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COLOR CHANGE, SURFACE ROUGHNESS, AND MICROHARDNESS OF FLUORIDE RELEASING RESTORATIVES AFTER EXPOSURE TO VARIOUS TEA SOLUTIONS AND BLEACHING TREATMENT

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ABSTRACT: Material preferences of dental practitioners for the restoration of cavities shifted in favor of fluoride (F) releasing materials as F is a powerful therapeutic and preventive agent against dental caries. Thus, this *in vitro* study aimed to examine whether various tea solutions and bleaching have an effect on the color, surface roughness, and microhardness of F releasing restoratives. Cylindrical 48 specimens of 3 high viscosity restorative glass ionomers (GIs); Riva self-cure HV, Fuji Bulk, and Equia Forte Fil (named also as glass hybrid) and a giomer (Beautifil II) were fabricated. Each group was split across 4 groups (n=12). The specimens were immersed in tea solutions (mixed fruit, black, green, and white) and then bleached with 40% hydrogen peroxide. Color, surface roughness, and microhardness measurements were done after immersing in distilled water for 24 hours (T0), tea solutions (T1) and bleaching (T2). Data were analyzed statistically ($p<0.05$). The highest color change was observed with mixed fruit tea; whereas the lowest was observed with white tea at T1 ($p<0.001$). At T2, the highest color change was recorded with Equia Forte Fil, Fuji Bulk, and Riva Self Cure HV immersed in mixed fruit tea and Fuji Bulk and Equia Forte Fil in black tea and the lowest color change was seen with Beautifil II and Riva Self Cure HV immersed in white tea. For surface roughness and microhardness, significant differences were found among groups at T0, T1, and T2 ($p<0.001$). Surface roughness increased whereas microhardness decreased at T1 and T2 in all groups. All tested materials were susceptible to tea staining and bleaching. Giomer showed better color stability, lower roughness, and higher microhardness values than the tested GIs.

Keywords: Bleaching; Fluoride releasing materials; Microhardness; Staining; Surface roughness.

INTRODUCTION

It is known that the longevity of restorations is directly associated with the durability of materials as well as their properties such as hardness, surface roughness, solubility, wear resistance, and integrity of tooth restoration interface.[1] However, from patients' point of view, aesthetic appearance is, almost always, attributed the most importance. So, discoloration and staining resistance have been major required features in restorative materials.

Currently, the material preference among the practitioners for the restoration of cavities and core build-ups is changed in favor of F releasing materials.[2] F is a powerful therapeutic and preventive agent against dental caries.[3,4] Therefore, the primary concern of many new studies is to search ways to maintain F in the oral environment, especially at the interface of the tooth and the bacterial biofilm.[4,5] F releasing restorative materials benefit from the constant interaction with the oral fluids, which influence the protective properties by release and recharge.[6] *Streptococcus mutans* and *Streptococcus sobrinus* species can be affected from F, as F has the ability to inhibit their metabolic activities related to induction of caries process.[6] Commercial F releasing restoratives are categorized as: glass ionomers

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(GIs), resin-modified GIs, and polyacid-modified composite resins (compomers) as their mechanical properties and F release capacities are different.[5]

Another F releasing restorative material group is Giomers; that combines the aesthetic, handling, and physical properties of resin composites with F release and recharge and anti-plaque effect of GIs.[7] Giomers, in which pre-reacted fluorosilicate particles were incorporated into resin matrix, exhibit similar finishing-polishing sequences and aesthetic properties with resin composites.[7,8]

Several studies have been conducted on changes caused by staining and of possible therapies either on teeth or restorative materials using various non-invasive methods.[8-10] When discoloration occurs, the common choice is usually tooth bleaching which is a conservative, low-cost, and effective technique. Hence, it is also a great importance to know the behavior of restorative materials after bleaching as they may be affected during this procedure. Carbamide peroxide and hydrogen peroxide (HP), which work the same way, are being used as the current bleaching materials.[11] There is not much evidence regarding staining followed by bleaching treatment on F releasing restoratives. Studies have been mostly focused on the effects of only bleaching agent /bleaching toothpastes on conventional or resin-modified GIs.[12-16] To the extent of authors knowledge, recently, limited number of studies were launched.[17,18] Amalavathy et al. investigated staining effect of various beverages of two F releasing tooth-colored restorative materials; Equia Forte Fil (coated with Equia Forte Coat) and Cention N (non-coated).[17] They reported that resin coated, high viscous GIs can delay the staining effect caused by various beverages. Tüzüner et al. investigated the effects of different pediatric medicines on the color stability of various restorative materials and reported that GIs seem to be more resistant to staining capacity of pediatric formulations.[18] However, none of the studies investigated the effects of bleaching after staining. Silva et al. published a review article on the mechanical and optical properties of conventional restorative GIs and stated that, it is not possible to compare the results due to the lack of standardization of the studies.[19] So, the objective of this study was to evaluate the effects of various tea solutions and following bleaching treatment on the color, surface roughness and microhardness of various F releasing restorative materials. The tested hypotheses were that: 1) immersing in tea solutions and bleaching would not have an effect on the color, surface roughness and microhardness of the tested materials and 2) there would not be significant differences among the groups.

