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ASSESSMENT OF SURFACE ROUGHNESS OF A CAD – CAM RESIN NANO-CERAMIC; A BULKFILL RESIN COMPOSITE AND A UNIVERSAL NANOHYBRID ORMOCER ESTHETIC RESTORATIVE MATERIALS WITH DIFFERENT PH CHANGES

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ABSTRACT

There has always been a continuous search for an ideal esthetic restorative material that can restore and replace the natural tooth structure and maintain mechanical and esthetic properties.

Materials and methods : in this study three recent esthetic restorative materials [a CAD – CAM resin nano ceramic (Lava Ultimate); a nano hybrid bulk fill resin composite (Aura) and a Universal nano hybrid ORMOCER]; were investigated in terms of surface roughness in relation to different pH incubation solutions.

Results : The CAD – CAM resin nano ceramic Lava Ultimate and the bulk fill nanohybrid resin composite, approximately, showed close values of average surface roughness and both materials were statistically significantly superior to the Universal nano hybrid ORMOCER regarding the resistance to surface roughness in respect to all incubation solutions. Except for the CAD – CAM resin nano ceramic and regarding the other two materials; the acidic immersion solution produced significant changes in surface roughness.

KEY WORDS : Esthetic restorative materials, surface roughness, pH changes

INTRODUCTION

The rapid development of esthetic tooth colored dental restorative materials have pointed the way for further investigations regarding those materials mechanical and physical properties. The search of an ideal esthetic restorative material that can restore and replace the natural enamel and dentin structures and show adequate mechanical, esthetic, self- adhesive and caries preventive properties; has sparked various scientific and clinical investigations

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regarding restorative materials technologies over decades of time [1–5]. Also, dental restorative materials should provide adequate years of clinical service without prominent deterioration in mechanical and physical properties. The ultimate esthetics of tooth colored restorative materials are strongly influenced by the adequacy of surface finish and polish [6–8]. Appropriate finishing and polishing of dental restorative materials are critical clinical procedures that enhance the esthetics as well as the long-term clinical service of restorations [6,9 –12]. Moreover the surface texture of dental restorative materials has an outstanding influence on stress concentration, wear, discoloration and plaque accumulation[6,13-15]. Recently, numerous efforts have been made to analyze the suitability of various systems available for finishing and polishing. Also, it was reported that the effect of polishing systems on surface finish was material dependent[16]. The changes in pH related to the consumption of certain food and beverages was found to possess a direct correlation with the development of surface roughness in restorative materials[17–21]. Moreover, researches have been conducted to develop new monomers for resin matrices as well as to focus on the filler content, loading, and type and size of filler particles [22–27]. In this study, the surface roughness of different recent esthetic restorative materials (a CAD – CAM resin nano-ceramic; a bulk fill resin composite; and a universal nanohybrid Ormocer); in relation to different pH immersion solutions was investigated.

MATERIALS AND METHODS

Three esthetic restorative materials, a bulkfill resin composite, a universal nanohybrid Ormocer and a CAD – CAM resin nanoceramic were used in this study. Materials, composition and manufacturers are listed in Table (1)

TABLE (1) Composition and manufacturers of the tested materials (data provided by the manufacturers)

Resin Composite	Composition	Manufacturers
Nano-Hybrid Bulk fill (Aura)	Resin matrix: UDMA, Bis-EMA, Bis-GMA,TEGDMA Filler: amorphous SiO2, Bariumaluminosilicate glass,pre-polymerized filler (72.71%)	SDI, Melbourne, Australia Lot# 200126
Universal nano hybrid ORMOCER restorative material, Admira fusion X-tra	Resin matrix: Organically modified silicic acid Filler: Silicon oxide nano filler,glass ceramics filler(1 μm)	VOCO GmbH, Cuxhaven, Germany Lot# 2128436
CAD – CAM Resin nanoceramic, Lava Ultimate (LT, A2)	80% w/w ceramic (69% silicon dioxide and 31% zirconium dioxide) 20% polymer (Bis-GMA, Bis-EMA, UDMA, and TEGDMA) silica nanomers:20 nm zirconia nanomers:4–11 nm	3M ESPE, Maplewood, USA

Bis-GMA: bisphenol A diglycidyl ether methacrylate. TEGDMA: triethylene glycol dimethacrylate; UDMA: urethane dimethacrylate

