ISSN : 0974 - 7486 Volume 14 Issue 10

Materials Science

An Indian Journal

Full Paper

MSAIJ, 14(10), 2016 [377-387]

Microstructure, surface properties and antimicrobial effectiveness of filtek and ceramic materials

Abu Bakr El-Bediwi[1]*, Reham Hamed Ebrahim[1], Afaf Sarhan Abdelrazek[2], Mutlu Özcan[3]

[1]Metal Physics Lab., Physics Department, Faculty of Science, Mansoura University, (EGYPT)

[2]Biological Advanced Materials, Physics Department, Faculty of Science, Mansoura University, (EGYPT)

[3]Clinic for Fixed and Removable Prosthodontics and Dental Materials Science, University of Zürich, (SWITZERLAND)

E-mail : baker_elbediwi@yahoo.com

ABSTRACT

Microstructure, molecular structure, hardness, surface roughness and antimicrobial efficiency of filtek, porcelain and zirconiarestorative dental materials have been evaluated. Specimens used in this study were dental porcelain (VitaVMK, Master, VITAZ ahnfabrik, Germany), zirconia (Ceramill zi, made in Austria) and Feltik 250 (3M ESPE, USA). The results show that, porcelain consists of a mixture of glassy matrix and crystalline phases imbedded it. Zirconia consists of ZrO_2 with different plans, intensity, broadness and orientation. Filtek consists of glassy matrix with other accumulated particles (very small peaks) imbedded it. Zirconia is very low surface roughness with high Vickers hardness compared to porcelain dental material. Alsobacterial growth was detected around filtek, porcelain and zirconia.

KEYWORDS

Zirconia;
Porcelain;
Filtek;
Hardness;
Roughness;
Antimicrobial efficiency.

INTRODUCTION

Dental restorative materials are specially fabricated materials, designed for use as dental restorations (fillings), which are used to restore tooth structure loss, usually resulting from but not limited to dental caries (dental cavities). Zirconia based ceramics have become a very popular type of all-ceramic restorations. It can be a near ideal choicefor restoring crowns, fixed partial dentures and implants in esthetic areas. In 1993 the first study wasrecorded on used zirconia for implants, when a group of researchersinserted experimental Y-TPZ implants in the mandible of dogs[1, 2]. Also adding stabilizing oxides is important because it allows themaintenance ofthe tetragonal format room temperature[3]. Zirconia is used to make ceramic knives because of its hardness; Zirconia based cutlery stays sharp longer than a stainlesssteel equivalent[4]. Dental ceramics are able to mimic natural teeth due to their excellent physical properties such as esthetics,biocompatibility, low thermal conductibility and wear resistance[5- 7]. Dental ceramics have been extensively used in several rehabilitation procedures, including inlays, onlays, crowns, and porcelain veneers[8]. One of the purposes of dentistry is to prevent the occurrence of dental caries and another one is to avoid the recurrence of marginal caries, because these are the main factors that influence the duration of dental restorations. Dental caries and candidiasis are common infections found in the oral cavity and create health problems for people. Dental caries is the destruction of hard tissue of teeth due to acids produced by bacteria. Streptococcus mutans and Lactobacillus spp. have been shown to have cariogenic potential owing to their acidogenic and aciduric abilities[9]. Streptococcus mutansappears to be important in the initiation of dental caries since its activities lead to colonization of the tooth surface, dental plaque formation and demineralization. Lactobacillihave been suspected to be secondary invaders that contribute to the progression of lesions. In the weakened, compromised host, oral candidal infection may spread to the gastrointestinal tract, trachea, lungs, liver and central nervous system, capable of causing septicemia, meningitis and endocarditis[10-12]. The greatestcommonCandida spp. isolated from oral cavity is Candida albicans with capability toadhere and colonize oral surfaces in large numbers[13]. In our knowledge, no information exists regarding the antimicrobial activities of filtek, porcelain and zirconia materials on the oral microorganism. The aim of this study was to evaluate the antimicrobial activity of filtek, porcelain and zirconiadental materials against oral microorganisms. Structure, hardness and roughness of these materials were also investigated.

MATERIALS AND METHODS

The dental restorative materials used in this study were dental porcelain (VitaVMK, Master, VITAZ ahnfabrik, Germany) were prepared in a standardized manner andaccording to the manufacturer's directions in rectangular stainless steel split mold (20mm-5mm. 3 mm), zirconia (Ceramill zi, made in Austria) and Feltik 250 (3M ESPE, USA). Unpolymerized Filtek was applied in Teflon mold with dimensions 20 mm in length $\times$ 2 mm in width $\times$ 2mm 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 surfacepolymerization 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. 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 60° 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, Spectral Analysis Unit, Chemistry Department, Faculty of Science, Mansoura University. 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 used specimens were measured by using surface roughness measurements device (surface test S.J 201.P). Data are measured numerically and get it from computer program, the program is calculating roughness parameters then plot the result to give roughness profile and different roughness parameters, after that data saved to be analyzed, by calculating the average surface roughness parameter Ra along the total sliding distance.

The dental restorative materials used in this study were zirconia, porcelain, and filtik 250. All these materials were made into suspension in sterile distilled water at different concentration 0.2g D 5ml, 0.3g D 5ml, 0.5g D 5ml and 1g D 5ml. The bacteria used in this study were staphylococcus aureus, streptococcus mutans, E-Choli and candida albicans (fungus). They have been provided by microbiology department, Faculty of medicine, Mansoura University, Egypt. Agar diffusion test was the method of antimicrobial activity for these four materials. This method is widely used among other researches (Tobias1988). Initially staphylococcus aureus and E-Choli were inoculated in maconky, streptococcus mutans was inoculated in blood agar and fungus (candida albicans) was inoculated in PDA (potato dextrose agar). All were incubated at 37 °C for 24 hour. From these cultures, bacterial suspension were prepared in sterile peptone solution, until measuring turbidity by nanophotometer (IMPLEN) equivalent to 10^8 CFU D ml for bacteria and 10^7 CFU D ml for fungi. Briefly 15 ml of nutrient agar was poured into 90 mm petri dish and plates were then inoculated with 10^8 CFUD ml of bacteria or 10^7 CFUD ml of yeast. By punching the agar container with a sterile cork borer and scooping out the punched part, agar cups of 4 mm diameter were made. The agar had already been divided into 4 sections. Each of the material suspension at different concentrations was placed in each cup. All the plates were incubated at 37 °C for 48 hours in incubator mode (Jsgi-100T). All the plates were prepared under laminar flow (unilab) and all instruments, glass mixing slab were sterilized. The antimicrobial activity was evaluated based on zones of growth inhibition (mm).

