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Effect of Artificial Saliva Storage on Microhardness and Fracture Toughness of a Hydrothermal Glass-Ceramic

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Abstract

Purpose: This study evaluated the effect of artificial saliva storage on the hardness, crack length, and fracture toughness of a glazed, polished, and bleached hydrothermal low-fusing glass-ceramic (Duceram LFC).

Materials and Methods: Forty ceramic discs were constructed. The discs were assigned to four groups (n = 10) according to their surface finish: Gp1—Autoglaze, Gp2—Autoglaze/ground/diamond-polished, Gp3—Overglaze, Gp4—Overglaze/ground/diamond-polished. Each group was further divided into two subgroups forming eight total subgroups (n = 5). Subgroup A was unbleached; Subgroup B was bleached. Testing was performed before and after 21 days of artificial saliva storage. Data were presented as means and standard deviation (SD). ANOVA was used, along with Duncan's post hoc test for pairwise comparison between the means when ANOVA test was found significant ($p \leq 0.05$).

Results: Surface treatments such as glazing, polishing, and bleaching, saliva storage, and the interaction between these variables had a statistically significant effect on mean values of microhardness, crack length, and fracture toughness of the specimens. There was a statistically significant increase in microhardness and fracture toughness mean values, while crack length values decreased after saliva storage. Polished specimens recorded the smallest crack lengths and fracture toughness, and highest hardness values before and after saliva storage. No difference in fracture toughness values was evident between glazed and polished specimens. Mean crack lengths decreased after saliva storage in all the tested specimens. Hardness values increased after saliva storage. The autoglazed group showed significantly higher fracture toughness, lower crack length, and microhardness than the overglazed group.

Conclusions: Surface finishing procedures and artificial saliva storage had a statistically significant effect on mean values of microhardness, crack length, and fracture toughness. This in vitro study suggests that fracture toughness of ceramics may be affected by different surface treatments such as glazing, polishing, bleaching, or a combination; however, in this study Duceram LFC proved its self-healing property after 3-week storage in artificial saliva.

Ceramic materials are susceptible to stress corrosion, dynamic fatigue, and surface degradation, which affect their strengths. Environment-assisted crack growth is manifested as a decrease in flexural strength on immersion in water. This is brought about by a reduction in the energy required at the crack surface due to the action of the aqueous environment to create vacancies at the crack tip, thereby decreasing the energy required for crack growth. Crack growth occurs by chemical degradation of the silicate network (-Si-O-Si-) in the ceramic material.[1] It has been postulated that water reacts with the molecules at the crack tip, breaking the (-Si-O-Si-) network to form hydroxyl ions.[2] The resultant OH^- ions act as catalysts during the hydrolysis of the silicate bonds. Stress corrosion theory predicts that basic solutions react strongly with silicate glasses, and therefore are expected to weaken rather than strengthen the glass.[3]

According to methods developed by Bartholomew at Corning Glass, more chemically resistant low-fusing glasses could be formed through the addition of hydroxyl groups to the glass.[4]

Ducera manufacturers claim to have commercialized this process in its Duceram LFC (low-fusing ceramic) dental porcelains. It sinters at 660°C, that is, 30% lower temperature than conventional ceramics and shows a 40% increase in its flexural strength after hydrolytic testing.[7] Its strength increase is attributed to a region of strongly hydrogen-bonded hydroxyl groups on the outer surface of the material, "a surface layer" formed through exchange of alkali cations and hydroxyl groups. Since this region would not be as rigid as a fully polymerized glass network, it is believed to be able to deform plastically instead of by brittle fracture, and this hydrolyzed layer is more ductile than bulk porcelain.[5-12] The SiOH region is expected to be approximately 3-μm thick and is thought to heal surface flaws and protect the surface against damage. Because of its manufacturing process, the company expects the SiOH region to be regenerated in the mouth after it is damaged or removed by mastication. Duceram LFC is said to be a leucite-free glass with claims of a 25% increase in flexural strength after immersion in boiling water for 24 hours or after 14 days in artificial saliva.[5,6] Moreover, this material is thought to possess lower hardness values when compared to other ceramics due to its leucite-free composition adding to its low abrasiveness.[7] This was also confirmed by other authors, who found that the flexural strength of Duceram LFC was increased after 16-hour exposure to 4% acetic acid. Scherrer et al[8] also reported a significant increase in its fracture toughness after aging in water. Such a behavior suggests an ion exchange mechanism, which modifies the surface's structural arrangement after exposure to specific environments.[8,9]

