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Effect of Aging on Microstructure and Mechanical Properties of Some Dental Materials

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Abstract

Effect of aging in saliva on microstructure, molecular structure, hardness and surface roughness of Filtek, glass ionomer, porcelain and zirconia dental materials have been evaluated. From X-ray and scanning electron microscope analysis microstructure of Filtek, glass ionomer, porcelain and zirconia dental materials changed after aging in saliva for one month. Vickers hardness of Filtek and glass ionomer increased but surface roughness decreased after aging in saliva for one month. Vickers hardness of porcelain and zirconia decreased but surface roughness increased after aging in saliva for one month. These results indicated that, there is a significant effect of saliva on microstructural and mechanical strength of used dental materials.

Keywords: Zirconia; Porcelain; Filtek; Glass ionomer; Hardness; Roughness; Saliva; Atomic force microscope

Introduction

Dental restorative materials are used to restore tooth structure loss, usually resulting from but not limited to dental caries. Zirconia can be a near ideal choice for restoring crowns, fixed partial dentures and implants in esthetic areas. In 1993 the first study was recorded on used zirconia for implants in the mandible of dogs [1,2]. Zirconia is used to make ceramic knives because of its hardness; Zirconia based cutlery stays sharp longer than a stainless steel equivalent [3]. Dental ceramics are able to mimic natural teeth due to their excellent physical properties such as esthetics, biocompatibility, low thermal conductibility and wear resistance [4-6]. They have been extensively used in several rehabilitation procedures, including inlays, onlays, crowns, and porcelain veneers [7]. The influence of different light sources on the roughness of Filtek Z250, Filtek P60, Charisma and Durafill with varying post-irradiation times, in an in situ experiment was evaluated [8]. Roughness was influenced only by post-irradiation times, presenting the 30 days period inferior behavior. The influence of two surface sealants and three application techniques on the roughness of two composites (Filtek Z250/Z350) after the tooth brushing test

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was evaluated [9]. The surface microhardness of Filtek Z250 and Filtek-A110 light cured with four different distances of polymerization was investigated by Bezerra et al. [10]. The microhardness of Filtek Z250 was higher at all distances analyzed when compared with Filtek-A110. The flexural strength and modulus of two nanohybrid Grandio and microhybrid Filtek Z250, commercial resin composites were compared at room temperature and 40°C, 45°C and 50°C prior to light polymerization with standard and step-cure protocols [11]. The mechanical properties of the tested materials did not changed by preheating. The effect of aging on roughness and hardness of microhybrid composites was investigated [12]. Hardness at the subsurface was higher than surface hardness and Z250 was harder than Charisma. Roughness ranged from 0.53 to 0.52 for Z250 and from 0.52 to 0.56 for Charisma, at 24 hours, 6 months and 1 year, with no significant differences among the materials. The bottom: top Vickers hardness ratio of Z250, Herculite and Heliomolar after using two LED light curing systems have been evaluated by Godoy et al. [13]. The results show, LED systems evaluated were not significantly different from each other and produced equivalent hardness on the composites tested. A significant softening effect on the surfaces of the Filtek Z250 and Charisma composites, although no significant deleterious alteration was detected for the subsurface hardness after 6 month [14]. In addition, the storage period had no significant effect on the surface roughness of the materials. Gomes et al. [15] studied the effect of artificially accelerated aging on the microhardness of Filtek Z250, Charisma, Durafill VS, Filtek Supreme for body and Filtek Supreme translucent. A significant increase in flexural modulus of Filtek A110, Filtek Z100 and Z250, poly-acid modified F2000 and flowable Filtek was observed with all composites after aging [16]. The surface roughness of glass ionomer restorative cement, two nanohybrid resin composites, a flowable resin composite and a silorane based composite was significantly increased after sonic instrumentation but it decreased after re-polishing [17]. Also the polishing of composite resins showed better results when performed without refrigeration water (dry grinding) and no difference in the hardness was detected [18]. The hardness of composite resin materials are strongly influenced by food simulating solutions [19]. The influence of various artificial ageing protocols (storage in distilled water/ ethanol/artificial saliva for 7/90/365 days) on the surface properties of dental composites materials was investigated [20]. The data indicates that, influences of the artificial ageing method on surface parameters, such as Ra, and hydrophobicity as well as microbial adhesion. A significant difference among hardness means recorded at the different aging time and the tested materials (Art glass; belle Glass) and (Filtek Z250; Alert) [21]. Also all materials presented hydrolytic degradation due to aging in aqueous environment. Resin-based composite materials with different chemical composition showed different surface roughness prior to and following mechanical and chemical aging [22]. The aim of this study was to evaluate the effect of aging in saliva on microstructure, molecular structure, hardness and surface roughness of Filtek, glass ionomer, porcelain and zirconia dental materials.

Materials and Methods

The dental restorative materials used in this study were porcelain, zirconia, Filtek and glass ionomer. Porcelain (VitaVMK, Master, VITAZ ahnfabrik, Germany) were prepared in a standardized manner and according to the manufacturer's directions in rectangular stainless steel split mold (20 mm- 5 mm- 2 mm). Zirconia was Ceramill zi, made in Austria. Feltik 250 (3M ESPE, USA), unpolymerized Filtek was applied in Teflon mold with dimensions 20 mm in length × 2 mm in width × 2 mm in height. To ensure that the resin composite would be well distributed within the mold, 0.5 kgf was applied for 30 s to the material. Glass slides were used to prevent inhibition of surface polymerization due to the presence of oxygen. The specimens were then photocured with a visible light curing unit (Visilux2, 3M Company, ST., Paul, MN, USA) for 40 s on each of the two covered slides. Glass ionomer restorative powder (Kavitan1 Plus; Lot number: 2015388, Spofa Dental, Czech

Republic) (code: GI) was used. The specimens were tested with respect to microstructure, molecular structure, roughness and Vickers microhardness. Microstructure of used specimens was performed on the flat surface of all specimens using an X–ray Diffractometer (Dx–30, Shimadzu, Japan) of Cu–Kα radiation with λ=1.54056 Å at 45 Kv and 35 mA and Ni–filter in the angular range 2θ ranging from 0 to 90° in continuous mode. Also scanning electron microscope was used to investigate surface structure of used specimens. Molecular structure of used specimens was performed using Mattson 5000 FTIR Spectrometer. Vickers hardness (Hv) was measured for all specimens by a digital Vickers microhardness tester (Model FM–7, Tokyo, Japan) at 10 and 100 gf indentation load for 5 sec indentation time. The roughness of Filtek, glass ionomer and porcelain were measured by using surface roughness measurements device (surface test S.J 201.P) but the roughness of zirconia was measured by atomic force microscope.

