



Investigation the Effect of Hydrothermal Aging on the Microstructural, Physical, and Color Stability of Different Dental Ceramics. (An in vitro Study)

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Abstract

Purpose: To evaluate the influence of thermocycling on the microstructural, physical, and color stability of three CAD/CAM ceramic materials.

Materials and Method: In total, 93 specimens (7×5×1.5mm) were prepared from lithium disilicate glass ceramic (IPS e.max CAD), Extra translucent zirconia (VITA YZ), and resin nanoceramic (Cerasmart 270). All the materials was A2 or equivalent shades. The samples were obtained as directed by the manufacturer. these were samples exposed to thermocycling at 5 to 55 C in distilled water for (10.000 cycles). Color stainability, surface roughness, microhardness and surface microstructure were measured and assessed prior and after thermocycling. The Statistical analysis were performed with a Wilcoxon Signed Ranks Test, independent Kruskal-wallis test and Pearsons's correlation analysis. The significant level was set at $P \leq 0.05$.

Results: prior and after hydrothermal aging Cerasmart presented the lowest hardness value . VITA XT exhibited the highest hardness value . The surface roughness had the maximum level of exhibition in VITA XT group . lithium disilicate exhibited the lowest surface roughness values . Cerasmart showed the highest color changes values (ΔE) after hydrothermal aging , lithium disilicate showed the lowest (ΔE) values . after the hydrothermal aging the hardness of VITA XT increased significantly . surface roughness of lithium disilicate increased significantly after aging . for stain ability all the materials used in this study was within the clinically accepted threshold < 3.3 .

Conclusion: Cerasmart showed least degree of hardness before and after aging and highest stainability after aging . Before and after aging, VITA XT showed the highest levels of hardness and surface roughness. lithium disilicate presented the lowest stainability , lowest surface roughness prior and after aging .

Introduction:

The Ceramic materials have become increasingly prevalent in modern dental clinics due to their aesthetically pleasing appearance, exceptional durability, and biocompatibility with oral tissues. Nonetheless, the clinical application of said materials in the posterior region remains restricted due to their susceptibility to brittleness and fracture. Consequently, the examination of the ceramic structure and hardness remains crucial in enhancing comprehension of the efficacy and durability of these restorative materials within the oral cavity, across varying circumstances ⁽¹⁾.

Since the oral biomechanical regime affects the prognosis and longevity of tooth restorative material, it is crucial to have a firm grasp on this topic, Like The mechanical properties of dental restorative materials and dental hard tissue should be similar or the same for optimal performance and to prevent damage to the dental tissue (if the material is harder) or restorative material fracture (if the restorative material is less stiffness than the opposite dental tissue). ⁽²⁾

The effects of intraoral conditions on dental ceramics like alterations in microhardness, surface roughness, temperature, and staining lead to Different degrees of ceramic degradation, absorption, and adsorption of pigments which occur in different ratio according to the composition of the ceramic and its surface treatment⁽³⁾.

When the ceramic surface is kept smooth, we will achieve greater aesthetics and patient comfort. While increasing roughness has an impact on gloss and plaque buildup has an impact on mechanical and physical qualities. The critical threshold for bacterial retention is an average roughness of 0.2 μm ⁽⁴⁾.

It has been observed that rougher surfaces increase the probability of crack development and propagation in ceramic restorations. Additionally, it has been demonstrated that the surface roughness of the restoration has a significant impact on the wear rate of an opposite teeth ⁽⁵⁾.

Hardness is one of the characteristics that influences how well and how long dental materials last. Therefore, a reduction in hardness causes materials to age more quickly and become fatigued ⁽⁶⁾.

Thermocycling is a common technique for artificially speeding up the aging process of ceramics since it mimics the mouth environment. The baths used in the water aging process typically range in temperature from 5 to 55 degrees Celsius, and the process is repeated multiple times. The thermocycling approach has been shown to have an effect on restoration longevity and to accurately mimic the behavior of ceramic in the mouth. ⁽⁷⁾. This study aimed to investigate the effect of thermocycling aging on the microstructure, surface roughness and stainability of different CAD/CAM dental ceramics.