Jestel et al[6] analyzed the surface of Duceram LFC specimens after exposure to water and 4% acetic acid. They demonstrated that structured zones containing metaborate, pyroborate, boroxol rings, and a mixture of borate and silicate tetrahedra developed on the outer surface of the glass. They hypothesized that the increase in strength after exposure to water or acetic acid was due to this newly developed 3-μm thick boron-containing surface layer;[9-11] yet a strengthening mechanism linked to the exposure to water would be unique among ceramic restorative materials, as most feldspar-based ceramics show strong tendencies toward stress corrosion,[13-18] a phenomenon originating in an increasing hydrolysis of the ceramic when subjected to stress application and resulting in a time-dependent reduction in flexural strength.[19-24] Kelly[25] reported that failure response of brittle materials was governed by load, contact area, and elastic moduli, in addition to the participation of water in the phenomenon of chemically assisted crack growth or static fatigue.

The limitations of laboratory strength testing as an indicator of the structural performance of brittle materials have been pointed out by Yilmaz et al.[14] In contrast, an appropriate parameter would be the ceramics' fracture toughness (KIc), that is, the material's intrinsic resistance to crack propagation.[14,25,26]

Dental restorative materials are subjected to intermittent forces during mastication with maximal occlusal forces ranging from 200 to 1000 N.[27-29] When forces of this magnitude are applied to minute surfaces, such as during tooth contact, substantial stresses are generated, and each stress is capable of creating a corresponding deformation or strain in contacting bodies. Brittle dental ceramics are incapable of absorbing appreciable amounts of elastic strain energy before fracture.[30] The resistance of a material to crack propagation is defined as fracture toughness and is one measure of the strain-energy-absorbing ability of brittle materials.[31] The fracture toughness of a material is related to the tensile stress that must be achieved in a crack tip before fracture is initiated.

Several techniques have been proposed to assess the fracture toughness of brittle materials.[31] These methods include the double cantilever beam, double torsion, notch bend, and indentation techniques. The theoretic concept that supports application of the indentation technique involves the direct measurement of radial crack length as a function of indentation load and is well established in the literature, particularly for homogeneous single-phase ceramic materials.[32-39]

Dental ceramics can fail through growth of microscopic surface flaws that form during processing or from surface impact during service.[37] Therefore, fracture toughness is a critical property to consider when selecting a dental ceramic restorative material as it indicates the serviceability of a material intraorally by using a crack growth parameter similar to those produced clinically. Flaws of controlled size, shape, and location are introduced, followed by direct measurements of radial cracks. In addition, any procedure undertaken by the operator, which may decrease the fracture toughness of a material during function, will precipitate its failure and limit its serviceability.[14,37]

Bleaching is a common procedure currently performed by many dentists without realizing the potential danger to ceramic restorations. The changes recorded in bleached enamel include pitting and erosion for lower pH solutions,[38] increase in enamel wear rate of bleached teeth,[39] decrease in fracture toughness, and decrease in hardness in the outer enamel.[40-42] According to McGuckin et al,[41] bleached enamel appeared to resemble acid-etched enamel. Regarding the bleaching effects on restorative materials, several studies showed contradicting results. Hunsaker et al[43] studied the effect of seven brands of bleaching gels on dentine, enamel, and restorative materials and concluded that no major changes were observed with scanning electron microscopic examination. Other authors agree,[44-46] while various others reported changes such as decreased hardness, increased roughness, and color change.[47-51]

Turker and Biskin[50,51] investigated the effects of three home-bleaching agents on the microhardness of various dental esthetic restorative materials. All the bleaching agents decreased the microhardness of the porcelain and increased that of the light-cured modified glass-ionomer cement.