Results and Discussions

Microstructure

X-ray analysis

The X-ray diffraction patterns of Filtek, glass ionomer, porcelain and zirconia dental materials before and after aging in saliva are shown in Figures 1-4. X-ray diffraction patterns, Figure 1a, of Filtek material show broad hump peak contains small peak with low intensity and small peaks outside which indicated the disturbance of filler particles in matrix. From X-ray analysis, Filtek consists of main matrix peak (glassy matrix started from 2Ө=18° to 37° with fine crystal appeared a round 2θ=31°) and other crystal phase from filler atoms outside appeared at 2Ө=42 and 51°. Figure 1b shows X-ray diffraction patterns of Filtek after aging in saliva for one month, which have new feature such as started base line, amorphous area and formed fine particles. The base line stated at 400 counts and glassy matrix goes to end around 27°. Also many fine crystals formed from filler particles appeared. That is meant that, aging in saliva effected the structure of Filtek (main matrix and particles appeared in surface) which caused change in its surface properties. Figure 2a shows X-ray diffraction patterns of glass ionomer which have broad peak (glassy matrix started from 2Ө=18° to 35°) contained fine crystal from filler particles. Also fine crystal appeared at 2Ө=43°. X-ray diffraction patterns of glass ionomer after aging in saliva for one month, Figure 2b shows that, there is a change on started base line, glassy area and crystal phases appeared. A high started hump at around 500 count and go to end a round 2θ= 26°. Many sharp crystal phases (θ=28, 30 and 40°) and other crystal formed from filler atoms. There is a clear change in glass ionomer structure (main matrix and disturbed crystals) which effects on its surface properties. X-ray diffraction patterns of porcelain shown in Figure 3a have glassy matrix and other crystalline phases. Glassy matrix (20-35°) contains sharp peaks for tetragonal $KAl(SiO_3)_2$ phase. Also sharp peak appeared at 16.5° for tetragonal $Na_2B_4O_7$ (011) with little intensity peaks for hexagonal (SiO_2) disturbed in the composite matrix. X-ray diffraction patterns and it is analysis for porcelain, Figure 3b, show that changed in amorphous feature and formed crystal phases inside or outside. High started hump ~ 500 counts and go to end ~ 2θ=25° with crystal phase of tetragonal $Na_2B_4O_7$ (011) and small glassy matrix contained $KAl(SiO_3)_2$ phase and hexagonal (SiO_2). Also hexagonal (SiO_2) phase disturbed in other place in matrix. The analysis of X-ray diffraction patterns in Figure 4a for zirconia showed that, it consisted of monoclinic ZrO_2 with different plans (hkl), intensity (crystallinty), broadness (crystal size) and orientation (2θ). After aging in saliva for one month zirconia matrix contain both monoclinic and tetragonal ZrO_2 phases with changing started base line and that is affected on its surface properties as shown in Figure 4b.

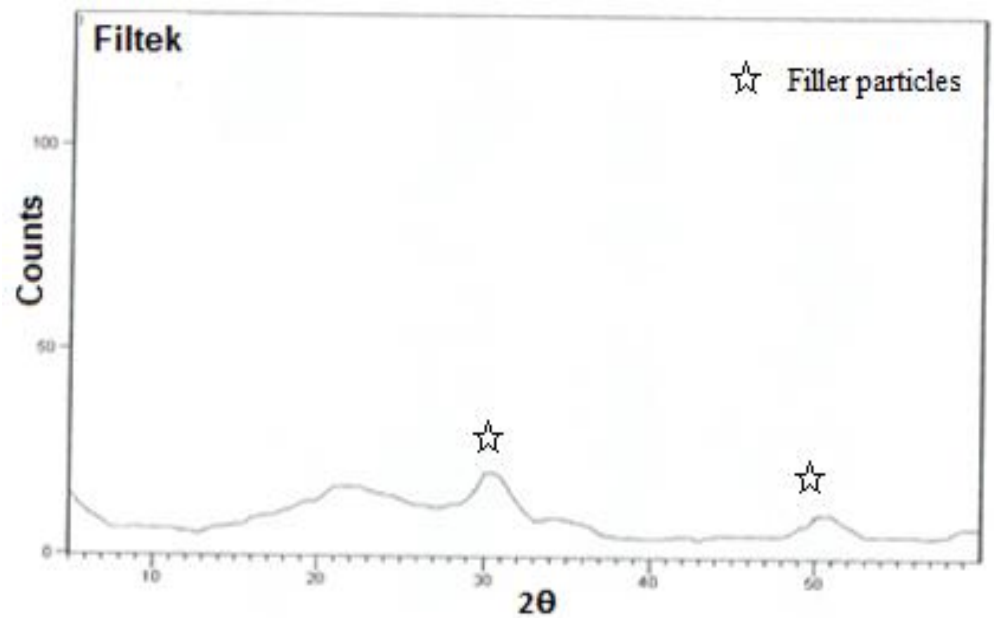


Figure 1a: X-ray diffraction patterns of Filtek

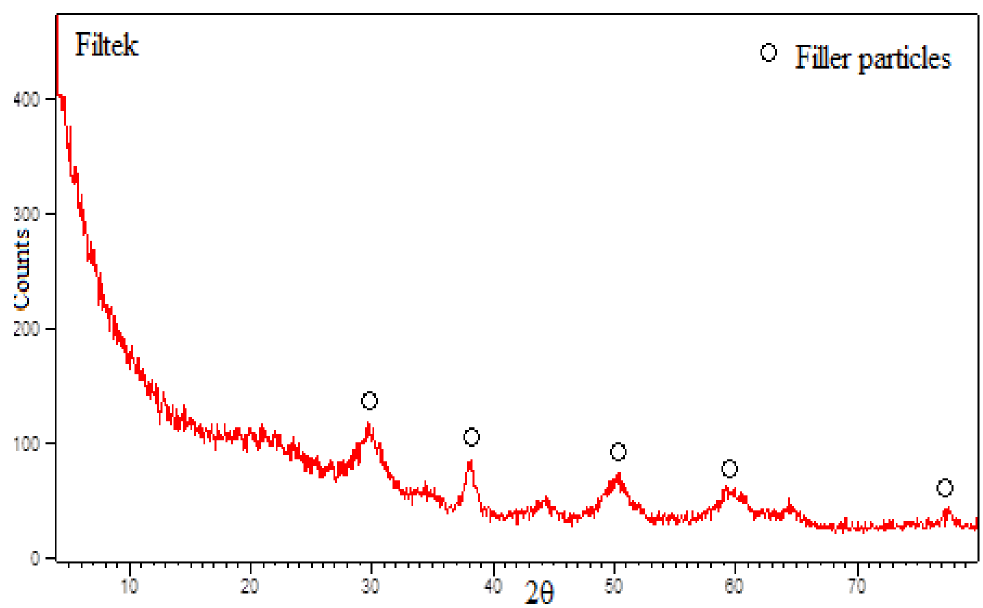


Figure 1b: X-ray diffraction patterns of Filtek after aging in saliva for one month

