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Original research article

Evaluation of tensile bond strength of nanoparticle reinforced soft liner materials: A pilot study

Esra Nur Avukat,[1] Miraç Berke Topcu Ersöz,[2] Canan Akay,[3,4,5] Emre Mumcu[4,5,6]

[1]Department of Prosthodontics, Faculty of Dentistry, The University of Osmangazi, Eskişehir, Türkiye, [2]Department of Prosthodontics, Faculty of Dentistry, The University of Osmangazi, Eskişehir, Türkiye, [3]Department of Prosthodontics, Faculty of Dentistry, The University of Osmangazi, Eskişehir, Türkiye, [4]Translational Medicine Research and Clinical Center, The University of Osmangazi, Eskisehir, Türkiye, [5]Advanced Material Technologies Application and Research Center, The University of Osmangazi, Eskisehir, Türkiye, [6]Department of Prosthodontics, Faculty of Dentistry, The University of Osmangazi, Eskişehir, Türkiye

Abstract

Objective: **The aim of this study is to evaluate the tensile bond strength between polymethyl methacrylate (PMMA) surfaces and autopolymerized silicon-based soft lining materials with 1% w/w Titanium dioxide (TiO_2) and Hydroxyapatite (HA) nanoparticles added.**

Materials and Methods: **For the tensile test, 60 pieces of acrylic (Meliodent, Bayer Dental, Newbury, England) samples of 30 × 10 × 10 mm3 dimensions were prepared using metal molds. Acrylic surfaces were sanded with silicon carbide sandpapers of 500, 1000, 1500, and 2000 grids to ensure standardization. After the samples were placed back in the metal mold, adhesive (Detax, Germany) was applied to the surfaces that would come into contact with the soft lining. Soft lining materials (Mollosil, Detax, Ettlingen, Germany) to which 1% by weight TiO_2 and 1% HA nanoparticles were added were polymerized by placing them between two acrylic blocks. For the tensile test, a total of 30 samples were obtained, with 10 samples in each group (n=10). The specimens were placed on the holder end of the universal test device and force was applied until failure occurred.**

Results: **The tensile bond strength (0,86 ± 0,21 MPa) in the TiO_2 nanoparticle-added group was found to be significantly higher than the control group (0,65 ± 0,14 MPa) ($p<0.05$). There is no significant difference between the control group and the HA nanoparticle-added group (0.65 ± 0.1 MPa) ($p>0.05$).**

Conclusion: **It was observed that the addition of nanoparticles increased the tensile strength. However, further studies are needed to evaluate the effect of nanoparticle addition on other mechanical and physical properties of soft liners.**

Keywords: **Nanoparticle; polymethylmethacrylate; soft lining; tensile bond strength.**

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Introduction

Polymethyl methacrylate (PMMA) is frequently used in the manufacturing of removable dentures because they are aesthetic, economical, easy to apply, have good fracture resistance, and are rigid.[1,2] Trauma occurs in the mucosa under the removable dentures, whose harmony with the tissue deteriorates over time. Soft liners are often preferred in the treatment of patients suffering from pain originating from traumatized oral mucosa. While they ensure that the forces coming during the function are evenly distributed to the soft tissues, they also absorb some of them. At the same time, they support healing and provide more comfortable use of the dentures.[3]

The main features that soft liners should have are biocompatibility, improved dimensional stability, adequate tear strength, long-term softness, viscoelasticity, easy cleaning, color stability, enhanced antimicrobial properties, strong bonding to the denture base material, low water absorption and solubility.[1] However, there is no ideal soft liner material. Generally, problems such as failure of adherence to the denture base, colonization of oral microorganisms, weak tear strength, and increased hardness over time occur.[3]

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*Corresponding author: Esra Nur Avukat, Department of Prosthodontics, Faculty of Dentistry, The University of Osmangazi, Eskişehir, Turkey; E-mail: dtesravukat@gmail.com

Soft liners must be well bonded to the denture base surface in order to function effectively. After it is used in the mouth, water and saliva absorption occur over time. This situation negatively affects the viscoelastic properties of soft liners, causing them to be more fragile and transmitting the incoming loads to the adhesion surface. Failures in the adhesion surface are among the most common clinical problems and cause an increase in dental calculus, plaque, and bacteria.[1] The clinically acceptable bond strength between the soft liner and denture base material should be 0.44 MPa.[3-5]