MATERIALS AND METHODS

Sample size calculation: G*Power software (Ver 3.1, Heinrich - Heine Dusseldorf University, Dusseldorf, Germany) was used to calculate sample size with a 95% confidence interval, an 90% power, and 0.30 effect size values according to repeated measures ANOVA-type power analysis for 3 measurements and 16 groups. For each group, a minimum of 12 specimens per group was assessed to be appropriate.

Experimental design: F releasing restorative materials tested are presented in Table 1.

Table 1. Description of the materials used in the study

Material	Category	Composition	Manufacturer
Riva Self-cure HV	High viscosity glass ionomer	Fluoro-aluminosilicate glass/Polyacrylic acid/Tartaric acid	SDI Limited, Victoria, Australia
Fuji Bulk	A strengthened glass ionomer restorative	Ultrafine highly reactive glass particles/Higher molecular weight Polyacrylic acid	GC corporation, Tokyo, Japan
Equia Forte Fil	Bulk fill glass hybrid restorative	Fluoro-alumino-silicate glass/Polybasic carboxylic acid/Polyacrylic acid/Distilled water	GC corporation, Tokyo, Japan
Beautifil II	Giomer	Bis-GMA 7.5 %, Triethylenglycol dimethacrylate 5%, Aluminofluoro-borosilicate glass 7.5%, Al2O3, DL-Camphorquinone.	Shofu Dental, Tokyo, Japan
Equia Forte Coat	Light-cured resin coating	Urethane methacrylate/Methyl methacrylate/Camphorquinone/Colloidal silica/Phosphoricester monomer	GC corporation, Tokyo, Japan

A total of 212 specimens were fabricated. Five specimens from each group were used for the calibration and training and 192 specimens (n=48, each) were used for the test purposes. In each group, the specimens were further split across 4 groups (n=12). The color, surface roughness and microhardness of the specimens were recorded at three stages: after immersing in distilled water for 24 hr (T0), after immersing in tea solutions (T1), and after bleaching treatment (T2) (Figure 1).

Preparation of specimens: All specimens were fabricated using standardized stainless-steel molds (8.0 mm diameter and 2.0 mm height). A transparent mylar strip located on a glass slide was placed under the mold. Capsulated GIs were mixed according to the manufacturers' instructions using an automatic mixing device (Softly, Satelec Acteon, Merignac Cedex, France) and inserted into the molds. The giomer restorative was inserted into the molds in one increment. The molds were filled slightly excessive and then another transparent mylar strip and a glass slide were placed on the top of the filled molds, an axial load of 500 g was applied during 20 s to extrude excess material and to obtain a smooth and flat surface. GI specimens were allowed to set for the manufacturers' recommended setting time at 37ºC before being removed from the molds. Giomer was polymerized using an LED curing unit at a wavelength range of 440–480 nm and an emitting light intensity of 1500 mW/cm^2 (Radii plus, SDI, Victoria, Australia) after removal of the glass slide from both directions for 20 s. Before each use, a radiometer was used to check the irradiance of the curing unit (SDS Kerr, Orange, CA, USA). After removal of specimens from the

respective molds, they were refined using surgical scalpel blades to remove flashing material from the edges. The specimens were polished using aluminum-oxide polishing discs (Sof-Lex, 3M ESPE, Minnesota, USA). For every session, 10 strokes were made using a low-speed handpiece in one direction. The polished specimens were then cleaned ultrasonically for 2 min in distilled water and any surface debris were removed (Eurosonic energy, Euronda SpA, Italy). The recommended resin coatings by the manufacturers were used to coat each GI specimens. (Equia Forte Coat, GC, Tokyo, Japan, Japan; Riva Coat, SDI, Victoria, Austria) as recommended by the manufacturers and light cured for 20 sec. Distilled water (37ºC) was used to keep the specimens throughout the experiment in an incubator at 37ºC.

