RESEARCH PAPER

Effect of Zirconium Oxide -Titanium Dioxide Nanoparticles on Mechanical and Physical Properties of Soft Denture Lining Materials

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ABSTRACT

This study sought to identify the effect of adding zirconium oxide and titanium dioxide nanoparticles on some mechanical and physical properties (hardness, roughness, and spectrophotometer UV-light absorption) of soft denture lining materials. For 3 experiments of this study, 315 specimens were prepared from soft liner by conventional methods (n of each experiment=105) and then divided into three groups as stated by the tests (roughness, hardness, and spectrophotometer color absorption); 15 specimens as a control group (pure soft liner), 45 specimens for nanoparticles as a two test group (reinforced with 2 wt %, 3 wt %, and 5wt % of Zirconium oxide and Titanium dioxide nanoparticles respectively. The mean hardness and surface roughness values of the soft-lining denture in the experimental groups of 2 %, 3%, and 5% of TiNPs and ZrNPs were significantly lower than the control group ($P< 0.001$). The hardness and surface roughness decreased significantly after adding nanoparticles, and the most decrease was seen in the 5% of both TiNPs and ZrNPs groups. The UV- light absorption values of the experimental groups were significantly higher than those of the control group ($P< 0.001$), and the maximum increase was seen was in the 5% of both TiNPs and ZrNPs groups. Incorporating the TiO_2 and ZrO_2 nanoparticles into soft liner improve the mechanical and physical properties; decrease surface roughness, hardness, and increased opacity of the liner material. This improvement was directly proportional to the concentrations of the nanoparticle.

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INTRODUCTION

Soft liner materials have been used as a cushion for the internal surfaces of the denture for patients with bony atrophy, injured oral mucosa, ridge undercuts, aptyalism, odontoprisis, obturator in case of some oral birth defects, and for enhancing denture retention by extension of denture flanges to engage an existing soft-tissue undercut [1]. Soft liners are often utilized to minimize the pressure of topical points. They usually act as a pillow for the distribution of the chewing forces that move to the underlying tissue but do not

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necessarily reduce the transmitted force [2]. The desired characteristics of lining materials should include: high strength bonding to the prosthesis, dimensional stability, durable softness or elasticity, minimum water absorption, colorfastness, easy processing, and biocompatibility [3]. The main defects of soft linings are the lack of firm bonding to the prosthesis. Disengagement of soft liners from a dental base is widespread clinical events. The tensile strength of the soft liner substance decides the bond strength of these materials. An efficient bond is necessary between the denture base and soft liner, in order to the denture works properly [4]. Bond failures between liner and denture base create a potential surface for bacterial growth, plaque, and calculus and also weaken the durability of the soft liner [5]. Factors that influence the bond between the denture base and resilient liner are Geometry of bond surface, etching, use of bonding agents, inherent bond strength, tear strength, and thickness of lining material [6]. It is also noted that piling up of stains, water absorption, disintegration of components, and degeneration of basic dyes are major causes of change of acrylic base materials after long times of utilization. Drinks, like tea, coffee, synthetic dyes as a portion of food, smoking, microbial colonization, and the tinctures in the liner also participate in a change of the color [7-9]. Preprocessing of the denture base surface is necessary to enhance the bond strength between the denture base resin and the soft liner [10,11].

Numerous approaches and technologies have been used to ameliorate the bonding strength and liner adhesion. Approaches made to roughen the acrylic/liners interface as a means of boosting the bond strength with the aid of lasers, alumina abrasion, and chemical etching [12]. Also, oxygen plasma treatment which is another surface roughening technique has shown an enhancement in the tensile bond strength between acrylic resin and soft liner [13].

