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Evaluation of Surface Characteristics of Denture Base Using Organic-Inorganic Hybrid Coating: An SEM Study

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Abstract

Statement of Problem: Despite the numerous positive features of acrylic denture base, there are a number of undeniable associated disadvantages. The properties of denture base have been improved through various interventions including application of different types of filler and coatings.
Objectives: This study aimed to evaluate the surface roughness, thickness and coating quality of organic-inorganic coating on the denture base through scanning electron microscopy. Moreover, the colour change was evaluated visually.
Materials and Methods: The organic-inorganic hybrid coatings were prepared. Acrylic discs of 10×10 mm were fabricated. The test discs were dipped in the hybrid coating and cured. In order to evaluate the surface roughness and coating thickness, the surface and cross-section of the samples in both coated and control groups were subjected to scanning electron microscopy. The colour change and transparency were visually evaluated with naked eyes. The data were statistically analyzed by student's *t* test.
Results: The hybrid materials perfectly covered all the surfaces of acrylic resin and established proper thickness. The coated group seemed smoother and flatter than the control group; however, the difference was not statistically significant (for all parameters $p > 0.05$). It was quite a thin coating and no perceptible colour change was observed.
Conclusions: The hybrid coating maintained good binding, caused no noticeable discoloration, and thoroughly covered the acrylic resin surface with uniform delicate thickness. It also slightly improved the acrylic resin surface roughness.

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Introduction

Among the several materials used for the denture bases, polymethyl methacrylate (PMMA) resins are the most favored ones [1]. They owe their popularity to their excellent working characteristics, pleasant physical and aesthetic properties, ease of fabrication, and cost-effectiveness [2]. Yet, like other denture base materials, there are some inherent limitations associated with PMMA resin which make it much different with an ideal denture base material [2,3].

When PMMA is polymerized, the compounds (monomers) of low molecular weight are converted (monomers) to polymers of high molecular weight [2,4]. Decreasing the processing temperature and time, results in increased residual monomer content within the processed denture base [2]. Its small size and hydrophilic nature causes the PMMA monomers to diffuse rapidly into the oral cavity and body [2,5], causing tissue irritation, hypersensitivity, or allergic reactions in some cases [4]. The mechanical properties and dimensional stability of dentures were found to be negatively influenced by the plasticizing effect of the excess monomer [6]. Meanwhile, a post-polymerization curing cycle is suggested to eliminate any remaining peroxides [7].

The surface-related features of denture-base materials including roughness, free energy, wettability, hydrophobicity, etc. are clinically important since they play roles in plaque accumulation and staining [1]. Surface roughness, particularly, affects the adhesion and retention of Candida Albicans [8]. Different species of Candida are opportunistic pathogens that reside in the oral cavity of even healthy individuals [9]. Candida biofilms contribute to the development and maintenance of denture stomatitis, which is known as the most common and clinically considerable lesion in denture wearers by virtue of their ability to adhere to acrylic resin [3].

The hybrid organic–inorganic materials are currently becoming so popular due to some particular features which promote them to the material of choice for a wide spectrum of technological applications [10]. Their employment in base resins may improve the colour stability and also reduce their water absorption and solubility, which can consequently extend the denture longevity [11].

At the molecular scale (nanometer), hybrid materials are composites comprised of two components [12], one of which is usually inorganic and the other is organic in nature. Accordingly, hybrids are different from the traditional composites whose components are at the macroscopic level (micrometer to millimeter) [12]. The material that is mixed at the microscopic level is generally so homogeneous that its properties are either between the two original phases or beyond the original one [13].

Interestingly, hybrids of adjustable characteristics can be designed or engineered for particular applications, based on the nature of their components [11,14]. Through the convenient process of sol–gel, hybrid materials can be prepared in the form of powders, monoliths or coatings. In this method, the chemical composition of the hybrid can be highly controlled at low temperature and large area processing [12].