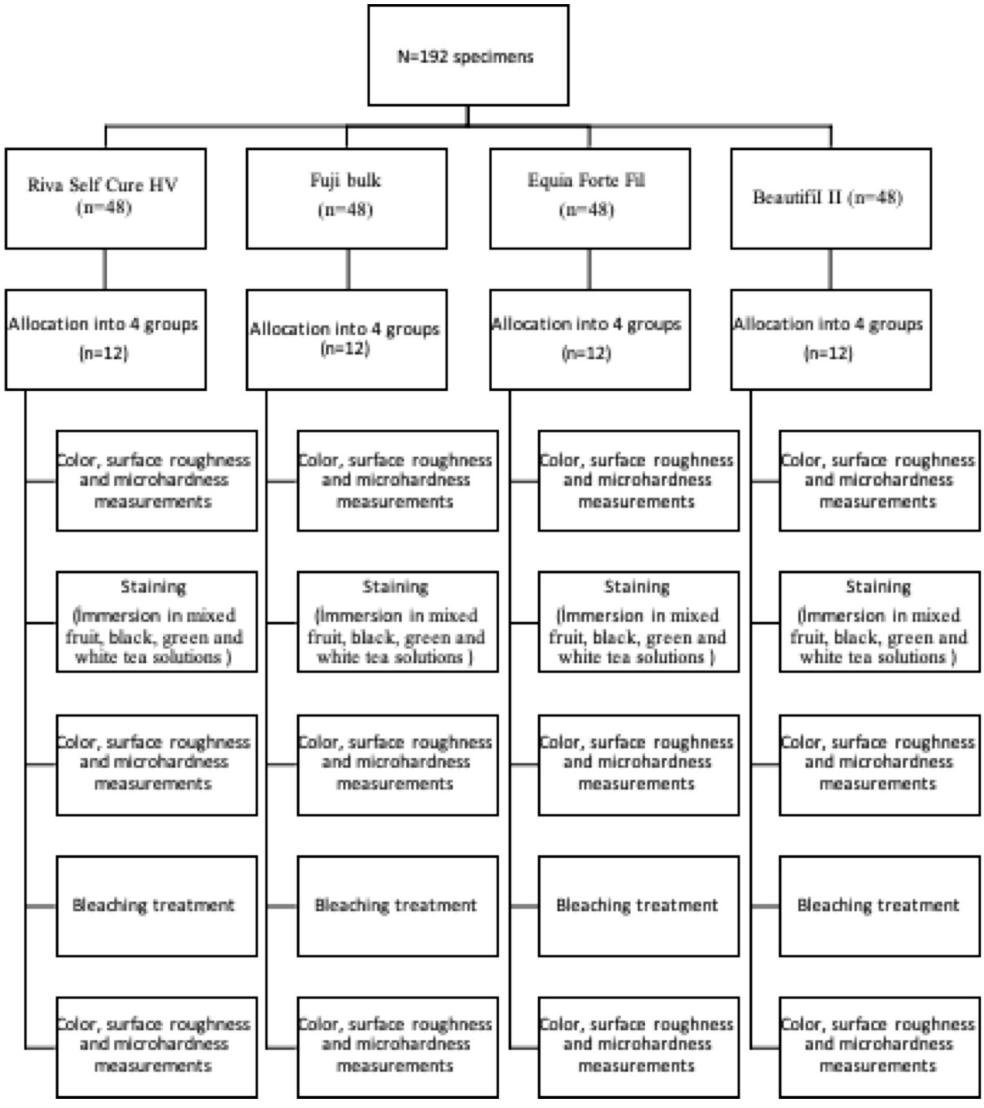

Figure 1. A flow chart representing the experimental design

Calibration: Two researchers (UKV and EM) conducted the laboratory analyses. The color, surface roughness and microhardness of 5 specimens from each group were recorded at two distinct times with a 10-day gap between records. The obtained data was used to calculate intra-examiner reproducibility and Kappa coefficient. The results revealed a high agreement for both researchers (0.916 and 0.946, respectively). One researcher (UKV) was responsible for the recording the color and surface roughness while the other researcher (EM) was responsible for conducting bleaching treatment and microhardness data of the specimens at T0, T1 and T2. The researchers were not aware of the material to remove potential bias.