Currently, zirconium oxide nanoparticles (nano-ZrO_2) and Titanium oxide nanoparticles (nano-TiO_2) are considerably utilized, owing to their non-toxic, chemically inert, low cost, high refractive index, good toughness and mechanical strength, corrosion resistance and, perfect biocompatibilities as well as their white color which makes it less prone to alter the esthetic when compared to other nanoparticles of metal oxide [14,15]. Literature has also shown that n-TiO_2 and n-ZrO_2 as a reinforcement agent bring new optical, electrical and, physiochemical properties attained at very low content of both (n-TiO_2/ n-ZrO_2). This will make these metal nanoparticles a promising new category of materials [12]. These nanoparticles (n-TiO_2/ n-ZrO_2) have been used as a reinforcement agent to ameliorate the mechanical characteristics of the polymethyl methacrylate (PMMA) matrix [15]. Excellent adherence and an identical scattering of these nanoparticles inside an acrylic matrix augmented the flexural characteristics of a polymer with nanoparticles complex [16]. The color at the forefront of the characteristics sought by the user and regards it as the basis for the quality of the dental base. One of the main reasons for the replacement of dental prosthesis is the change in color due to obsolescence or the use of detergents or disinfectants and other reasons mentioned earlier [17]. All TiO_2 and ZrO_2 nanomaterials are distinguished from other materials by their small size, large surface area, and strong interaction with the organic polymer. Thus, the physical and optical characteristics of the polymer can be enhanced, helping it to resist aging and cracking due to environmental stress [18]. A group of studies has shown that the addition of some nanomaterials (TiO_2 / ZrO_2) to the soft lining material leads to remarkable resistance to environmental UV irradiation and more stability in the color of these materials [2,19]. The current study hypothesized that the addition of nanoparticles (TiO_2 / ZrO_2) would reduce the hardness and surface roughness and increase the UV- light absorption of the soft denture lining materials.

MATERIALS AND METHODS

Study Protocol

Three hundred and fifteen specimens (Vertex Soft, Heat-polymerized acrylic resin-based resilient liner, Vertex-Dental BV, Zeist, The Netherlands) were used as a matrix component and TiO_2 / ZrO_2 powder with a medium grain size of 200 nm as a reinforcing agent (TiO_2/ZrO_2 NPs, Changsha Santech Co., China) were manufactured and prepared by conventional methods and divided into 3 groups based on the tests [shore A hardness, roughness, and light absorption, each experiment =105, and then divided into three groups; 15 specimens as a control group (unreinforced soft liner), 45 specimens for both n-ZrO_2 and n-TiO_2 as two test group (reinforced with 2 Wt%, 3 wt%, and 5wt% of n-ZrO_2 and n-TiO_2 respectively, 15 specimens for each concentration)] (Fig.1). Traditional flasking procedure for complete dentures was followed during the mould preparing. An insulating medium was used to paint the metal model and then allow it to dry, before investing the down part of the metal flask filled with dental stone and mixed according to the manufacturer's instructions using vibration to dispose of the trapped air, and then let it adjust. The metal model was inserted to approximately one half of their depth as to be easily removed after setting of the stone. The experimental samples (Vertex –soft heat polymerizing acrylic-based soft liner) were prepared by mixed n-ZrO_2 and n-TiO_2 with soft liner monomer in three different concentrations by weight with the powder. Then the resilient lining liquid was added to the powder and the material was mixed in accordance to the manufacturer's instructions, the weight of the n-ZrO_2 and n-TiO_2 powder that had been added to the monomer soft liner was taking into account and subtracted from soft liner monomer volume to achieve correct P/L ratio. The conventional method has been followed to investing, packing, curing, and finishing for samples [20]. After finish curing, the flask was opened and the specimens were removed from the flask and remove the excess of the material by using a sharp blade, and then all specimens were kept in packages of distilled water in an incubator at 37Cº for 24 hours [20].

Scanning electron microscopy (SEM)

The specimens were sputtered with a thin gold layer using a sputter coater (HITACHI, E-1010, Japan) to overcome the non-conductive nature of the material. Subsequently, the morphology of the fractured specimens for control and modified groups was conducted to show the degree of TiO_2 NPs/ ZrO_2 NPs dispersion within the poly ethyl methacrylate (PEMA) matrix by using a scanning electron microscope (SEM) (HITACHI, S-3700N, Japan). To obtain a better visual inspection, images were taken at different magnifications to note important features about the nature of the failure.

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis (IR Prestige -21Shimadzu, Japan) was conducted to assess the chemical interaction between n- ZrO_2 / n-TiO_2 and soft denture lining.