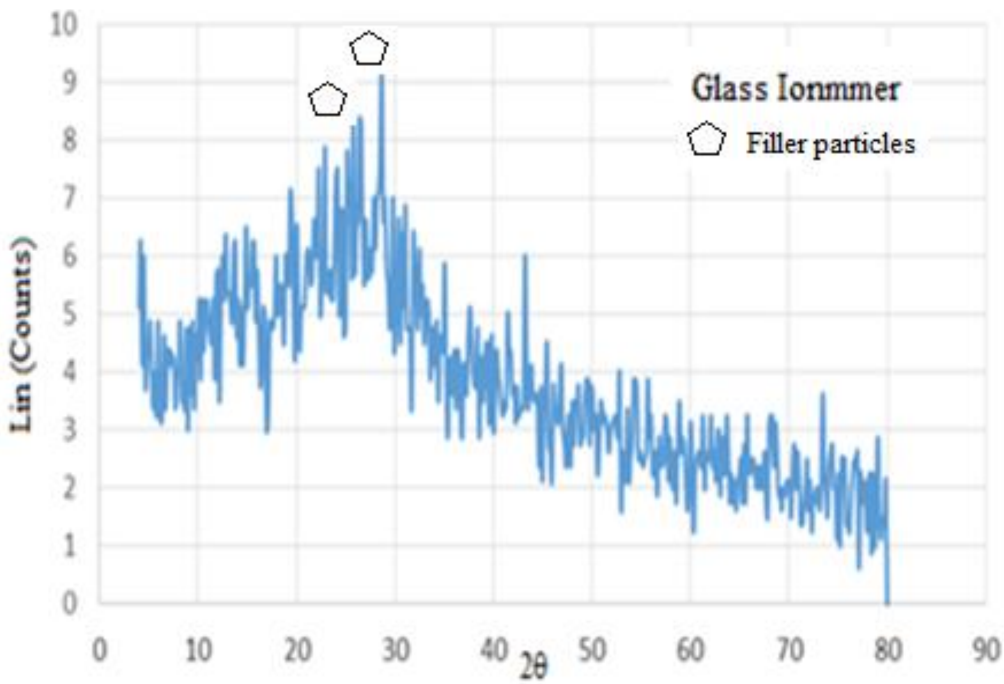


Figure 2a: X-ray diffraction patterns of glass ionomer

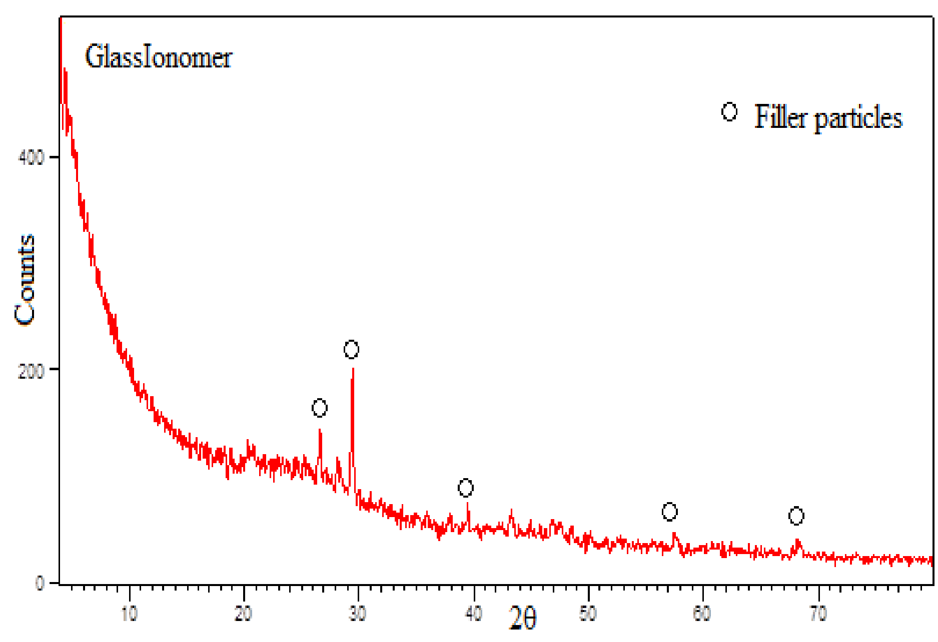


Figure 2b: X-ray diffraction patterns of glass ionomer after aging in saliva for one month

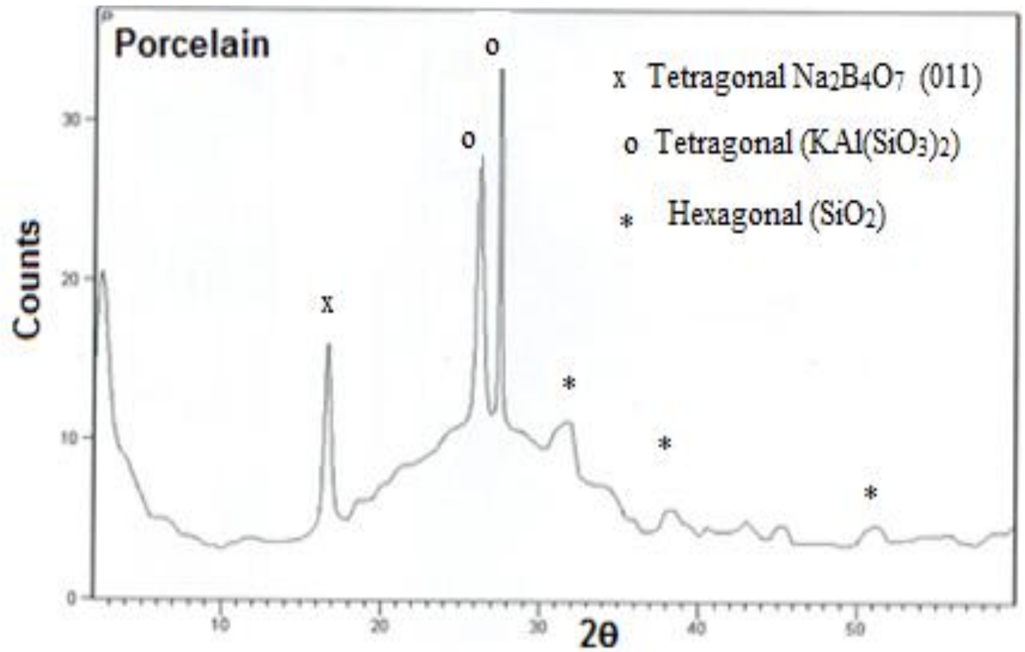


Figure 3a: X-ray diffraction patterns of porcelain

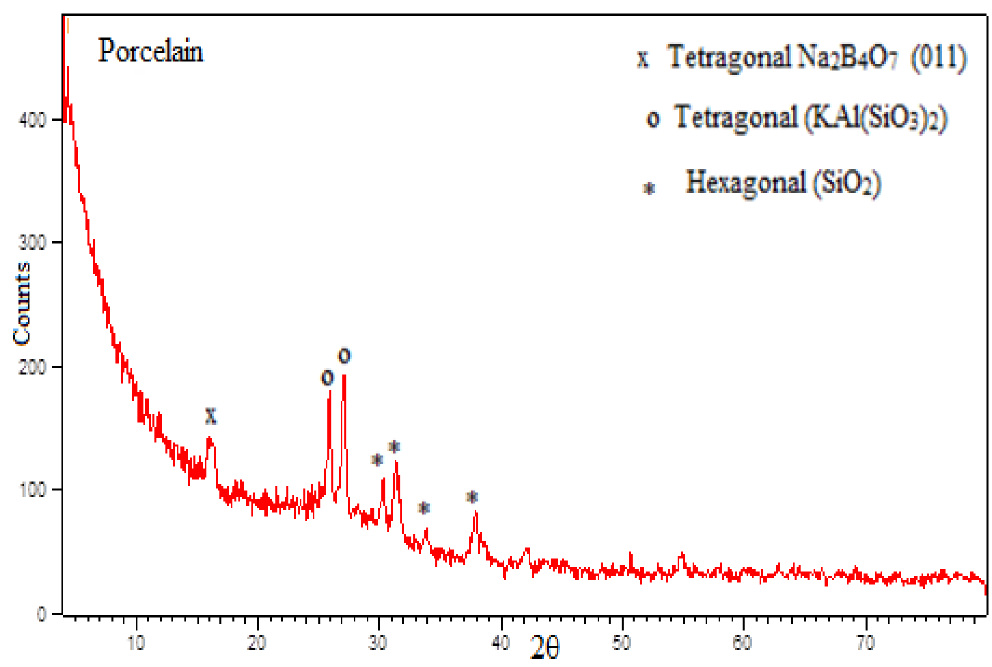


Figure 3b: X-ray diffraction patterns of porcelain after aging in saliva for one month