Samples preparation

For bulk fill resin composite (Aura) and ORMOCER, disc-shaped samples (10.0mm diameter × 2.0mm thick) were prepared for each material (n=30/group). Each material was positioned in the appropriate Teflon mold and hand-pressed between two transparent matrix strips and glass plates to obtain a flat surface. The materials were light cured for 40 seconds by means of an LED curing unit (Demi Plus, Kerr, Orange Co., USA) with a spectral range of 450-470 nm wave length and 1200mW/cm2 intensity; according to the manufacturers instructions. Each sample was; then; extruded from the mold by applying positive pressure using a pestle of 9.5 mm diameter to allow equal distribution of pressure. The guide of the light curing unit was kept perpendicular to surface and the distance between the unit and the sample was standardized using a 1 mm thick glass slide. All the samples were incubated in distilled water for 24h at 37˚C to ensure complete polymerization. Resin nanoceramic samples (n=30) were prepared from CAD/CAM blocks using a water-cooled low-speed diamond saw (IsoMet®; Buehler, Lake Bluff, USA). The surfaces of all samples were polished under water cooling conditions with P400, P600, P800, P1000, and P1200 silicon carbide papers at 300 rpm. The thickness of all samples was confirmed using a digital micrometer (Electronic Caliper; Tire Corporation Ltd., Canada) to be 2.0 ±0.01 mm. All samples were then ultrasonically cleaned in distilled water for 10 min.

Baseline roughness evaluation

Roughness measurement of each sample (T_0) was performed using non-contact computational optical profilometry technique with a combination of a digital camera, and an image processing software technique [28]. The images were taken with the following image acquisition system;

1. Digital camera (*U500x Digital Microscope, Guangdong, China*) with 3.6 Mega Pixels of resolution, placed vertically at a distance of 2.5cm from the samples. The angle between the axis of the lens and the sources of illumination was approximately 90^o.
2. Illumination was achieved with 8 LED lamps (Adjustable by a Control Wheel), with a color index close to 95 %.
3. The images were taken at maximum resolution and connected with a compatible personal computer using a fixed magnification of 90X. The images were recorded with a resolution of 1280 × 1024 pixels per image.
4. Digital microscope images were cropped to 350 x 400 pixels using the Microsoft office picture manager to specify/standardize the areas of roughness measurement.
5. The cropped images were analyzed using a WSxM software *(Ver 5 develop 4.1, Nanotec, Electronica, SL)*[29]. Within the WSxM software, all limits, sizes, frames and measured parameters are expressed in pixels. Therefore, the system calibration was done to convert the pixels into absolute real world units. Calibration was made by comparing an object of known size (a ruler in this study) with a scale generated by the software.

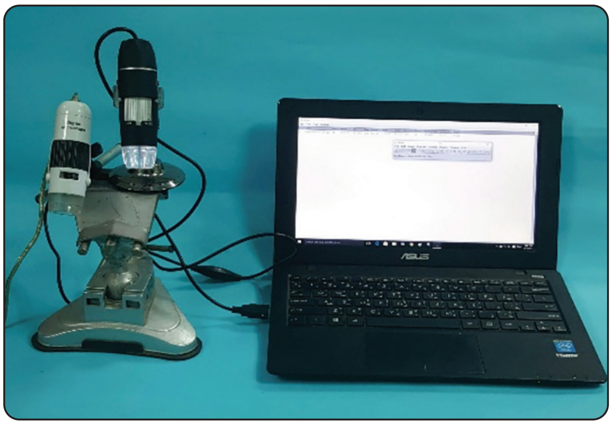

Fig. (1) Digital Microscope

Subsequently, 3D images of the surfaces profiles of the specimens were created. Three 3D images were collected for each specimen, in the central area and at the sides at an area of 10 μm × 10 μm. WSxM software was used to calculate the average of heights (Ra) expressed in µm, which could be considered as reliable indices of surface roughness [30].

The disc-shaped samples of each material were subdivided randomly into three subgroups (n=10) based on different types of immersion solutions (alkaline, acidic or neutral solutions). The samples of each material were individually immersed in glass vials containing the alkaline solution (Licorice, pH,9; *Ramsy Products, Damascus, Syria*), the acidic solution (Lemon juice, pH,1.5; Kemal Kukrer, Turkey) or the neutral solution (Distilled water, pH, 7;Health Aqua, Alexandria, Egypt). The pH of the solutions was measured using a pH meter (AD11; Adwa Instruments, Romania). All samples were kept in the incubator between measurements at 37°C, for forty eight hours.