Materials and Methods

Specimens (ceramics) Preparation : 3 types of dental ceramic (N=93) made by milling . lithium disilicate glass ceramic (IPS emax CAD, Ivoclar vivadent, USA), VITA YZ Extra translucent Zirconia (VITA, Germany) and CERASMART 270 (hybrid ceramic, GC dental products. Japan), (n=31) Were examined for their optical, physical and degree of roughness properties prior to and following thermal cycling. Materials evaluated and their respective compositions are detailed in Table (1). The samples (rectangular shape) prepared by sectioning CAD/CAM blocks of (7*5*1.5) mm dimensions (confirmed by a digital micrometer) utilizing a low-speed saw cooled by water (Isomet 1000, Buehler, IL) and then polished with wet 400-, 600-, and 800-grit silicon carbide papers. After polishing, the lithium disilicate specimens were glazed and crystallized in a Programat EP 3010 furnace (Ivoclar Vivadent, Schaan, Liechtenstein), while the zirconia specimens were sintered in a ZETIN furnace (ZTCF-30B), both according to the manufacturers' guidelines.

Color Change Measurement Procedure:

One experienced operator used a spectrophotometer (VITA Easyshade Advance; Vita Zahnfabrik) to measure the

CIE $L^*a^*b^*$ values of the polished surfaces of each specimen against a white background. Each and every measurements was taken in accordance with the specifications provided by the supplier. The spectrophotometer was checked for accuracy before and after taking readings from each sample. Each specimen was measured three times for CIELab values, and the average was used to determine the specimen's mean color value. After taking measurements before and after thermal aging, the (ΔE) values were determined using the following equation. ⁽⁸⁾.

$$\Delta E^* = \sqrt{(L_2 - L_1)^2 + (a_2 - a_1)^2 + (b_2 - b_1)^2}$$

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$$

Surface Roughness Measurements:

Three separate measurements of roughness were taken for each specimen by using an AFM(NaioAFM) in noncontact mode. 20 *20 mm scan sizes were taken and the scan rate was 0.8 Hz. AFM provide 3D topographical images of surface roughness at nanometer resolution. Sa and Sq values were obtained as extension of Ra (the calculation of the heights at all points on the surface) and Rq (the average of root mean square of the Hight deviation taken from the mean image data plane. ⁽⁹⁾.

Microhardness Testing: Before and after thermocycling , the microhardness of the samples was measured by using the indentation technique (digital microhardness tester, LARYEE) using a load of 9.81 N to determine an average hardness value.. Three measurements were performed on each sample and the average of these three measurements was accepted as the surface hardness of the samples. ⁽¹⁰⁾.

How Artificial Aging Of The Samples was done. The samples were aged in a thermocycler for 10 thousand cycles at 5 and 55 degrees Celsius with a dwell period of 30 seconds at each temperature. ((Dorsa; Karaj, Iran)), ⁽¹¹⁾, After initial measurements for optical, microhardness and roughness parameters, the samples were aged for 10,000 thermocycles in

distilled water. to estimate ten years of oral conditions.⁽⁷⁾.

Scanning Electron Microscopy (SEM): In the current investigation, the samples underwent evaluation utilizing scanning electron microscopy (SEM), which facilitated a qualitative characterization of the surface properties. The ceramic samples, which were studied and had varying compositions, were observed both before and after undergoing thermocycling.

Method of Statistical Analysis: the data were analyzed with a Wilcoxon Signed Ranks Test and the correlation between the color change, surface roughness and microhardness were statically analyzed using Pearson's correlation analysis. The correlation were made between ΔE (color change prior and after thermocycling), ΔSa , ΔSq (Sa, Sq before and after thermocycling) and microhardness prior and after thermocycling .

Results

Color change parameters values are summarized in Table (2).

In Table (2), mean values for the color changes parameters and show the differences between the mean and standard deviation and error. After measurements the lowest ΔE value was in the Lithium disilicate followed by vita and. the highest ΔE value was observed in the Cerasmart group and there is no a significant difference statically between all these groups .For acceptability all the ceramic groups was lower than the acceptability threshold < 3.3.