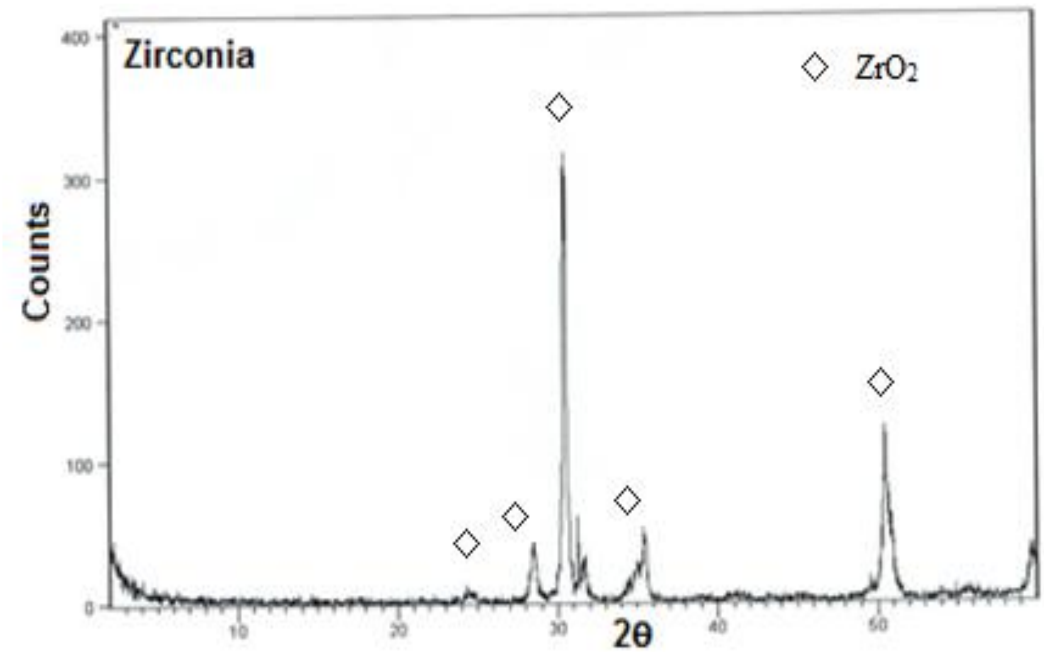


Figure 4a: X-ray diffraction patterns of zirconia

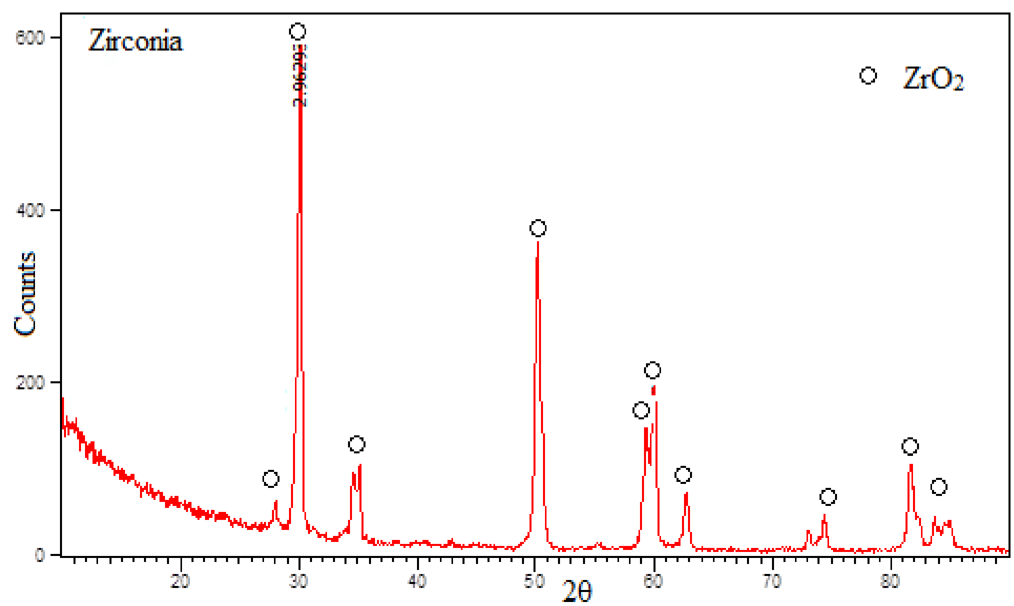


Figure 4b: X-ray diffraction patterns of zirconia after aging in saliva for one month

Scanning electron microscope analysis

Scanning electron microscope graphs, SEM, of Filtek, glass ionomer, porcelain and zirconia dental materials before and after aging in saliva for one month are shown in Figures 5a-5h. SEM, Figure 5a, of Filtek shows glassy matrix (main structure) which have fine resin particles ~ 0.4 µm (white color) or cluster of these particle ~ 2 µm disturbed at different places. Also it has different shape and size of diamond black color. SEM, Figure 5b, of Filtek after aging in saliva for one month glassy matrix (main structure) which have fine resin particles ~ 0.3 µm (white color) and cluster of these particle disappeared and Fungi and Bacteria from saliva appeared in surface (the surface may be appeared as homogenous and smooth). SEM, Figure 5c, of glass ionomer showed fine particles appeared (disturbed in surface of large and condensed chain of molecules or between) in composite matrix with average diameter ~0.2- 0.5 µm. SEM, Figure 5d, of glass ionomer after aging in saliva for one month showed that, fine particles disappeared or very little appeared and more condensed moecules appeared (very colsed or more bonded). SEM, Figure 5e, of porcelain showed that it has sandwich microstructure with different shape, orientation and fine particles disturbed around\or in it. SEM, Figure 5f, of porcelain after aging in saliva for one month showed that, smooth and clear sandwich shape with different size, shape and orientations (vertical and horizontal). SEM, Figure 5g, of zerconia showed that, arrangement of condensed Zo_2 particles in small size or large size and the size ~ 0.3 µm to 2 µm. SEM, Figure 5h, of zerconia after aging in saliva for one month showed that, Zo_2 particles in lamellar structure (different size strip) and other large\or small particles distributed in matrix. These SEM results agreed with X-ray analysis results. Also scanning electron micrograph showed that, Fungi and\or Bacteria stick on the surface of Filtek, glass ionomer, porcelain and zirconia with different sticking area.

Molecular structure

FTIR spectrum

Figures 6a-6h shows FTIR spectra of Filtek, glass ionomer, porcelain and zirconia before and after aging in saliva for one month. Figures 6a and 6b shows IR spectra of based Filtek before and after aging in saliva which exhibited strong sharp peak at 471 cm^{-1}, strong sharp peak at 1100 cm^{-1}, weak-sharp peak at 1725 cm^{-1} and weak-broad peak at 3427 cm^{-1}. IR spectra, Figures 6c and 6d, of glass ionomer before and after exhibited strong sharp peak at 456 cm^{-1}, strong sharp peak at 1044 cm^{-1} and weak-broad peak at 3445 cm^{-1}. IR spectra of porcelain, Figure 6e and 6f, exhibited strong sharp peak at 447 cm^{-1}, strong sharp peak at 1026 cm^{-1} and weak-broad peak at 3439 cm^{-1}. IR spectra, Figures 6g and 6h, of zirconia exhibited weak-wide peak at 1426 cm^{-1} and weak-broad peak at 3425 cm^{-1}.

The results of FTIR spectrum show that, the characteristics of IR bands for used dental materials are changed intensity which related to strong, broad which related to weakness and position which related to molecular type bond which dependent on material molecular structure caused due to diffusivity of water in it and interact of OH with material matrix.

