

IN VITRO ASSESSMENT OF MECHANICAL AND OPTICAL PERFORMANCE OF ADVANCED LITHIUM DISILICATE CERAMICS BEFORE AND AFTER IMMERSION IN DIFFERENT PH MEDIA

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ABSTRACT

Purpose: This study was conducted to assess the impact of different immersion solutions on hardness, fracture toughness and color stability of advanced lithium disilicate (ALD) ceramics.

Methods: Forty-four plates of ALD ceramic (Cerec Tessera) measuring 14x12x1 mm were randomly divided into 4 groups (n=11) according to the immersion solution; Artificial saliva (AS), Soft Drink (SD), Energy Drink (ED) and Mouthwash (MW). Each plate was immersed in a tightly sealed container filled with 5 ml of solution inside an incubator at 37°C for 12 days simulating one year of intraoral use. The solutions were renewed every 12 hours to obtain freshly solutions. The plates were rinsed using distilled water for five minutes and then gently dried using tissue paper. Surface microhardness was measured via a Vickers hardness test followed by fracture toughness calculation using the indentation technique. While color stability was measured using a spectrophotometer. All the outcomes were obtained before immersion as baseline readings and after immersion. Data were statistically analyzed using One Way ANOVA test followed by Tukey's Post Hoc test for multiple comparisons. While comparison between before and after was performed by using Paired t test with the significance level set at $p \leq 0.05$.

Results: Regarding hardness, only group MW exhibited a statistically significant reduction in hardness ($P < 0.0001$). Regarding fracture toughness, there was a highly statistically significant decrease in fracture toughness values in SD, ED and MW groups ($P < 0.0001$). Regarding color stability, group ED recorded the highest ΔE followed by group SD, group MW while the lowest color change was recorded in group AS.

Conclusions: Acidic media have negative effect on the fracture toughness of ALD ceramics. Meanwhile mouthwash has a deteriorating effect on their hardness. ALD ceramics are susceptible to color change after being subjected to acidic media.

KEY WORDS: Advanced lithium disilicate, Hardness, Fracture toughness, Color stability

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INTRODUCTION

Recent advances in digital technology have enabled the validation of restorative and prosthetic restorations fabricated using computer-aided design and computer-aided manufacturing (CAD/CAM) systems. This development resulted in a growing preference for monolithic materials which offer high mechanical properties, less interfaces, more conservative preparation, more simplified procedures in addition to accepted esthetic outcomes in comparison with bilayered ceramic restorations ^[1,2].

Recently, Dentsply Sirona introduced a new type of monolithic CAD/CAM ceramic block called advanced lithium disilicate (ALD) (Cerec Tessera). It is composed of a dual-crystalline structure of Lithium disilicate ($\text{Li}_2\text{Si}_2\text{O}_5$) and Virgilite (lithium aluminum silicate, $\text{Li}_{0.5}\text{Al}_{0.5}\text{Si}_{2.5}\text{O}_6$) incorporated within a zirconia-reinforced glassy matrix. The key feature of this type is the significant rapid grinding and firing making it a suitable choice as a chair-side restoration ^[3]. Currently, it may be considered one of the strongest glass ceramic blocks with flexural strength reaching 700 MPa in addition to delivering the tooth-like esthetics of glass ceramics. For aforementioned reasons, ALD ceramics have a wide variety of applications in fixed prostheses ^[4].

One of main factors that may affect the success of dental ceramics is the pH media of the oral cavity. The normal pH of resting saliva is 6.8-7.2 which can be frequently altered to be more acidic or alkaline not only by food or drink consumption but also with daily habits as mouthwash rinsing. This change in pH levels in the oral cavity may have a negative impact on dental ceramics leading to stress corrosion, surface degradation, potential phase transformation and subsequently alteration in mechanical and optical properties ^[5,6]. Fracture toughness (K_{IC}) is a key macroscopic property that reflects the brittleness and fragility of ceramics, indicating how readily a crack or flaw can spread through the material. While hardness is the ability of a material to endure a permanent indentation

or perforation. Therefore they may be considered useful gauges of the mechanical nature of dental materials ^[7].

Therefore, the objective of this study was to assess the impact different immersion media on hardness, fracture toughness and color stability of ALD ceramics. According to the null hypotheses, no difference was expected in mechanical or optical properties of ALD ceramics after immersion in intraoral simulating solutions.

