

Evaluation the Effects of Different Silica Coating Methods and Silane Types on the Bond Strength Between Polyamide and Polymethylmethacrylate

Farklı Silika Kaplama Yöntemleri ve Silan Tiplerinin Poliamid ve Polimetilmetakrilat Arasındaki Bağlanma Dayanımına Etkisinin Değerlendirilmesi

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ABSTRACT

Objectives: The aim of this study is to evaluate the effect of different silica coating methods and silane applications on the bond strength between polyamide and polymethylmethacrylate (PMMA).

Methods: Forty-five polyamide specimens were divided into two main groups based on the coating method applied: sol-gel silica coating (S) and tribochemical silica coating (T). Each main group was further divided into two subgroups based on the type of silane used: Bisco Porcelain Primer (BPP) and Clearfil Primer Plus (CPP) (n=9). The control group (C) received no silica coating or silane application. PMMA was then bonded to all specimens, and shear bond strength was evaluated.

Results: The highest shear bond strength was observed in group T with BPP application, while the lowest value was recorded in group C. Statistically significant differences were found between S-T and T-C groups ($P<.05$). Although BPP application in group T resulted in higher bond strength than CPP, the opposite trend was observed in group S.

Conclusion: The shear bond strength between polyamide and PMMA can be enhanced by applying silane after silica coating using either the tribochemical or sol-gel method.

Keywords: Polyamide, Polymethylmethacrylate, Silica

Öz

Amaç: Bu çalışmanın amacı, farklı silika kaplama yöntemleri ve silan uygulamalarının poliamid ile polimetilmetakrilat (PMMA) arasındaki bağlanma dayanımı üzerindeki etkisini değerlendirmektir.

Yöntemler: 45 poliamid örneği uygulanacak kaplama yöntemine (Sol-jel silika kaplama (S) ve tribokimyasal silika kaplama (T)) göre iki ana gruba ayrılmıştır. Her ana grup ise uygulanan farklı silan türlerine (Bisco Porcelain Primer (BPP) ve Clearfil Primer Plus (CPP)) göre iki alt gruba ayrılmıştır (n=9). Kontrol grubu (C) için ise herhangi bir silika kaplama veya silan uygulaması yapılmamıştır. Ardından hazırlanan tüm örnekler PMMA bağlanarak makaslama bağlanma dayanımı değerlendirilmiştir.

Bulgular: En yüksek makaslama bağlanma dayanımı grup T'de BPP uygulanan örneklerde bulunurken, en düşük değer grup C'de gözlenmiştir. Gruplar arasında S-T ve T-C arasında istatistiksel olarak anlamlı fark bulunmuştur ($P<.05$). Grup T'de BPP uygulanan örneklerin makaslama bağlanmadayanımı CPP uygulanan örneklerden daha yüksek olmasına rağmen, bu durum grup S'te ters yönde izlenmiştir.

Sonuç: Poliamid ile PMMA arasındaki makaslama bağlanma dayanımı, tribokimyasal silika kaplama veya sol-jel yöntemiyle silika kaplama sonrası silan uygulaması ile artırılabilir.

Anahtar kelimeler: Poliamid, Polimetilmetakrilat, Silika.

INTRODUCTION

Dental treatments of the geriatric patients are generally aimed at the prosthetic rehabilitation of their missing teeth with partial or complete dentures, for which many materials and manufacturing techniques are used in the construction of these dentures.¹ Currently, patients demand that their prosthetic restorations perform an excellent aesthetics in addition to good function.² Polyamide polymer denture bases, also described as nylon, were used for the first time in 1950 and became one of the remarkable denture base materials due to their advantages such as sufficient strength, high elasticity, no residual monomer and aesthetic appearance.³⁻⁷ In addition to the many advantages of polyamide-based prostheses, the weak bond strength to autopolymerizing resins is one of the ongoing problems.⁸⁻¹⁰ Denture fractures may develop due to temporary and large force such as an accident or repetitive and small forces

during chewing and in such cases, the repair of polyamide prostheses is more difficult than polymethylmethacrylate dentures. Fixing a broken tooth or clasp, repairing a denture fracture or relining a denture with polyamide materials is difficult and expensive. Sometimes making a new denture is a more appropriate approach than repairing the old one.^{6,11} Unlike polymethylmethacrylate (PMMA), polyamide materials are resistant to chemicals due to their dense crystalline content. Therefore, it is difficult for polyamides to react and repair with chemical binders. The bond strength between polyamide and other materials can be increased with some surface preparations.¹² While many surface improvement methods have emerged and proven to be effective, in order to provide compatibility and repair between conventional heat-polymerizing resins and autopolymerizing resins, which are routinely used in prosthetic treatments today, a few studies have aimed to develop polyamide bases.^{8,13} There is a need for scientific studies on repair and strengthening of these prostheses, which have exhibiting better features compared to traditional dentures, in order to avoid the burden of time and cost by making them again in case of any failure. For these reasons, it was decided to conduct such a study.