Shore A hardness test

The dimensions of the samples were prepared for the hardness according to ISO 20795-1: 2013. [21]. Soft liner samples were prepared in disc shape with a diameter of about 30 mm and a thickness

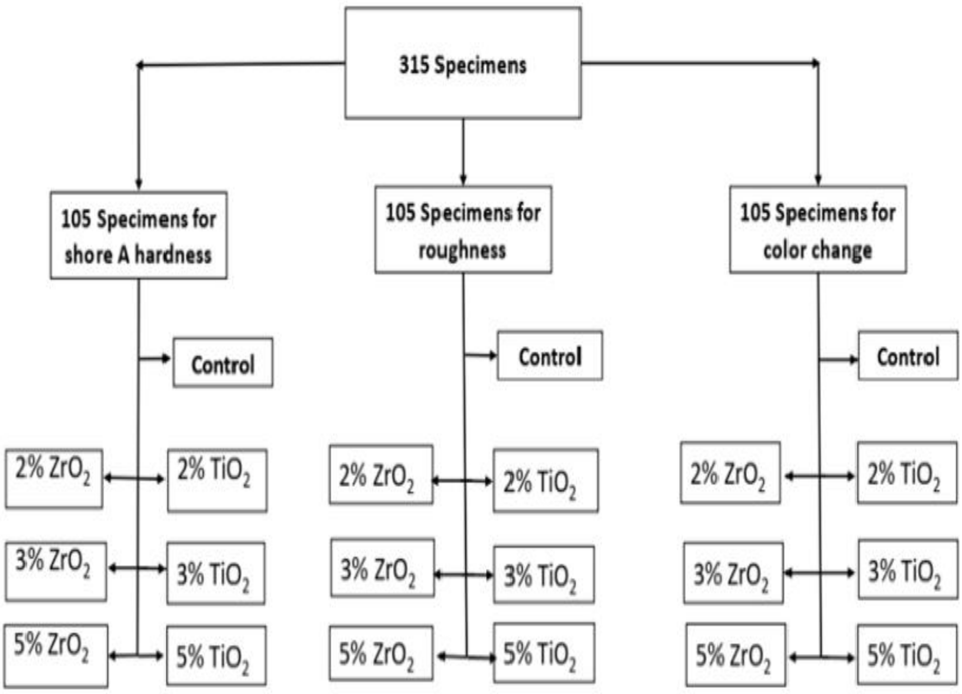

Fig. 1. Classification of test specimens according to the concentration of TiO_2 and ZrO_2 nanoparticles.

of 3 mm to measure the hardness of soft denture lining using Shore A durometer (Time group-TH200, China). 20 mm was the distance between the durometer indenter and the sample, 5 seconds was the contact duration after penetration, an indentation was performed for each sample and their average was obtained [21].

Surface roughness test

The dimensions of the samples were constructed for the roughness according to ADA specification No.12, 1999 [22]. The test was performed using a profilometer (Time 3200 / TR 200 China). It is provided with a surface analyzer (stylus) made from diamond to trace the profile of the surface irregularities and recording all the peaks and cavities which distinguish the surface. This stylus shifted 4 mm on the tested samples. To secure that the measurements were conducted in one place for all samples, a pencil sign has been made on the base of each sample. The final value of the surface roughness for each sample is the average of the two recorded readings. After treatment, all samples were kept in distilled water containers in an incubator at 37 ° C for 24 hours. The first estimation of surface roughness was performed 24 hours later (readings were considered as a baseline value) [23].

Spectrophotometer color absorption

The light absorption percentage was measured by a UV-Visible Spectrophotometer (UV-1800, Department of Biotechnology Engineering). Soft denture lining specimens were disk-shaped in diameter and thickness (50± 1mm, 0.5 ± 0.05mm, respectively) as stated by ADA specifications No.1228 [22].

Statistical Analysis

The collected data were evaluated using IBM statistical package for social science (SPSS, Chicago, IL) version 20.0. The data were Quantitative, expressed as mean and standard deviation, and the Student t-test was used to test the difference between each two independent groups. A P-value lower than (0.05) was defined as a statistical significance.

