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EFFECT OF MOUTH WASHES ON HARDNESS AND ROUGHNESS **OF LITHIUM DISILICATE AND ZIRCONIA REINFORCED LITHIUM** SILICATE CERAMICS: AN IN VITRO STUDY

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ABSTRACT

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AIM: The purposes of this study were to evaluate hardness, surface roughness and analysis the surface elements of lithium disilicate (LDS) and zirconia reinforced lithium silicate (ZLS) ceramics submitted to different mouth washes.

Materials and Methods: A total of sixty disc shaped samples (5mm diameter and 2mm thick) were fabricated of the two types of ceramic; 30 discs for group A, LDS (IPS e.max ceram) and 30 discs for group B, ZLS (VITA SUPRINITY). Each group were subdivided into 5 subgroups I-V (n=6) according to the type of immersion solution: distilled water, chlorhexidine based, povidone iodine based, green tea based and whitening mouth wash, for subgroups I-V respectively. Each sample was stored in 20 ml of distilled water which renewed daily for 90 days. For subgroups II-V samples were immersed with agitation in 10ml of fresh corresponding mouth wash solution for 1 minute/12 hours. All samples were evaluated quantitatively for hardness, surface roughness and Energy Dispersive Analytical X-Ray (EDAX) before and after immersion.

Results: All subgroups reveal statistically significant decrease in mean for hardness after immersion except for subgroup I. Subgroup I reveal the highest statistically significant mean comparing to other subgroups for both types of ceramics after immersion. Furthermore, all subgroups disclose statistically significant increase in mean for roughness after immersion except for subgroup I. Subgroup I reveal the lowest statistically significant mean compared to the other subgroups for group A.

Conclusion: Using mouth washes for 90 days affects surface properties of LDS and ZLS ceramics.

KEYWORDS: Energy Dispersive Analytical X-Ray (EDAX), Hardness, Roughness, lithium disilicate and zirconia reinforced lithium silicate.

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INTRODUCTION

All ceramic dental restorations are recommended from both dentists and patients for their high esthetic and longevity⁽¹⁾. Dental ceramic represents excellent biocompatibility, mechanical and physical properties in an optimal environment. Thus, it has become the material of choice for restoring dental structures. However, fluctuation of stresses, temperature and pH of the oral environment may cause changes in ceramics properties.⁽²⁾

Now a days, computer-aided design/ computeraided manufacturing (CAD/CAM) lithium disilicate (LDS) based ceramics blocks are commonly used in esthetic bearing areas⁽³⁾. In addition, lithium silicate ceramic was reinforced by zirconia particles to form zirconia reinforced lithium silicate ceramic (ZLS). Zirconia particles can improve the mechanical properties of the ceramic hindering the crack propagation^(4, 5).

Mouth washes are not only used on account of professional recommendations, but they are used as adjunctive tool in oral hygiene routine measurements in conjunction to brushing for many patients. Patients used mouth washes for many purposes such as cooling effect, reducing halitosis, anti-microbial control, desensitization, or whitening ⁽⁶⁾.

According to American dental association (ADA) mouth washes can be classified into two major types: cosmetic and therapeutic. Cosmetic mouth washes can control halitosis and leave a pleasant taste after usage, but it should not have other chemical or biological applications. On the other hand, therapeutic mouth washes should have active ingredients that can control or reduce conditions like bacteria which cause halitosis, gingivitis and plaque formation. Moreover, therapeutic mouth washes may have other active ingredients such as antimicrobial agents, herbals, essential oils, whitening agents and desensitizing agents. Cosmetic and therapeutic mouth washes are available commercially over the counter (OTC) or by dentist prescription.⁽⁷⁾

In addition to the ability of colored foods and beverages to stain both teeth, composite and ceramics restorative materials, they can also change their surface properties expressed by hardness and roughness⁽⁸⁾. Mouth washes as well can have the same effect. Several studies investigated the effect of mouth washes on surface properties of teeth and composite restorative materials⁽⁹⁻¹¹⁾. However, a few studies studied the effect of mouth washes on surface properties of ceramic restorative materials. For this reason, this study was conducted to evaluate that effect. The null hypothesis of this study stated that there were no significant changes in hardness and roughness and surface composition would be observed after immersion in chlorhexidine (CHX) based, iodine based, green tea based and pentasodium triphosphate based mouth washes.