RESULTS AND DISCUSSIONS

Structure

X-ray analysis

The x-ray diffraction patterns of filtek, porcelain and zirconia dental materials are shown in Figure 1(a, b and c). From x-ray diffraction analysis, Filtek consists of main matrix peak, glassy matrix, and other formed crystal peaks, (accumulated filler particles).

X-ray diffraction analysis of porcelain, TABLE (1a), showed that, it consisted of a mixture of glassy matrix and other crystalline phases. Glass matrix, (20º-35º), of porcelain contained irregularly shaped of crys-

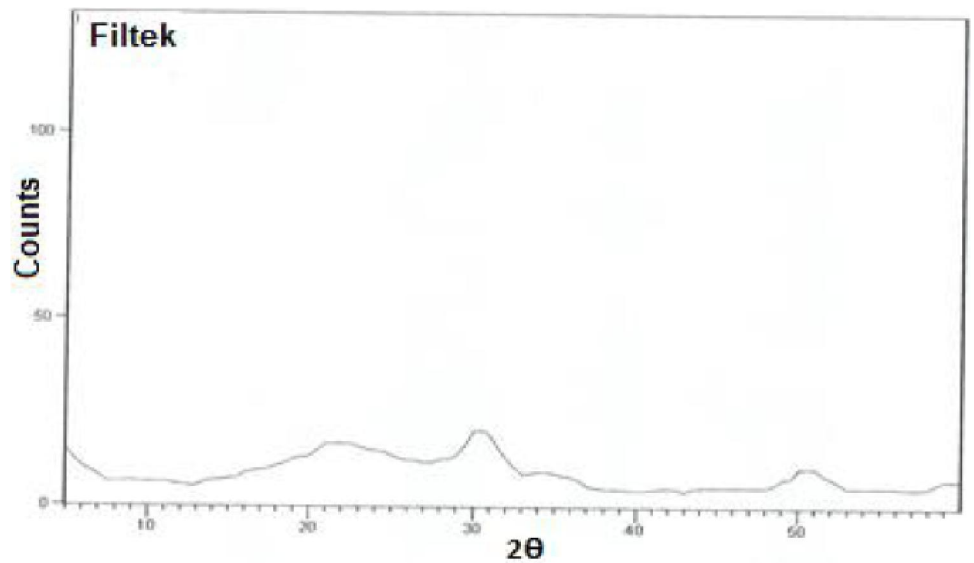

Figure 1a : x-ray diffraction patterns of filtek

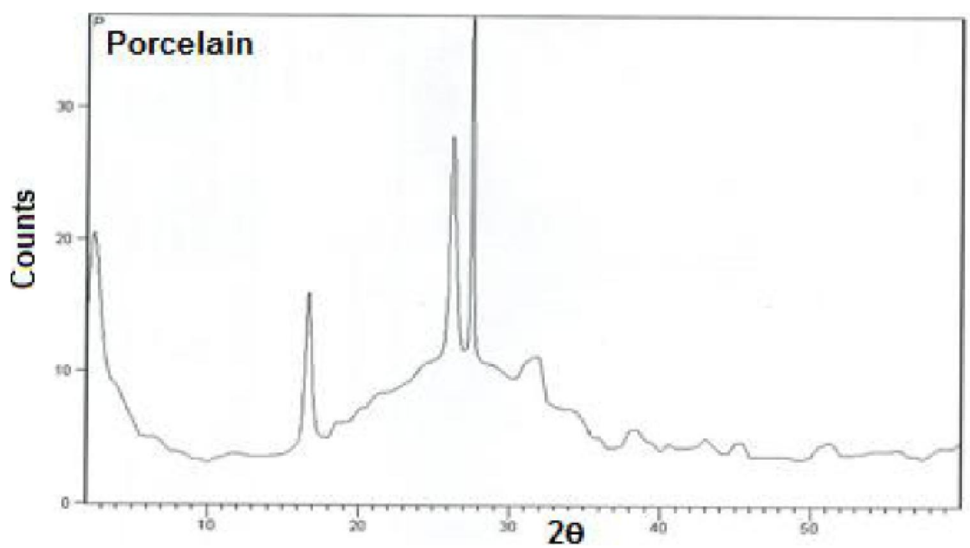

Figure 1b : x-ray diffraction patterns of porcelain

Full Paper

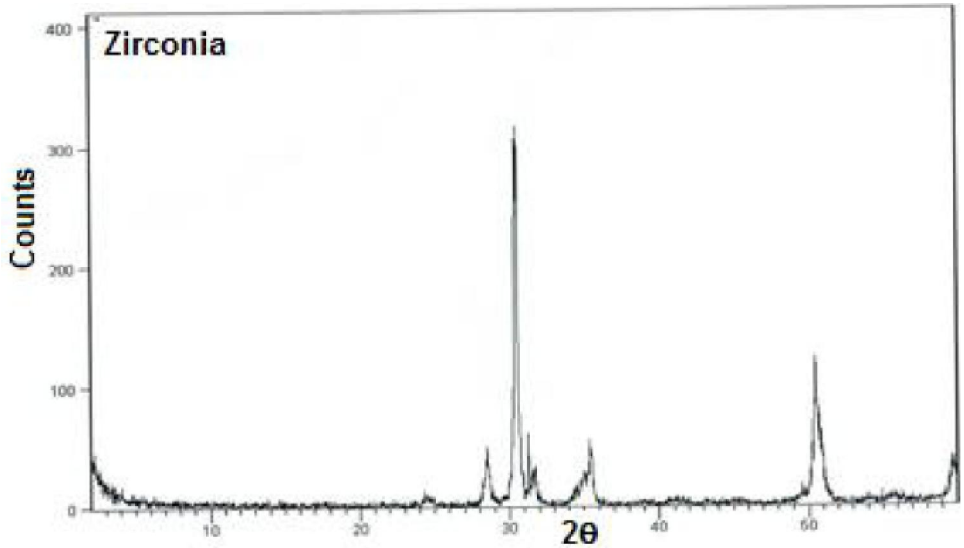

Figure 1c : x-ray diffraction patterns of zirconia

TABLE 1a : x-ray diffraction analysis of porcelain

2θ	d Å	hkl	phase
16.5	5.4476	011	Tetragonal ($Na_2B_4O_7$)
25.95	3.4374		Tetragonal ($KAl(SiO_3)_2$)
27.25	3.29		Tetragonal ($KAl(SiO_3)_2$)
31.5	2.85368		Tetragonal ($KAl(SiO_3)_2$)
38.2	2.3806	102	Hexagonal (SiO_2)
42.25	2.1616	200	Hexagonal (SiO_2)
51	1.79	112	Hexagonal (SiO_2)

TABLE 1b : x-ray diffraction analysis of zirconia

2θ	d Å	FWHM	Int. %
24.5379	3.62792	0.6298	1.66
28.4988	3.13207	0.2755	13.08
30.4852	2.93236	0.2362	100
31.2898	2.85877	0.0590	19.82
31.6041	2.83105	0.4723	7.55
35.4618	2.53143	0.2362	17.43
41.2147	2.19040	0.9446	1.02
50.4941	1.80750	0.1968	33.53

talline particles, tetragonal $KAl(SiO_3)_2$. Also other crystalline phases are tetragonal $Na_2B_4O_7$ and Hexagonal SiO_2.

The analysis of x-ray, TABLE (1b),of zirconia showed that, it consisted of ZrO_2 with different plans (hkl), intensity (crystallinty), broadness (crystal size) and orientation (2θ).

Scanning electron microscope analysis

Scanning electron microscope analysis, SEM, of filtek, porcelain and zirconia dental materials are shown in Figure 2 (a, b and c). SEM of filtek shows two separate structure, glassy matrix and accumulated particles, and that is agreed with x-ray analysis results. SEM of porcelain shows two separate structures, glassy matrix and other different phases, and that is agreed with x-ray analysis results. SEM of zirconia shows the shape structures with different orientations, and that is agreed with x-ray analysis results