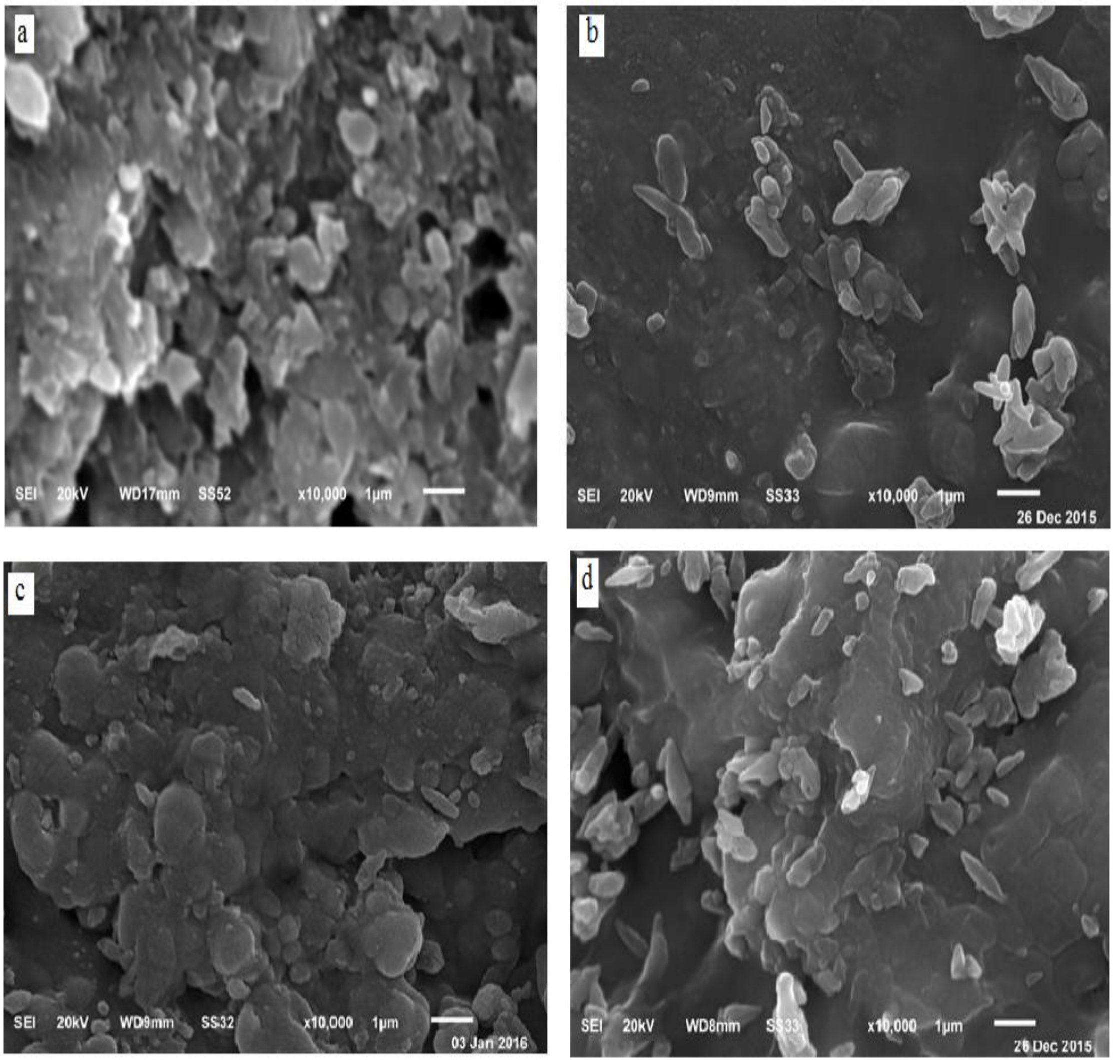


Figure 5: scanning electron microscope Filtek before aging b) Filtek after aging for one month c) Glass ionomer before aging d) Glass ionomer after aging for one month

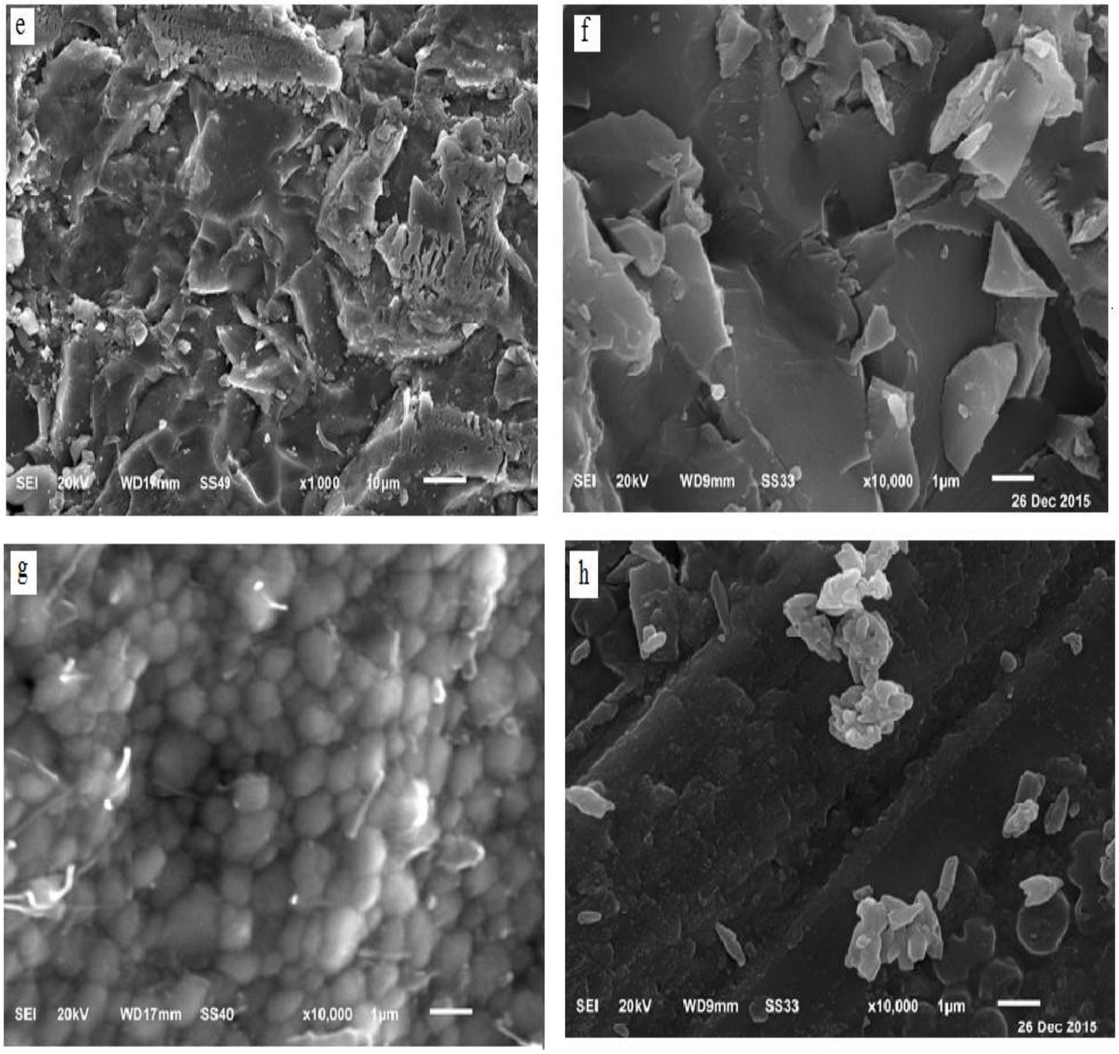


Figure 5: scanning electron microscope e) Porcelain before aging f) Porcelain after aging for one month g) Zirconia before aging h) Zirconia after aging for one month