RESULTS AND DISCUSSION

The results obtained from FTIR analysis indicated that there was no chemical reaction between the Soft denture lining and nanoparticles (TiNPs and ZrNPs) in all percentages. FTIR spectra of control soft liner group and reinforced specimens (TiNPs and ZrNPs) are shown in (Fig. 2) respectively.

Moreover, SEM detected a perfect dispersion of ZrNPs and TiNPs in the soft liner matrix, demonstrating a more uniform fracture surface and showing that 2% of the TiO_2 and ZrO_2 nanoparticles were distributed within the matrix without clumping; However, with increasing the concentration of TiO_2 and ZrO_2 nanoparticles to 3% and 5% (w / w), the polymer matrix was filled with nanoparticles, producing a stronger material, and good dispersion of the nanoparticles in the resin matrix to fill the serial spaces between the polymer, which indicates the importance of the additional content of nanoparticles. In addition, the phenomenon of nanoparticle agglomeration was evident in the soft lining as the percentage of the added nanoparticles increased (Fig. 3-5).

the test groups of both TiNPs and ZrNPs for different percentages was significantly lower than the control group (P< 0.001). The hardness and surface roughness decreased significantly after adding the nanoparticles, and the largest decrease was seen in the 5% group (Table. 1, 2).

The statistical results of this study evidenced that the UV light absorption values of the samples combined with TiNPs and ZrNPs were significantly increased compared to the control group (P <0.001), and the highest absorbance value in the group was 5% for both TiNPs and ZrNPs (Table. 1, 2).

The present study aims to find out the effect of the addition of TiNPs and ZrNPs on the Shore A hardness, surface roughness, and UV- light absorption properties of the soft denture liners. The results uncovered that all the reinforced groups showed significantly decreased hardness and surface roughness and increased UV-light absorption in direct proportion to the concentrations (2%, 3%, 5%) of both n-TiO_2 and n-ZrO_2; according to the results obtained, the hypothesis of this study has become accepted.

Soft denture liners are subject to changes in hardness during clinical use, which is an important characteristic because the ability to absorb impacts increased as the elasticity enhanced, [24] therefore, low hardness is one of the most important properties that should be characterized by these materials. [24,25]. One of the most complicating agents when using soft denture liners is maintaining their hardness within the