MATERIALS AND METHODS

Sample size calculation:

Using G*Power software (G*Power 3.1.9.7, Heinrich-Heine, Dusseldorf Germany) sample size was calculated. The effect size was (f=0.5928) by considering Soygun K et al 2017(12) study. As a result, a minimum of 60 samples (n=6 for each subgroup) was found to be sufficient, with a power of 80% and 0.05 significance level.

Sampling and grouping:

Lithium disilicate ceramic (LDS) (IPS e.max ceram, Ivoclar- Vivadent AG, Germany) and zirconia reinforced lithium silicate (ZLS) (VITA SUPRINITY pc, VITA Zahn fabric, Bad Säkingen, Germany) were selected for this study and considered as groups A and B respectively. The chemical composition and supply of both types was illustrated in table 1. Sixty disc shaped samples (5mm diameter, 2mm thickness) were fabricated 30 discs for each group. Ceramic blocks were shaped in a cylindrical shape of diameter 5mm using a lathe cutting machine (CNC, centroid, USA) then sectioned in to 2 mm thick discs using precision saw (ISOMET, Buehler, USA). After that, the discs of both groups were fired for crystallization, finished and polished according to manufacturer instructions. By the end of this step, all discs were ultrasonically cleaned in distilled water bath for 10 minutes to remove debris.

Samples of each group were then divided randomly into 5 subgroups (n=6) according to the type of immersion solution mentioned as follows:

- Subgroup I: Distilled water (control)
- Subgroup II: Chlorhexidine (CHX) based mouth wash (Orovex mouth wash, Macro capital, Egypt).
- Subgroup III: Povidone Iodine (PVI) based mouth wash (BETADINE; El- Nile Co. Cairo)
- TABLE (1) Composition and supply of used ceramics.

- Subgroup IV: Green Tea based mouth wash (Listerine, green tea, Listerine[®], Johnson & Johnson, Italy)
- Subgroup V: Whitening mouth wash (Listerine, Advanced white, Listerine[®], Johnson & Johnson, Italy)

The pH of all mouth washes used in the present study were measured using pH meter (AD 1030, Adwa instrument, Hungary, Romania) by inserting the pH meter electrode in 50 ml of the mouth wash at room temperature. The chemical composition and pH of the mouth washes used are showed in table(2).

Materials	Composition	Supply
Lithium disilicate (IPS e. max CAD)	$\begin{aligned} \mathbf{SiO}_{2} 57.0 - 80.0, \mathbf{Li}_{2}\mathbf{O} 11.0 - 19.0, \mathbf{K}_{2}\mathbf{O} 0.0 - 13.0, \mathbf{P}_{2}\mathbf{O}_{5} 0.0 \\ -11.0, \mathbf{ZrO}_{2} 0.0 - 8.0, \mathbf{ZnO} 0.0 - 8.0, \mathbf{Al}_{2}\mathbf{O}_{3} 0.0 - 5.0, \mathbf{MgO} \\ 0.0 - 5.0, \text{ and Coloring oxides } 0.0 - 8.0 \end{aligned}$	Blocks (18x14x12 mm)
Zirconia reinforced lithium silicate (VITA Suprinity pc)	SiO ₂ 56 - 64, Li ₂ O 15 - 21, K ₂ O 1 - 4, P ₂ O ₅ 3 - 8, Al ₂ O ₃ 1 - 4, ZrO ₂ 8 - 12, CeO ₂ 0-4, La ₂ O ₃ 0.1, and Pigments 0 - 6	Blocks (14.4×14.4×18mm)

TABLE (2) Composition and pH of the mouth washed used.