Roughness Measurements: Sa and Sq parameters the mean value and statistical analysis of the surface roughness (Sa and Sq) are shown in figure. before hydrothermal aging extra translucent zirconia presented the highest Sa and Sq values, and the lithium disilicate exhibited the lowest. after hydrothermal aging all materials show increase in surface roughness with same order extra translucent zirconia the highest and the lithium disilicate the lowest. Only lithium disilicate show significant increase in value after hydrothermal aging.

Microhardness Measurements: the

microhardness values of the ceramic groups are shown in Table (2). Before hydrothermal aging The highest hardness values was found

in extra translucent zirconia, the lowest was the Cerasmart group . after hydrothermal aging extra translucent zirconia exhibited statically significant increase in microhardness, the lithium disilicate exhibited non-significant increase in microhardness while Cerasmart show statically non-significant decrease in microhardness as shown in Figure (2).

Scanning Electron Microscopy:the lithium disilicate samples show needles like crystals. After thermocycling the samples show visible change so that crystals (needle like crystals) cannot seen clearly. The vita SEM pictures show more homogenous grain size distribution before hydrothermal aging than after hydrothermal aging.

Discussion

The study found that after being subjected to thermocycling, the materials' surface roughness, microhardness, and stainability were significantly different from their pre-cycling counterparts.

Before thermocycling the greater color stainability found in this study with a Cerasmart samples and the lowest one was lithium disilicate samples.

IPS e.max CAD contain small lithium disilicate grains, A more uniform microstructure, provided by a smaller crystal size, is said to improve optical and chemical properties. Thus, the EMX's increased stain resistance could be attributable to its finer grains and more uniform microstructure.. Because CERASMART has a resin matrix composed of Bis-MEPP, UDMA, and DMA monomers, the material has the ability to absorb force, which is why CER was the most discolored material in the testing. It has been claimed that resin materials are discolored more than ceramic materials due to the hydrophilicity of the matrix, making UDMA and triethylene glycol dimethacrylate (TEGDMA) undesirable additives..⁽¹²⁾.

Previous research has shown that PS e.max CAD discolors less than

CERASMART and our findings confirm this.⁽¹³⁾.Another study tested the effects of Staining and aging-dependent changes in color of CAD-CAM materials lithium disilicate based ceramics, exhibited better color stability as compared with resin nanoceramics and PICN, because of polymer up taking water and higher surface energy for resin CAD/CAM materials⁽¹⁴⁾. The color change for the VITA YZ XT was less than CERASMART and more than the lithium disilicate and all groups was with in the clinical acceptance . this finding for VITA XT can be associated to increase grain sizes and more cubic phase which is optically isotropic.

Kanpalta, Burduroğlu (15) concluded in their study the artificial aging affected the optical properties in VITA XT , but no effects on lithium disilicate. **Kurt and Turhan Bal (16)** investigated the effect of artificial aging on color stability of zirconia and lithium disilicate ceramics , the results of The aging process affected the color of the specimens, especially the zirconia specimens, which were found to be clinically unacceptable ($\Delta E=5.03$) , they explained the cause to tetragonal to monoclinic phase transformation which called low temperature degradation (LTD) , this lead to increase in volume and stress formation and increase the roughness of surface. Also reduced alumina content to increase translucency lower its resistant to (LTD). **Dikicier, Ayyildiz (17)** found the mean color difference of the zirconia specimens was($\Delta E = 1.29$) . this value was so close to the value in this study. But for lithium disilicate was higher than the value in this study. Surface roughness is crucial for plaque accumulation capacity and discoloration of dental materials. Surfaces of restorations are susceptible to deterioration and loss of shine when in intraoral service due to factors including tooth cleaning and food acidity. ⁽⁹⁾. Surface roughness can be measured in a number of ways. Optical and scanning electron microscopy, as well as quantitative techniques like surface profile analysis, have been used in the study of surface roughness in dental materials. Surface topography can be more precisely defined by AFM, as evidenced by differences

between SEM and AFM. ⁽¹⁸⁾. Before hydrothermal aging VITA XT exhibited the highest Sa value while the lithium disilicate was the lowest surface roughness and this appear in other studies.⁽¹⁹⁾. Another previous study showed that Cerasmart presented higher Sa values than lithium disilicate before and after aging. ⁽⁹⁾. Modern study on CAD/CAM composite materials showed Sa, Sq value for Cerasmart so close to the Sa, Sq values in this study.⁽²⁰⁾. All the materials used in this study exhibited increase in surface roughness after hydrothermal. but all the Sa values was equal or less than 0.2 μm As a result, clinical acceptability of the materials is assumed.