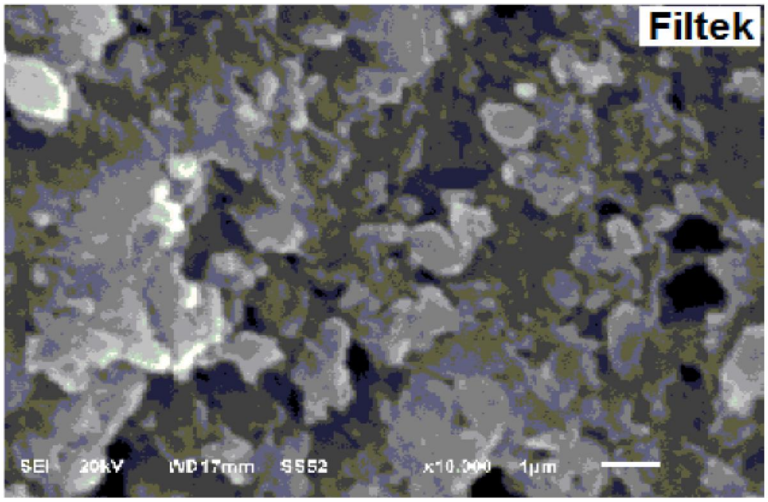

Figure 2a : SEM of filtek dental material

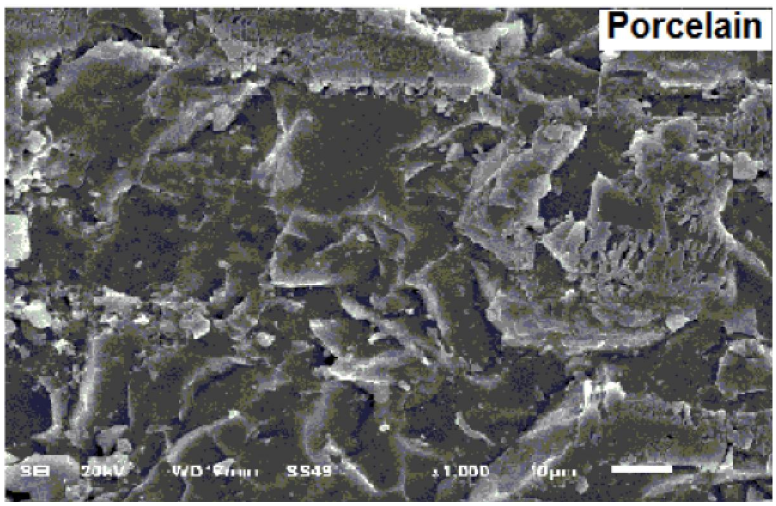

Figure 2b : SEM of porcelain dental material

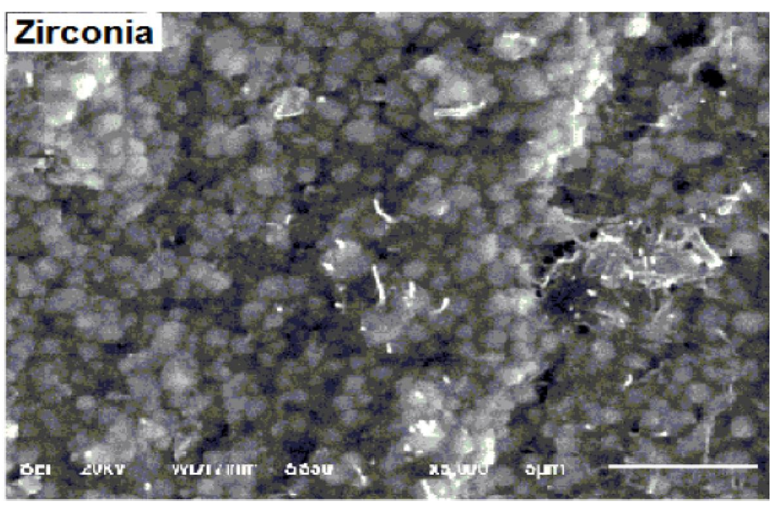

Figure 2c : SEM of zirconia dental material

FTIR spectrum

Figure (3a) shows IR spectra of filtek dental material exhibited, strong IR band at 470 cm^{-1} corresponding to O-Si-O, IR band at 602 cm^{-1} corresponding to M–N vibrations, strong band at ~ 1100 corresponding to C-O, The region <d1633–1515 cm^{d1} is due to H–

O–H bending vibrations, representing the presence of a coordinated water molecules and weak- sharp IR band at 1726 cm^{-1} corresponding to COOCH3, strong/or weak- very broad IR band at ~ 3420 cm^{-1} corresponding to C-H. Figure (3b) shows IR spectra of filtek dental material exhibited, strong IR band at 447 cm^{-1} corresponding to O-Si-O, strong band at ~ 1026 corresponding to C-O, IR band at 1619 cm^{d1} is due to H–O–H bending vibrations, representing the presence of a coordinated water molecules and weak- very broad IR band at ~ 3439 cm^{-1} corresponding to C-H. Figure (3c) shows IR spectra of zirconia dental material exhibited, IR band at 1625 cm^{d1} is due to H–O–H bending vibrations, representing the presence of a coordinated water molecules and weak- very broad IR band at ~ 3425 cm^{-1} corresponding to C-H. The results of FTIR spectrum show that, the characteristics of IR bands for used dental materials are changed (strong, broad and position) which dependent on material molecular structure.