MATERIALS AND METHODS

The materials used in the study and their composition are described in Table 1.

Specimens Preparation

A total number of forty-four plates of advanced lithium disilicate ceramic (Cerec Tessera, Dentsply Sirona, USA) were cut using a slow speed diamond saw under water coolant (Isomet-4000, Buehler, Dusseldorf, USA) with 14x12x1 mm dimension. All the plates were carefully examined where the damaged or chipped plates were discarded. The plates were cleaned ultrasonically and air dried. They were glazed according to the manufacturer's instructions utilizing Ivoclar Programat CS furnace (Preheating 400°C, Closing time 2 min, Temperature gradient 55°C, Holding temperature 760°C, Holding time 2 min and 0 Vacuum) ^[4]. The plates' dimension were double checked using a digital caliber (digital Vernier caliper, Hogetex) to ensure a uniform 1 mm thickness.

Aging Procedures

The plates were randomly and equally divided into 4 main groups (n=11) according to the aging solution. Group AS (Artificial Saliva), Group SD (Soft Drink) (Sprite, Coca Cola Co., Egypt), Group ED (Energy Drink) (Red Bull, GmbH, Salzburg, Austria) and Group MW (Mouthwash) (Listerine Cool Mint, Lambertville, New Jersey, USA). Each plate was immersed in a tightly sealed container

Table (1). Materials used in this study and their composition

Commercial name	Description	Chemical Composition	Manufacturer
Cerec Tessera CAD/CAM blocks	Advanced Lithium Disilicate (ALD) (A2 MT C14) LOT: 16012883	$\text{Li}_2\text{Si}_2\text{O}_5$: (90%), Li_3PO_4 : (5%), $\text{Li}_{0.5}\text{Al}_{0.5}\text{Si}_{2.5}\text{O}_6$ (virgilite): (5%), embedded within zirconia-reinforced glassy matrix	Dentsply Sirona, York, PA, USA
Artificial saliva	Artificial saliva pH = 6.8	Sodium chloride (0.4 g/L), Potassium chloride (0.4 g/L), Calcium chloride dehydrate (0.795 g/L), Potassium dihydrogen (0.69 g/L), mucin (2.0 g/L) and traces of Magnesium chloride and sodium hydroxide.	Faculty of Pharmacy, Cairo University, Egypt
Sprite	Carbonated soft drink pH = 2.81	Carbonated water, sugar, Citric acid, Sodium citrate, Lemon flavor and Sodium benzoate.	Coca Cola Co., Egypt
Red Bull	Energy drink pH = 3.18	Water, Glucose, citric acid, carbon dioxide, taurine, caffeine, sodium bicarbonate, plain caramel, riboflavin, Vitamins B3, B5, B6 and B12.	Gmbh, Salzburg, Austria
Listerine (Cool Mint) Mouth wash	Antiseptic mouthwash pH = 4.3	Water, Alcohol, Sorbitol, Poloxamer, Benzoic acid, Sodium benzoate and essential oils such as Eucalyptol, Menthol, Methyl Salicylate and Thymol.	Lambertville, New Jersey, USA

filled with 5 ml of solution inside an incubator at 37°C to maintain and simulate the ideal temperature of the oral cavity throughout the research. The plates were incubated for continuous 12 days simulating one year of intraoral use [8-10]. The solutions were renewed every 12 hours to obtain freshly solutions [11]. The pH measurement was performed three times using a pH meter (Inolab pH meter Level 1, WTW Company, Germany) to ensure accuracy where the pH of the aging solutions recorded: Artificial saliva = 6.8, Sprite = 2.81, Red Bull = 3.18 and Listerine = 4.3. After aging, all the plates were rinsed using distilled water for five minutes and then gently dried using tissue paper.

Measuring the Outcomes

The measurements of color, hardness and fracture toughness were performed before aging in the solutions to record baseline readings. While after aging they were measured again to investigate the effect of these solutions on the optical and mechanical properties of ALD ceramics.