The functional surfaces of porcelain are often ground to adjust occlusion. This procedure introduces flaws within the material.[37] From the presented data it would be fair to assume that ceramic failure is induced from flaws in the material in addition to chemically assisted crack growth. This study was undertaken to evaluate the effect of artificial saliva storage on the hardness and fracture toughness of a glazed, polished, and bleached hydrothermal low-fusing glass ceramic (Duceram LFC).

Materials and methods

Forty ceramic discs, Duceram LFC (Ducera, Rosbach, Germany) 12-mm diameter, 2-mm thick, were constructed for this study using a circular split Teflon mold ring for standardization. Disc-shaped specimens were produced by a condensation

technique using predetermined proportions of dentine powder and modeling liquid according to the manufacturer's instructions. The slurry was packed and vibrated into the ring while the excess liquid was removed using an absorbing tissue. The discs were fired in a programmable and calibrated vacuum furnace, according to the manufacturer's recommended firing cycles: air-fired at 450°C for 360 seconds, vacuum-fired at 660°C for 60 seconds, and air-fired at 660°C for 60 seconds.

Two corrective firings were performed after grinding. Caliber control and defective specimens were adjusted by porcelain powder addition and corrective firings. A final enamel firing was done. The fired discs were allowed to air cool to room temperature then ground flat with diamond stones (30 μm, Komet-Brasseler, Lemgo, Germany), followed by progressively finer abrasives (15 μm, Komet) using a slow-speed hand piece (KaVo Model K9; KaVo America, Lake Zurich, IL) at 20,000 rpm. The ceramic discs were sonicated in distilled water for 10 minutes. The discs were assigned to four groups (n = 10) according to their surface finish:

- Gp1. Autoglaze,
- Gp2. Autoglaze/ground/diamond-polished,
- Gp3. Overglaze, and
- Gp4. Overglaze/ground/diamond-polished.

Specimens were ground with diamond stones and polished with diamond paste according to the following regimen:

- Diamond stones (125 μm) were used to simulate intraoral corrective grinding followed by progressively finer abrasives at 10,000 to 20,000 rpm.
- Finishing was done using diamond wheels (30 to 15 μm), (Dialite, Brasseler USA, Savannah, GA), followed by Sof-lex disks (3M Sof-lex 1982-C, 1982-M, and 1982-F, 3M ESPE, St. Paul, MN) for 30 seconds each at 20,000 rpm.
- Polishing was done using felt wheels coated with a diamond paste (Diafinish, Renfert, Hilzingen, Germany) for 30 seconds until a satin finish was produced. All the polishing was done on a single surface.

Each group was divided into two subgroups (n = 5). Subgroup A was composed of unbleached specimens, while Subgroup B specimens were bleached according to the following aggressive protocol: the specimens were exposed to 2-hour bleaching (Opalescence Bleaching Gel 35%, Ultradent Products, Inc, South Jordan, UT) followed by six applications of 8-hour bleaching (Opalescence Bleaching Gel 15%). Each two bleaching applications were interrupted by a 10-hour application of Flor-Opal, (a 1.1% neutral sodium fluoride NaFl, Ultradent Products, Inc.). After each application, the treated specimens were washed and cleaned under running water.

Microhardness, crack length determination, and fracture toughness testing was performed for all eight subgroups before and after 21 days artificial saliva storage.

Microhardness testing

Microhardness was measured using a computerized microhardness tester (Shimadzu Microhardness Tester, Shimadzu, Inc., Kyoto, Japan). Testing consisted of making a dent in the disc sample with a load of 5 N (500 grams) in a time of 20 seconds. The Vicker indenter is a square, pyramid-shaped diamond, which leaves a square-shaped indentation on the surface of the material being tested. Hardness was determined by measuring the diagonals of the square, d_1 and d_2, and calculating the average of the dimensions. The machine was calibrated against a force transducer, which is used as a reference standard force transducer with relative expanded uncertainty $\pm$ 0% to 1% and coverage factor K = 2, with confidence level 95%. The reference standard force transducer is traceable to the primary standard machine.