METHODS

Since the relevant study is a material research, there is no need for an ethics committee decision. Table 1 shows the composition of the materials used in this experiment. Table 2 shows the experimental and control groups.

Table 1. Materials used in the study

Material	Chemical content	Brand-Manufacturer
Polyamide base	Poliamid	Deflex Nuxen S.R.L Buenos Aries, Argentina
Autopolymerizing acrylic resin	Polimetilmetakrilat	Blau Cryl, Efes Dental Bursa,Turkey
Silane coupling agent	3 Trimethyloxysyl propyl-2 Methyl propenoic acid	Bisco Porcelain Primer, Bisco, Schaumburg II, USA
Silane coupling agent	3Trimethyloxypropyl methacrylate 10- Methacryloyloxydecyl dihydrogen phosphate	Clearfil-Primer, Kuraray, Osaka, Japan
Silica-containing sand	Silica and 30 µm Al ₂ O ₃	Cojet Sand, 3M ESPE, Seefeld, Germany
Cylindrical tube with an inner diameter of 17 mm, an outer diameter of 25 mm and a height of 30 mm		
Cylindrical tube with an inner ring diameter of 4mm and a height of 2mm		
#100, #400, #600, #800 and #1000grid size silicone carbide abrasive papers	Silicone carbide	Waterproof silicon carbide paper, UK

Table 2. Experimental and control groups in the study

Polyamide samples (n=45)			
T:Tribochemical silica coating group (n=18)	S:Sol-gel silica coating group (n=18)	C:Control group (n=9)	
TC: Clearfil Primer Plus (CPP) group (n=9)	SC: Clearfil Primer Plus (CPP) group (n=9)	No silica or silane coupling agent was applied	
TB: Bisco Porcelain Primer (BPP) group (n=9)	SB: Bisco Porcelain Primer (BPP) group (n=9)		

Preparation of polyamide samples

For use in the study, 45 disc-shaped polyamide samples with a diameter of 10 mm and a thickness of 2 mm were manufactured from injection molded thermoplastic polyamide resin (Deflex, Nuxen SRL,

Argentina). The formed polyamide samples were fixed in previously prepared plastic pipes (Inner diameter of 12 mm, outer diameter of 24 mm and a height of 30 mm. cylinder shaped) with the help of autopolymerizing acrylic (BLAU CRYL, Efes Dental, BURSA). Fixed samples were polished under water using #100, #400, #600, #800 and #1000 grid size silicone carbide abrasive papers. The prepared samples were kept in an ultrasonic cleaner (VEVOR, China) for 10 minutes at 25°C. The samples were removed from the ultrasonic cleaner and dried with air.

Dividing the samples into groups according to the surface treatment to be applied

The samples were divided into two main groups (n=18) to apply sol-gel silica coating (S) and tribochemical silica coating (T) treatment, and each main group was divided into two subgroups (n=9) to apply different silane material. No silica coating or silane application was applied for the control group (C).

Silica coated group with sol-gel (S)

Sol solution was prepared by acid-catalyzed hydrolysis of tetraethylorthosilicate (TEOS). TEOS was dissolved in ethanol and deionized water, then acetic acid was added until a molar ratio of 1 TEOS: 30 ethanol: 5 water: 2 acetic acid was reached. This reaction mixture was stirred continuously for 2 hours until a clear and homogeneous solution was obtained and finally the solution was aged at 20 to 25°C for 24 hours. The samples were immersed in this prepared TEOS sol and UV irradiation was carried out under O₂ environment and approximately 1 cm below the light source for 30 min. TEOS coated samples were divided into two subgroups (n=9) and silane coupling agent with different chemical content was applied to each subgroup. 1st group (SB): 3-(Trimethoxysilyl) propyl-2-Methyl-2-Propenoic Acid containing silane (Bisco Porcelain Primer, Bisco, Schaumburg II, USA), 2nd group (SC): 3-trimethoxysilylpropyl methacrylate, 10-Methacryloyloxydecyl dihydrogen phosphatic silane (Clearfil-Primer, Kuraray, Osaka, Japan) was applied.