Forty-eight hours after immersion, the surface roughness were re-evaluated (T_{fin}) using the previously described protocol[31].

RESULTS

Data were presented as mean and standard deviation (SD) values. Data were explored for normality by checking the data distribution and using Kolmogorov-Smirnov and Shapiro-Wilk tests. After homogeneity of variance and normal distribution of errors had been confirmed, one-way analysis of variance was performed followed by Tukey's post-hoc test if showed significance. Two-way analysis of variance ANOVA test of significance was performed for comparing variables affecting mean values (material group and solutions). Sample size (n=30/group) was large enough to detect large effect sizes for main effects and pair-wise comparisons, with the satisfactory level of power set at 80% and a 95% confidence level. The results were analyzed using Graph Pad Instat (Graph Pad, Inc.) software for windows. A value of P < 0.05 was considered statistically significant.

Roughness average(μm) results

Roughness average (μm) results (Means ± SD) for all material groups as function of different pH immersion solution are summarized in table (2) and figures (2, 3, 4, 5).

At baseline, there were non-significant differences among material groups as indicated by one-way ANOVA test (p=0.3461 >0.05) where (Bulk fill ≥ Lava Ultimate ≥ ORMOCER).Table (2) and figures (2, 3, 4, 5).

With the alkaline pH solution, there were significant differences among material groups as indicated by one-way ANOVA test (p=0.0002 >0.05) where (Bulk fill ≥ Lava Ultimate > ORMOCER). Table (2) and figures (2, 3, 4, 5).

With the acidic pH solution, there were **significant** differences among material groups as indicated by one-way ANOVA test (p=0.0419 >0.05) where (Lava Ultimate > ORMOCER ≥ Bulk fill). Table (2) and figures (2, 3, 4, 5).

With the neutral pH solution, there were **significant** differences among material groups as indicated by one-way ANOVA test (p=0.0001<0.05) where (Bulk fill > Lava Ultimate ≥ ORMOCER). Table (2) and figures (2, 3, 4, 5).

N.B. (>) sign means statistically significantly higher (≥) sign means statistically non-significantly higher

Within the Bulk fill composite group, **alkaline** and **neutral** pH immersion solutions subgroups affected on roughness average means values were **non-significant** as indicated by one way ANOVA test; while the **acidic** pH immersion solution subgroup was **significantly** affected on roughness average means values compared to **baseline** mean values (p=0.0001 <0.05); where (**baseline ≥ neutral ≥ alkaline > acidic**). Table (2) and figures (2, 3).

Within the ORMOCER composite group, the **neutral** pH immersion solution subgroup affected on roughness average means values was **non-**

significant as proved by the one way ANOVA test; while the **alkaline** and **acidic** pH immersion solutions subgroups were **significantly** affected on roughness average means values compared to **baseline** mean values (p=0.0046 <0.05); where (**baseline ≥ neutral > alkaline > acidic**). Table (2) and figures (2, 4).

Within the CAD – CAM Lava Ultimate material group, **all the** pH immersion solutions subgroups affected on roughness average means values were **non-significant** as verified by the one way ANOVA test compared to **baseline** mean values (p=0.2482 >0.05); where (**baseline ≥ neutral ≥ alkaline ≥ acidic**). Table (2) and figures (2, 5).

Total effect of the material group; regardless of the different pH immersion solutions, there were significant differences among material groups as demonstrated by the two way ANOVA test (p=0.0006<0.05); where (Bulk fill ≥ Lava Ultimate > ORMOCER).

Total effect of the different pH immersion solutions; irrespective of the materials groups, there were significant differences among the different pH immersion solutions as indicated by the two way ANOVA test (p=<0.0001<0.05); where (**baseline ≥ neutral ≥ alkaline > acidic**).

Therefore, the CAD – CAM resin nano ceramic Lava Ultimate and the Bulk fill nano hybrid resin composite demonstrated almost matching average surface roughness results and both materials were significantly superior to the Universal nano hybrid ORMOCER in terms of the resistance to surface roughness; respective to all immersion solutions (i.e with different pH changes). Meanwhile, at base line measurements, the three types of restorative materials showed compatible roughness average results. Regarding the immersion solutions, the neutral and alkaline solutions produced no significant effects on the surface roughness of any of the three tested materials. However, the acidic immersion solution produced significant changes in the surface roughness of the Bulk fill resin composite and the ORMOCER restorative material. The CAD – CAM resin nano ceramic Lava Ultimate did not demonstrate any significant changes in surface roughness in relation to the acidic immersion solution.