Hybrid materials can be employed in decorative coatings in which the organic dyes are embedded in hybrid coatings. They are also used in scratch-resistant coatings that have hydrophobic or anti-fogging properties. Moreover, hybrids are employed in devices with nanocomposite base for electronic and optoelectronic applications (e.g. light-emitting diodes, photodiodes, solar cells, gas sensors and field effect transistors) [15].

In construction industry, the hybrids are included in fire retardant materials. In dentistry, they are used in nanocomposite-based dental filling materials. Likewise, they are used in proton conducting membranes of fuel cells, antistatic/anti-reflection coatings, as corrosion protection, porous hybrid materials, and antibacterial properties [12,15]. Besides being quite thin and uniformly covering all surfaces, a suitable coating should resist noticeable colour changes in the denture base. The present study aimed to apply hybrid coatings on denture base and identify the main structural changes through scanning electron microscopy (SEM). The null hypothesis was that inorganic-organic hybrid coating would not influence the surface roughness, thickness and colour of PMMA used in the denture base.

Materials and Methods

Two sheets of resin plates were made of heat polymerized material (PMMA - SR Triplex Hot Acrylic Resin, Ivoclar Vivadent Inc; England) through a classical press-pack dough moulding technique of 2-mm thick wax-plate flaked based on the polymerization regime as suggested by the manufacturer. The flask was immersed in cold water, tempered up to 100°C, and maintained at that temperature for 60 minutes. The plates were all sectioned into 10×10-mm samples. Metallographic abrasive paper (MATADOR; Germany) at 600 and 800 mesh were sequentially used to polish the samples. Then, they were rinsed in ethanol and deionized water for 3 times (each time 3 minutes), and were stored until used.

For the hybrid coatings, 10.4g tetraethyl orthosilicate, 30g ethylene glycol diethyl ether, 5.85g deionized water, and 0.39g glacial acetic acid were stirred in a flask for 1.5 hours in water bath at 40°C. Then, 4.72g (2, 3-epoxypropoxy) propyltrimethoxysilane (all by Kimia-Gostar; Tehran, Iran) was added to continue the reaction for 6 hours at 60°C and, then, left for another 24 hours. The flask was supplemented with 4.26g glycidylmethacrylate, 2.16g acrylic acid, 9g methyl methacrylate, and 147g EGME (all by Kimia-Gostar; Tehran, Iran), and then placed in an oil bath. The flask was connected to a condenser and stirring apparatus. Stirring began after nitrogen was added to the flask. The nitrogen gas was infused for 15 minutes.

The oil bath was heated and stabilized at 70°C. Then, 0.10 g of azobisisobutyronitrile was dissolved in 3g ethylene glycol monomethyl ether and injected into the flask. The complex was allowed to progress for 10 hours and after it cooled down to the room temperature, the stirring and nitrogen gas influx were stopped. 2 -methylimidazole (Kimia-Gostar; Tehran, Iran) was then added at 10% of the molar mass of the epoxy group. The mixture was stirred at room temperature for 24 hours to obtain organic-inorganic hybrid coatings. The hybrid coating was applied over the surface of the resin plates, dried, placed in oven at 70°C for 1 hour, and polymerized at 90°C for 2 hours.

The prepared specimens were divided into an experimental (n=5) and a control group (n=5). The selected samples were subjected to SEM evaluation (Phenom ProX; Netherlands). SEM photomicrographs were taken at different magnifications for visual inspection. This system was also used to measure the surface roughness on 3 locations of each surface sample (Ra and Rz as profile roughness parameters). To calculate the Ra using an algorithm, we measured the average distance between the peaks and valleys, as well as the deviation from the mean line on the entire surface within the sampling length. To calculate the Rz, we also measured the vertical distance from the highest peak to the lowest valley within five sampling lengths; the distances were finally averaged.