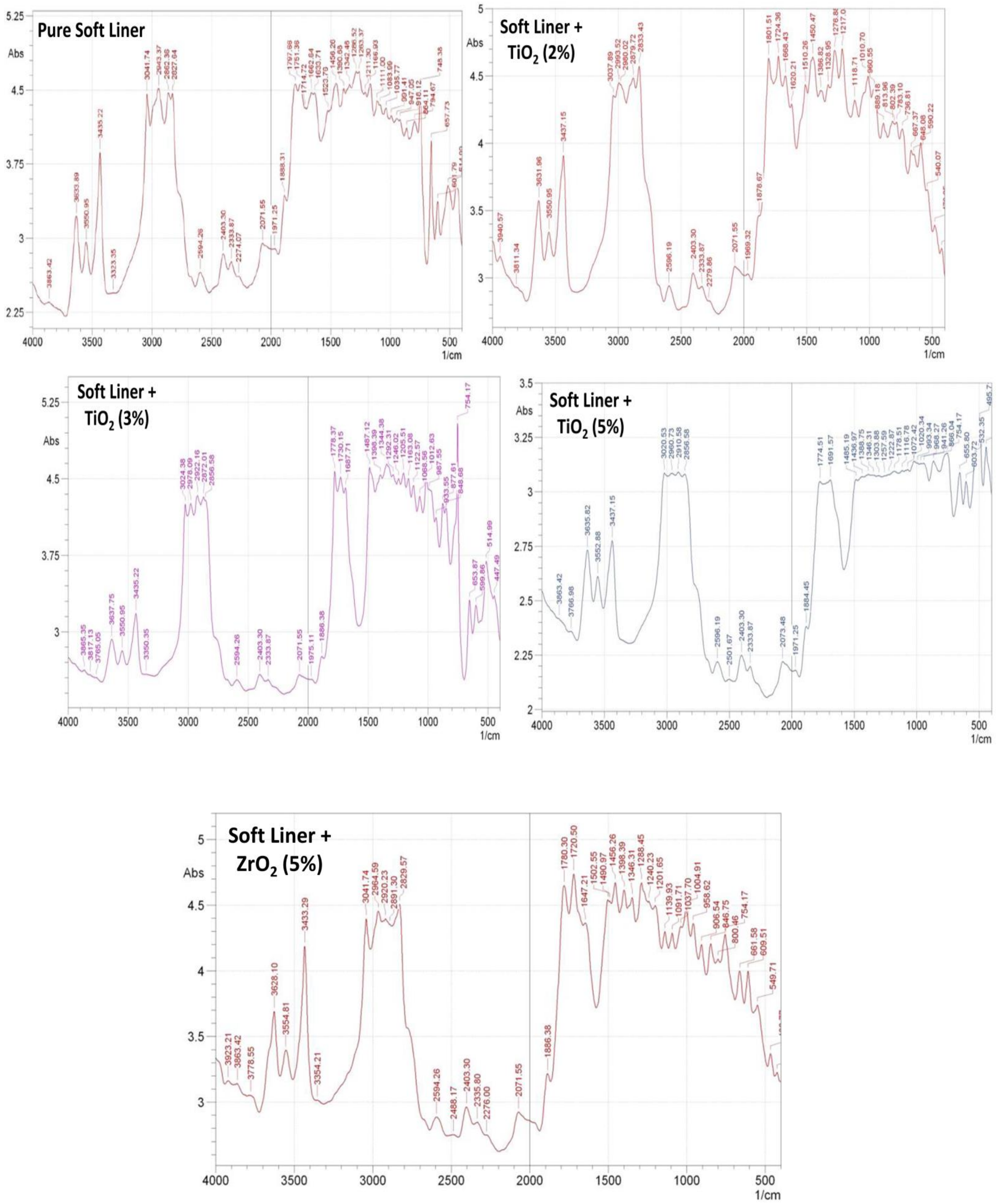

Fig. 2. FTIR spectrum for pure soft liner, soft liner specimens reinforced with 2%, 3%, 5% of TiNPs, and soft liner specimens reinforced with 2%, 3%, 5% of ZrNPs.

favorable requirements, due to their instability in the aquatic environment [26]. During the use of these materials, they will be submerged in saliva, food, water, and hygiene a product, which exposes its ingredients to leaching and water absorption [25,27]. The loss of balance between the components of the soft-lining materials and their absorption of liquids directly affects the performance and stability of the dimensions of these materials, [28] and these phenomena are often affiliated with the increase in hardness and expansion, distortion, the emission of bad

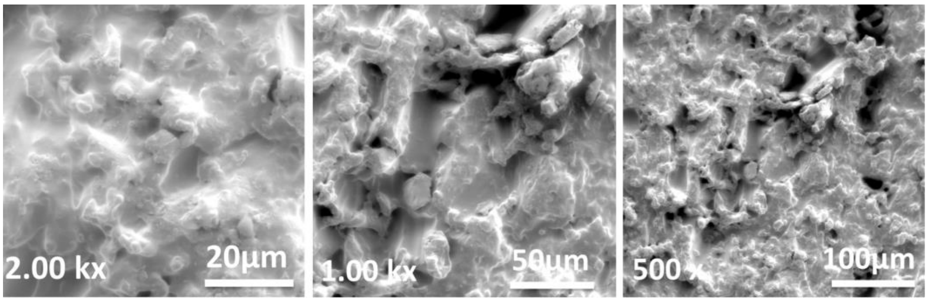

Fig. 3. Scanning electron microscope images for pure soft liner at different magnification and scale bare.