Regarding surface microhardness, it was performed using a digital Vickers (Model HVS-50,

Laizhou Huayin Testing Instrument Co., Ltd., China) equipped with a 20X objective lens and a Vickers diamond indenter surface microhardness was evaluated. The specimen surface was subjected to a static load of 300 grams for a standardized duration of time 20 seconds. To minimize measurement variability, three indentations were created on each specimen surface, strategically positioned in an equidistant circular pattern with a minimum inter-indentation distance of 0.5 mm to prevent interference. Using a built-in scale microscope, the lengths of the indentation diagonals were measured, and the Vickers Hardness Number was subsequently calculated using the established formula: $\text{HV} = 1.854 \text{ P}/\text{d}^2$; Where HV = Vickers hardness in kgf/mm², P = the applied load in kgf and d = the average length of the diagonal indentations in mm.

Regarding fracture toughness, Fracture toughness was evaluated using the indentation technique, which involved examining crack patterns generated around a Vickers diamond indenter under applied load. The fracture toughness was calculated using the formula: $K_{\text{IC}} = 0.016(E/H)^{0.5} (P/C^{1.5})$; where K_{IC} = the fracture toughness, C = the crack length measured from the center of the indentation, P = is the

applied indenter load, H = Vickers hardness and E = the elastic modulus of Cerec Tessera. Crack length was measured with a measuring microscope (Nikon Eclips E600, Tokyo, Japan) at 40 \times and 110 \times magnification. The crack should be measured immediately to avoid recovery of the cracks after unloading [12].

Regarding color change, the staining susceptibility is defined as a shading variation measured by comparing the results to the initial input. At both the start and the end of the 12 days, the colors of the plates were estimated using a spectrophotometer (X-Rite, model RM200QC, Neu-Isenburg, Germany). An aperture size of 4 mm was selected, and the samples were precisely positioned in alignment with the device. A white background was selected, and measurements were made according to the CIE $L^*a^*b^*$ color space relative to the CIE standard illuminant D65. The measurements were repeated 3 times for each sample, and the mean values of L^* , a^* , b^* were calculated. The color change (ΔE) of the plates were calculated using the following equation $\Delta E = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{1/2}$; Where L = lightness (0-100), a = change in color on the red/green axis and b = color on the yellow/blue axis [13]. A lower ΔE value indicates reflects minimal color change, whereas a higher ΔE value indicates a more noticeable color change. ΔE values greater than 1.2 were considered visually detectable, while those exceeding 3.3 were considered as clinically unacceptable [14-15].

Statistical Analysis

Statistical analysis was performed with SPSS 27® (Statistical Package for Scientific Studies), Graph pad prism & windows excel. Exploration of the quantitative data was performed using Shapiro-Wilk test and Kolmogorov-Smirnov test for normality which revealed that all data originated from normal distribution except difference between before and after v/ percentage of change. Accordingly, Comparison between groups in normal data was performed by using One Way ANOVA test followed by Tukey's Post Hoc test for multiple comparisons,

while comparison between before and after was performed by using Paired t test. In non-parametric data, comparison between groups was performed by using Kruskal Wallis test. The significant level was set to be at $P \leq 0.05$.

RESULTS

Hardness

Vickers hardness before and after immersion in different groups and percentage of change within each group were presented in Table 2 and Figure 1. After immersion in different intra-oral simulating solutions, no significant difference in hardness was observed among the groups either ($P = 0.48$), with Group AS measuring 458.31 ± 5.44 , Group SD 454.81 ± 14.82 , Group ED 451.72 ± 12.46 , and Group MW 452.50 ± 7.72 . Meanwhile, within the same group, when comparing the mean difference in hardness before and after immersion within each group, only Group MW exhibited a statistically significant reduction in hardness ($P < 0.0001$). The mean difference in Group MW was 9.76 ± 6.76 , which was significantly higher than those recorded in the other groups. Group AS showed a minimal change of 0.02 ± 0.03 , while Group SD and Group ED demonstrated changes of 3.18 ± 11.31 and 3.37 ± 12.96 , respectively.

Fracture Toughness

The data for fracture toughness before and after immersion in the different groups, as well as the percentage of change within each group, are presented in Table 3 and Figure 2. After immersion, there was significant difference among the groups ($P < 0.0001$). The recorded means were 5.83 ± 0.09 for Group AS, 4.90 ± 0.06 for Group SD, 4.96 ± 0.07 for Group ED, and 4.60 ± 0.04 for Group MW. Meanwhile, within the same group, there was no statistically significant difference was observed in Group AS, where the mean toughness decreased slightly from 5.88 ± 0.03 before immersion to 5.83 ± 0.09 after immersion ($P = 0.14$). In contrast,

there was a highly statistically significant decrease in fracture toughness values in the other groups. Group SD exhibited the largest reduction with a mean difference of -0.86 ± 0.07 , followed by Group ED with -0.55 ± 0.07 , and Group MW with -0.43 ± 0.04 .