Nanoparticles are particles ranging in size from 1 to 100 nm. Since they have a large surface area, their physical and chemical properties are also developed. This allows them to bind strongly to different surfaces such as bacteria, plaque, and protein.[6-8] Nanoparticles are used in many areas of dentistry. Implantology (coating of dental implant surfaces with hydroxyapatite (HA) nanoparticles), improvement of dental material (addition of HA nanoparticles to cement, addition of silicon dioxide (SiO_2) nanoparticles to the soft liner material), and treatment of bone defects are the areas where nanoparticles are used.[6,7,9] It has been reported in previous studies that the addition of different nanoparticles (for example, silver, silicon dioxide, yttrium oxide, fluorescent carbon nanoparticles) to soft liners can affect physical, mechanical, and biological properties.[9-12] Han *et al.*[13] observed that the addition of titanium dioxide (TiO_2) nanoparticles to dental materials increased the tensile bond strength. However, it has been reported that the nanoparticle concentration should not be over 2.5%.[13] The addition of nanoparticles at increased concentrations causes the agglomeration of particles. Agglomeration points, as areas where stress is concentrated, negatively affect the mechanical properties of the material. Therefore, the nanoparticle concentration to be added should be carefully determined.[13] Gad *et al.*[9] reported that *Candida albicans* (*C. albicans*) adhesion, contact angle, and surface roughness decreased in their study where they added different concentrations of SiO_2 nanoparticles to the soft liner material. It was observed that the increase in nanoparticle concentration caused agglomeration, and an even distribution occurred when nanoparticles were added at low concentrations.[9]

There is limited information in the literature about the effects of adding TiO_2 and HA nanoparticles to soft liner materials on their physical and mechanical properties. The aim of this study is to evaluate the tensile bond strength between PMMA surfaces and auto polymerized soft liner materials with 1% by weight TiO_2 and HA nanoparticles added *in vitro*. The null hypothesis of the study is that the addition of 1% by weight TiO_2 and HA nanoparticles will not affect the tensile bond strength between PMMA and soft liner materials.

Materials and Method

For this *in vitro* study, 30 pairs (60 pieces) of acrylic specimens (Meliodent, Bayer Dental, Newbury, England) with dimensions of 30 × 10 × 10 mm^3 were first prepared using a specially manufactured metal mold. Acrylic surfaces were sanded with 500, 1000, 1500, and 2000 grit silicon carbide sandpapers in order to ensure standardization. After the specimens were placed back in the metal mold, adhesive (Detax, Ettingen, Germany) was applied to the surfaces that would come into contact with the soft liner. For the control group, soft liner material (Mollosil, Detax, Ettingen, Germany) was mixed in accordance with the manufacturer's instructions and applied to the gap between two acrylic blocks in the metal mold. During the preparation of nanoparticle-added groups, 1% by weight nanoparticle powders were included in the catalyst and base. After the materials were mixed homogeneously, they were pressed into a metal mold to ensure equal size and polymerized at room temperature in accordance with the manufacturer's instructions.

The appropriate number of specimens for the research was decided by taking into consideration the previous studies.[10] For the tensile test, a total of 30 specimens were obtained, with 10 specimens in each group (Table 1, Table 2). The specimens were stored in distilled water at 37°C for 1 week before the test. The specimens were placed on the holder end of the universal test device (Jinan Hensgrand Instrument, China) and force was applied until rupture occurred. Head speed was set as 5 mm/min as stated in the literature. Tensile was applied until rupture occurred (Figure 1). Values were recorded in MPa.[1]