Randomization: First, all specimens were numbered according to a list generated by RANDOM.ORG.; then, the sequence of the immersing into the tea solutions was randomized.

Color measurements: The color measurements were done with a spectrophotometer (CM-700d, Konica Minolta, Tokyo, Japan) equipped with Spectra Magic^{TM} NX (Konica Minolta) software. The values were expressed as CIE L*a*b* color space. L* is lightness, from white (100) to black (0), a* is red – green and b* is yellow – blue chromatic coordinates. In the spectrophotometer, D65 illumination light from pulsed xenon lamp (with UV cut filter) was used to irradiate specimens and Ø3 mm measuring aperture was selected. The device was run in the specular component exclusion (SCE) mode to remove any specular reflected light. A white reflectance standard (CM-A117, Konica Minolta) and the zero-calibration black box was used to calibrate the device. Color differences induced by immersing in tea solutions and bleaching treatment were calculated by the following formula:

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

Where

ΔE^* = Color change

ΔL^* = Change in lightness from white to black

Δa^* = Change in red-green chromatic coordinates

Δb^* = Change in yellow-blue chromatic coordinates

Surface roughness (Ra, µm) measurement: Ra was measured using a contact type profilometer device (Perthometer M2, Mahr GmbH, Gottingen, Germany). After the specimens were fixed with a special jig to ensure their position, multidirectional readings were taken in five areas of each specimen one in the center and one in every quadrant and averaged. The profilometer device was calibrated at every 5 readings.

Microhardness (VHN) measurement: A digital microhardness tester (HMV-2, Shimadzu Corp., Kyoto, Japan) equipped with a Vicker's diamond indenter was used to measure microhardness and a constant load of 100 g for 10 s was applied (20). The testing machine was calibrated at every 3 readings. Five indentations were obtained; one in the center and one in every quadrant with at least 0.5 mm distance between

each indentation and not at the margins. For each specimen, mean microhardness value was calculated for the analysis.

Staining: A concentrated solution of mixed fruit (Doğadan, Ankara, Turkey; pH=2.86±0.01), black (Earl Grey, Lipton, Rize, Turkey; pH =5.09± 0.01), green (Doğadan, Ankara, Turkey; pH=5.06±0.01) and white (Doğadan, Ankara, Turkey; pH=5.33±0.01) tea solutions were prepared by the infusion of 500 mL of water and 16 g of tea (10 sachets).[21] The pH of the tea solutions was detected by a pH meter (ISOLAB, Laborgerate GmbH, Wertheim, Germany). The specimens were immersed vertically in 10 mL of the tea solutions at 37±1ºC for 48 hr in the dark with a minimum 3 mm space between the specimens. Tea solutions were refreshed daily. After 48 hr, the specimens were removed from the respective solutions, gently rinsed with distilled water and dried with an absorbent paper.

Bleaching: The bleaching gel (40% HP, Opalescence Boost, Ultradent, South Jordan, UT, USA) was applied in a thin and even layer of about 1.5 mm on the specimen surfaces as recommended by the manufacturer and remained for 20 min in 37ºC humidity. Three consecutive applications were performed which resulted in 60 min of application time. After each session, the bleaching gel was washed with distilled water for 1 min and the specimens were then kept in distilled water at 100% relative humidity at 37ºC for 24 hr.

Statistical Analysis: SPSS 23.0 software (SPSS Inc., Chicago, IL, USA) was used to perform statistical analysis. All property mean testing results were submitted first to the Kolmogorov-Smirnov and Shapiro-Wilk tests to identify irregularities in the distribution and variance of the data. Data for the color change (ΔE), surface roughness and surface microhardness in relation to evaluation time, material and type of tea and their interactions were analyzed by three-way mixed analysis of variance (ANOVA). As the color difference data was not normally distributed, a log-transformation was applied. The Shapiro-Wilk test showed the normality of the data ($p>0.05$). The test of homogeneity of variances was performed by Levene's test ($p>0.05$). The Mauchly's sphericity test indicated that the assumption of sphericity was not met ($p<0.05$), thus Greenhouse-Geisser correction was used. Fisher's least significant difference (LSD) test was used for multiple comparisons.