TABLE (2) Roughness average (μm) results (Mean values ± SDs) for all groups as function of different pH immersion solutions

Variable		*Material group*			*Statistics*
		Bulk fill	***ORMOCER***	***Lava Ultimate***	***P value***
Baseline		0.2516^{A}_{a}±0.002	0.2501^{A}_{a}±0.002	0.2503^{A}_{a}±0.002	0.3461 ns
pH immersion solution	***Alkaline***	0.2501^{A}_{a}±0.001	0.2477^{B}_{b}±0.0007	0.2495^{A}_{a}±0.0005	0.0002*
	Acidic	0.2471^{B}_{b}±0.003	0.2472^{B}_{b}±0.001	0.2491^{A}_{a}±0.001	0.0419*
	Neutral	0.2514^{A}_{a}±0.001	0.2485^{B}_{a}±0.002	0.2496^{A}_{a}±0.0009	<0.0001*
Statistics	***P value***	0.0001*	0.0046*	0.2482 ns	

Different subscript small letter in the same column indicating statistically significant difference between subgroups (p< 0.05

Different superscript large letter in the same raw indicating statistically significant difference between materials (p< 0.05

****; significant (p< 0.05)   ns; non-significant (p>0.05)***

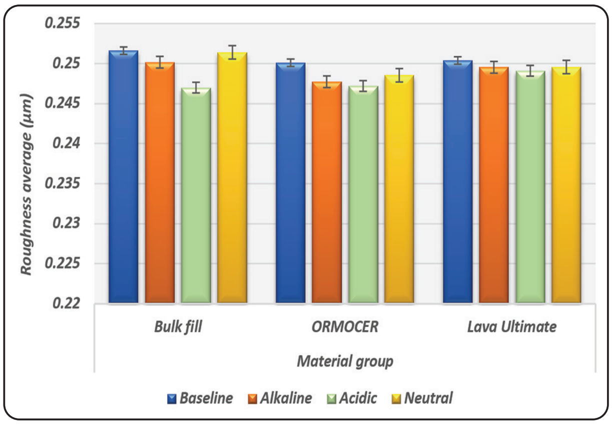

Fig. (2) Column chart of roughness average(μm) mean values for all material groups as function of different pH immersion solution