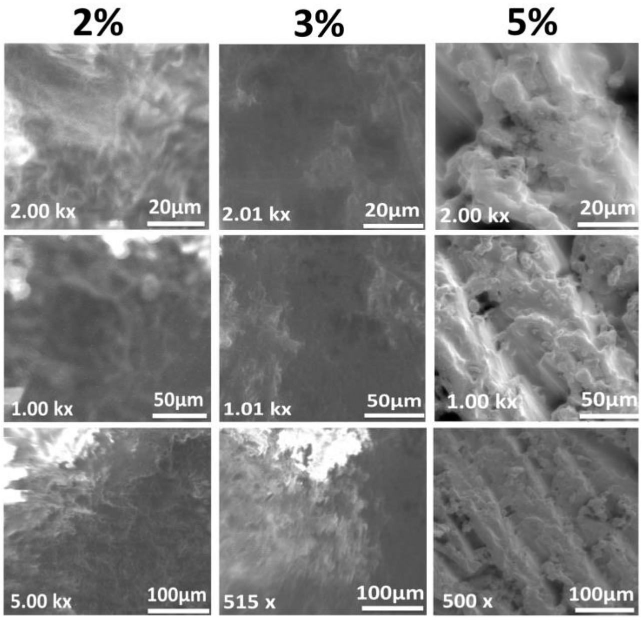

Fig. 4. Scanning electron microscope images for soft liner specimens reinforced with 2%, 3%, 5% of TiNPs at different magnification and scale bare.

odors, and the change of color, in addition to the adhesion and growth of microorganisms [25].

Lack of the elastic viscosity of soft denture lining may causes irritation in the denture-bearing area, thus result in destruction to the supportive tissues and accelerating damage of these materials. [29] The addition of zirconia and titania nanoparticles has been proposed to enhance the mechanical properties of PMMA. Many studies observed that the incorporation of TiNPs and ZrNPs into PMMA significantly increased hardness, flexural strength, and fracture toughness. Still, a scanty decrease in the flexural strength has been reported that could be caused by the aggregation of particles within the resin, debilitating the material. But some recent studies indicated a significant increase in the hardness, fracture toughness, and impact strength of PMMA after strengthening with ZrO_2 [17, 14, 30]. Besides, it was observed that adding TiO_2 to PMMA significantly increases the impact strength [31] and significantly reduces water sorption and solubility [32]. These data are not in agreement with the results of some other studies that have demonstrated that TiO_2 does not improve the flexural strength of PMMA. This is due to the agglomeration of the particles inside the resin, which causes its weakening [33,34]. Incorporation of Zirconia NPs into PMMA increased their hardness [35-37]. Contrary to the results of another study, which observed a scanty increase in the hardness of n-ZrO_2 / PMMA with an insignificant change in the surface roughness [38].

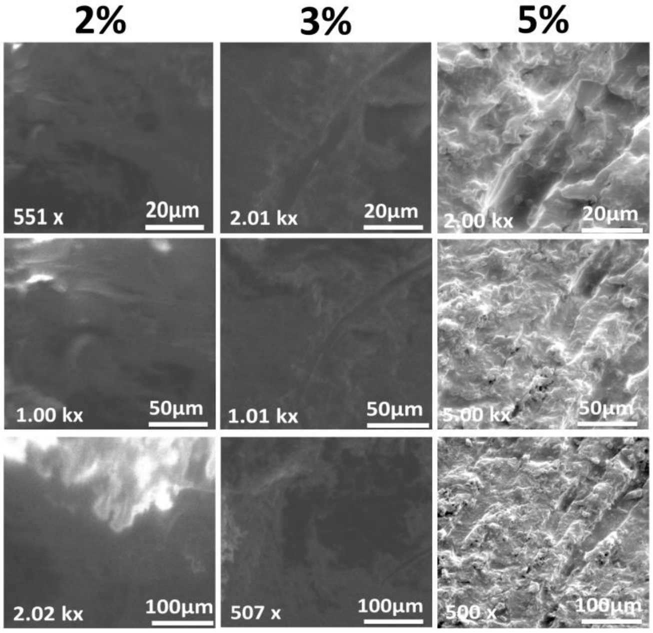

Fig. 5. Scanning electron microscope images for soft liner specimens reinforced with 2%, 3%, 5% of ZrNPs at different magnification and scale bare.