RESULTS

Color change (ΔE): The mean ± standard deviation (SD) of LSD test results for ΔE are shown in Table 2. ANOVA showed significant differences among evaluation times, ($p<0.001$), materials ($p<0.001$) and tea solutions ($p<0.001$). Evaluation time and material ($p<0.001$), evaluation time and tea ($p<0.001$), material and tea ($p<0.001$) and evaluation time, and material and tea interactions ($p<0.001$) were also found significant.

At T1, all tea solutions produced color change in all materials. The highest color change was calculated after immersing in mixed fruit tea, while the lowest color change was seen after immersing in white tea solution in all groups ($p<0.001$). The highest color change was recorded in Fuji Bulk and Equia Forte Fil immersed in mixed fruit tea and the lowest color change was observed in Beautifil II immersed in white tea.

At T2, the highest color change was recorded in Equia Forte Fil, Fuji Bulk, Riva Self Cure HV immersed in mixed fruit tea, followed by Fuji Bulk, Equia Forte Fil immersed in black tea. The lowest color change was seen in Beautifil II and Riva Self Cure HV immersed in white tea.

After bleaching treatment, the highest color change was seen in Riva Self Cure HV and Equia Forte Fil immersed in mixed fruit tea, followed by Fuji Bulk and Equia Forte Fil immersed in black tea. The lowest color change was seen in Beautifil II immersed in white tea ($p<0.001$).

Significant differences were found among calculated ΔE1, ΔE2, and ΔE3 values of each group except Riva Self-cure HV immersed in mixed fruit tea, Equia Forte Fil immersed in green tea and Beautifil II immersed in mixed fruit tea ($p=0.512$, $p=0.670$, $p=0.167$, respectively).

Surface roughness (Ra, μm): The three-way mixed ANOVA showed significant differences among evaluation times ($p<0.001$) and materials ($p<0.001$). The results showed that tea solution ($p=0.648$), evaluation time, and tea solution interaction ($p=0.314$) were not statistically significant. Thus, a two-way mixed ANOVA was applied to investigate evaluation time and material and their interactions. The mean ± SD of the groups are shown in Table 3.

The LSD test showed significant differences in Ra among the groups at T0 ($p<0.001$). The highest Ra was detected as; Fuji Bulk and Riva Self-cure HV > Equia Forte Fil > Beautifil II. Ra increased in all groups at T1. The highest Ra was recorded as; Riva Self-cure HV > Fuji Bulk > Equia Forte Fil > Beautifil II ($p<0.001$). The highest Ra was detected at T2 in all groups. The highest Ra was seen as; Fuji Bulk and Riva Self-cure HV > Equia Forte Fil > Beautifil II ($p<0.001$). Significant differences were seen among Ra of each group at T0, T1 and T2 ($p<0.001$).

Microhardness (VHN): The mean ± standard deviation (SD) and LSD test results for VHN of the groups are shown in Table 4. The three-way mixed ANOVA showed significant differences among evaluating times ($p<0.001$), and materials ($p<0.001$). Evaluation time and material interaction was found significant ($p<0.001$). However tea solutions had no significant effect on microhardness and interactions with material and evaluation time ($p>0.05$). Thus, a two-way mixed ANOVA was applied to reveal the effects of evaluation time and material.

Significant differences were observed among the groups in surface microhardness at T0 ($p<0.001$). The highest VHN was recorded as; Beautifil II > Equia Forte Fil and Fuji Bulk > Riva Self-cure HV.

VHN decreased at T1 in all groups. A significant difference was seen between T0 and T1 in all groups.

The lowest VHN was detected in all groups at T2 compared to T0 and T1. The highest VHN was seen as; Beautifil II > Equia Forte Fil and Fuji Bulk > Riva Self-cure HV.

Beautifil II showed the highest surface microhardness at T0, T1, and T2. The results indicated that, the VHN of the groups decreased after immersing in tea solutions and after bleaching treatment in all groups ($p<0.05$).