Tribochemical silica coated group (T)

The samples in this group (n=18) were sandblasted by containing silica and 30 µm Al₂O₃ particles (Cojet Sand, 3M ESPE, Seefeld, Germany) at 4.2 psi pressure from 10 mm. Distance for 15 s with an intraoral blasting device (Prophy-neo Partner, Liang ya Dental, China). Sandblasted samples were divided into two subgroups (n=9) and a different silane coupling agent was applied to each subgroup. 1st group (TB): 3-(Trimethoxysilyl) propyl-2-Methyl-2- Propenoic Acid containing silane (Bisco Porcelain Primer, Bisco, Schaumburg II, USA), 2nd group (TC): 3-trimethoxysilylpropyl methacrylate, 10-Methacryloyloxydecyl dihydrogen phosphatic silane (Clearfil-Primer, Kuraray, Osaka, Japan) was applied.

Connecting polyamide and autopolymerizing resin

A cylindrical tube with an inner circle diameter of 4 mm and height of 2 mm will be placed in the center of the polyamide discs for the standard application of autopolymerizing acrylic and this tube will be expected to be polymerized by placing autopolymerizing acrylic with a brush inside. After polymerization, the pipe around the autopolymerized acrylic was cut vertically with the help of a scalpel and removed precisely.

Thermal cycle application

All of the samples (n=45) were kept in an automatic thermal cycle device (Mod Dental, Ankara, Turkey) for 20 seconds at each temperature (5-55°C) and for 5 seconds between two temperatures for 1000 cycles.

Measuring shear bond strength

Acrylic molds are fixed to the test device in such a way that they do not move and the breaking apparatus comes into contact with the polyamide discs and the autopolymerizing acrylic junction at a right angle. Shear tests were performed with a 0.5 mm/min approach speed using a universal test device (Mod dental, Ankara, Turkey) and the test was monitored as tension and percent elongation graphs on the computer screen. The test was stopped automatically when separation occurred and the results were calculated by the computer in MPa.

Statistical analysis

In the statistical analysis of the study SPSS V25. (IBM SPSS Corp., Armonk, NY, USA) package program was used. Descriptive statistics of categorical variables in the study; descriptive statistics for frequency and percentage and continuous variables; are given with mean, standard deviation, median, minimum and maximum values. The conformity of continuous variables to the normal distribution was examined using the Shapiro Wilk test. The Oneway Anova Test was used to compare groupings with more than two options with normal distribution. In the 3-group comparisons of continuous variables that do not show normal distribution; Kruskal Wallis test and Mann-Whitney U tests were used in comparison of two groups. Relationships between continuous variables were examined by Spearman correlation analysis. In all statistical comparisons in the study, comparisons with a p value below 0.05 were considered statistically significant.

RESULTS

The results of the shear bond test for the main and subgroups are shown in Figure 1 and 2, respectively. The results of the Kruskal Wallis test performed to compare the main groups are given in the Table 2. According to the analysis results, there was a statistically significant difference between the main groups ($P < .05$). Bonferroni Corrected Mann Whitney test results show that there was a statistically significant difference between groups S-T and T-C. Shear bond values of the subgroups are given in Table 3, according to the table, the highest bond strength was found in group TB and the lowest bond strength is in control group. The results of the Kruskal Wallis test performed to compare the average shear bond strengths of the subgroups are given in Table 4. According to the analysis results, there was a statistically significant difference between the groups. Bonferroni Corrected Mann Whitney test results show that the difference between the SB group and the TB group and the difference between the TB and C groups were statistically significant. Mann Whitney test results are given in table 5 in order to compare all groups directly with each other. According to the results of the analysis, there were statistically significant differences between SC-TB, SB-TC, SB- TB, TC-C and TB-C.