Trial indentation tests identified that loads less than 0.5 kg produced indents that were difficult to measure accurately due to failure to distinguish the edges of each indentation. Three readings were calculated for each specimen ensuring that the surfaces of the specimens were represented. Microhardness was measured as Vicker hardness numbers (VHNs) at baseline and after storage.

Fracture toughness testing

Hardness and fracture toughness were determined by the indentation technique.[32,34-36] Three indentations were made on each specimen at widely separated locations with a load of 19.6 N for 20 seconds in a microhardness tester (Shimadzu Microhardness tester). The basis of the indentation technique is a series of cracks that form under heavy loading in a brittle material around a Vickers diamond indenter. When viewed superiorly the cracks appear to emanate from each of the corners of the indentation. The size of these cracks, expressed by the surface dimension "c," increases with an elevating indentation load and is an inverse function of fracture toughness. The fracture toughness was calculated with the following formula:[8]

$$K_{IC} = 0.016(E/H)^{0.5}(P/c^{1.5})$$

where K_{IC} is the fracture toughness, c is the crack length (measured from the center of the indentation), P is the applied indenter load, H is the Vickers hardness, a is the half diagonal of the indentation, and E is the elastic modulus. The elastic modulus (E) for each material was determined from the work of Griggs et al.[11] Optimal testing load was determined before and after storage by comparing the crack length from the center of the indent to the length of the half diagonal. A load of 19.6 N was chosen.

Statistical analysis

Data were presented as mean and standard deviation (SD) values. A regression model with repeated measures ANOVA was used in testing significance for the effect of glazing, polishing, and their interactions on hardness, crack length, and fracture toughness. Duncan's post hoc test was used for pairwise comparison between the means when ANOVA was significant. The significance level was set at $p \leq 0.05$. Statistical analysis was performed with SPSS 14.0® (SPSS Inc., Chicago, IL) for Windows.

Results

ANOVA showed there was a statistically significant difference among the surface treatments and saliva storage on the microhardness, crack length, and fracture toughness and their interactions (Table 1).

Table 1 Effect of saliva storage and surface treatments and their interactions on microhardness, crack length, and fracture toughness

Variable	Source of variation	Sum of squares	df	Mean square	*f*-value	*p*-value
Microhardness	Saliva storage	44,331.341	1	44,331.341	30.449	<0.001*
	Glaze	61,885.278	1	61,885.278	113.491	<0.001*
	Surface treatment	78,663.407	3	26,221.136	48.087	<0.001*
	Saliva storage/glaze/surface treatment	43,744.249	3	14,581.416	10.015	<0.001*
Crack length	Saliva storage	354.381	1	354.381	47.326	<0.001*
	Glaze	312.971	1	312.971	148.539	<0.001*
	Surface treatment	95.922	3	31.974	15.175	<0.001*
	Saliva storage/glaze/surface treatment	615.803	3	205.268	27.413	<0.001*
Fracture toughness	Saliva storage	0.024	1	0.024	10.298	0.002*
	Glaze	0.165	1	0.165	219.508	<0.001*
	Surface treatment	0.012	3	0.004	5.229	0.004*
	Saliva storage/glaze/surface treatment	0.221	3	0.074	32.293	<0.001*

*Significant at $p \leq 0.05$.

There was a statistically significant increase in microhardness and fracture toughness mean values while crack length values decreased after saliva storage (Table 2).

Polishing significantly increased microhardness compared to glazing; however, bleaching significantly decreased the microhardness of both groups. Saliva storage increased all the numeric values of microhardness of the groups while changing their ranking with the exception of the polished/bleached group. The polished group maintained the highest values followed by glazing, polished and bleached, and finally the glazed and bleached group (Table 3).

As for crack lengths, the polished specimens showed the lowest significant mean value, while no statistically significant difference was evident between glazed, glazed/bleached, and polished + bleached specimens. Saliva storage showed the same statistical ranking between the groups (Table 3).