Table 1. *Schematic representation of groups*

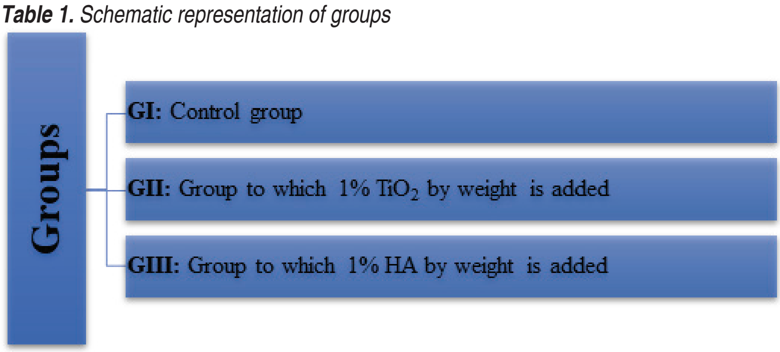

Table 2. *Materials and manufacturers used in the study*

	Manufacturer
PMMA	Meliodent (Bayer Dental, Newbury, UK)
Soft Lining	Mollosil (Detax, Ettlingen, Germany)
TiO_2 nanoparticles (20 nm)	Nanografi (Nanografi, Ankara, Türkiye)
HA nanoparticles (50 nm)	Nanografi (Nanografi, Ankara, Türkiye)

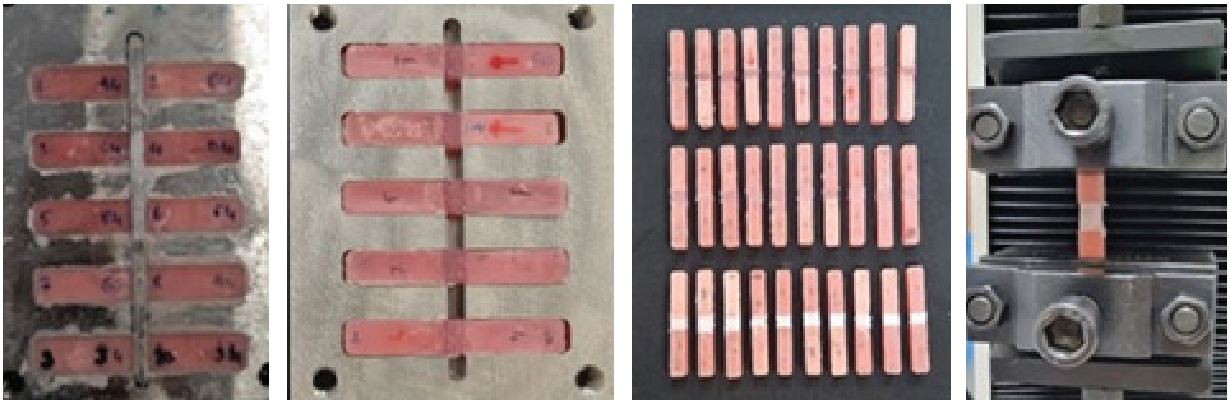

Figure 1. *a: Preparation of PMMA samples, b: Application and polymerization of the soft liner material, c: Test samples, d: Application of tensile bond strength test.*

Table 3. *Tensile bond strength values according to groups (Mean±Standard Deviation) (p<0.05)*

	GI	GII	GIII	Test statistic	p
TBS (MPa)	0,65 ± $0,14^{ac}$	0,86 ± $0,21^{b}$	0,65 ± $0,1^{c}$	6,475	**0,009**

One-Way ANOVA, (Welch) $^{a-c}$: There is no difference between groups with the same letter. (Tamhane's T2)

Table 4. *Failure modes in each group of specimens.*

Groups	Adhesive	Cohezive	Mix
GI	1	-	9
GII	2	-	8
GIII	1	-	9

The tensile bond strength (MPa) was calculated according to the following formula:

Tensile bond strength=Fmax/A, where Fmax is the maximum force (N) recorded during debonding, and A is the bonding surface area between the tested materials and PMMA resin (mm^2).

Statistical Analysis

IBM SPSS Statistics 22 program was used for statistical analysis while evaluating the findings obtained in the study. The Shapiro-Wilk test was used to test normality. Descriptive statistical methods were used while evaluating the study data (mean, standard deviation). One-way analysis of variance was used to compare the groups. Significance was evaluated at the $p < 0.05$ level.

Results

The tensile bond strength values for each group are presented in Table 3. The tensile bond strength in the TiO2 nanoparticle-added group (0,86 ± 0,21 MPa) was found to be significantly higher than the control group (0,65 ± 0,14 MPa) ($p \leq 0{,}019$). There is no significant difference between the tensile bond strength in the HA nanoparticle-added group (0.65±0.09 MPa) and the control group ($p \geq 0.976$). The tensile bond strenght in the TiO_2 nanoparticle-added group was found to be significantly higher than the HA nanoparticle-added group ($p \leq 0{,}011$). According to the results of the failure modes, mostly mixed failure was seen in all groups (Table 4).