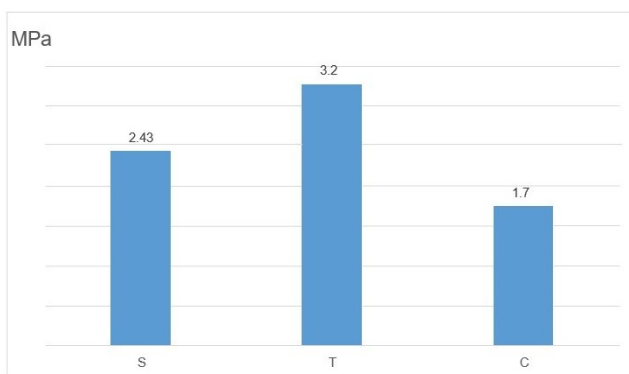


Figure 1. Mean shear bond values of main groups

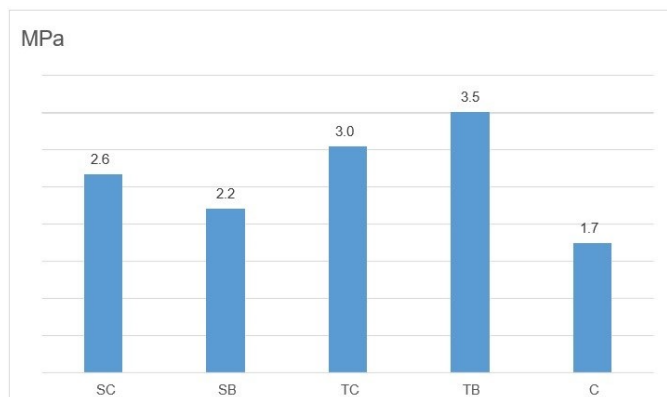


Figure 2. Mean shear bond values of subgroups

Table 3. Comparison of shear bonding values of main groups with Mann-Whitney U test with Bonferroni Correction

	Min	Max	X	ss	Median	X ²	P	Difference
S	0.45	7.39	2.43	1.45	2.22	15.359	<.01	T-C, S-T
T	1.98	4.50	3.27	0.70	3.22			
C	0.55	3.84	1.74	1.11	1.08			

*Statistical significance

S: Silica-coated group with sol-gel, T: Tribochemical silica-coated group, C: Control group

Table 4. Comparison of shear bonding values of main groups with Mann-Whitney U test with Bonferroni Correction

	Min	Max	X	ss	Median	X ²	P	Difference
SC	0.45	7.39	2.66	1.94	2.17	16.262	<.01 *	SB-TB, TB-C
SB	0.58	3.01	2.20	0.77	2.33			
TC	1.98	4.50	3.04	0.74	3.09			
TB	2.72	4.31	3.50	0.60	3.62			
C	0.55	3.84	1.74	1.11	1.08			

*Statistical significance

SB: Group coated with silica by sol-gel method followed by Bisco Porcelain Primer, SC: Group coated with silica by sol-gel method followed by Clearfil-Primer, TB: Group with tribochemical silica coating followed by Bisco Porcelain Primer, TC: Group with tribochemical silica coating followed by Clearfil-Primer, C: Control group, Min: Minimum, Max: Maximum, SD: Standard deviation

Table 5. Direct comparison of the mean shear bonding values of all subgroups with each other

	Min	Max	X	ss	Median	Mann-Whitney U Test (P < .05)
SC	0.45	7.39	2.66	1.94	2.17	SC-TB
SB	0.58	3.01	2.20	0.77	2.33	SB-TC
TC	1.98	4.50	3.04	0.74	3.09	SB-TB
TB	2.72	4.31	3.50	0.60	3.62	TC-C
C	0.55	3.84	1.74	1.11	1.08	TB-C

SB: Group coated with silica by sol-gel method followed by Bisco Porcelain Primer, SC: Group coated with silica by sol-gel method followed by Clearfil-Primer, TB: Group with tribochemical silica coating followed by Bisco Porcelain Primer, TC: Group with tribochemical silica coating followed by Clearfil-Primer, C: Control group, Min: Minimum, Max: Maximum, SD: Standard deviation

DISCUSSION

This study was conducted to repair polyamide-based prostheses, which are superior to PMMA prostheses in terms of aesthetics, biocompatibility, and mechanical properties. While PMMA-based prostheses applied today have an acceptable repair application with fast

and inexpensive methods when they fail mechanically for several reasons, this is not the case for polyamide prostheses. This is because polyamide-based dentures resist chemical substances due to their dense crystalline content.¹²

PMMA-based materials are applied with increasing interest in prosthetic treatments due to their simple manipulation technique, low toxicity, low water absorption and solubility, repairability and aesthetic appearance.¹⁴ In addition to these advantages, dentures have disadvantages, such as insufficient surface hardness, weak brittleness, and low fracture toughness.¹⁵ To repair the PMMA dentures, heat-polymerized, auto polymerized or light-polymerized acrylic resins can be used.¹⁶ The prosthesis is exposed to repetitive bending due to the chewing function in the mouth, and cracks or fractures may occur in the denture base due to fatigue developed. To reduce this, dentures must have high bending strength and resistance.¹⁷ Polyamide-based dentures have high flexibility capacity and high impact strength, but when some mechanical problems develop in polyamide dentures, repairing these prostheses is more complicated than PMMA.^{6,18}