Color Stability

The color changes (ΔE) measured in the different groups are summarized in Table 4 and Figure 3. The intergroup comparison was performed using the Kruskal-Wallis test. The highest mean color change was recorded in Group ED, with a mean ΔE

value of 6.50 ± 4.30 , ranging from 2.91 to 16.32, followed by Group SD with a mean of 5.66 ± 3.20 and a range between 1.10 and 11.64. Group MW demonstrated a comparable mean color change of 5.60 ± 3.16 , ranging from 2.11 to 10.36. The lowest mean color change was observed in Group AS, with a mean ΔE value of 3.23 ± 1.04 , ranging from 1.38 to 4.82. Despite the mean values of color change between groups showed no statistically significant difference, but group AS is the only group considered a clinically accepted color change as the value of ΔE didn't exceed 3.3.

TABLE (2). Vickers hardness before and after immersion in different groups, percentage of change within each group:

Hardness	Group								P value
	Group AS		Group SD		Group ED		Group MW		
	Mean	Standard Deviation	Mean	Standard Deviation	Mean	Standard Deviation	Mean	Standard Deviation	
Before	458.33 ^a	5.42	457.99 ^a	10.89	455.08 ^a	7.98	462.26 ^a	8.30	0.26
After	458.31 ^a	5.44	454.81 ^a	14.82	451.72 ^a	12.46	452.50 ^a	7.72	0.48
Difference	0.02 ^a	0.03	3.18 ^a	11.31	3.37 ^a	12.96	9.76 ^b	6.76	0.01*
P value	0.07		0.37		0.41		<0.0001*		
% of change	0.0027 ^a	0.01	0.75 ^a	2.59	0.72 ^a	2.88	2.10 ^b	1.47	0.01*

*Significant difference as $P \leq 0.05$. Means with different superscript letters per row were significantly different as $P < 0.05$

TABLE (3). Fracture toughness before and after immersion in different groups, percentage of change within each group:

	Group								P value
	Group AS		Group SD		Group ED		Group MW		
	Mean	Standard Deviation	Mean	Standard Deviation	Mean	Standard Deviation	Mean	Standard Deviation	
Before	5.88 ^a	0.03	5.76 ^b	0.10	5.51 ^c	0.05	5.02 ^d	0.04	<0.0001*
After	5.83 ^a	0.09	4.90 ^b	0.06	4.96 ^b	0.07	4.60 ^c	0.04	<0.0001 *
Difference	-0.05 ^a	0.10	-0.86 ^b	0.07	-0.55 ^c	0.07	-0.43 ^c	0.04	<0.0001*
P value	0.14		<0.0001*		<0.0001*		<0.0001*		
% of change	-0.81 ^a	1.66	-15.00 ^b	1.09	-9.94 ^c	1.30	-8.52 ^c	0.68	<0.0001*

*Significant difference as $P \leq 0.05$. Means with different superscript letters per row were significantly different as $P < 0.05$.

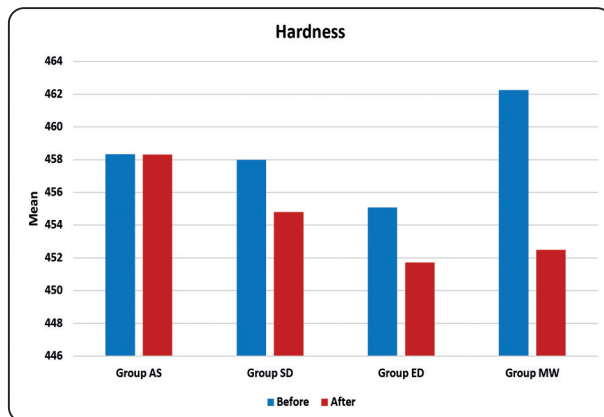


Fig. (1) Bar chart showing Vickers' hardness before and after immersion in different groups.