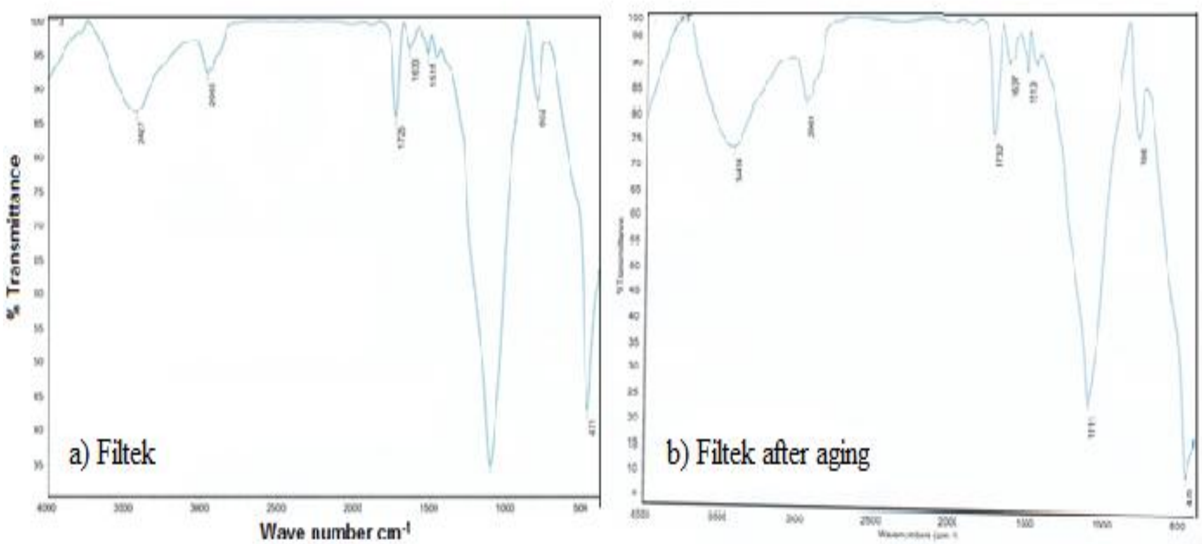


Figure 6: a and b) IR spectra of Filtek before and after aging in saliva

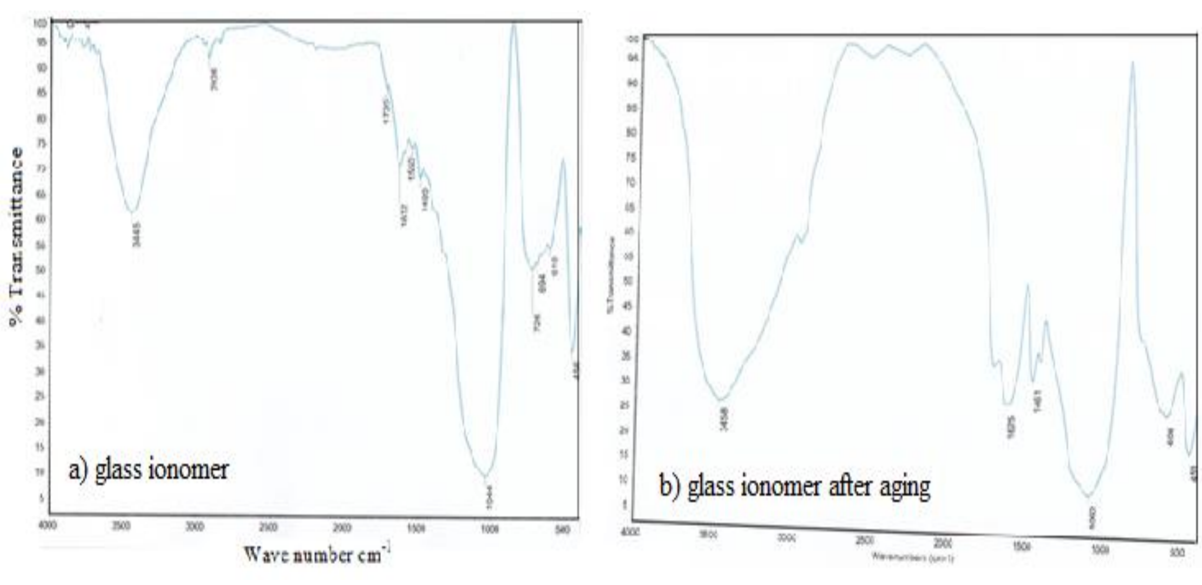


Figure 6: c and d) IR spectra of glass ionomer before and after aging in saliva

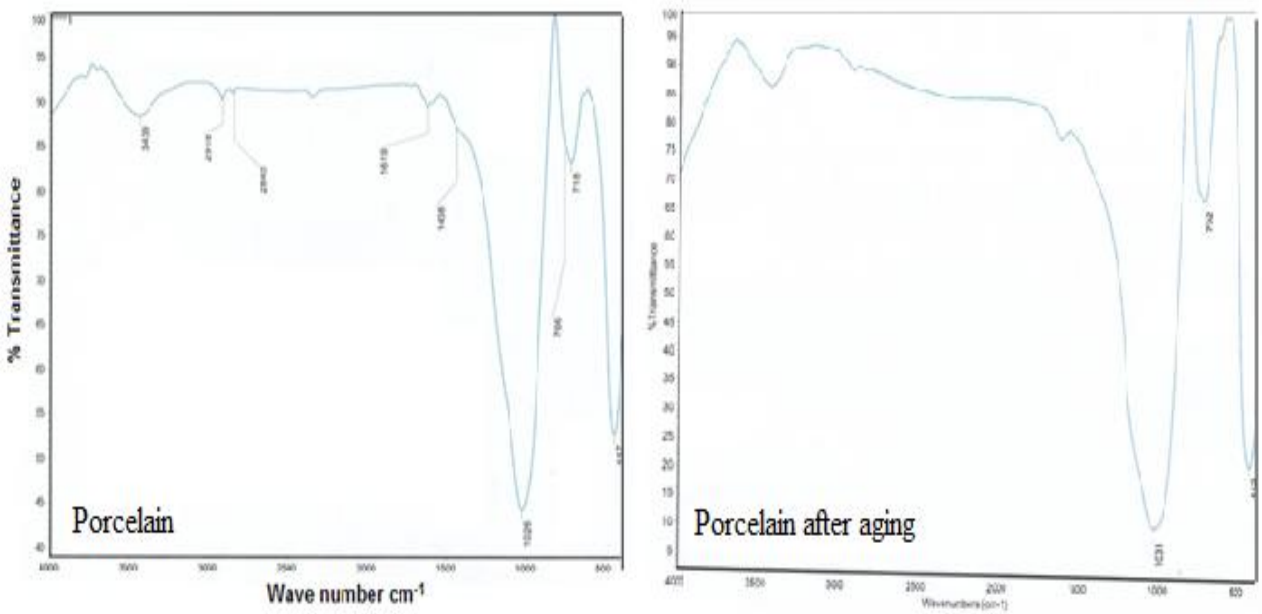


Figure 6: e and f IR spectra of porcelain before and after aging in saliva

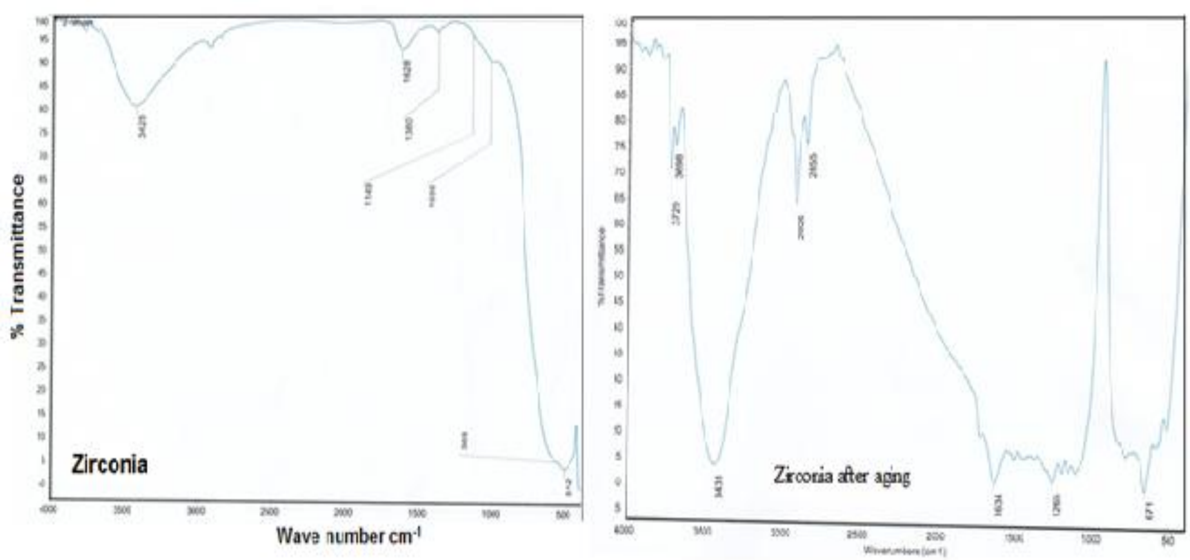


Figure 6: g and h) IR spectra of zirconia before and after aging in saliva

Vickers hardness

The hardness is the property of material which gives it the ability to resist being permanently deformed when a load is applied. The microhardness value was conducted using a digital Vickers microhardness tester, applying a load of 10 g for 5 s, for Filtek and glass ionomer dental materials which is listed in Table 1. Also calculated maximum shear stress (τ_m) values [23] are listed in Table 1. Vickers hardness of Filtek and glass ionomer dental materials increased after aging for one month.

That is because aging in saliva released relief stress\ or fine particles which disturbed in composite matrix increased Vickers hardness and strength.

Vickers hardness of porcelain and zirconia was conducted at applying load of 100 g for 5 s and it's shown in Table 2. The maximum shear stress (τ_m) value of porcelain and zirconia was calculated and then listed in Table 2. The results show that, Vickers hardness of porcelain and zirconia decreased after aging in saliva for one month. That is because porcelain structure changed small sandwich shape with fine particles to large sandwich shape without\ or less fine particle. Also zirconia structure changed from condensed (bonded) ZO_2 particles to large lamellar structure.

Table 1: Vickers hadness and maximum shear stress of Filtek and glass ionomer before and after aging in saliva

Samples	H_v Kg/mm^2	(τ_m) kg/mm^2
Filtek (base)	27.1 ± 1.57	8.943
Filtek in saliva	36.8 ± 2	12.14

Samples	H_v kg/mm^2	(τ_m) kg/mm^2
GI base	21.95 ± 2	7.244
GI in saliva	33.2 ± 2	10.956