Polished and glazed groups had statistically similar mean fracture toughness values (Table 3). In addition, no statistically significant difference was found between bleached/polished and glazed/bleached specimens, which showed statistically significantly lower mean values. Saliva storage showed highest values for polished specimens while no difference was evident between the other groups.

Table 2 Comparisons among microhardness (VHN), crack length (μm), and fracture toughness (Mpa· $m^{0.5}$) of subgroups before and after saliva storage

	Before saliva storage		After saliva storage		
Test	Mean	SD	Mean	SD	*p*-value
Microhardness	558	57.3	572.1	48.7	<0.001*
Crack length	97.9	3.1	94.9	5	<0.001*
Fracture toughness	1.07	0.08	1.1	0.08	0.002*

*Significant at $p \leq 0.05$.

The effect of saliva storage on microhardness, crack length, and fracture toughness of subgroups with different surface finish is shown in Table 4. Within each of the glazed, glazed/bleached, and polished groups a significant increase was detected in the microhardness values by saliva storage while the polished/bleached group mean values remained unchanged. A significant decrease in crack length was apparent by saliva storage in all the tested groups while no significant change was evident regarding the fracture toughness within all groups (Table 4).

The autoglazed group showed significantly higher fracture toughness, lower crack length, and lower microhardness than the overglazed group (Table 5).

Discussion

Hardness is one of the most frequently measured properties of a ceramic. Its value helps to characterize resistance to deformation, densification, and fracture.[14] There was a statistically significant increase in mean hardness values after saliva storage (Table 2). This finding disagrees with that of Scherrer et al[8] who reported a decrease in the hardness of Duceram LFC after 8 weeks aging in water. The differences might be attributed to key experimental differences between studies, such as testing duration and storage medium. In this study, aging was done in artificial saliva for 3 weeks. This duration was chosen because the manufacturer's claims were that this was the period of maximum change in properties induced by moisture environment. Moreover, variation exists in the aging solution itself, which was water in their study and artificial saliva in this study. Their compositions and mineral contents are different, which could have accounted for an ionic exchange through the allegedly formed Si-OH layer causing increase in resistance to surface indentation.

The polished specimens showed the highest hardness and fracture toughness values along with the lowest crack length values before and after saliva storage (Table 3). Several authors investigated and described different polishing techniques of

Table 3 Comparisons among microhardness (VHN), crack length (μm), and fracture toughness (Mpa· $m^{0.5}$) values with different surface finish before and after storage in saliva

Test /before or after saliva storage	Glazed		Glazed/bleached		Polished/bleached		Polished		
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	*p*-value
Hardness before	555.2[c]	46.2	524.5[d]	27.6	580.2[b]	25.7	603.8[a]	30.8	<0.001*
Hardness after	611.1[b]	26.5	537.3[d]	22.7	575.1[c]	19	642.1[a]	43.2	<0.001*
Crack length before	97[a]	2.6	99.5[a]	2.8	98[a]	2	93.6[b]	3.1	<0.001*
Crack length after	95.7[a]	1.6	95.7[a]	7.6	94.5[a]	1.8	92.5[b]	3	<0.001*
Fracture toughness before	1.12[a]	0.1	1.04[b]	0.07	1.04[b]	0.04	1.13[a]	0.1	0.001*
Fracture toughness after	1.09[a]	0.06	1.09[a]	0.1	1.09[a]	0.04	1.12[b]	0.05	0.037*

*Significant at $p \leq 0.05$, means with different letters are statistically significantly different according to Duncan's test.

ceramic restorations and supported the use of polishing as an alternative to glazing with higher strength values;[52–58] however, surface cracks are induced by machining and grinding with flaw sizes varying from 20 to 50 μm. Ceramic failure originates from the most severe flaws;[59] however, many ceramists prefer polishing to glazing as it controls the amount and distribution of luster and gloss.[60]

Dental ceramics can be smoothed by two methods: polishing or glazing. The literature is full of advocates for each procedure. Grinding and polishing ceramic restorations involve mechanical removal of ceramic from the surface. These finishing procedures are thought by some to provide smoother surfaces and induce residual compressive stresses. It has been suggested that an area of compressive stress is created below the ground area, thereby preventing crack extension and improving strength;[56] however, others disagree strongly and recommend glazing,[60] while Wiley[57] regarded them as comparable.