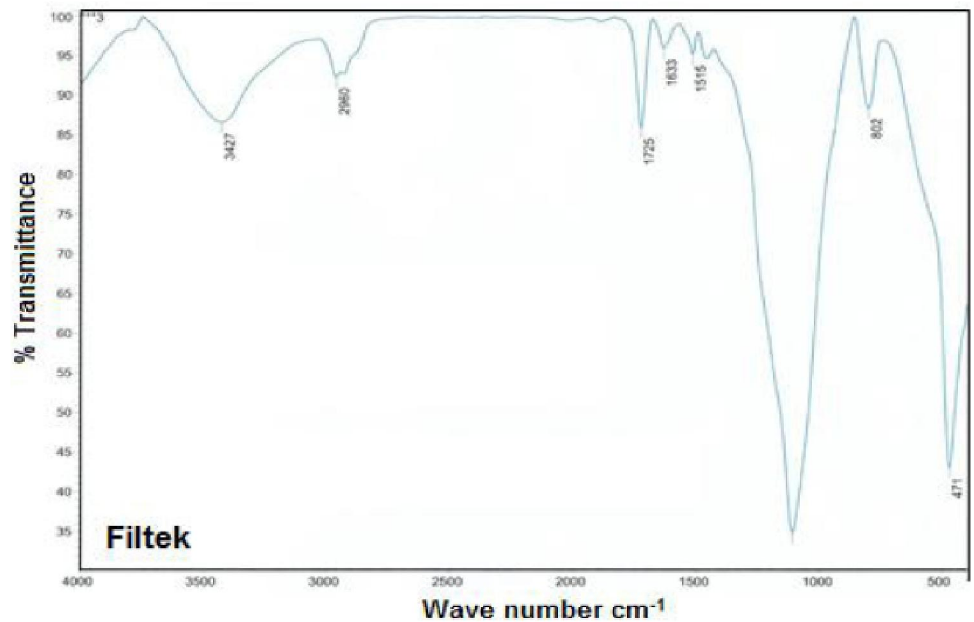

Figure 3a : IR spectra of filtek dental material

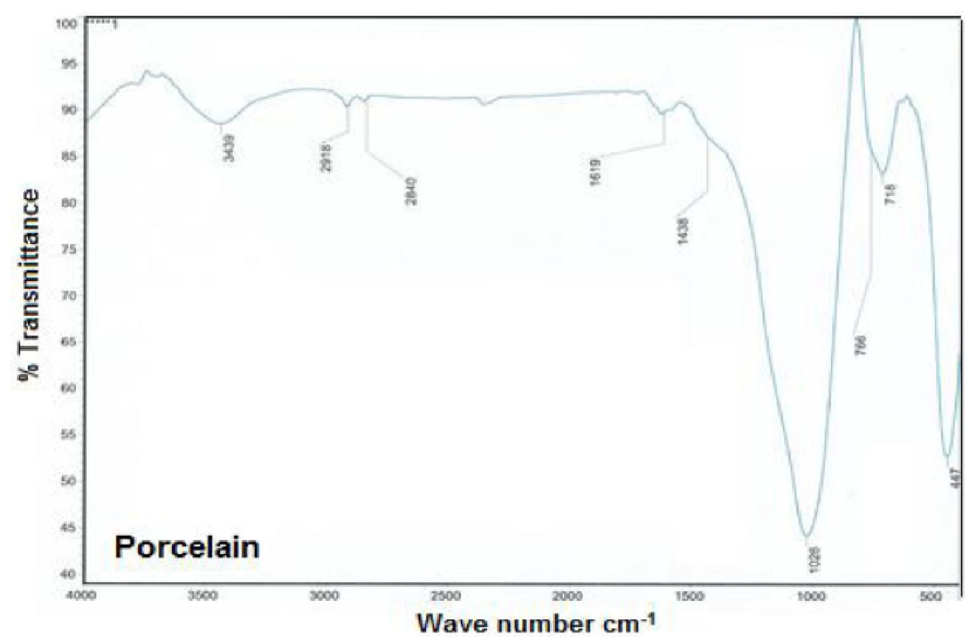

Figure 3b : IR spectra of porcelain dental material

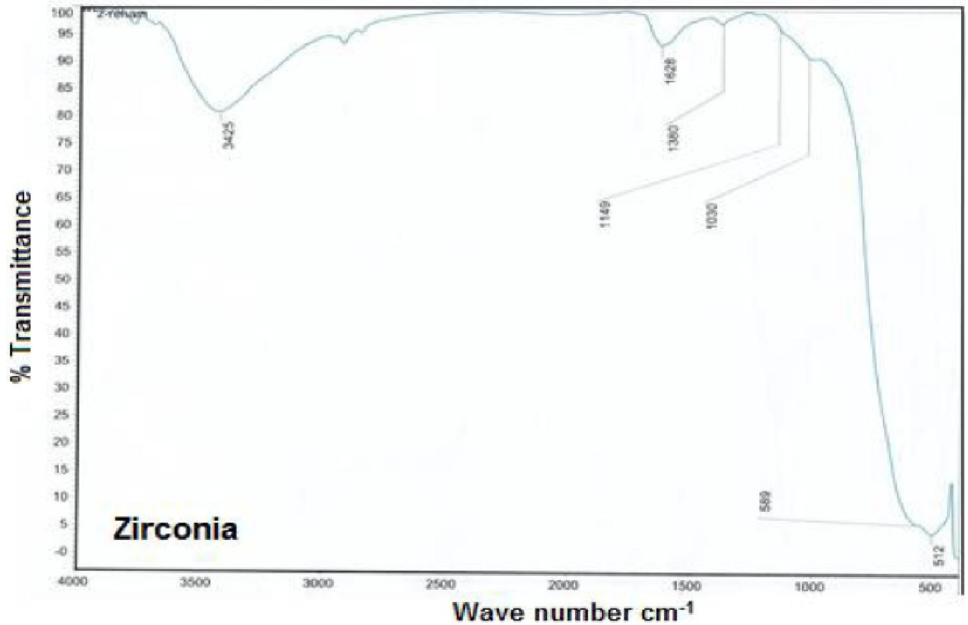

Figure 3c : IR spectra of zirconia dental material

TABLE 2 : Vickers hadness and maximum shear stress of used dental materials

Sample	H_v kg/mm^2	μ kg/mm^2
Filtek	25.1±1.47	8.28
Porcelain	500±50	165
Zirconia	950±40	313.5

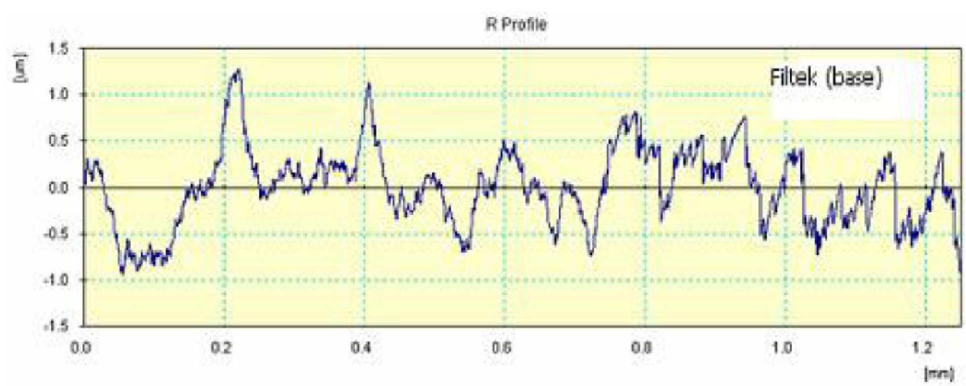

Figure 5a : Roughness profile of filtek material

Surface properties

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 dental material which is listed in TABLE (2). But for porcelain and zirconia microhardness value was conducted using a digital Vickers microhardness tester, applying a load of 100 g for 5 s and it's shown in TABLE (2). The maximum shear stress (τ_m) value of filtek, porcelain and zirconia dental materials was calculated using the equation[16]:-

$$\tau_m = \frac{1}{2}H_v\left\{\frac{1}{2}(1-2\nu)+\frac{2}{9}(1+\nu)[2(1+\nu)]^{\frac{1}{2}}\right\}$$

Where $\nu$is Poisson's ratio? The results show that, Vickers hardness of filtek, porcelain and zirconia dental materials dependent on its structure, micro and molecular structure.