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Table 2. Mean ± standard deviations (SD) of ΔE values of the groups. (ΔE* : color change; ΔE1*: distilled water - tea solutions; ΔE2** : tea solutions - bleaching treatment; ΔE3***: distilled water- bleaching treatment)

Material	Tea solution	Color change after the various treatments with ΔE1*: distilled water - tea solutions; ΔE2** : tea solutions - bleaching treatment; and ΔE3***: distilled water- bleaching treatment			p+
		ΔE1* Mean±SD	ΔE2** Mean±SD	ΔE3*** Mean±SD	
Riva Self-cure HV	Mixed Fruit	21.73 ± 7.27^{axX}	20.45 ± 4.46^{axX}	18.67 ± 5.74^{axX}	0.512
	Black	15.08 ± 2.74^{ayX}	9.49 ± 3.00^{byX}	6.44 ± 2.43^{cyX}	<0.001
	Green	19.32 ± 3.28^{axW}	7.55 ± 2.23^{byW}	12.76 ± 3.43^{czX}	<0.001
	White	10.77 ± 2.65^{azX}	4.31 ± 1.14^{bzX}	8.38 ± 2.23^{ayX}	<0.001
Fuji Bulk	Mixed Fruit	36.42 ± 8.60^{axY}	27.78 ± 12.24^{bxX}	12.97 ± 6.69^{cxY}	<0.001
	Black	29.10 ± 8.72^{ayX}	24.20 ± 10.20^{bxY}	15.46 ± 6.54^{cxY}	<0.001
	Green	27.30 ± 14.17^{ayX}	18.95 ± 7.74^{byX}	12.22 ± 8.55^{cxX}	<0.001
	White	23.05 ± 14.68^{azY}	15.83 ± 5.57^{byY}	13.38 ± 19.45^{bxY}	<0.001
Equia forte fil	Mixed Fruit	31.07 ± 11.69^{axY}	27.86 ± 11.05^{axX}	16.27 ± 2.88^{bxX}	<0.001
	Black	24.40 ± 8.05^{ayY}	22.58 ± 7.92^{axY}	14.42 ± 3.14^{bxY}	0.001
	Green	13.65 ± 5.51^{azY}	12.86 ± 6.02^{ayY}	13.85 ± 2.00^{axX}	0.670
	White	12.49 ± 5.03^{azY}	8.72 ± 5.39^{bzZ}	10.48 ± 3.02^{abxX}	<0.001
Beautifil II	Mixed Fruit	9.97 ± 2.55^{axZ}	8.38 ± 2.66^{axZ}	9.92 ± 3.22^{axZ}	0.167
	Black	9.11 ± 2.16^{axZ}	4.99 ± 1.54^{byZ}	5.63 ± 1.54^{byX}	<0.001
	Green	8.55 ± 2.22^{axZ}	4.43 ± 1.59^{byZ}	9.20 ± 2.40^{axY}	<0.001
	White	6.84 ± 2.09^{ayZ}	3.43 ± 1.17^{bzX}	4.97 ± 1.41^{cyZ}	<0.001

a, b, c for each row imply significant differences among evaluation times; for given time and material, x, y, z imply significant differences among teas; for given time and tea, W, X, Y, Z imply significant difference among materials +p<0.05.

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Table 3. Mean and standard deviations (SD) of surface roughness (Ra, µm) of the groups. (T0: after immersion in distilled water for 24 hr; T1: after immersion in tea solution; T3: after bleaching treatment)

Material	Tea solution	Surface roughness, Ra (µm), at the different treatment stages T0, T1, and T2			p+
		T0	T1	T2	
		Mean±SD	Mean±SD	Mean±SD	
Riva Self-cure HV	Mixed Fruit	0.48±0.14	0.70±0.13	0.75±0.15	>0.05
	Black	0.41±0.12	0.74±0.13	0.76±0.15	
	Green	0.47±0.10	0.68±0.26	0.79±0.24	
	White	0.46±0.10	0.75±0.16	0.78±0.16	
Fuji Bulk	Mixed Fruit	0.50±0.17	0.71±0.26	0.95±0.26	>0.05
	Black	0.43±0.19	0.55±0.24	0.57±0.28	
	Green	0.53±0.16	0.66±0.24	0.92±0.40	
	White	0.51±0.17	0.46±0.21	0.68±0.41	
Equia forte fil	Mixed Fruit	0.27±0.08	0.43±0.14	0.46±0.15	>0.05
	Black	0.26±0.19	0.40±0.16	0.55±0.15	
	Green	0.27±0.08	0.32±0.16	0.42±0.12	
	White	0.28±0.11	0.50±0.14	0.54±0.23	
Beautifil II	Mixed Fruit	0.09±0.07	0.05±0.02	0.14±0.09	>0.05
	Black	0.09±0.04	0.11±0.09	0.12±0.07	
	Green	0.09±0.08	0.13±0.10	0.18±0.08	
	White	0.05±0.03	0.08±0.04	0.19±0.22	

+ $p<0.05$.