The 3D Roughness Reconstruction software helped the Phenom desktop SEM systems to generate three-dimensional images and submicrometer roughness measurements. The coating thickness was measured on the cross-section of the resin-coated samples. Meanwhile, the major discolourations were merely checked with naked eyes since the minor colour changes in the denture did not matter. The two groups were visually compared visually.The data related to changes from the baseline of wrinkles as well as the roughness features were analyzed using SPSS software version 18.0 (SPSS Inc., Chicago, IL, USA). Student's *t* test was performed and the significance level was set at $\alpha= 0.05$.

Results

The SEM inspection of the hybrid-coated acryl resin plates and control group revealed a coarser topography in the latter (Figure 1, a-d). However, the results of t test showed no statistically significant difference between the two groups (for Rz, $p = 0.053$ and for Ra $p = 0.110$) (Table 1). The cross-section view of the coated resin plates showed the thickness of the coating material (20μm**)** (Figure 2). Moreover, a perfect junction was observed between the acrylic resin and hybrid coating, i.e. it was quite uniform and had no gap. According to the SEM, the hybrid coating thoroughly covered the acryl surfaces. The hybrid coating was more homogeneous and compact than the acrylic resin plates. No dramatic discolouration was observed in the coated and control groups, as shown with naked eye (Figure 3).

Table 1: Surface roughness (Rz, Ra) of specimens in each group. Data are mean ±standard deviation (n=5)

group	Rz (μm)	Ra (nm)
Control	5.1 ± 0.15	518 ± 47
Experimental	4.48 ± 0.59	474 ± 27
p-value	0.053	0.110

The result was not significant at $p > 0.05$.

Discussion

So far, various options like fillers, plasticizers and coating material have been used to improve the properties of denture-base [17,18]. Such interventions could change the mechanical and antibacterial properties of the denture base. Denture coatings are often used as the antimicrobial agent [17,19-22]. Sato