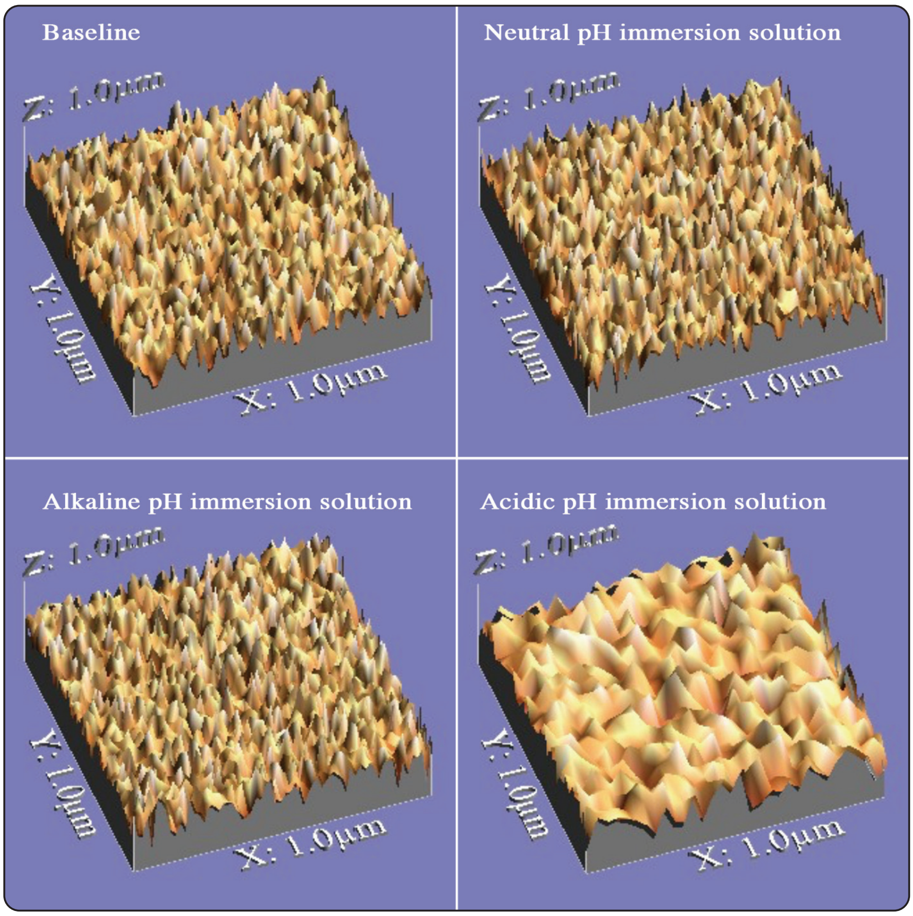

Fig. (3) Representative 3 dimensional images for the ***Bulk fill*** composite group showing surface topography with different pH immersion solutions

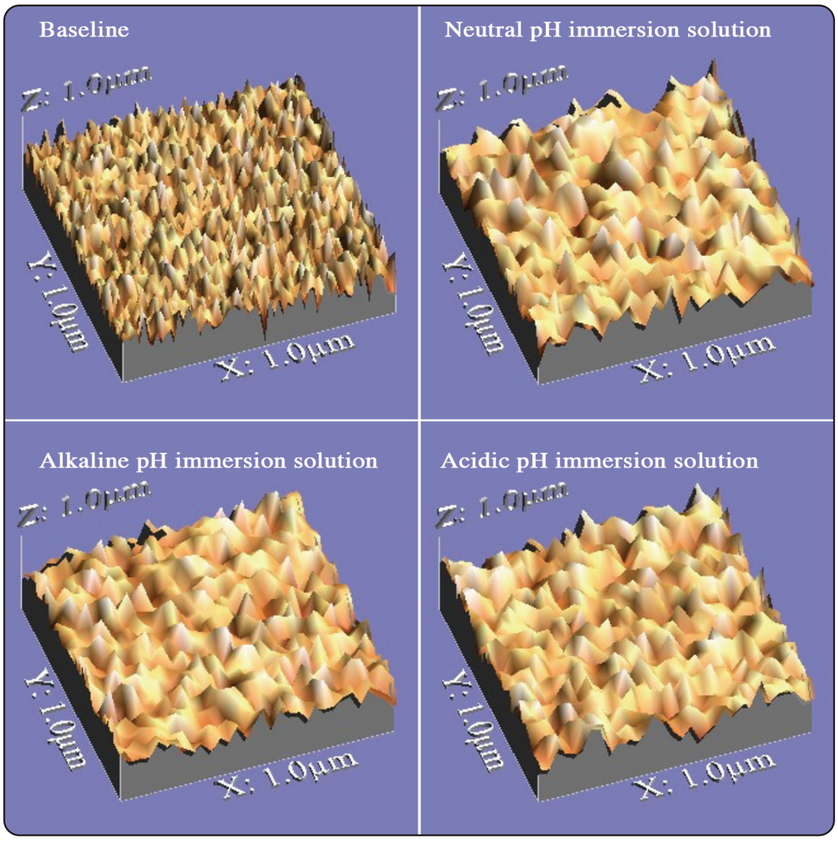

Fig. (4) Representative 3 dimensional images for the ***ORMOCER*** composite group showing surface topography with different pH immersion solutions