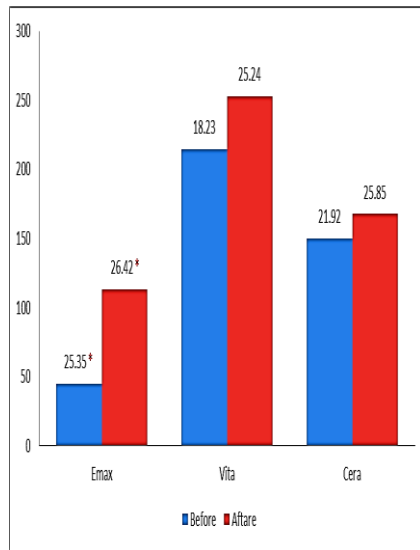
In the SEM observations conducted in previous studies, showed that Cerasmart presented the most consistence appearance prior and after aging, while lithium disilicate showed more random irregularities in lithium disilicate particles after aging however these irregularities were not significant and no changes in surface composition were observed and this appeared in this study also. ⁽²¹⁾. Another study investigated the effects of thermocycling on lithium disilicate showed the milled samples of Emax CAD presented more significant changes after aging.⁽²²⁾. Because of its direct correlation with the material's stiffness and mechanical strength, hardness has a direct impact on the clinical success and survival rates of the restorations. A Vickers indenter with a 98 N load was used to measure the material's hardness and fracture toughness over the course of 10 seconds. In this study lithium disilicate show non significant increase in hardness after aging while Cerasmart show slight decrease in hardness after aging only Vita show a significant increase in microhardness. ⁽¹⁾. In this study the lithium disilicate and Cerasmart showed slight variations in hardness after hydrothermal aging in (Table 4) only VITA XT showed statically significant increase in hardness after hydrothermal aging.

The result of previous studies exhibited that ceramic materials show significantly higher hardness values than materials with polymer resin components, even after aging, materials with ceramic network

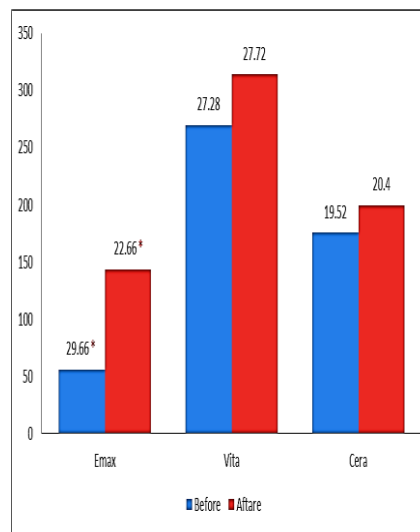
structures like lithium disilicate and VITA XT have been reported to be less affected by aqueous conditions and thermocycling ⁽²³⁾. Previous study showed that the hardness value of VITA XT higher than lithium disilicate. Higher Vickers hardness value reflects milling difficulty, being less prone to marginal chipping, and less permanent deformation of the material surface. This is likely because zirconia, which has a finer-grain structure, would be more resistant to permanent deformation compared glass-based ceramic materials. ⁽¹⁹⁾. Ceramic characteristics are affected by the material's microstructure. The mechanical and optical capabilities of ceramics are strongly influenced by their microstructural factors, such as grain size and porosity. ⁽²²⁾. Cerasmart contain uniform sized filler particles that are less than 500 nm. ⁽²⁴⁾, the filler loading of Cerasmart is 71 mass% and the filler particles are very small with rather narrow distribution. ⁽²⁵⁾ after hydrothermal aging Cerasmart show clear alteration in their surface microstructure whereas lithium disilicate and extra translucent zirconia show relatively slight alteration and this proven by other studies.⁽⁹⁾. The grain size for extra translucent zirconia 815 nm, the grain boundaries are clear and homogenous. ⁽¹⁾. The highest fracture toughness was observed for extra translucent zirconia followed by lithium disilicate and Cerasmart. ceramic materials exhibited significantly higher value than materials with polymer resin components.⁽⁹⁾. In this study After hydrothermal aging only extra translucent zirconia exhibited significant increase in microhardness.