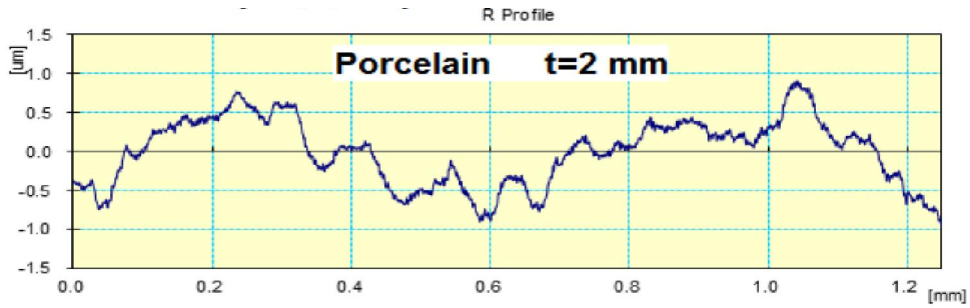

Figure 5b : Roughness profile of porcelain material

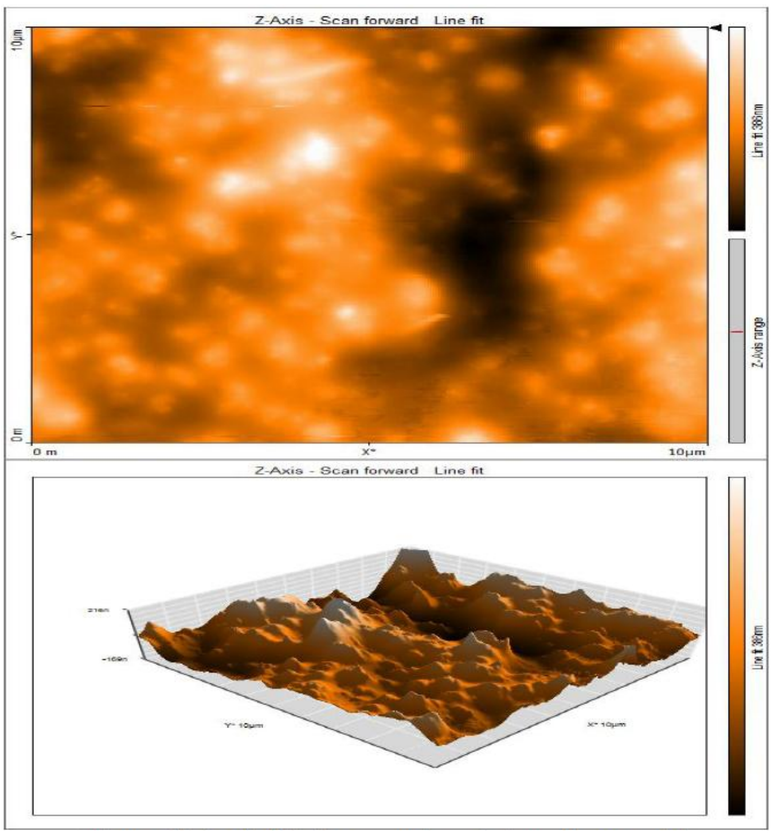

Figure 5c : AFM photographs of zirconia material

TABLE 3a : Roughness parameters of filtek material

Roughness parameter	Ra um	Rz um	Rq um	Rt um	Rp um
Filtek	0.341	1.527	0.403	2.233	0.832

TABLE 3b : Roughness parameters of porcelain material

Roughness parameter	Ra um	Rz um	Rq um	Rt um	Rp um
Porcelain	0.36	1.27	0.42	1.87	0.60