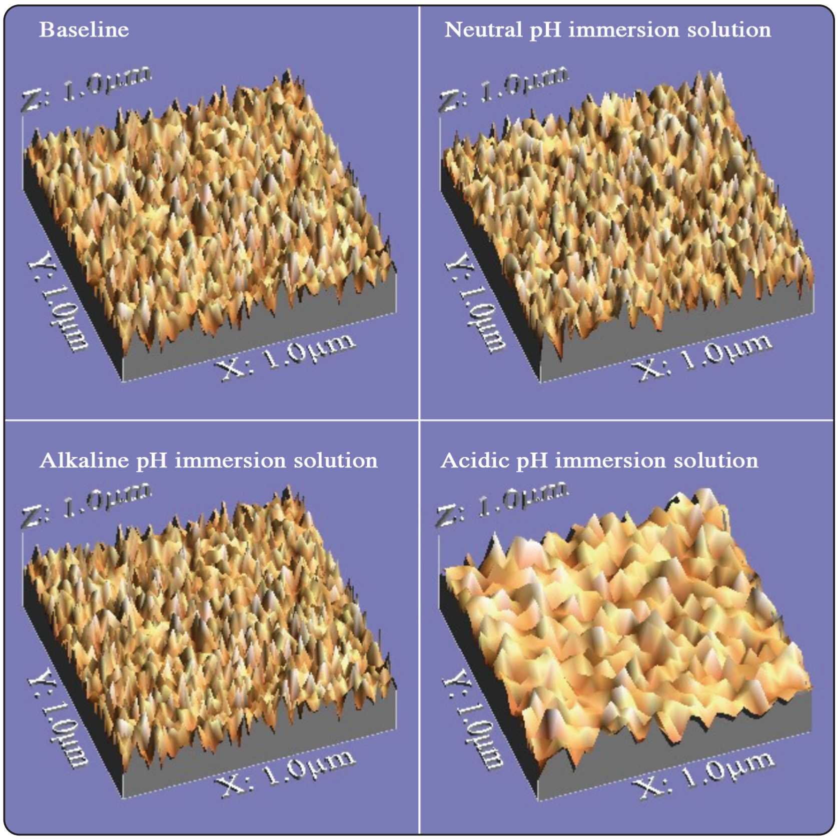

Fig. (5) Representative 3 dimensional images for the CAD - CAM ***Lava Ultimate*** group showing surface topography with different pH immersion solutions

DISCUSSION

The ultimate esthetics of tooth colored restorative materials are massively influenced by the adequacy of the restoration finishing and polishing[6–8]. Appropriately finished and polished restorative materials surfaces should be resistant to any scratches or penetrations. This property is related to the material strength, toughness, elastic stiffness, plasticity, strain, and viscoelasticity [11,14]. Regarding the restorative materials used, the surface roughness have been reported to have direct correlation with the filler loading level as well as the type and size of filler particles [22 – 27]. Food and beverages in the oral cavity expose the esthetic restorative materials to pH changes leading to acidic – base environmental roughness [5, 17 – 21]. The results of this study revealed that, at base line measurements, the surface roughness of the three tested materials was comparable probably due to the standardized finishing and polishing procedures. However, after the immersion procedures, the threshold of surface roughness for the three materials changed. The CAD – CAM resin nano ceramic Lava Ultimate and the Bulk fill nano hybrid resin composite demonstrated comparable average surface roughness results and both materials were statistically significantly superior to the Universal nano hybrid ORMOCER regarding the resistance to surface roughness and relative to the different immersion solutions. That could be attributed to the type and size of the filler particles as well as the filler loading levels. That in addition to the type and chemical composition of the matrices[22–27]. Both materials; the CAD – CAM resin nano ceramic and the Bulk fill nano hybrid resin composite possess comparable filler content weight and volume. Also, being nanohybrid, both materials show high and matching filler loading levels. Moreover, the resin matrix of both materials, being composed of UDMA, Bis – EMA, Bis – GMA and TEGDMA demonstrate a rigid back bone. The CAD – CAM resin nano ceramic has a resin – ceramic combination in a network structure that combines the positive characteristics of resin and ceramics. The material has a rigidity, a hardness and tensile properties that are comparable to those of the natural tooth structure; meanwhile it possesses high flexibility, fracture toughness and wear resistance. That could provide an explanation to the fact that it was the only material of the three tested materials that was not significantly affected by immersion in the acidic solution. Conversely, the Bulk fill nano hybrid resin composite demonstrated significant surface roughness changes related to the acidic immersion solutions and that could be attributed to the fact that the organosilane bonding the filler to the resin matrix may be a weak link where micro cracks can form and cause degradation of the surface. The Universal nano hybrid ORMOCER exhibited the least resistance to surface roughness compared to the other two materials and the condition was even more worse after incubation in the acidic solution. The surface topography results revealed deep scratch lines and interrupted surfaces with multiple projections (figure 4). Ormocers (organically modified ceramics) are manufactured by combining organic and inorganic co polymers with ceramic materials. That combination of the complex network matrix and the larger filler particles results in an inhomogeneous structure after the polishing procedure leading to different wear values between the resin and filler[22–27].

CONCLUSION

The surface roughness of esthetic restorative materials is influenced by intrinsic and extrinsic factors. The intrinsic factors include the particles type, size and filler loading levels in addition to the matrix composition; while the extrinsic factors include the different finishing and polishing techniques.

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