Discussion

In the present study, the effectiveness of nanoparticles, which have recently increased in use in dental materials, was examined. As a result of, it was observed that the addition of nanoparticles increased the tensile strength and the null hypothesis was rejected.

TiO_2 nanoparticles are white, economical, low toxicity, and have enhanced photocatalytic and optical properties. In addition, they have enhanced antimicrobial features against bacteria and fungi. For this reason, TiO_2 nanoparticles are frequently added to dental materials.[14-17] TiO_2 nanoparticles added to the soft liners provided a reduction in surface roughness and hardness. While the decrease in roughness reduces the microorganism colonization, the decrease in the hardness increases patient comfort.[16]

HA is the main component of dental and bone tissues in humans.[7,8] HA nanoparticles are biocompatible. They are bioactive and thus their ability to adhere to surrounding tissues has improved. HA nanoparticles are used as glass ionomer cement in restorative dentistry and in the treatment of bone defects in oral surgery. They are also added to toothpaste due to their enamel-repairing effect.[6,8] In this study, the effect of TiO_2 and HA nanoparticles on the tensile bond strength between the soft liner and PMMA was evaluated. According to the data obtained, the hypothesis that the addition of nanoparticles to the soft liner material would not affect the tensile bond strength between the soft liner material and the denture base material was rejected.

The ideal soft liner thickness should be 2-3 mm.[3] Therefore, in this study, the soft liner thickness was prepared to be 3 mm. Bond strength values are commonly evaluated by tensile (tensile), shear, and peel bond strength test methods.[12,18] McCabe *et al.*[19] reported that the tensile test method is a suitable method to evaluate the bond strength of soft liners.[19] In this study, as in previous studies, the tensile test was applied.[20-22]

The most common problem encountered during the use of soft liner materials is the separation of the soft liner material from the denture base surface. This situation affects both function and hygiene negatively. For this reason, it has been reported that the tensile bond strength of materials for clinical use should be more than 0.45 MPa.[23] In this study, the tensile bond strength values obtained in all groups were found to be higher than 0.45 Mpa. According to the values obtained, it was observed that the addition of 1% by weight of TiO2 and HA nanoparticles to the soft liner material improved the bond strength. TiO_2 nanoparticles have strong surface interaction with the organic polymer. In addition to improving the physical and optical properties of the organic polymer, they are also resistant to environmental stresses that cause cracking and aging.[13] It can be thought that the increase in tensile bond strength in the group to which TiO_2 is added is due to this situation.

It is thought that the other reason for the increase in the tensile bond strength is due to the large surface area of the nanoparticles and the increased contact surface with the materials. This result Abdul-Baqi *et al.*[12] was found to be consistent with his work. It has been reported that the increase resulting from the addition of yttrium oxide nanoparticles may be due to the new bonds formed between the polymer and nanoparticles.[12]

Habibzadeh *et al.*[24] in their study, in which they added silver nanoparticles at different concentrations to the silicon-based soft liner material and evaluated the tensile bond strength, reported that the strength decreased as the nanoparticle concentration increased in the group that did not undergo thermal cycling. While the tensile bond strength obtained in the group to which 1% silver nanoparticle was added was 0.97 MPa, this value was higher than the values obtained in the 3 groups in our study. This may be due to the use of different materials. However, the different nanoparticles added and the different nanoparticle sizes may also have affected the situation. In addition, nanoparticles can aggregate. Aggregation acts as stress concentration points, which adversely affects the mechanical properties of the material.[24]

Ahmed *et al.*[15] reported that there was no change in shear bond strength values after the addition of 2% by weight TiO_2 nanoparticles. However, there was a significant reduction in *C. albicans* adhesion and hardness. The use of acrylic-based heat-polymerized soft liner material in this study may have led to different results. At the same time, it was reported that aggregation areas were observed in the SEM examination. The increase in the number of nanoparticles led to the formation of aggregation areas.[15]

The mean tensile values obtained in the present study were found to be close to the values of the study conducted by Köseoğlu *et al.*[25] which evaluated the tensile bond strength values between conventional and additive-produced base materials and different soft liner materials. The tensile bond strength of the group using silicone-based chairside soft liner and heat-polymerized PMMA was reported as 0.61 MPa. The average tensile bond strength between the additive-produced base material and the auto-polymerized soft liner was reported as 0.51 MPa. In our study, the tensile bond strength, which was 0.64 MPa in the control group, was found to be higher in the nanoparticle-added groups.[25] Today, there is a need for studies evaluating the tensile bond strengths between the current materials Computer-aided design / Computer-aided manufacturing (CAD / CAM), and additive production denture base materials.