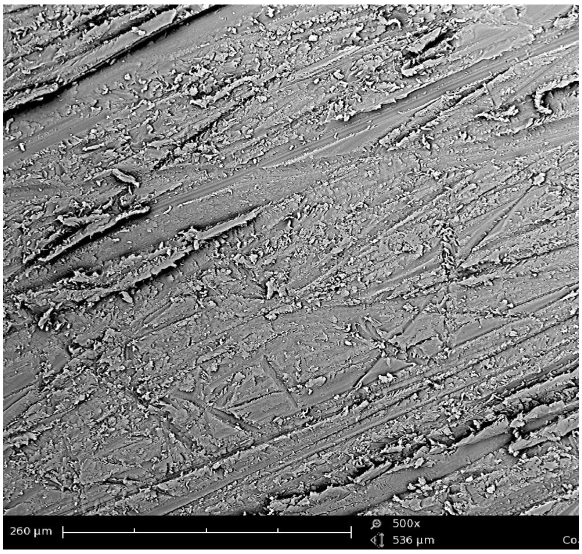

Figure 1-a: The surface roughness of a specimen in the control group at 500X

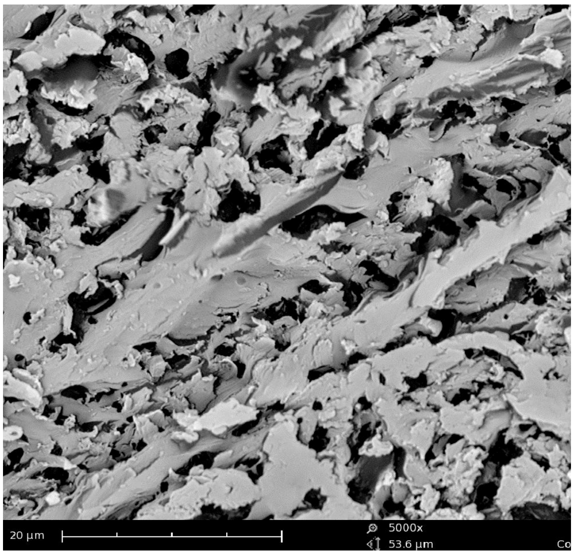

Figure 1-b: The surface roughness of a specimen in the control group at 5000X

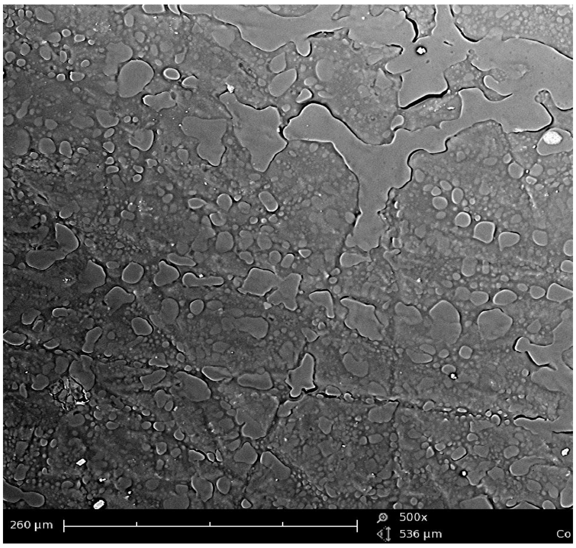

Figure 1-c: The surface roughness of a specimen in the coated group in 500X

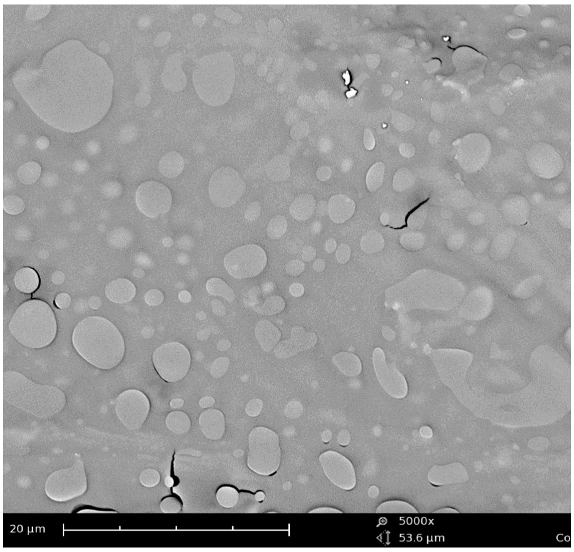

Figure 1-d: The surface roughness of a specimen in the coated group in 5000X

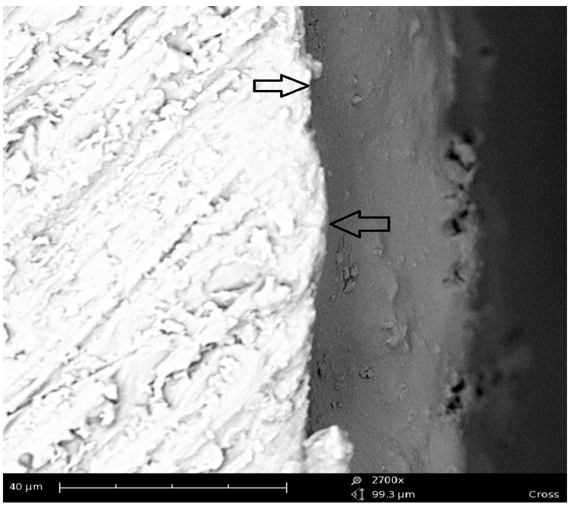

Figure 2: The cross-sectional view displaying the uniform junction. 20 µm thickness of coating (the coating is seen darker than the denture base).

et al. found that the use of mannan (the polysaccharide found in yeasts cell wall) coating on the denture base could prevent the adhesion of candida albicans [21]. Hybrid coating is also believed to improve the denture base properties [11,14,23]. It prevented water uptake into the base resin [11].

Zuo *et al*. noted that the hybrid coating effectively reduced the water uptake and solubility of the base resins and also improved the colour stability of the denture base. Moreover, they observed that this coating decreased the release of residual monomers and other particles of the resin, and prevented degradation of the denture base [11]. The network structure in hybrid coating is so dense that it decreases the adhesion and penetration of microorganisms like ceramic [23].