TABLE 3c : Roughness parameters of zirconia material

Roughness parameter	Sa nm	Sq nm	Sy nm	Sp nm
Zirconia	53.702	68.645	607.64	437.08

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

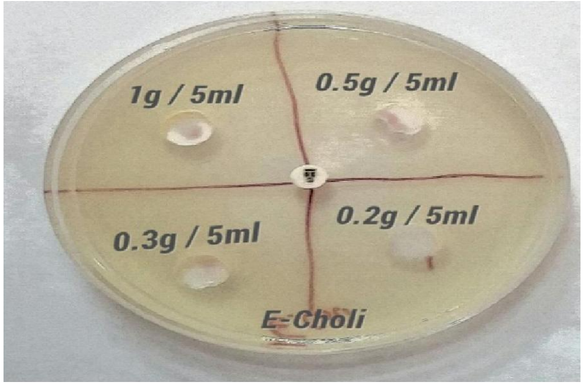

Figure 4a : E-Choli growth around filtek material

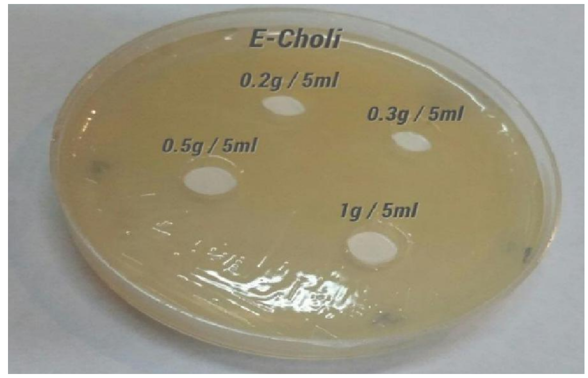

Figure 4b : E-Choli growth around porcelain material

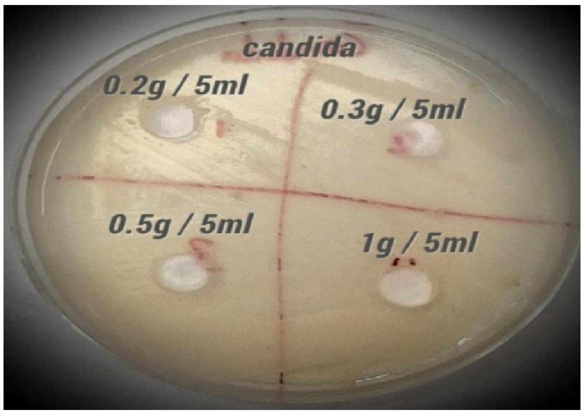

Figure 4a : Candida growth around filtek material

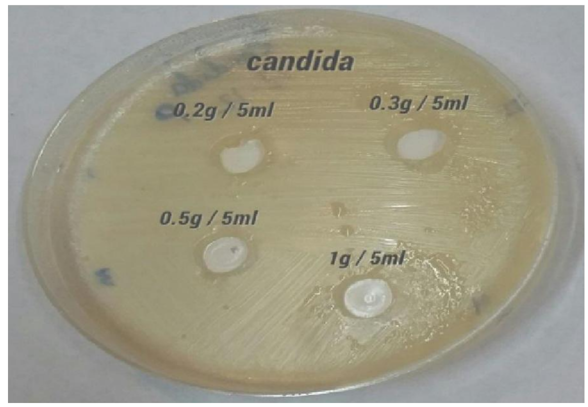

Figure 4b : Candida growth around porcelain material

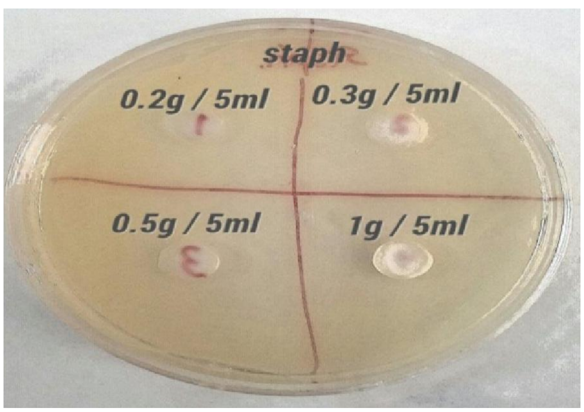

Figure 4a : Staph aureus growth around filtek material

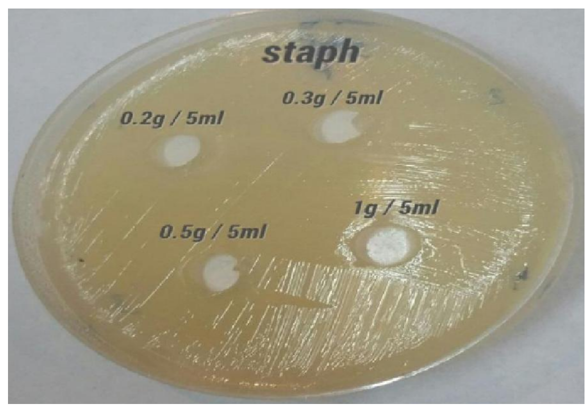

Figure 4b : Staph aureus growth around porcelain material

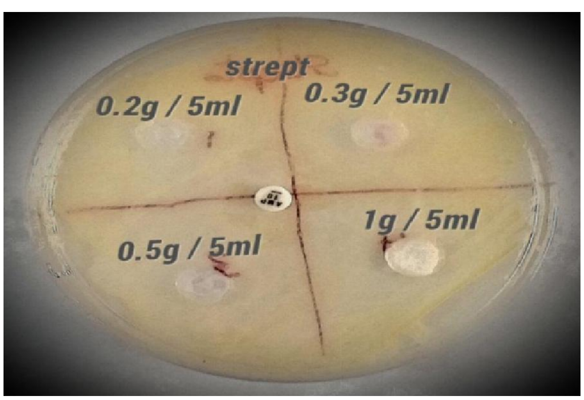

Figure 4a : Strept growth around filtek material

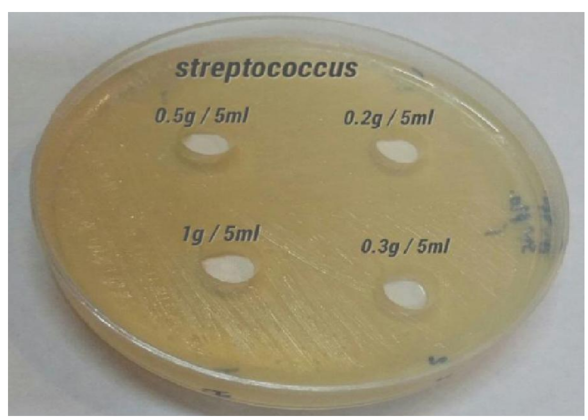

Figure 4b : Strept growth around porcelain material tissues with the implant on a macroscopic scale. This important consideration in overall biologic response to implants was discussed and from this discussion results, the concept of surface topography refers to the surface texture on a microlevel. It is on this microscopic level that the intimate cell and tissue interactions leading to osseointegration. The effects of surface topography on in vitro and in vivo cell and tissue responses have been a field of intense studies in recent years. The overall goal of these studies is to identify surface topographies which mimic the natural substrata in order to permit tissue integration and improve clinical performance of the implant. Increased surface roughness produced by such techniques as sand or grit blasting or by rough polishing, provided the rugosity necessary for optimum cell behavior. 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 profile of Filtek and porcelain materials is shown in Figure 5 (a and b). 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 TABLE 3 (a and b).

Atomic force microscope, AFM, was used to identify roughness parameters of zirconia. Figure (5c) shows the surface topography of zirconia materials in two and three dimensions. The roughness parameters of zirconia material are listed in TABLE (3c).

Antimicrobial activity

Antimicrobial activity of filtek, porcelain and zirconia dental materials for E-Choli,staphylococcus aureus, streptococcus mutans, Strept and candida albicans (fungi) are shown in Figure 4 (a, band c). The results in Figure (4a) show that, filtek has no activity, no resis-