The adherence between the resin matrix and the filler particles plays a critical role in enhancing the properties of the resulting composite. Both of the hardness and surface roughness were significantly improved when the silanized zirconia NPs integrated into the acrylic resin [39,40]. The incorporating of TiNPs into PMMA was useful in improving the characteristics of acrylic resin matrix [41].

One study had found that the incorporation of silanized titanium with PMMA led to a marked improvement in the surface hardness of the resin,

Table 1. Shore A Hardness, surface roughness, and UV light absorption of pure soft liner (control) and reinforced soft liner with n-ZrO_2 in three concentrations.

Test	Control N = 15	ZrO_2 (2%) N =15	ZrO_2 (3%) N=15	ZrO2 (5%) N=15
Shore A Hardness				
Mean± SD	77.56±0.87	69.32±1.23	68.38±1.25	66.72±0.74
Range	76.19 – 79.37	67.69 – 70.95	65.99 – 70.78	65.12 – 66.33
Test (P value)		8.75 (<0.001)[1]	19.75 (<0.001)[1] 15.70 (<0.001)[2]	27.18 (<0.001)[1] 24.68 (<0.001)[2] 0.89 (0.38)[3]
Surface roughness				
Mean± SD	2.26±0.09	1.50±0.15	1.26±0.02	1.42±0.33
Range	2.07 – 2.40	1.23 – 1.72	1.23 – 1.29	1.12 – 1.96
Test (P value)		17.7 (<0.001)[1]	41.31 (<0.001)[1] 6.16 (<0.001)[2]	9.39 (<0.001)[1] 0.85 (0.41)[2] 1.80 (0.08)
Light absorption				
Mean± SD	2.28±0.38	4.22±0.53	5.73±0.53	7.46±0.56
Range	1.68 – 3.11	3.45 – 4.98	4.89 – 6.57	6.7 – 8.5
Test (P value)		11.59 (<0.001)[1]	20.38 (<0.001)[1] 7.81 (<0.001)[2]	28.55 (<0.001)[1] 15.88(<0.001)[2] 8.41 (<0.001)[3]

1 = comparing with control group
2 = comparing with the group with ZrO_2 2%
3 = comparing with the group with ZrO_2 3%

Table 2. Shore A Hardness, surface roughness, and UV light absorption of pure soft liner (control) and reinforced soft liner with n-TiO_2 in three concentrations.

Test	Control N = 15	TiO2 (2%) N =15	TiO2 (3%) N=15	TiO2 (5%) N=15
Shore A Hardness				
Mean± SD	77.56±0.87	74.09±1.02	68.52±4.10	65.54±0.74
Range	76.19 – 79.37	73.1 – 75.29	64.06 – 74.12	68.38 – 70.99
Test (P value)		10.01(<0.001)[1]	8.36 (<0.001)[1] 5.11 (<0.001)[2]	40.45 (<0.001)[1] 27.72 (<0.001)[2] 4.06 (0.001)[3]
Surface roughness				
Mean± SD	2.26±0.09	1.32±0.39	1.18±0.24	1.10±0.16
Range	2.07 – 2.40	1.02 – 2.10	1 – 1.6	1 – 1.4
Test (P value)		9.08 (<0.001)[1]	16.01(<0.001)[1] 1.16 (0.26)[2]	24.25(<0.001)[1] 2.0 (0.06)[2] 1.06 (0.30)[3]
Light absorption				
Mean± SD	2.28±0.38	4.42±0.57	6.33±0.72	8.46±0.69
Range	1.68 – 3.11	3.45 – 5.1	5.16 – 7.30	7.3 – 9.5
Test (P value)		12.05 (<0.001)[1]	19.28 (<0.001)[1] 8.03 (<0.001)[2]	30.37 (<0.001)[1] 17.44 (<0.001)[2] 8.29 (<0.001)[3]

1 = Comparing with control group
2 = comparing with the group with TiO_2 2%
3 = comparing with the group with TiO_2 3%

but it was associated with an increase in surface roughness by adding 3% w of TiNPs to acrylic resin [42].

Since the maximum benefit of the soft liner materials represented in their softness and ability to act as a cushion to absorb the forces of occlusion, so it is very preferable that the hardness of these materials be low [43].