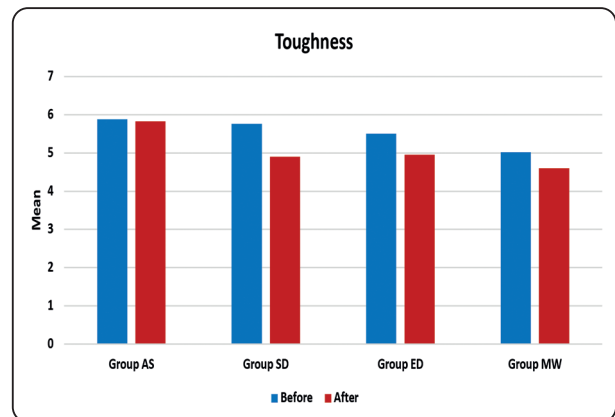


Fig. (2) Bar chart showing Fracture toughness before and after immersion in different groups.

TABLE (4). Color changes after immersion in different groups:

ΔE	Minimum	Maximum	Median	Mean	Standard Deviation	P value
Group AS	1.38	4.82	2.81	3.23	1.04	0.07
Group SD	1.10	11.64	6.10	5.66	3.20	
Group ED	2.91	16.32	4.86	6.50	4.30	
Group MW	2.11	10.36	3.65	5.60	3.16	

*Significant difference as $P \leq 0.05$. Means with different superscript letters per row were significantly different as $P < 0.05$.

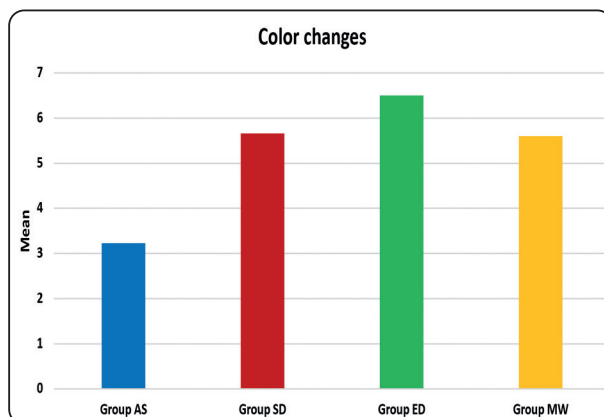


Fig. (3). Bar chart showing color changes after immersion in different groups.

DISCUSSION

ALD ceramic is considered one of the recent ceramic materials in the dental field. It is composed of a combination of two types of needle-like $0.5 \mu\text{m}$ long crystals, which are the lithium disilicate and virgillite embedded in a zirconia enriched glass matrix. The CEREC Tessera's compact and interwoven crystal configuration creates a robustly reinforced, high density restorative material where the lithium disilicate provide compression strength, while the newly formed virgillite increases the pre-compression strength. Moreover, this crystals combination enhances its esthetic qualities and dynamic light refraction, such as transmission, absorption, opalescence and fluorescence closely resembling the natural appearance of teeth^[4].

Dental ceramics commonly contain flaws and defects due to laboratory and clinical procedures such as: milling, adjustment of occlusal, proximal contacts and axial contours of dental restorations. Even at microscopic level, the damage induced by these procedures creates surface flaws, which can promote crack initiation and dramatically reduces strength and fatigue life of all-ceramic restorations. Therefore, fracture toughness, which describes the resistance of brittle materials to crack propagation, is more predictive and relevant to clinical performance than flexural strength, which has been widely used as a key parameter for the reliability assessment of dental ceramics [16]. Meanwhile hardness is a crucial mechanical property of dental ceramics, contributing to their resistance to surface wear and abrasion, thereby enhancing their longevity and esthetic appeal [17].

The thickness of the plates used was 1mm to be clinically relevant according to the manufacturer recommendation and to allow indentation test without fracture of the samples in line with the 10% Bückle's rule. It includes that the plate thickness must be at least 10 times greater than the indentation depth to ensure precise and valid testing procedures [18].

Several studies [19,20] have demonstrated that fluctuations in pH levels over time can affect both the mechanical and optical performance of ceramic restorations during their function intraorally. The normal pH value of saliva is 6.8–7.2. Nevertheless, multiple factors contribute to fluctuation in oral pH levels [19]. Consumption of acidic media may lower the pH value to as low as 3. Carbonated beverages and energy drinks are considered among the most consumed beverages in the world especially between young people [21–22]. On the other hand, mouthwashes have been advised as a supplementary treatment for antimicrobial control. In addition they are routinely used as a preventive measure after teeth brushing. Therefore, artificial saliva was selected as the

comparator group. Meanwhile soft, energy drinks and mouthwash were used to determine their effect on hardness, fracture toughness and color stability of ALD.