Table 2: Vickers hadness and maximum shear stress of porcelain and zirconia before and after aging in saliva

Sample	**H_v kg/mm^2**	**(τ_m) kg/mm^2**
Porcelain	579 ± 40	191.07
Porcelain in saliva	536 ± 35	176.88

Sample	**H_v kg/mm^2**	**(τ_m) kg/mm^2**
Zirconia based	970 ± 60	320.1
Zirconia in saliva	938 ± 65	309.54

Roughness

The effects of surface topography are different than the overall three-dimensional design or geometry of the implant, which is related to the interaction of the host tissues with the implant on a macroscopic scale. Every surface has some form of texture that takes the form of a series of peaks and valleys. These peaks and valleys vary in height and spacing and have properties that are a result of the way the surface was produced. The roughness profiles of Filtek and glass ionomer before and after aging in saliva for one month are shown in Figures 7a and 7b. Also the average surface roughness parameter Ra along the total sliding distance and other roughness parameters, (R_z, R_q, R_t and R_p), of Filtek and porcelain materials are listed in Tables 3a and 3b. Ra of Filtek and glass ionomer decreased after aging in saliva for one month. That is because chemical and biological interactions of saliva due smoothing surfaces\ or decreased height valley which caused by fine particles of these materials.

The roughness profile of porcelain before and after aging in saliva for one month is shown in Figure 7c and the average surface roughness parameters are listed in Table 3c. Atomic force microscope, AFM, was used to identify roughness parameters of zirconia. Figure 7d shows the surface topography of zirconia materials in two and three dimensions. The roughness parameters of zirconia are listed in Table 3d. Ra of porcelain and Sa of zirconia increased after aging in saliva for one month. That is meant that, chemical and biological interactions of saliva caused cracks and pits on the surface of these smooth materials.

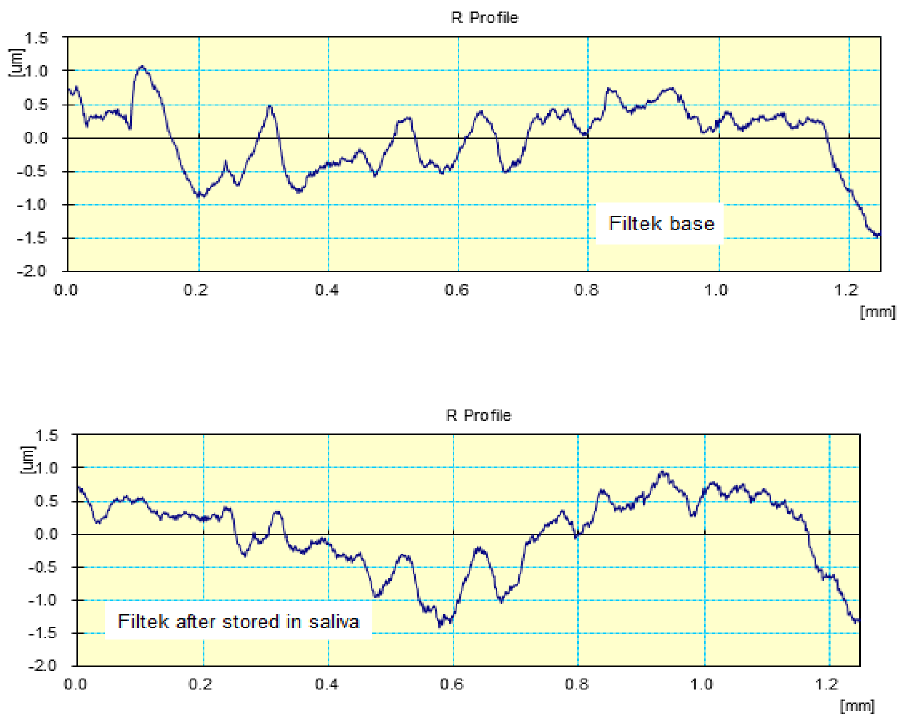
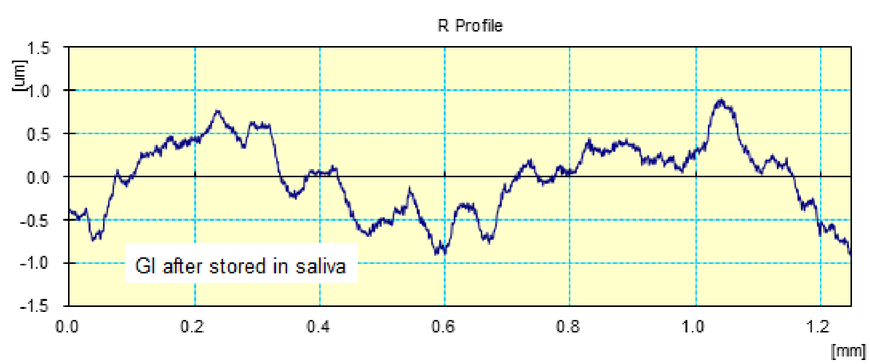