Mouth rinse	Composition	рН
Chlorhexidine based mouth wash (Orovex)	Thymol, Menthol, Glycerin, Sodium Fluoride, Chlorhexidine and Sodium Saccharine.	6.6
Povidone Iodine based mouth wash (BETADINE)	Povidone Iodine 1%, Glycerin, Sodium Saccharine, Ethyle alcohol, Sodium hydroxide, Methyl salicylate, Menthol and Purified water	3.5
Green tea based mouth wash (Listerine, Green tea)	Aqua, Propylene Glycol, Sorbitol, poloxamer 407, Sodium Lauryl Sulfate, Sodium Saccharin, Aroma, Eucalyptol, Benzoid Acid, Sodium Benzoate, Methyl Salicylate, thymol, Sodium Fluoride, Menthol, Sucralose, Camellia Sinensis Leaf Extracts, Caffeine, CI 47005, CI 42053, contains sodium fluoride (220 ppm F)	4.8
Whitening mouth wash (Listerine, Advanced white)	Aqua, Sorbitol, Propylene Glycol, Tetrapotassium Pyrophosphate, Pentasodium Triphosphate, Citric Acid, Poloxamer 407, Aroma, Sodium Methyl Cocoyl Taurate, Caprylyl Glycol, Eucalyptol, Thymol Sodium, Saccharin, Menthol, Sodium Fluoride, Sucralose, Contains Sodium Fluoride (220 ppm F)	6.5

Immersion protocol:

Each sample in each subgroup was stored in 20 ml of distilled water which renewed daily for 90 days. Moreover, for subgroups II-V samples were immersed in 10ml of fresh corresponding mouth wash solution for 1minutes/12hours with agitation to resample the usage of the mouth wash twice daily for 1minutes each time for three months ⁽¹³⁾.

Evaluation of surface composition elements by energy dispersive Analytical X-ray (EDAX).

The examination was carried out to obtain chemical analysis regarding the elemental composition of the surface. The surface of samples were carefully evaluated with EDAX (JEOL,JXA-840A, Electron probe microanalyzer, Jaban) at 30KV before and after immersion.

Hardness measurement

The hardness of each sample was determined before and after the end of immersion period. The measurements were done using digital display Micro-hardness tester (Model HVS-50, Laizhou Huayin Testing Instrument, China) with a Vickers pyramid square based diamond indenter, and objective lens 20X. A load of 200 Kgf was applied perpendicular to the surface of the disc for 15 seconds. Three indentations were made in each disc and the mean value of them was recorded as the sample reading. The indentations of each disc were equally placed over a circle with a distance not closer than 0.5mm to the border or to the adjacent indentation. The diagonals length of the indentations were measured and hardness values were calculated by the following equation: VHN= 1.854 P/d^2 where, VHN is Vickers hardness in Kgf/mm², P is the load in Kgf and d is the mean length of the diagonals of each sample in mm.

Roughness measurement

The surface roughness of each sample was determined before and after the end of immersion

period using an optical non-contact method. Three dimensional (3D) images for the surface of the samples (U500X Capture Digital Microscope, Guangdong, China) were captured perpendicular to the surface under fixed illumination and magnification of 90X to obtain surface topography for quantitative measurements. Three captures of each sample were taken one on the center and two 1mm away from the border of the disc at area of $10\times10 \,\mu$ m. The images were analyzed using WSxM software (Ver 5 develop 4.1, Nanotec, Electronica, SL) to calculate average roughness (Ra) which expressed in μ m.^(8, 14)

Statistical analysis:

The data was collected, tabulated and statistically analyzed using software SPSS version 26, Descriptive statistics was done for parametric quantitative data by mean and standard deviation. Hardness and roughness values were analyzed with two- way ANOVA, Post- hoc LSD comparison was done and $P \le 0.05$ was accepted as statistically significant. Moreover, comparison between after and before measurements were done using dependent sample t- test.

RESULTS

All subgroups reveal statistically significant decrease in mean for hardness after immersion except for subgroup I. When comparing subgroups to each other, all subgroups for both groups show insignificant mean values before immersion. On the other hand, after immersion subgroup I reveal the highest statistically significant mean comparing to other subgroups for both types of ceramics (Table 3, figure 1). Furthermore, all subgroups disclose statistically significant increase in mean for roughness after immersion except for subgroup I. When comparing subgroups to each other, all subgroups for both groups show insignificant mean values before immersion. On the other hand, after immersion subgroup I reveal the lowest statistically significant mean compared to the other subgroups for group A. Moreover, in group B, subgroup I showed lower statistically significant mean than subgroup IV. (Table 4, figure 2,3,4).