Based on the findings of this study, the null hypotheses of this study were rejected, as immersion of ALD ceramic in acidic media resulted in a negative effect on fracture toughness and color stability. Meanwhile regarding hardness, acidic media had negative effect only in MW group.

Regarding hardness, there was a non-significant difference among the groups ($P = 0.48$). But within the same group, only the MW group exhibited a statistically significant reduction in hardness before and after immersion ($P < 0.0001$). This may be due to the negative effect of alcohol (ethanol = 25%) in the composition of the mouthwash in addition to the acidic nature of the mouthwash used. They promote hydrolysis of the ceramic glass matrix and ion exchange, where hydrogen ions (H^+) replace alkali ions (Al^+ , Si^+ , Li^+ and Na^+) in the glass matrix. This process weakens the silica phase of lithium disilicate leading to surface degradation and reduced hardness [23]. These findings were consistent with a previous systematic review in 2022 which found that Listerine mouthwash causes increase in the roughness of ceramic due to surface erosion and dissolution, which in turn have a negative impact on wear, hardness and surface integrity [24].

Regarding fracture toughness, AS group recorded the highest mean fracture toughness among the other groups and the lowest reduction of fracture toughness value ($-0.81 \pm 1.66\%$). This may be attributed to its neutral pH and ionic balance which minimized any chemical reactions with the ceramic surface. This in turn, helps to maintain the integrity of ceramic composition and the material's microstructure. In addition, the shortest crack length was recorded in AS group which indicates more resistance to crack propagation and explains the higher mean value of its fracture toughness. Meanwhile the

three other groups (SD, ED and MW) showed a significant statistical lower mean values of fracture toughness ($P < 0.05$). SD group exhibited the largest reduction of fracture toughness ($-15.00 \pm 1.09\%$) followed by ED group ($-9.94 \pm 1.30\%$) and MW group ($-8.52 \pm 0.68\%$). This may be explained by presence of citric acid in the composition of Sprite and Redbull used in our study. This acid is responsible for lowering the pH into critical levels reaching 2.5 to 3.2. Frequent or prolonged exposure to this acid may cause deteriorating effects on the surface of ceramics due breakage of silica-oxygen bond (Si-O-Si) at the crack tip and leaching out of ions leading to increase crack length and decreasing fracture toughness [25]. Another explanation is the increased surface roughness after acid exposure which may initiate microcracks or flaws at the surface [26]. These finding was consistent with many studies [27,28] who concluded that acidic media significantly reduce fracture toughness of dental ceramics through the same mechanism by breaking down the silicate network in the glassy matrix which results in reducing the material's ability to resist crack propagation.

Regarding Color stability, despite of the non-statistical significant difference between the tested groups, but group AS recorded the lowest ΔE and it was within the clinically accepted threshold as the value of ΔE didn't exceed 3.3. This may be attributed to the neutral pH of the saliva which doesn't significantly affect the optical properties of ALD ceramic. Meanwhile the other groups demonstrated higher ΔE values exceeding the clinically accepted limit. This may be attributed to the surface disintegration leading to diffusion of pigments and, consequently, discoloration of the material [29]. Multiple studies have highlighted the impact of acidic solutions on changing the optical properties and surface integrity of various CAD/CAM dental restorative materials [30-32].

At the end, this in-vitro study tried to simulate the acidic exposure of ALD ceramics that may occur intraorally. However, this simulation does not entirely reflect real intraoral circumstances such as neutralizing effect of saliva, the duration and frequency of acidic exposure, patients' oral hygiene habits and fluctuating temperature. These factors influence the impact of acidic media on dental restorations. Therefore, the findings of our study are only applicable to the same conditions.

RECOMMENDATIONS

As this was an in-vitro investigation, further long-term clinical studies are necessary to thoroughly assess the optical performance of CAD-CAM materials and their durability when exposed to various acidic environments.

Patients with ALD ceramics esthetic restorations are advised to limit their intake of acidic beverages such as Sprite and Redbull in addition to Listerine mouthwash to improve the restorations' durability and optical properties.

CONCLUSIONS

Within the limitations of our study and according to its findings:

1. Acidic media have negative effect on the fracture toughness of ALD ceramics. Meanwhile mouthwash has a deteriorating effect on their hardness.
2. ALD ceramics are susceptible to color change after being subjected to acidic media.

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