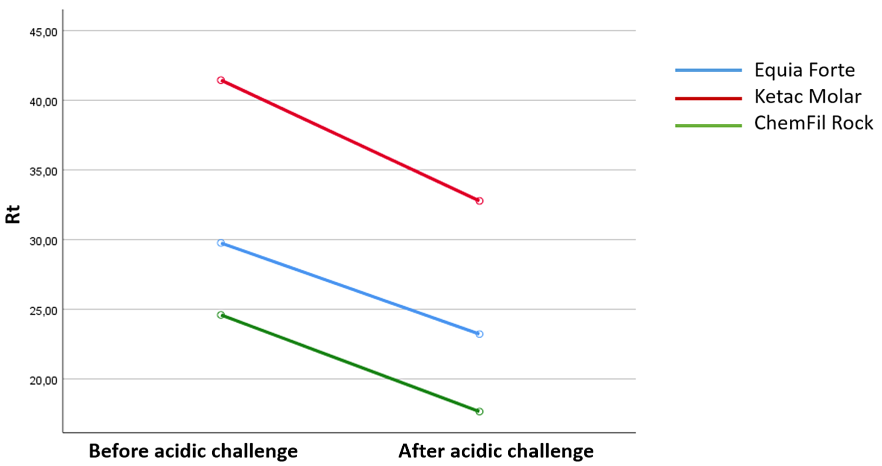

Figure 3. Change of Rt values.

material due to clinical use. In these studies, it was stated that the compressive strength, flexural strength, and tensile strength values of ChemFil Rock restorative materials were high.[24,25]

This material, according to the manufacturer, contains a unique reactive zinc-modified fluoroaluminumsilicate glass filler as well as high-molecular-weight polyacids to improve the gelation caused by hydrogen bond formation.[26] The zinc-polyacid complexes that are created by the leached zinc ions are more powerful than those made by bivalent calcium or strontium cations.

ChemFil Rock was shown to have the lowest surface hardness when compared to Equia Fil, Fuji IX GP Extra, and Ketac Molar Quick Aplicap in a study comparing the mechanical properties of the 4 materials following tooth brushing abrasion.[3] In another study, it was stated that ChemFil Rock showed superior macromechanical properties when compared to Equia Fil, Riva Self Cure, and Fuji IX GP Fast but lower micromechanical properties in Vickers hardness and elasticity modulus.[27] Kumar et al[28] reported that ChemFil Rock's values were lower than Equia and Ketac Molar quick applicap in terms of surface hardness values. In our study, ChemFil Rock showed lower values than Equia Forte and Ketac Molar in terms of surface microhardness values before and after acidic aging.

The type, size, shape, distribution, and amount of filler particles in the matrix have been observed to have an impact on the surface hardness of glass ionomers.[29] In this study, the low microhardness of ChemFil Rock is thought to be due to its hardness, gap size between the filler particles, morphology, and chemical composition of the material, and calcium aluminum-zinc fluorosilicate glass may be less strong.[30] Additionally, it is possible that the material's

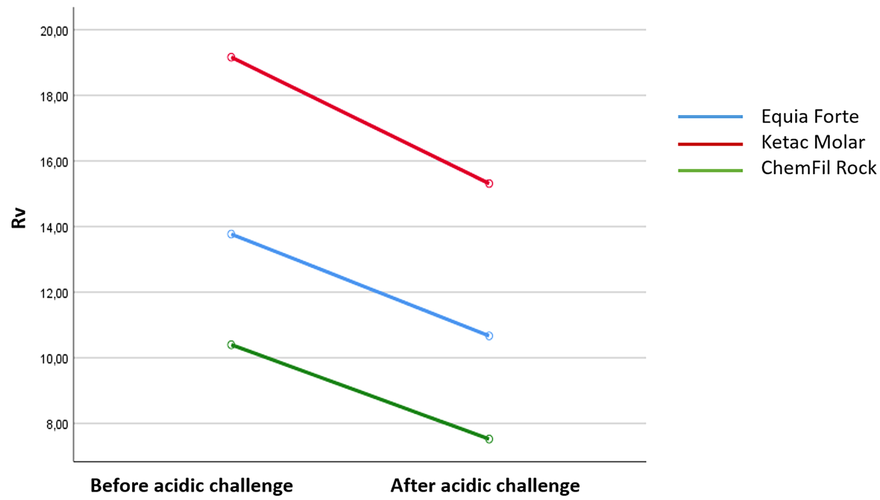

Figure 4. Change of Rv values.

microhardness was negatively impacted by the inadequate zinc dispersion on the glass particles.[31] However, the greatest change in microhardness values after acidic aging was observed in the Equia Forte material. It has been reported that the absorption of acidic beverages by glass ionomer-based materials may cause degradation and dimensional changes in the material.[32] Additionally, it is reported that water sorption and solubility of restorative materials may decrease the mechanical properties and surface coating protects initial water contamination.[24] In a study examining the weight loss of acidic drinks in glass ionomer-based restorative materials, the highest weight loss on the seventh day was detected in the Equia Forte group without coat, and the least weight loss was detected in the coat-applied Equia Forte group and ChemFil Rock group, respectively.[33] In this study, the fact that no surface coating was applied to the Equia Forte material suggests that the microhardness value of the material changed the most in this material due to the increase in liquid absorption of the material after acidic aging. In our study, the surface roughness values (*Ra*, *Rt*, *Rv*) before and after aging were found to be lower in the ChemFil Rock group compared to the other groups. In the study of Kumar et al,[28] in which they evaluated the surface roughness before and after brushing abrasion, ChemFil Rock material exhibited lower Ra values than Ketac Molar quick applic and Equia Forte. In another study measuring the weight loss on glass ionomer cements after acidic wear, the lowest weight loss was demonstrated in Equia Forte and ChemFil Rock groups.[33] The roughness sections of our analysis are supported by these earlier investigations. As a result, it is possible that the filler's size and the form of the particles account for the ChemFil Rock group's low roughness values.[34] The fact that ChemFil Rock has a very low mean particle size compared to other conventional glass ionomer cements provides another rationale for its relatively low surface roughness.[34,35]

This study's primary limitation is that it was in vitro, which means that saliva's ability to buffer acids and prevent their corrosive effects was not considered. Additionally, it has been shown that the composition of each restorative material alone determines how severe the alterations brought on by

Table 5. Comparison of Difference in Roughness (ΔRa, ΔRt, ΔRv) Values of Materials

Roughness	Material group	N	Mean	SD	*F*	*P*
ΔRa	Equia Forte[1]	24	−1.06	2.82	0.054	.947
	Ketac Molar[2]	24	−1.02	4.78		
	ChemFil Rock[3]	24	−0.77	1.54		
ΔRt	Equia Forte[1]	24	−6.54	22.25	0.051	.950
	Ketac Molar[2]	24	−8.67	33.03		
	ChemFil Rock[3]	24	−6.94	14.58		
ΔRv	Equia Forte[1]	24	−3.10	8.54	0.059	.942
	Ketac Molar[2]	24	−3.85	14.68		
	ChemFil Rock[3]	24	−2.88	5.38		

One-way analysis of variance test.
*Significant *P*-value at .05 level.

acidic solutions will be; nevertheless, other in vivo variables, including dietary practices and dental hygiene practices, must also be taken into account.

Ethics Committee Approval: This paper did not need ethical approval since the author did not perform any experiments using humans or animals in it.

Informed Consent: N/A.

Peer-review: Externally peer-reviewed.

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