Conclusion

1. The aging process affected the resin ceramic more than glass or polycrystalline ceramics.
2. Microstructure have direct effects on the optical and mechanical properties of cad cam ceramics.
3. The lithium disilicate was the least stainability after aging and the VITA XT was the highest Vickers value.



A.



B.

Figure (1): A: Sa B. Sq (surface roughness before and after hydrothermal aging)

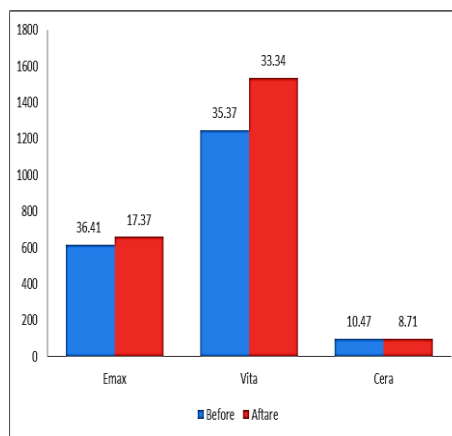


Figure (2): Average microhardness values for ceramic groups

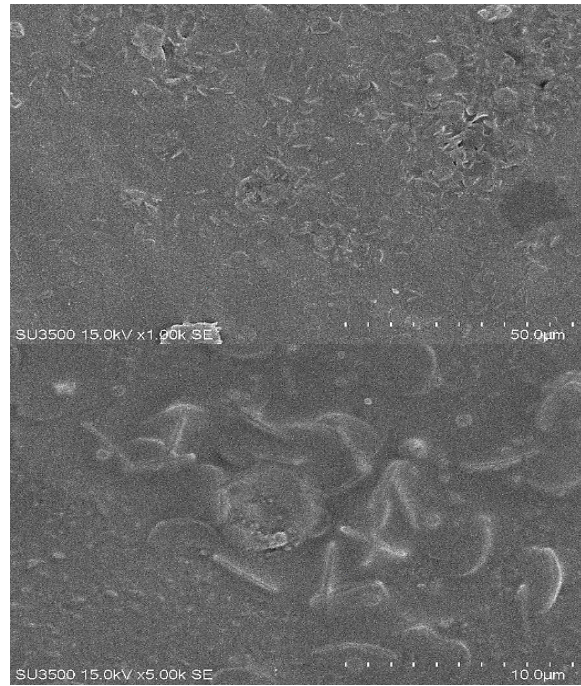


Figure (3): Lithium disilicate before hydrothermal aging

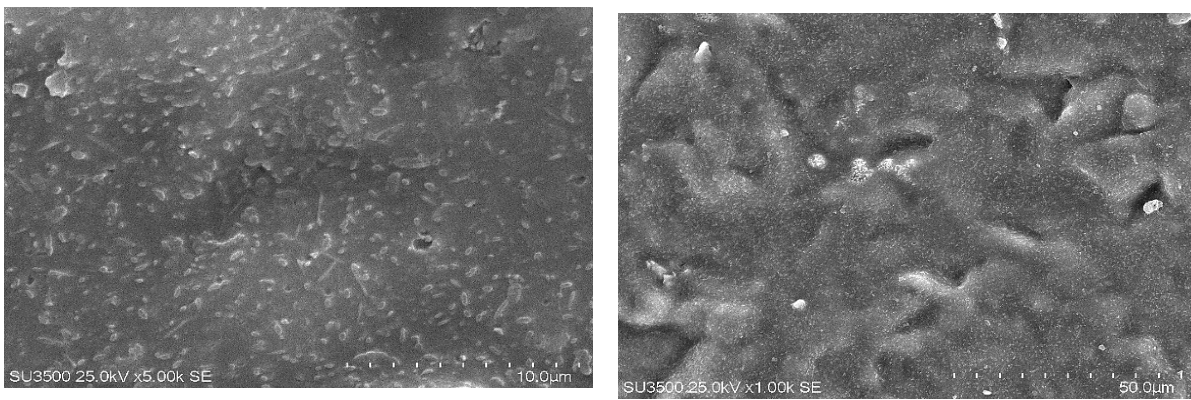


Figure (4): Lithium disilicate after hydrothermal aging

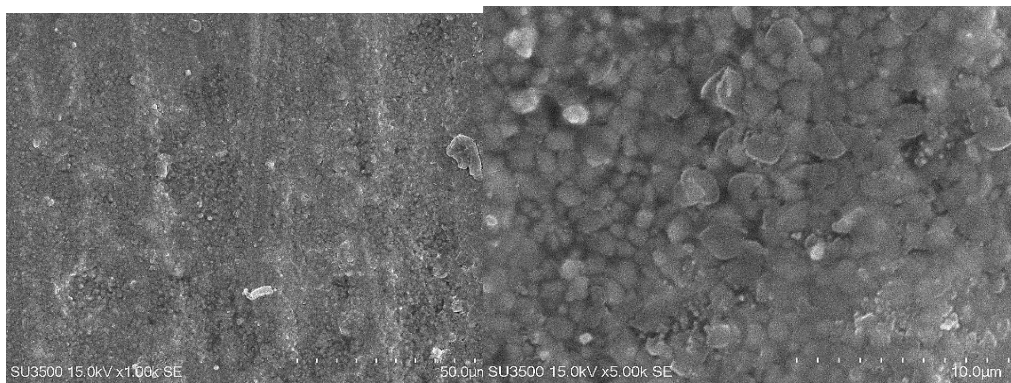


Figure (5): Vita before hydrothermal aging