The types of failures that occur are taken into account when interpreting the tensile strength test results. Failures in the bonding surface are classified as adhesive, cohesive, and mixed. Adhesive failures occur when the tensile strength of the soft liner is greater than the bond strength of PMMA. Cohesive failures occur when the tensile strength of the soft liner is less than the bond strength of PMMA. Mixed failures occur when the bond strength to PMMA is nearly equal to the tensile strength of the liner.[19] The different chemical contents of the materials lead to the appearance of adhesive failures. The reason for the adhesive rupture may be due to the soft liner material being silicone-based.[10]

Although there are studies in the literature evaluating the effect of HA nanoparticles on dental materials to the authors' knowledge there is no study evaluating the effect of soft liner materials on tensile bond strength. Therefore, an effective comparison of test results could not be made. In addition to being a pilot study with a limited number of samples and experimental groups, the use of a single soft liner material and PMMA and the absence of a thermal cycle are among the limitations of the study.

Conclusion

The tensile bond strength values obtained in the nanoparticle-added groups are above the clinically accepted bond strength value. In light of these data, further studies are needed to evaluate the effect of nanoparticle addition on other mechanical and physical properties of soft liners for clinical use.

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Nanopartikül ile güçlendirilmiş yumuşak astar materyallerinin çekme bağlanma dayanımının değerlendirilmesi: Pilot çalışma

Özet

Amaç: Bu çalışmanın amacı ağırlıkça %1 oranında Titanyum dioksit (TiO_2) ve Hidroksiapatit (HA) nanopartikülü ilave edilmiş otopolimerize silikon esaslı yumuşak astar materyalleri ile polimetil metakrilat (PMMA) yüzeyleri arasında meydana gelen çekme bağlanma dayanımını *in vitro* olarak değerlendirmektir.

Gereç ve Yöntem: Çekme testi için 30 × 10 × 10 mm3 boyutlarında 60 adet akrilik (Meliodent, Bayer Dental, Newbury, İngiltere) örnek metal kalıp kullanılarak hazırlandı. Akrilik yüzeyleri standardizasyonu sağlamak amacıyla 500, 1000, 1500 ve 2000 gridlik silikon karbid zımparalar ile zımparalandı. Örnekler metal kalıba tekrar yerleştirildikten sonra yumuşak astar ile temas edecek yüzeylerine adeziv (Detax, Almanya) uygulandı. Ağırlıkça %1 oranında TiO2 ve %1 oranında HA nanopartikülü ilave edilen yumuşak astar materyalleri (Mollosil, Detax, Ettlingen, Almanya) 2 akrilik blok arasına koyularak polimerize edildi. Çekme testi için her grupta 10 örnek olacak şekilde toplam 30 adet örnek elde edildi. Örnekler

evrensel test cihazının (Jinan Hensgrand Instrument, China) tutucu ucuna yerleştirilerek kopma meydana gelene kadar kuvvet uygulandı.

BULGULAR: TiO_2 nanopartikül eklenen grupta çekme bağlanma mukavemetinin (0.86 ± 0.21 MPa) kontrol grubuna (0.65 ± 0.14 MPa) göre anlamlı derecede yüksek olduğu belirlendi ($p<0.05$). Kontrol grubu ile HA nanopartikül eklenen grup arasında anlamlı bir fark görülmemektedir (0.65 ± 0.1 MPa) ($p>0.05$).

SONUÇ: Nanopartikül ilavesinin çekme dayanımını artırdığı gözlemlenmiştir. Bununla birlike nanopartikül ilavesinin yumuşak astarların diğer mekanik ve fiziksel özelliklerine olan etkisini değerlendiren ileri çalışmalara ihtiyaç duyulmaktadır.

ANAHTAR KELİMELER: Çekme bağlanma dayanımı; nanopartikül; polimetilmetakrilat; yumuşak astar