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Table 4. Mean and standard deviations (SD) of microhardness (VHN) of the groups. (T0: after immersion in distilled water for 24 hr; T1: after immersion in tea solution; T3: after bleaching treatment)

Material	Tea solution	Microhardness, VNH (µm), at the different treatment stages T0, T1, and T2			p+
		T0	T1	T2	
		Mean±SD	Mean±SD	Mean±SD	
Riva Self-cure HV	Mixed Fruit	58.33±11.45	56.91±9.97	48.98±8.69	>0.05
	Black	59.56±8.74	56.76±8.22	46.06±6.80	
	Green	61.31±9.78	59.72±10.15	50.31±9.31	
	White	61.38±11.74	61.30±11.36	55.92±6.67	
Fuji Bulk	Mixed Fruit	75.53±9.61	75.92±9.16	68.86±9.97	>0.05
	Black	76.39±15.68	73.41±12.42	64.02±9.59	
	Green	79.94±13.29	77.16±13.96	71.26±14.90	
	White	84.67±9.46	82.98±8.68	78.44±6.88	
Equia forte fil	Mixed Fruit	78.28±15.26	78.28±15.26	72.80±22.89	>0.05
	Black	82.85±20.17	79.12±20.35	73.78±15.21	
	Green	86.83±25.69	86.06±24.99	76.99±18.10	
	White	89.51±22.54	88.86±21.41	86.36±18.91	
Beautifil II	Mixed Fruit	95.39±9.68	88.54±11.04	76.45±7.38	>0.05
	Black	95.51±14.53	86.86±16.32	74.75±15.81	
	Green	97.05±10.75	97.13±10.81	83.95±11.23	
	White	99.01±24.83	98.75±25.97	93.55±15.77	

+p<0.05

DISCUSSION

In the present study, the effects of various tea solutions and bleaching treatment on the color, surface roughness, and microhardness of novel F releasing restorative materials, including three GIs and a giomer were tested. The first hypothesis claiming tea solutions and bleaching would not have an effect on the color, surface roughness, and microhardness of the tested materials was rejected as tea solutions and bleaching significantly affected the color of the materials tested. The second hypothesis was also rejected as significant differences were found in surface roughness and microhardness among materials tested after immersing in distilled water for 24 hr and after immersing in tea solutions or bleaching treatment.

Staining susceptibility of restorative materials might be credited to the variations in their compositions and setting reaction, degree of water sorption and hydrophilicity of the matrix resin; the water, presumably acting as a penetration vehicle. It was shown in previous studies that both resin composites and GIs are susceptible to staining in various staining media but the degree of color change is material-dependent.[22,23] Hydrophobic materials, such as resin composites, are known to have higher stain resistance and color stability than hydrophilic materials such as GIs or compomer.[24,25] In line with the current literature,[24-26] our results showed that, different tea solutions, which were used due to their major consumption by the population, caused significant and clinically perceptible color changes (>3.3) after immersion. Additionally, the color change in giomer was lower than the values obtained for GIs tested. Mixed fruit and black tea produced the highest increase in all groups. This could be explained by the high amount of black, purple and red pigments in mixed fruit and black tea solutions. On the contrary, white tea solution showed the lowest color change. This tea was purely produced only silvery leaves of the upper buds of the tea plants and the absence of dark pigments may have caused the materials to be less colored.

Another factor that may facilitate staining mechanism, is the porosity of the material. The GIs may have inner porosities due to the formation of air bubbles during the mixing or placing process.[27] Nomoto et al. reported that the method of mixing created very small differences on porosity of high viscosity GIs.[28] GIs with higher viscosity tend to have more inner porosity which is related to the strength of the material and superficial roughness.[27,29] In this study, the GIs were coated with the respective coating agents that the manufacturers recommended. However, Thongbai-on et al. reported that coating had no effect on decreasing the materials' porosity.[29] Therefore, it is reasonable to hypothesize that the inner porosities of the GIs had an influence on the materials' staining susceptibility, surface roughness and microhardness in the current study.