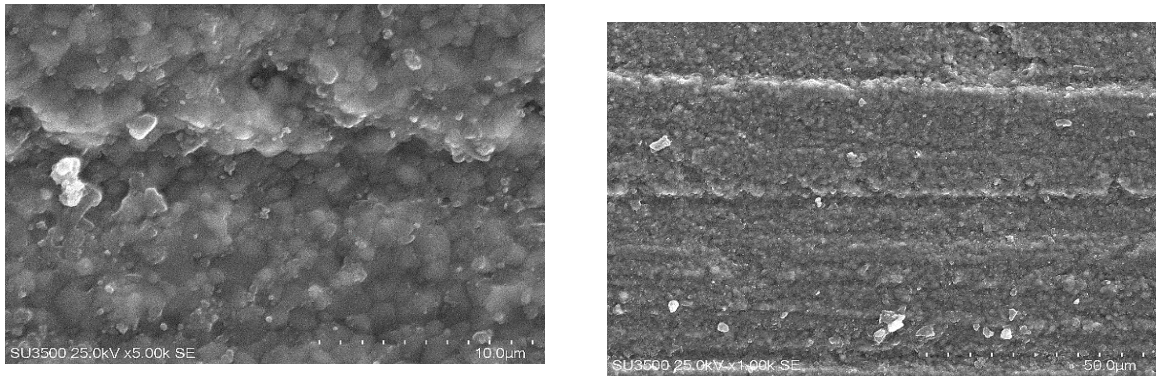


Figure (6): vita after hydrothermal aging

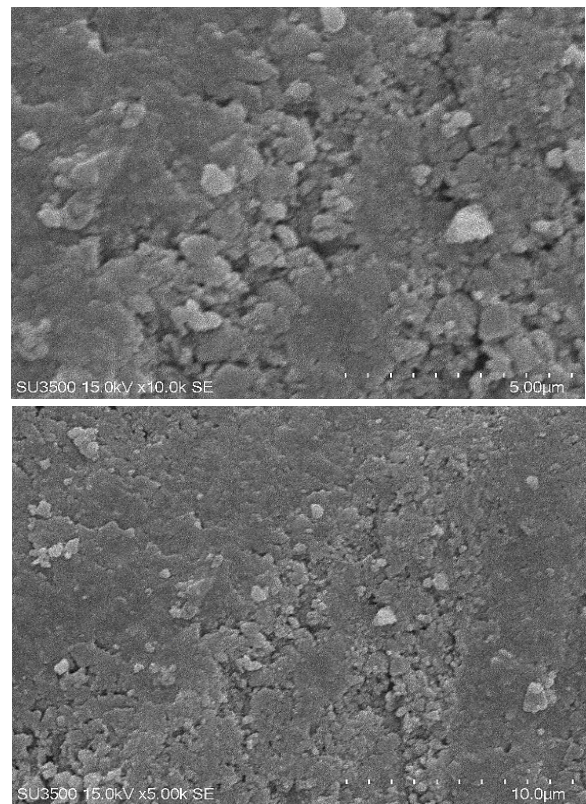


Figure (7): Cerasmart before hydrothermal aging

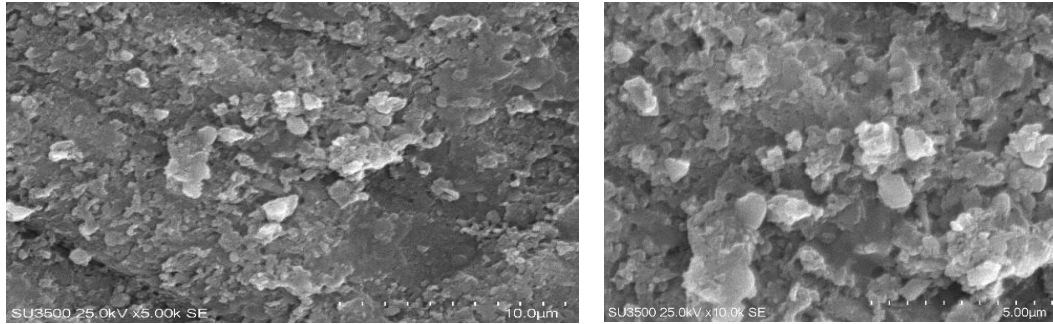


Figure (8):Cerasmart after hydrothermal aging

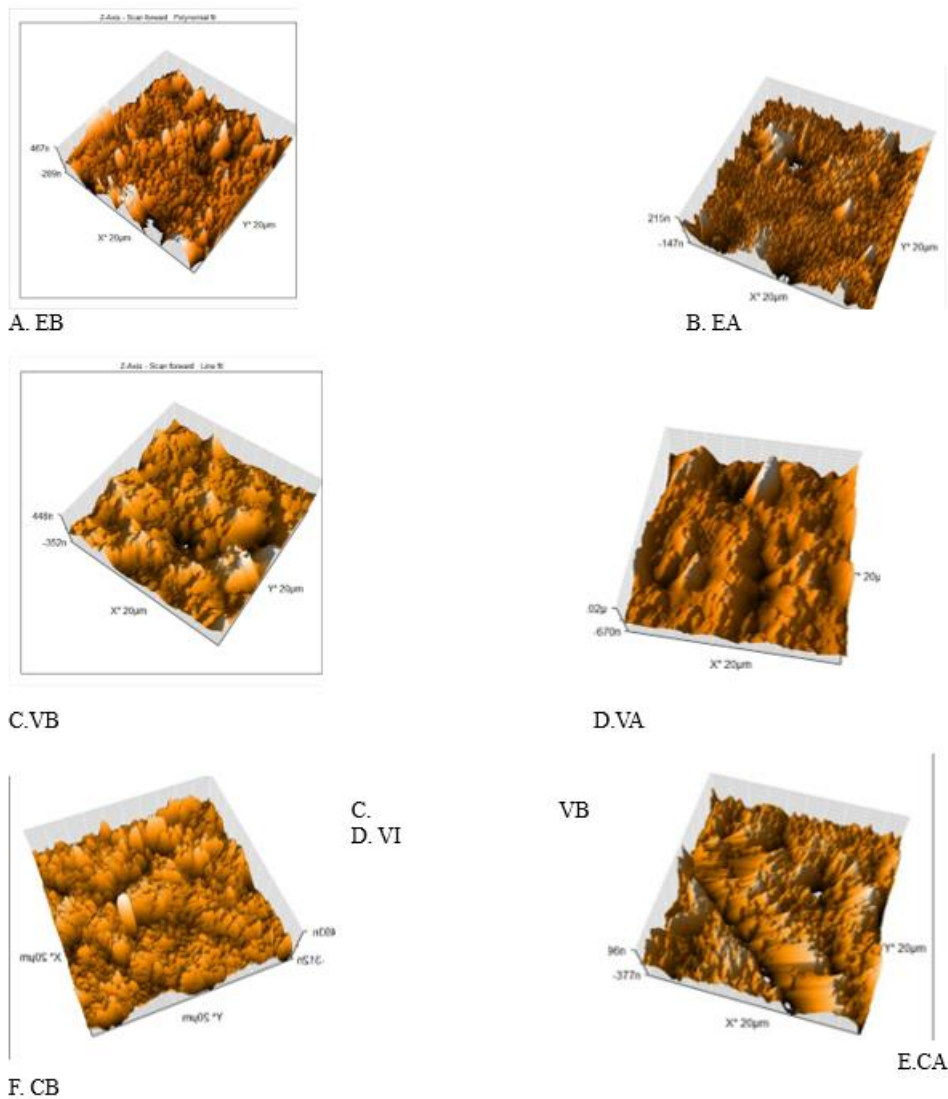


Figure 9: Atomic Force Microscopy Images. A: lithium disilicate before aging. B: Lithium disilicate after aging. C: VITA Before aging. D: Vita after aging. E: Cera before aging. F: Cera after aging.

Table (1): CAD/CAM Materials

Materials	Composition	Manufacturer
Cerasmart	Composite resin materials (UDMA,BisMEPP,DMA)with 71wt.%SIO2(20nm)and barium glass(300nm) nanoparticles	Cerasmart (GC dental products tokyo. japan)