The results of the current study showed that the values of both hardness and surface roughness of the reinforced samples were much lower compared to the non-reinforced samples (control group). Noting the SEM results of our study, there were some areas of TiNPs and ZrNPs agglomeration inside the acrylic resin matrix, because the TiNPs and ZrNPs are difficult to disperse, due to the existence of potent forces between the particles of the nanomaterials named Van der Waal's forces [44]. The fair dispersion of TiNPs and ZrNPs in PEMA may be the reason for the decrease in hardness, as the degree of conversion is adversely affected and thus leads to an increase in the amount of residual unreacted monomer which acts as a plasticizer, [31,12] and also TiNPs agglomerated within the resin matrix can act as stress concentration centers that negatively affect the mechanical features of the material of the polymerized resin [45]. The results of FTIR analyses of the current study showed there was no chemical reaction between TiNPs and ZrNPs with the soft lining materials and this is supported by many previous studies that showed that TiO_2 and ZrO_2 are inert chemicals and rarely interact with other materials, so it was not a chemical reaction that might change the chemical composition of TiO_2 and ZrO_2 or the chemical composition of other mixed material or the surrounding environment [46- 49]. The absence of a chemical reaction between TiNPs and ZrNPs with the base material for acrylic dentures (PMMA) makes the added TiNPs and ZrNPs work as a barrier that can interfere to some degree with the chemical reaction between PEMA and PMMA, especially if TiNPs and ZrNPs are mixed with PEMA monomer, it will affect the monomer ability of diffusion into the PMMA [50, 51].

Surface roughness account as an important characteristic of polymers, especially if taking into account their role in the adhesion of microorganisms. The attachment of microorganisms to the basic materials of the denture base is the precursor to the colonization and development of diseases such as stomatitis. Therefore, these materials must be with smooth surfaces to prevent the adhesion of microorganisms and the formation of biofilms and the concomitant pathogenesis of oral mucositis [52]. The results of current study shown surface roughness of reinforced specimens with ZrO_2 NPs and TiNPs decrease comparing to the control group, this result may be due to that the surface roughness test is concerned with outer surface and not with inner surface using ZrO_2 and TiO_2 nanoparticles which are very small size and they were well dispersed in the polymer matrix, avoid agglomeration, and improves interfacial adhesion of the soft liner to the polymer matrix [53].

The experimental groups in this study exhibited a significant increase in the absorption of ultraviolet (UV) light comparing with the control group, and this can be assigned to the being of both TiNPs and ZrNPs in the polymer matrix, which have good optical and photocatalytic characteristics, besides their high efficiency to absorb ultraviolet light, especially since these nanoparticles were capable to absorb 95% of UV- light [54, 55]. According to the results of a study that examined the effect of ZrNPs on the transparency of the PMMA, the values of the reinforced groups were less than those of the control group [17]. Acrylic resin fortified with TiO_2 nanoparticles showed a considerable increase in opalescence without any change in the fluorescence spectra, and thus led to a significant decrease in transparency and an increase in color stability ($p < 0.05$) [56].

Some studies have shown that adding nanoparticles increases the lifespan of the reinforced materials, preserving their color and aesthetics, and this is due to the ability of TiNPs to inhibit the passage of ultraviolet light through them, and thus reduce color degradation of the pigments within the polymer and prevent discoloration [18,56]. The crystalline nature enjoyed of n-ZrO_2 (high opacity) contributed to absorbing ultraviolet light and preventing their passage, which in turn reduced the translucency, in addition to the fact that the cluster formations of n-ZrO_2 prevented the transmission of light and thus reduced the translucency. These data are consistent with the results of a study, which indicated the necessity of preventing the agglomeration of the used particles within the matrix, which in turn disrupts the diffuse reflection of the UV beam, which reduces the translucency [12].

CONCLUSION

The physical properties of soft liner denture base are influenced by the addition of nanoparticles as the reinforcing agent and yield a promising restorative material with favorable physical and anti-aging properties. A significant decrease in both hardness and surface roughness and a significant increase in light absorption. This effect is directly proportional to the concentrations of nanoparticle used.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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