EDAX revealed that the main elements in composition of both types of ceramic found with the nearly same concentrations before and after immersion in subgroup I. Moreover, Precipitation of mouth washes elements like carbon, sodium and phosphorus were founded on the surface of ceramic discs were observed on other subgroups. Iodine precipitates were found on surface of both ceramic types in subgroup III with concentration of 0.03% by mass. Subgroups IV and V showed precipitation of fluoride on surfaces of the samples with concentration of 0.58% and 0.29% by mass respectively for group A and 0.0.9% and 0.17% by mass respectively for group B. (Figure 5,6)

TABLE (3) Means, standard deviation (SD) and P values of VHN

			Group A	L				Group B		
	Hardness I	Before	Hardnes	s After	P value After Vs Before	Hardness	Before	Hardness	Hardness After	
Sub groups	Mean	SD	Mean	SD	-	Mean	SD	Mean	SD	Before
Ι	550.88 ª	4.08	536.05	20.01	0.082	543.98 ª	2.21	511.84	40.59	0.091
II	556.36 ª	9.89	486.93 ^b	45.08	0.005	549.53 ª	5.93	431.16 ^b	9.64	0.000
III	554.67 ª	2.37	479.44 ^b	18.96	0.000	549.98 ª	6.85	434.86 ^b	9.17	0.000
IV	557.15 ª	4.81	470.39 ^b	23.34	0.000	547.49 ª	3.88	436.40 ь	8.10	0.000
V	547.77 ª	9.44	463.13 ^b	30.69	0.000	547.78 ª	5.12	432.03 ^b	12.75	0.000

Subgroups with different litter in the same column are significant



Fig. (1) Means of Hardness of groups and subgroups

			Group A					Group H	3	
Sub	Roughnes	s Before	Roughnes	ss After	P value After	Roughnes	s Before	Roughness After		P value After
groups .	Mean	SD	Mean	SD	Vs Before	Mean	SD	Mean	SD	Vs Before
Ι	0.2853ª	0.0032	0.2878	0.0033	0.096	0.2894ª	0.0067	0.2930 ^b	0.0049	0.107
II	0.2815 ª	0.0015	0.2909 ^b	0.0027	0.001	0.2817ª	0.0007	0.2911 ^{b,c}	0.0018	0.000
III	0.2816ª	0.0024	0.2901 ^b	0.0038	0.003	0.2815ª	0.0010	0.2908 ^{b,c}	0.0045	0.005
IV	0.2819ª	0.0006	0.2906 ^b	0.0019	0.000	0.2801ª	0.0048	0.2943 °	0.0016	0.000
V	0.2821ª	0.0040	0.2931 ^b	0.0015	0.004	0.2832ª	0.0016	0.2928 ^{b,c}	0.0012	0.000

TABLE (4) Inicalis, standard deviation (SD) and 1 values of 1	E (4) Inicalis, standard deviation (SD) and T	alues of Ro
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Subgroups with different litter in the same column are significant



Fig. (2) Means of Ra of groups and subgroups



Fig. (3) Three dimensional (3D) topography obtained for group A, where A represents an example for before emersion topography, B,C,D,E and F represent an example of subgroups I-V after immersion.



Fig. (4) Three dimensional (3D) topography obtained for group B, where A represents an example for before emersion topography, B,C,D,E and F represent an example of subgroups I-V after immersion.



Fig. (5) EDAX obtained for group A, where A represents an example for before emersion topography, B,C,D,E and F represent an example of subgroups I-V after immersion.



Fig. (6) EDAX obtained for group B, where A represents an example for before emersion topography, B,C,D,E and F represent an example of subgroups I-V after immersion.