lithium disilicate glass ceramic(IPS emax CAD	57–80%SiO2,11-19% Li2O,K2O,MgO, Al2O3, P2O5 and other oxides	Ivoclar vivadent,schaan,liechtenstein
Extra translucent zirconia	Zro2(86-91%),Y2O3(8-10%),HFO2(1-3%),AL2O3(0-1%) pigments(0-1%).	Vita zahnfabrik germany

Table (2): Mean and standard deviation of Color Change (ΔE) for all sub-groups

	Materials	N	Mean	Std. Deviation	Std. Error
1	EMAX	31	0.4	3.81067	1.55570
2	VITA	31	1.24	2.25826	0.92193
3	CERA	31	2.3	0.98680	0.40286

Table (3): Surface roughness values (Sa),(Sq) measured by AFM

	Mean	N	Std. Deviation	Std. Error Mean	Sig.	
Emax, Sa	Sa0_EMB	44.5783	31	25.34772	10.34816	0.028 *
	Sa1_EMA	113.2750	31	26.41821	10.78519	
Emax, Sq	Sq0_EMB	55.4183	31	29.65638	12.10717	0.028 *
	Sq1_EMA	143.2933	31	22.65641	9.24944	
Vita, Sa	Sa0_VIB	214.7000	31	18.22548	7.44052	0.075
	Sa1_VIA	252.9000	31	25.24306	10.30544	
Vita,Sq	Sq0_VIB	269.6500	31	27.28258	11.13807	0.116
	Sq1_VIA	313.8833	31	27.72381	11.31820	
Cera,Sa	Sa0_CERB	149.9150	31	21.92310	8.95007	0.345
	Sa1_CERA	167.5833	31	25.85261	10.55429	
Cera,Sq	Sq0_CERB	175.3833	31	19.52326	7.97034	0.173
	Sq1_CERA	199.4833	31	20.39955	8.32808	

Table (4): average microhardness values and SD of the ceramic groups Table 2. average microhardness value and SD of the ceramic groups

	Mean	N	Std. Deviation	Std. Error Mean	
Pair 1	emax bef	616.0833	31	36.41266	14.86541
	emax aft	658.5833	31	17.36922	7.09095
Pair 2	vita befo	1247.6500	31	35.37450	14.44000
	vita afte	1536.1333	31	33.33980	13.61000
Pair 3	cera befo	97.5333	31	10.47123	4.27486
	cera afte	96.9833	31	8.70642	3.55438

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