Figure 7a: Roughness profiles of Filtek before and after stored in saliva



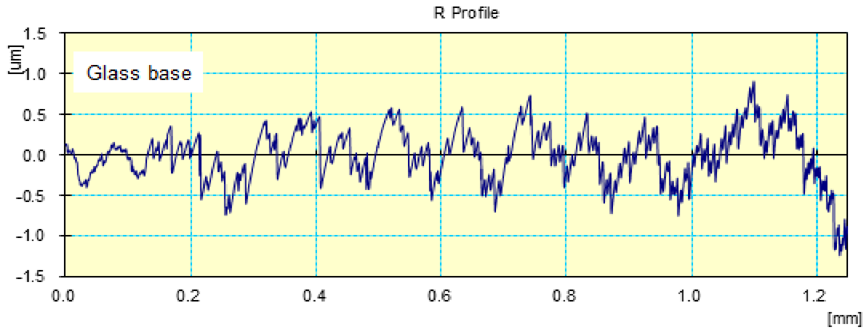


Figure 7b: Roughness profiles of glass ionomer before and after stored in saliva

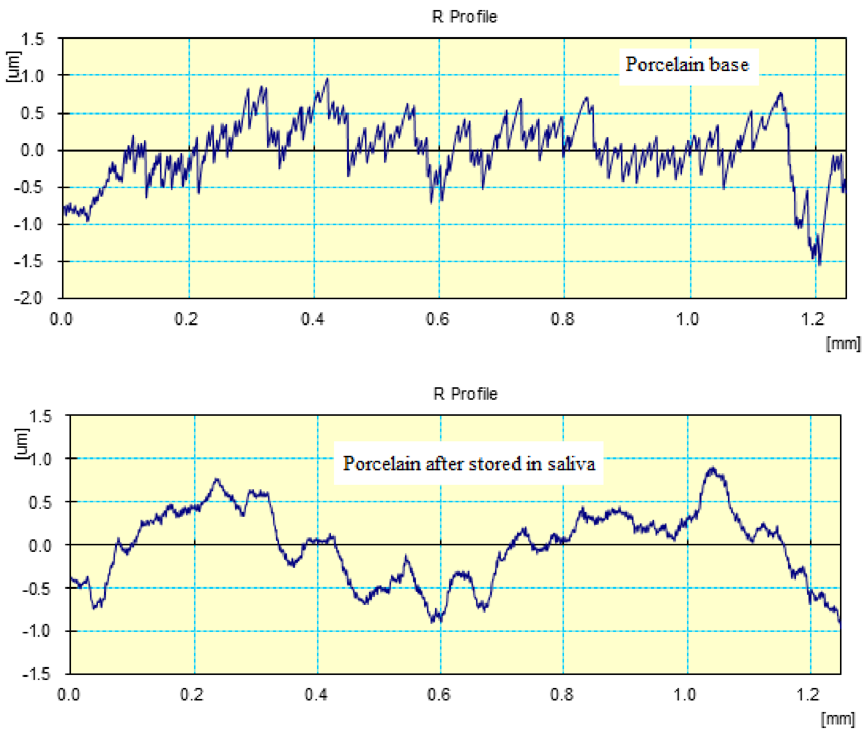
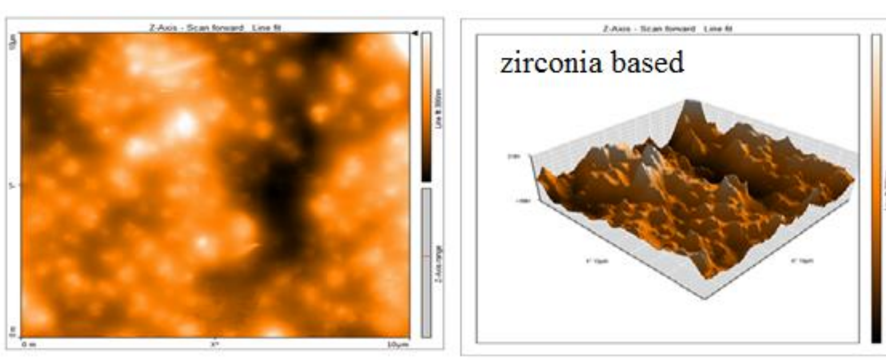


Figure 7c: Roughness profiles of porcelain before and after stored in saliva



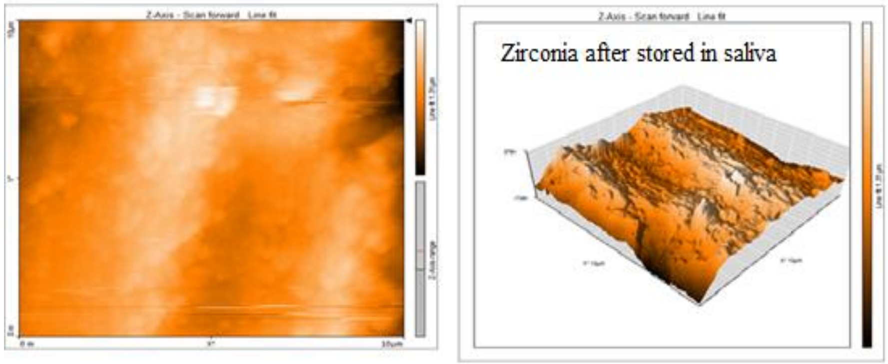


Figure 7d: AFM photographs of zirconia before and after stored in saliva

Table 3a: Roughness parameters of Filtek before and after stored in saliva

Roughness parameter	Ra um	Rz um	Rq um	Rt um	Rp um
Filtek base	0.48	1.34	0.55	2.38	0.60
Filtek in Saliva	0.42	1.38	0.49	2.57	0.63

Table 3b: Roughness parameters of GI before and after stored in saliva

Roughness parameter	Ra um	Rz um	Rq um	Rt um	Rp um
GI base	0.36	1.27	0.42	1.87	0.60
GI in Saliva	0.25	1.41	0.31	2.15	0.61

Table 3c: Roughness parameters of porcelain before and after stored in saliva

Roughness parameter	Ra um	Rz um	Rq um	Rt um	Rp um
Porcelain base	0.33	1.50	0.41	2.52	0.70
Porcelain in Saliva	0.36	1.27	0.42	1.87	0.60

Table 3d: Roughness parameters of zirconia before and after stored in saliva

Roughness parameter	Sa nm	Sq nm	Sy nm	Sp nm
Zirconia	53.702	68.645	607.64	437.08
Zirconia in saliva	148.6	185.25	1381.5	576.38

Conclusion

From our results, it is concluded that there is a significant effect of saliva on microstructural of Filtek, glass ionomer and porcelain (glassy matrix and formed crystal) and their molecular structure (intensity, broadness and position of IR band) which effects on its surface properties (hardness and surface roughness). After aging in saliva surface roughness of porcelain and zirconia increased but surface roughness of Filtek and glass ionomer decreased. Also Vickers hardness of porcelain and zirconia decreased but Vickers hardness of Filtek and glass ionomer increased after aging in saliva.

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