DISCUSSION

Nowadays, patients are highly demanding esthetic restorations. All ceramic restorations had the potential to be more efficiently selected when compared to porcelain fused to metal restoration⁽¹⁵⁾. However, the success of the restorations is not only related to its esthetic appearance but also its mechanical properties and durability ⁽¹⁶⁾. Lithium disilicate ceramic, IPS e.max ceram, (LDS) consist of crystalline phase of lithium disilicate and amorphous glassy matrix. Moreover, zirconia reinforced lithium silicate, VITA SUPRINITY, (ZLS) also consists of crystalline phases of lithium silicate and zirconia and amorphous glassy matrix. The glassy matrix interacts with the surrounding environment differently than crystalline phase. Glassy matrix is less stable and has more tendency to dissolute considering the alkaline ions like (Si, Ca, K and Mg) in comparison to a crystalline phase. Dissolution of ions affect the durability and surface properties of the restorations.⁽¹⁷⁻²⁰⁾

Surface characteristics play an important role in the clinical durability of dental ceramic materials,⁽¹¹⁾ as superficial biodegradation, in the form of hydrolysis, ion exchange and micro-cracks, resulting from contact with chemical solutions have detrimental effect on the esthetics, mechanical and biological properties ^(2, 10, 21, 22). Preventive media, such as mouth washes, may cause changes on the surface of ceramic restorative materials due to their pH and chemical ingredients^(23, 24) The current study aimed to evaluate hardness, roughness and EDAX of LDS and ZLS ceramic immersed in different mouth washes; containing either chlorhexidine, povidone iodine, green tea and pentasodium triphosphate.

Varied mouth washes are commercially available for patients as OTC. For that, selection of mouth washes in this study was done regarding the most commonly purposes patients used the mouth washes. Chlorhexidine (CHX) is the most common mouth wash used for preventing dental plaque and halitosis. It is considered the gold standard for antiseptic mouthwashes. Even though CHX has been found to be very powerful in decreasing bacterial dental plaque, it can change the surface properties leading to discoloration in both teeth and restorations^(5,9,25,26). The ADA emergency guidelines for dental procedures during the COVID-19 pandemic have the recommendation of using preprocedural mouthwashes containing oxidizing agents such as 0.2% Povidone-iodine (PVI) to minimize the risk of COVID-19 transmission.⁽²⁷⁻²⁹⁾

Green tea, Camellia sinensis, which is not fermented at all during the drying process, has numerous medicinal benefits due to antibacterial and antioxidant behavior⁽³⁰⁾. Herbal based mouth washes are preferable by dentist and patients for the reason of offering beneficial effect with minimal shortcomings that could be happened with other chemically artificially prepared mouthwashes. Mouth washes based on green tea are newly introduced natural extract mouthwash that claimed to offer antibacterial and antioxidant effect. Different whitening agents are applied to treat discoloration. In Office whitening products are usually performed by the dentist. On the other hand, mouth washes, toothpastes, gels, chewing gums and whitening strips are easy used, low cost and commonly used by patients at home aiming to prevent or remove teeth discoloration⁽³¹⁾.

Despite the dentist and manufacturers' instructions for usage period of mouth washes, they have been widely used by patients for a longer period of time ⁽¹⁰⁾. The immersion cycle used in this study was done to resample the usage of the mouth wash twice daily for one minute each time for three months.

Hardness and roughness are very important surface properties that can give an indication to the longevity and clinical success of dental ceramics. Hardness is a property of the material reviles the resistance to surface indentation and scratching. It is an essential property to compare materials and can give an indication to finishing and polishing ability of the materials, wear resistance as well as abrasiveness for the opposing⁽³²⁾. Meanwhile, surface roughness is an important parameter that contributes to judgment on aesthetics, initial bacterial colonization and plaque accumulation as well as it had a deleterious effect on antagonist wear⁽³³⁾.

Surface roughness measurements of Ra can be performed by two main methods; contact and noncontact. The contact method is done by the moving of a stylus across the surface to follow the profile. The measurements of the contact method are affected by the diameter of the stylus tip and pressure applied by it as well as the tip might scratch or alter the surfaces and measuring time is extended due to the direct physical contact between the stylus and the surface^(34, 35). For this reasons, noncontact optical method was used in this study as it give a quantitative characterization of surface topography⁽¹⁴⁾ and detect the profile more accurately through the penetration capability of light into smaller amplitudes without alternation of the surfaces as happened in the contact method⁽³⁵⁾.