Silica, found in two main types crystalline and amorphous, is the final product formed by the polymerisation of silicon dioxide monomers.¹⁹ There are various silica coating methods, such as tribochemical silica coating or silica coating with sol-gel, to increase the bonding of dental materials. Studies show that the silica-containing layer increases the reactivity of the polyamide material without impairing its mechanical properties.^{13,20} Tribochemical silica coating, a chemical bonding method with mechanical energy, is frequently used in the laboratory or clinic to provide the surface roughness of dental materials. In this method, silica-coated Al₂O₃ particles with an average particle size of 30 or 110 µm are used for 15 seconds at a pressure of 280 kPa (2.8 bar) and from a distance of 10 mm. With the high heat energy generated, a rough area up to 15 µm in size occurs within the material. The next step after roughening is silanisation. The aim is to form a thin layer of silica-containing enough free hydroxyl groups to bond with the resin. To increase the bond of the roughened area with the resin, a silica layer containing free hydroxyl groups should be formed; for this purpose, silanisation should be provided after the tribochemical silica coating process.^{21,22} Tetra alkoxysilane sol-gel polymerisation is a method used to synthesise amorphous silica gel that transforms into monolithic silica glass under hypercritical conditions.¹³ In the literature, there are studies in which silica coating was applied to the polyamide surface by tribochemical or sol-gel method to increase the bond strength between polyamide and PMMA. In their study, Kumbuloglu et al.²³ concluded that the tribochemical silica coating applied to the surface of polyamide samples increased the bonding with autopolymerizing PMMA. Hamanaka et al.²⁴ in their study on the adhesion strength of autopolymerizing acrylic and injection moulded thermoplastic denture bases, concluded that the best surface improvement method was tribochemical silica coating and 4-META/MMA-TBB application. In their study, Hafezeqoran and Koodaryan reported that the repair strength between autopolymerizing acrylic and polyamide increased with the application of aminofunctional silica coating.¹³ Although such studies are in the literature, studies comparing different silica coating methods or different silane types applied after silica coating have not been found. In this study, polyamide samples were divided into two main groups, tribochemical silica coating and sol-gel silica coating, and each main group was divided into two subgroups according to the type of silane to be applied to it. In the literature, autopolymerizing PMMA was used in a study on the transverse strength of polyamide-based prostheses and another study on the repair of polyamides.^{25,26}

Autopolymerizing PMMA resin, used in repairing dental prostheses, was also used in our study. The thermal cycle process applies thermal changes in the intraoral region under in-vitro conditions.²⁷ ISO TR 11450 (1994) standards state that the thermal cycle applied with 500 cycles in water tanks with a temperature of 5-55 °C is appropriate; sometimes, this cycle number is not enough. To imitate the temperature in the oral environment, the polyamide specimens with autopolymerizing acrylic bonding were kept for 20 seconds at each temperature and 5 seconds between two temperatures (5-55 °C) for 1000 cycles by the literature.