Polishing of the ceramic entails the use of diamond paste and stones, which probably caused a reduction in initial surface flaws and defects, inhibiting further crack propagation, thereby increasing resistance to surface indentation. Polishing might have also produced residual compressive stresses, thereby inhibiting crack growth, causing areas of heat generation on the surface increasing its surface hardness as suggested by Palin et al and Alkhiary et al.[61,62] Palin et al[61] investigated claims that a low-fusing hydrothermal ceramic gained strength by surface polishing. They also suggested that polishing may produce residual compressive stresses, thereby inhibiting crack growth and increasing strength. Alkhiary et al[62] confirmed these findings in their investigations. Albakry et al[63] simulated treatments during laboratory and clinical adjustments and tested their effect on two pressable ceramics. They concluded that polishing showed the highest flexural strength, while heat treatment had no effect on the strength of the tested specimens.

Fracture toughness and microhardness of both the glazed and polished groups was decreased by bleaching (Table 4). However, after saliva storage, no difference was apparent between the tested groups except for the polished group, which recorded the highest fracture toughness values (Table 3). This would suggest that bleaching decreased hardness and fracture toughness of both glazed and polished groups, probably due to dissolution of some of its components by the etching effects of bleaching and fluorides as suggested by Türken and Biskin;[50,51] however, saliva storage decreased crack length (Table 5), thus increasing the fracture toughness values.

The increase in the microhardness of Duceram LFC after storage in saliva (Table 4) may be attributed to the formation of a region of strongly hydrogen-bonded hydroxyl groups on the outer surface of the material. "A surface layer" as claimed by the manufacturer is formed through the exchange of alkali cations

Table 4 Effect of saliva storage on hardness (VHN), crack length (μm), and fracture toughness (Mpa· $m^{0.5}$) of subgroups with different surface finish

Saliva storage	Glazed		Glazed/bleached		Polished/bleached		Polished	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Hardness before	555.2	46.2	524.5	27.6	580.2	25.7	603.8	30.8
Hardness after	611.1	26.5	537.3	22.7	575.1	19	642.1	43.2
p-value	0.013*		0.015*		0.626		0.010*	
Crack length before	97	2.6	99.5	2.8	98	2	93.6	*3.1*
Crack length after	95.7	1.6	95.7	7.6	94.5	1.8	92.5	*3*
p-value	0.016*		0.003*		<0.001*		*0.024*	
Fract Toug before	1.12	0.1	1.04	0.07	1.04	0.04	1.13	0.1
Fract Toug after	1.09	0.06	1.09	0.1	1.09	0.04	1.12	0.05
p-value	0.561		0.051		0.117		0.420	

*Significant at $p \leq 0.05$.

Table 5 Comparisons of hardness (VHN), crack length (μm), and fracture toughness (Mpa· $m^{0.5}$) between autoglazed and overglazed ceramic specimens before and after saliva storage

	Autoglaze		Overglaze		
Saliva storage	Mean	SD	Mean	SD	*p*-value
Hardness before	526.5	34.8	579.9	60	<0.001*
Hardness after	552.4	34	585.7	52.1	<0.001*
Crack length before	95.6	2.5	100	1.9	<0.001*
Crack length after	91.7	4.3	97.9	3.7	<0.001*
Fracture toughness before	1.15	0.06	1.02	0.03	<0.001*
Fracture toughness after	1.17	0.07	1.05	0.05	<0.001*

and hydroxyl groups. This region is thought to not be as rigid as a fully polymerized glass network. It was believed to deform plastically instead of by brittle fracture, thus this hydrolyzed layer is more ductile than bulk porcelain.[5-12] The SiOH region is expected to be approximately 3-μm thick and is thought to heal surface flaws and protect the surface against damage. Because of its unique manufacturing process, the company expects this SiOH region to be regenerated in the mouth after it is damaged or removed by mastication.