The present study confirmed the hypothesis and rejected the null hypothesis as the VHN and Ra results revealed that the immersion in mouth washes had a statically significant effect on decreasing the VHN and increasing Ra for both types of ceramic. These results may denote deterioration of the surface for both types of ceramic. The deterioration can be explained by the acidic nature of the mouth washes. Acidity has a great impact on glass ceramics. It is well known that the chemical stability of the glass ceramics declined in the acidic solutions due to the affinity of the acids to alkaline ions leading to its diffusion from the glassy matrix to the solutions producing pores and channels lead to impairment of the ceramic structure⁽³⁶⁾. The pH of mouth washes used in this study were 6.6, 6.5, 4.8 and 3.5 for

CHX, whitening mouth wash, green tea based and PVI mouth wash respectively. Despite of CHX and the whitening mouth washes used in this study were slightly acidic, the extended exposure for a time equivalent to 90 days could exacerbate their effect.

Moreover, the results also can be explained by exposure to sodium fluoride (NaF) which is a component in the mouth washes in subgroups II, IV and V. Fluoride released from NaF can weaken the bond between oxygen and silicon in silicon dioxide, which is a basic composition in both types of ceramic, increasing the chance of bond breakage. Bond breakage may be associated with formation of SiF4 which affected hardness and roughness^{(37, 38).}

Despite the presence of NaF on composition of subgroup II, Fluoride may not be the reason for declination of surface properties in this subgroup. Previous studies on the combination of fluoride compound and CHX in mouth wash showed that monofluorophosphate and CHX combination led to elimination many free CHX due to reactions happened between them. However, NaF and CHX combination did not showed this drawback due to formation of chlorohexidine difluoride which did not jeopardise the effect CHX's on plaque and bacteria⁽³⁹⁾. On the other hand, CHX might affects the properties of fluoride^(40, 41). This finding is in harmonious with the EDAX results which showed that no fluoride precipitations were found on the surfaces of the samples immersed on subgroup II while subgroups IV and V showed precipitation of fluoride on surfaces of the samples with concentration of 0.58% and 0.29% by mass respectively for group A and 0.0.9% and 0.17% by mass respectively for group B.

Nevertheless, besides the exposure to slightly acidic media for prolonged time for subgroup II, another explanation for declination of the hardness and increasing surface roughness after immersion in CHX could be stated. Chlorhexidine is a highly cationic compound, which might breakdown the glassy matrix, facilitate releasing and reactions with ions like Na, K, Ca, Li and phosphate in the matrix^(39, 42, 43).

Furthermore, subgroup III for PVI mouth wash is the most acidic immersion solution used in this study and acidity has a great impact on deterioration of glassy phase, there is other explanation for its effect on hardness and roughness. Iodine ions present in PVI have a high oxidative potential (44) that might be able to oxidise the ceramic surface in acidic media leads to surface degradation. This finding is in harmonious with the EDAX results which showed that Iodine precipitates were found on surface of both ceramic types in subgroup III with concentration of 0.03% by mass. Moreover, the EDAX results did not showed the Li element in the reading for both ceramic types as it can not be detected by EDAX. The low energy of Li could explain that as it is too low to be detected by a standard EDAX detector (45,46)

The present study potential limitation was incompetence to simulate the actual orally environment as it's an in vitro study. In spite of immersion in cycles was a trial to mimic actual use of the mouth washes, but other factors like fluctuation in pH, temperature and stress which normally present in the oral cavity did not be applied. Other studies simulating the oral cavity environment are recommended in confirmation to this study.

CONCLUSION

Using chlorhexidine, povidone iodine, green tea and pentasodium triphosphate based mouth washes for 90 days affect hardness and roughness of LDS and ZLS ceramics.

CLINICAL RECOMMENDATION

Patients with LDS and ZLS ceramic restorations should be instructed to use mouth washes carefully and do not exceed the recommended instruction for usage.

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