The effects of bleaching on the restorative materials have been largely investigated with a wide variation in results but these studies were mostly focused on resin composites.[30-33] However, little is known about the effects of bleaching on GIs and giomers. In this study, HP was applied as it is known to have high capacities for oxidation and generate free radical species.[34] The application of 40% HP resulted in the bleaching of the tested restorative materials in agreement with previous studies reporting higher color changes in compomer and GIs than the resin composites.[25,31,35,36] Although the mechanism of color changes of the restorative

materials induced by bleaching is still not clear, it was proven that, the success of a bleaching treatment is directly related to the diffusion capacity of peroxides.[37] This may explain the higher color changes in compomer and GIs than the resin composites in the present study. Inner porosities of GI material could probably allowed better diffusion of hydrogen peroxide.

It was shown that, bleaching affected not only the color of the materials, but also their surface roughness and microhardness.[34,38] Taher reported that after bleaching with 35% HP, the microhardness of the resin modified GI was decreased 23.1%.[36] Yu et al. investigated the effect of bleaching on compomer, conventional GI and resin composite surfaces and reported that surfaces of all materials were roughened and softened after bleaching with 40% HP.[34] This may also be due to the detachment of some fillers leading to produce an irregular matrix surface. So, a decrease in microhardness and an increase in surface roughness observed in all groups in this study after bleaching are in line with the current literature.[34,38]

Although it is claimed that the polishability of contemporary GIs is increasingly approaching resin composites; one of the major flaws of today's GIs is weaker polishing properties compared to their predecessors.[32] A surface roughness of 0.2 μm is accepted as a threshold value for bacterial retention.[39] The results of present study showed that, the surface roughness of the giomer was below the 0.2 mm threshold, while all GIs tested were above, indicating the importance of need of more research to improve surface characteristics of the GIs. The results of this study correlates with the study of Bayrak et al.[40] They showed smoother surface structure of giomer than GIs, but rougher than resin composite. This may be due to the particle size of the materials as it has been shown to have a powerful impact on the polishability of the materials. Some studies reported high surface roughness values for the materials having larger particle sizes.[41,42] GIs tested in this study are composed of glass particles of 4–25 μm in size whereas giomer is composed of 1–10 μm sized pre-reacted fluorosilicate particles which allow a smoother surface profile than GIs. This may explain the differences in surface roughness at baseline, after immersing in tea solutions and after bleaching among the groups. Additionally, differences such as shape, distribution and number of fillers, interfacial bonding between fillers and matrix of the materials and size of glass particles can play a role.[42]

Microhardness is a crucial physical property of a restorative material and may be defined as the resistance of the material to wear, indentation and/or penetration. The results of this study revealed that immersing in tea solutions and bleaching significantly reduced the microhardness of all groups with significant differences among materials and evaluating times.

The resistance of the GI restorative depends on various factors; such as the chemical composition, glass structure, molecular weight, concentration of the polycarboxylic acid, and the powder/liquid ratio. Additionally, it is well known that, after the initial hardening, although the details of these processes are unknown, further reactions, which occur slowly and are together known as maturation, take place. Strength, translucency and the proportion of tightly-bound water within the material typically increase.[43] The present study showed that the microhardness of the test specimens in all groups decreased after immersing in tea solutions. This may be the result of the water sorption by the matrix that could cause in plasticization,

softening, and hydrolysis of the materials.[44] Considering giomer, this immersion may have altered the resin matrix, causing the exposure of filler particles.[45] As well as water sorption, the erosive potential of the tea solutions should not be disregarded. The erosive potential of acidic drinks depends on chemical factors (e.g., pH, titratable acidity, mineral content, and clearance on tooth or restoration surface) and also calcium-chelation properties. Reddy et al. examined the erosive potential of some herbal and fruit teas on the market and highlighted the effect of lower pH which have erosive potential on dentition.[46] In the current study, the pH of the all tea solutions tested were ranged between 2.86–5.33. This may explain the reduction in the microhardness and increase in the surface roughness of GIs in line with the current literature.

As the present study was conducted *in vitro*, it does not simulate clinical conditions precisely by the absence of the influence of saliva and masticatory stresses on restorative materials. So, *in vivo* studies are needed to achieve more precise results and to confirm the capability of these materials.

CONCLUSIONS

Within the limitations of the present *in vitro* study; it can be concluded that 1) tea solutions caused significant color changes on the tested three GIs and the giomer. The higher color changes were seen when the tested materials were immersed in mixed fruit tea and lowest color change was seen with white tea in all groups; 2) bleaching decreased the color of the tested materials stained with tea solutions; 3) tea solutions and bleaching increased the surface roughness of the tested materials with no significant difference among tea solutions; and 4) tea solutions and bleaching decreased the microhardness with no significant difference among tea solutions.

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