The denture roughness affects the adhesion

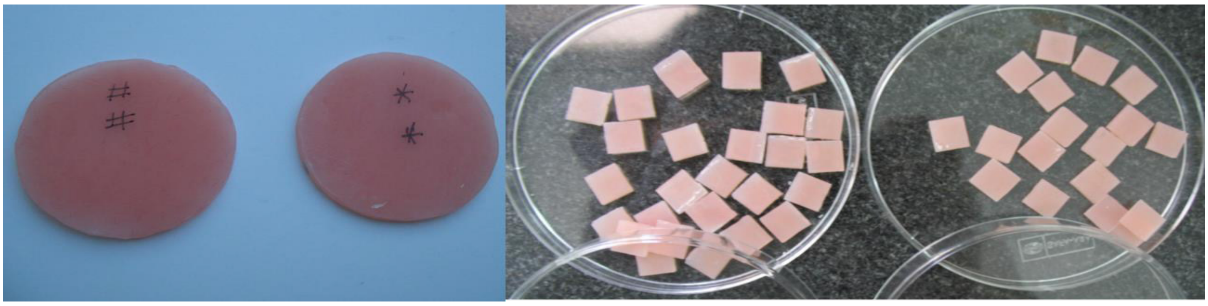

Figure 3: The control and coated groups. No significant discolouration was observable with naked eye. Visual comparison of the images confirms the absence of any significant difference in the discolouration between the two groups.

and retention of microorganisms, stain, plaque, and calculus [1]. Although the present study noted no statistically significant decrease in the surface roughness, more density and consistency was observed. This might be attributed to the reduced biofilm and plaque formation on the denture surface; however, it needs to be confirmed by more detailed studies. On the other hand, the wettability of the denture base is a determining factor in denture retention, especially in the maxilla [24]. It seems that the decreased surface roughness which is associated with lower surface contact results in lower wettability. This coating was hydrophilic, as Toselli *et al*. found that the hybrid coating improved the wettability since the coated acryl was more hydrophilic than pure acryl [23]. This coating can be used in nano science, as in coatings with nano-titania or nano-silver particles [17]. However, Wady *et al*. reported that the coating of silver nano-particles had no effect on candida albicans adherence [25].

The organic-inorganic hybrid used in the current study for coating of acryl resin disks provided a very thin coated layer of 20 µm thick (Figure 2). This feature can be an advantage for this coating as it does not interfere with the denture fit in denture users. Proper attachment of the hybrid layer to the resin is considerable because of chemical attachment. SEM evaluation showed continuous connection between the coating and the resin (Figure 2). Similarly, long-term coating should be insoluble.

The hybrid coatings are anti-scratch, as well [11,23]. The surface roughness promotes the denture base against candida adhesion, plaque accumulation, and stain. It was also reported by Toselli *et al*. and Mammeri *et al*. that the hybrid coating could resist the scratch and increase the surface hardness [10,23]. The employed hybrid layer formulation was transparent, so it did not alter the appearance and texture of the denture base [11]. Likewise, Toselli *et al*. and Mammeri *et al*. visually compared the coating polymer films with control groups and found no colour change in the coating resin [10,23]. In those studies, the coating was so transparent that the writing on polymer films was readable after the coating.

Polymerization process of this coating layer was considered a repolymerization for the denture base and reduced the amount of residual monomer in denture base [2,11]. Likewise, Shim and Watts found that additional heat curing reduced the residual monomer in the resin [26]. Hybrid coating was found to have covered the microcracks and microporous, which can, in turn, make up for poor polymerizing or polishing, and improve the denture properties [10,11].

Conclusions

The hybrid coating employed in this study created a very delicate layer of 20µm thick with ideal junction on the acryl resin disks. Seemingly, this coating increases the quality and roughness of the denture base, and controls the microbial plaque formation. Yet, further studies are recommended to confirm the findings of the present study.

Conflict of Interest: None declared.

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