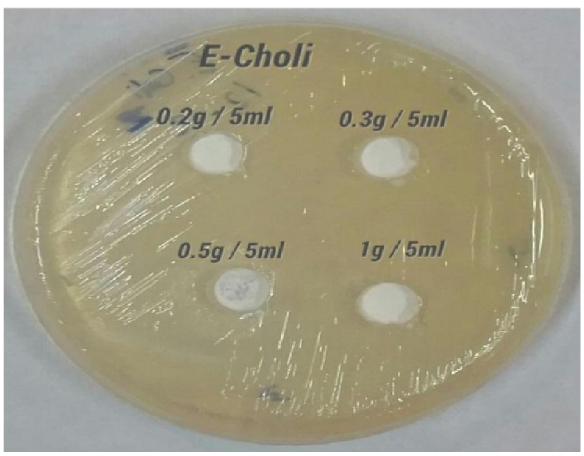

Figure 4c : E-Choli growth around zirconia material

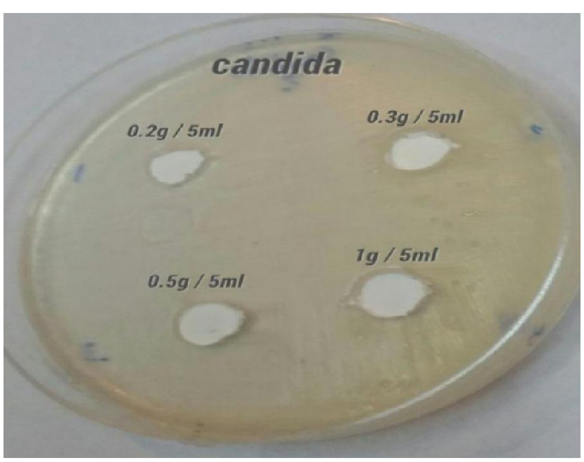

Figure 4c : Candida growth around zirconia material

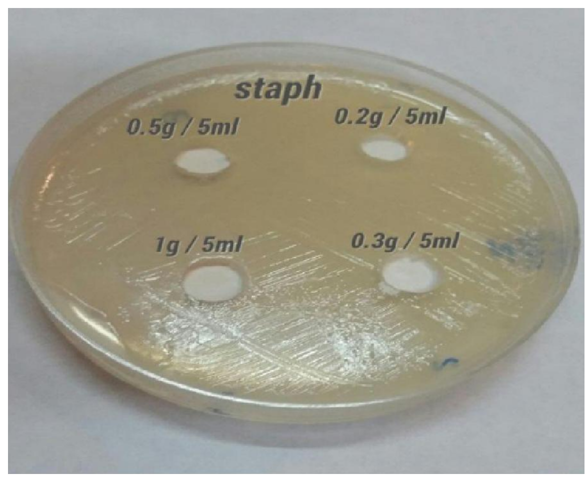

Figure 4c : Staph aureus growth around zirconia material

Figure 4c : Strept growth around zirconia material

tance, for E-Choli,staphylococcus aureus, streptococcus mutans and candida albicans. That is mean these bacteria stick on the surface which effects on all surface properties such as roughness and hardness and that is agreed with pervious work[14]. Also Figure (4b) shows porcelain no activity, no resistance, for E-Choli,staphylococcus aureus, streptococcus mutans and candida albicans. That is mean these bacteria stick on the surface which effects on all surface properties such as roughness and hardness and that is agreed with pervious work[15]. Figure (4c) also shows zirconia has no activity, no resistance, for E-Choli,staphylococcus aureus, streptococcus mutans and candida albicans. That is mean these bacteria stick on the surface which effects on all surface properties such as roughness and hardness and that is agreed with pervious work[15].

CONCLUSION

From our results it is concluded that:-

1. Filtek has amorphous structure with impeded some clusters or accumulated filler particles inside main matrix.
2. Porcelain consisted of glass matrix contained ~ 30% crystal with other formed crystal.
3. Zirconia has a monoclinic structure which consisted of ZrO_2 crystal.
4. Zirconia has low roughness and high hardness compared to porcelain.
5. Filtek, porcelain and zirconia dental materials have antimicrobial effect for staphylococcus aureus, streptococcus mutans and candida albicans.
6. From our research zirconia has beast properties for dental applications such as crown and removable denture but it has high coast.

REFERENCES

[1] A.N.Cranin, P.A.Schnitman, S.M.Rabkin, E.J.Onesto; J.Biomed.Mater.Res., **9**, 257-262 **(1975)**.
[2] C.Miani, C.Piconi, D.Piselli, M.Ponti; Rev.Ital.Osseoint, **3**, 23-34 **(1993)**.
[3] R.H.J.Hannink, P.M.Kelly, B.C.Muddle; J.Am.Ceram.Soc., **83**, 461-487 **(2000)**.
[4] Papaspyridakos, Panos Kunal La; J.Prosth.Dent., **100(3)**, 165-172 **(2008)**.
[5] Dental porcelain, In: W.J.O.Brien, (Ed); Dental materials and their Selection, 3rd Edition, Chicago: Quintessence, 210 **(2002)**.
[6] El A.O.E.Karaksi, G.I.Shehab, M.E.Eskander; Egyp.Dent.J., **39**, 485-90 **(1993)**.
[7] G.B.Camacho, D.Vinha, H.Panzeri, T.Nonaka, M.Gonçalves; Braz.Dent.J, **17**, 191-4 **(2006)**.
[8] K.J.Anusavice; J.Am.Dent.Assoc., **124**, 72-84 **(1993)**.
[9] T.M.Parisotto, W.F.King, C.Duque, Mattos R.O.Graner, Steiner C.Oliveira, Nobre M.Dos-Santos et al; Caries Res., **45**, 377-385 **(2011)**.
[10] M.Santos, G.Thiene, H.H.Sievers, C.Basso; Eur Heart J., **32**, 2265 **(2011)**.
[11] I.M.Ancalle, J.A.Rivera, I.García, L.García, M.Valcárcel; Bol AsocMed P R, **102**, 45-48 **(2010)**.
[12] P.Badiee, A.Alborzi; Exp.Clin.Transplant, **9**, 355-362 **(2011)**.
[13] R.Rautemaa, G.Ramage; Crit.Rev.Microbiol, **37,** 328-336 **(2011)**.
[14] El A.B.Bediwi, El A.Fallel; Abu El-Naga OH, MSIJ, **(2011)**.
[15] El A.B.Bediwi, El T.Helaly, A.Sakrana, M.Kamal; Submitted paper, MSIJ, Oct., **(2015)**.
[16] S.Timoshenko, J.N.Goddier; Theory of elasticity, 2nd Edition, McGraw-Hill, New York, 277 **(1951)**.