There was a statistical decrease in the mean crack length after saliva storage in all the tested specimens (Table 4). This phenomenon may be explained on the basis of the assumption by Griggs et al,[11] who declared that Duceram LFC formed a modified surface layer. They described the surface as a "remodeled surface," because the severity of the surface flaws seemed to be decreased through a selective dissolution mechanism of the material adjacent to the tips of surface cracks. The driving force for crack tip blunting might have lead to reduction in the concavity of crack tips to eliminate local solubility differences that are caused by capillarity effects.

Ceramic materials are susceptible to stress corrosion, dynamic fatigue, and surface degradation, which all affect their flexure strength. It was unclear whether their toughness was also affected by exposure to an accelerated aging environment. The results showed that storage, glaze, surface treatment, and the interaction between the variables had a statistically significant effect on mean fracture toughness (Table 1).

Bleaching of glazed and polished ceramic groups decreased the specimens' fracture toughness, which was changed after storage in saliva, except for the polished group (Table 3). There was a statistically significant increase in mean fracture toughness after saliva storage (Table 2). This finding agrees partially with that of Scherrer et al[8] who reported a significant increase in toughness of Duceram LFC. They suggested that in their 8-week water storage specimens, there was slower crack growth due to the presence of water molecules within the LFC surface; consequently the residual stresses were partially relieved, which in turn increased the fracture toughness. However, they reported a decrease in hardness from 6.4 to 4.6 VHN, which is contrary to the results of our study (Table 3).

The term overglazing describes the firing of a low-fusing colorless glass on the veneering porcelain. This thin layer of about 4 μm of glass produced after 60 seconds of hold time at the final temperature, reduces the size of the flaws present on the surface, thus increasing the strength of the material. The coefficient of thermal expansion of the overglaze is lower than the ceramic, which means that on cooling the underlying material shrinks more than the overglaze, placing the latter under compressive forces. This is a strengthening method to inhibit the propagation of cracks in veneering ceramics. Thus, a large number of defects present on the veneering materials introduced during sintering are repaired by the overglaze material.[58,60,64]

In this study, the mean fracture toughness of autoglazed specimens showed statistically significantly higher mean values than overglazed specimens before and after storage (Table 5). Crack length was also significantly smaller for the autoglazed group. This finding disagrees with the findings of Isgro et al[65] and Fahmy et al.[66] Fahmy et al[66] reported that glazing appeared to increase the strength of the low-fusing ceramic used, while finishing and or diamond-polishing alone showed lower strength values (71.4 and 63.13 MPa). Autoglazing showed numerically higher mean values than overglazing in the latter study, but the difference was insignificant in the case of Finesse ceramic. The difference in the recorded results is probably due to the difference in the composition of the ceramics used, because even though both ceramics are LFC, Finesse ceramic is not a hydrothermal ceramic and additionally, the polishing in this study was done after glazing and not as a form of surface finish as in the previous study. In addition, Duceram LFC is a glass with a leucite-free composition.[7]

Moreover, the limitations of flexural strength as an indicator of the structural performance of brittle materials have been pointed out by Kelly[25] and Yilmaz et al.[14] By contrast, an appropriate parameter would be the ceramics' fracture toughness (KIc); that is, the material's intrinsic resistance to crack propagation.[14,25,26]

Conclusions

1. Surface treatment and saliva storage had a significant effect on the microhardness, crack lengths, and fracture toughness of the tested ceramic.
2. Saliva storage increased microhardness and fracture toughness mean values of the tested groups, while crack length values decreased.
3. Polishing significantly increased microhardness compared to glazing; however, bleaching significantly decreased the microhardness of both groups.
4. The polished specimens showed the lowest crack length and highest hardness and fracture toughness values before and after saliva storage.
5. Polished and glazed groups had statistically similar mean fracture toughness values, which were both decreased significantly by bleaching.
6. The autoglazed group showed significantly higher fracture toughness and lower crack length and microhardness than the overglazed group.

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