The shear test, frequently used to measure the bond strength, is an in-vitro test method created by applying a force to the joint area at a speed of 0.5mm/min until the joint is separated between two bonded materials. The shear bond value is found by dividing the maximum force applied by the joint area.²⁸⁻³⁰ The samples in which the thermal cycle process was completed were subjected to a shear bond strength test in the universal testing device as stated in the literature. The group with the lowest shear bond strength was found to be C, while the highest value belonged to the TB group. The reason for the lowest value in group C is that PMMA is attached to the surface of the polyamide samples without any surface preparation. Some surface treatments should be applied to polyamides, which are resistant to chemical substances due to their dense crystalline content, to increase their bond with other materials.¹² Another reason for the low shear bond value seen here is that PMMA and polyamide are polymers with different chemical contents. In the TB group treated with Bisco silane after tribochemical silica coating, the average shear bond value was 3.49 MPa. Hamanaka et al.²⁴ in their study in which they examined the bond strength between polyamide and autopolymerizing PMMA, applied ESPE Sil silane and 4-META/MMA-TBB to the samples after tribochemical silica coating and reported the average shear bonding value as 16.44 Mpa. The mean shear bond value was 13.41 MPa in the group where only tribochemical silica and ESPE silane were applied. These values are well above the highest shear bond value we found in our study, and this difference is thought to be due to the use of ESPE-Sil silane and 4-META/MMA-TBB-based resin cement after tribochemical silica coating. In the same study, the average shear bond value was found to be 0.44 MPa in the group that did not undergo any surface treatment; this value was 1.73 MPa on average in group C in our study, which did not undergo any surface treatment, and the values were more compatible with each other. In another study evaluating the bond strength between autopolymerized resin and polyamide base, the average shear bond strength of polyamide samples applied ESPE-Sil silane after tribochemical silica coating was 13.18 MPa.⁸ This value was much higher than the average values found in our study; in this case, the reason might be the ESPE-Sil silane application. Kumbuloglu et al.²³ evaluated the bond strength of polyamide samples, on which ESPE-Sil silane applied to tribochemical silica coated samples, with two different autopolymerizing resins, found the bond strength value of the autopolymerizing resin in one group was 4.99MPa. In contrast, the bond strength in the other group was 3.27 MPa. In our study, in the TB group treated with Bisco silane after tribochemical silica coating, the average shear bond value was found to be 3.49 MPa. In comparison, the same value was found as 3.03 MPa in the group treated with Clearfil silane, and the values found agree with the results reported by Kumbuloglu et al.²³ When the literature is examined, reports state that applying tribochemical or sol-gel silica coating increases the bond strength between polyamide and autopolymerizing PMMA.^{13,23,24} As a result of the study, shear bond values between autopolymerizing PMMA and polyamide samples coated with silica by tribochemical or sol-gel were higher than the

control group without any surface treatment. It is reported in the literature that the homogeneity and purity of sol-gel-derived silica film are comparable or even superior to surface coatings obtained by other methods. Hafezeqoran and Koodaryan, in their research to improve the adhesion of polyamides with autopolymerized resins, formed a double layer silica layer with aminosilane and tetraethylorthosilicate (TEOS) on polyamide surfaces by sol-gel method. As a result of the study, they found that the mean shear strength value of double-layer silica-coated polyamide bases with autopolymerized resin was 20.86 MPa.¹³ In our study, the mean shear bond values of the silica-coated sol-gel groups were 2.20MPa for the SB group and 2.66MPa for the SC group. It can be thought that this situation is caused by the formation of a single-layer silica surface instead of two layers in our study and the chemical differences in the autopolymerizing resins used.

The silane forms a chemical bond with the silica-coated surface.²³ Silane application after silica coating improves the bond strength between non-silica-based and various materials.²⁰ In a study evaluating the bond strength of silica-coated titanium surfaces with adhesive cement, a total of five different silane groups were studied, and the group in which the Bisco porcelain primer was applied was one of the groups that showed the best bond strength.³¹ In another study examining the bond strength of composite resins with zirconium and titanium, it was concluded that applying Clearfil Ceramic Primer on titanium and zirconium samples increased the bond strength with the composite.³² In the study, silica-coated samples with either the tribochemical method or the sol-gel method were divided into different subgroups, and silane coupling agents with different chemical contents (Bisco Porcelain Primer and Clearfil-Primer), which were proven to have a positive effect on the bond strength of dental materials, were applied to each subgroup. In the tribochemical sandblasting group, the shear bond value of the TB subgroup was higher than the TC subgroup. In the sol-gel coating group, the shear bond value of the SC subgroup was higher than the SB subgroup, but the difference between them was not statistically significant. Htami et al.³³ in their study examining the bond strength between composite and zirconia, concluded that Bisco and Clearfil had a similar effect on bond strength.

Within the limitations of this study, the following conclusions and recommendations can be reached;

1. The bond strength between polyamide and polymethylmethacrylate can be increased by surface treatments.
2. The shear bond value between polyamide and polymethylmethacrylate was increased by tribochemical silica coating or silica coating with sol-gel followed by silane application.
3. Tribochemical silica coating and silane-applied polyamide samples were found to have higher shear bond strength with PMMA than sol-gel silica-coated, and silane-applied samples.
4. Although the shear bonding value of BPP-applied samples in group T was higher than the applied samples, this situation was opposite in group S.
5. In future studies, the bond strength between polyamide and polymethylmethacrylate can be evaluated with different silanes applied after different surface coating techniques.

Etik Komite Onayı: Bu çalışma etik kurul onayı gerektirmemektedir.

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