



Effect of Thermocycle and Bonding Agents on the Bond Strength of Titanium-resin Cements

Titanyum-rezin Siman Bağlanma Dayanımına Isıl Döngü ile Yaşlandırmanın ve Bonding Ajanlarının Etkisi

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ABSTRACT

Objective: This research aimed to evaluate the effect of different resin cement (RC) types, compare the effect of surface treatments and bonding applications and evaluate the effect of thermal cycling on bond strengths to Titanium (Ti) surfaces.

Methods: A total of 240 Ti discs (10x3 mm) were randomly divided into two groups. Half of the specimens were sandblasted with 110 µm Al₂O₃ particles, whereas the other half had no surface treatments (non-treated). Both sandblasted and non-treated specimens of each surface treatment type were divided into five subgroups, which received one of the following surface conditions and luting self-adhesive resin cement: (a) Panavia SA Cement, (b) Clearfil SE Bond + Panavia SA Cement, (c) RelyX U200, (d) Single Bond Universal + RelyX U200 and (e) MIS Crown Set Cement. A mould with a 4-mm diameter and 2-mm thickness was applied to the central region of the specimens. Each group was divided into subgroups, according to whether performing thermocycling or not. The shear bond tests were conducted at a crosshead speed of 1 mm/min. Data (N) were analysed using one-way analysis of variance and Tukey's honestly significant difference tests (p<0.05).

Results: The sandblasted + bonding agent groups provided higher shear bond strength than the non-treated groups for all RC types (p<0.05). Sandblasted Clearfil SE Bond + Panavia SA Cement (non-thermocycled) showed the highest values (182.761±41.55), whereas the MIS Cement (17.681±9.33) and Panavia SA Cement

ÖZ

Amaç: Bu çalışmanın amacı farklı rezin simanların, kumlama yüzey işleminin, bonding ajanlarının ve yaşlandırmanın Titanyum (Ti)-rezin siman (RS) arasındaki makaslama bağlanma dayanımına etkisinin değerlendirilmesidir.

Yöntemler: İki yüz kırk adet Ti disk (10x3 mm) yüksekliğinde olacak şekilde Ti bloklardan kazınarak hazırlandı ve akril reçine içerisine gömüldü. Rastgele olarak 2'ye ayrılan Ti disklerin yarısına 110 µm Al₂O₃ ile kumlama işlemi yapıldı, yarısına herhangi bir yüzey işlemi uygulanmadı. Hem kumlanmış hem de yüzey işlemsiz olan bu iki grup da 5 alt gruba ayrıldı. a) Panavia SA Cement (Kuraray) b) Clearfil SE Bond (Kuraray) + Panavia SA Cement (Kuraray) c) Rely X U-200 (3M-Espe) d) Single bond Universal + Rely X U-200 (3M-Espe) and Mis Crown Set Cement (MIS). RS'ler özel bir kalıpla 4 mm çapında ve 2 mm kalınlıkta olacak şekilde Ti disklerin ortasına yerleştirildi. Sonrasında her grup kendi içerisinde yaşlandırma uygulanıp uygulanmamasına göre 2 alt gruba daha ayrıldı. Makaslama bağlanma dayanımı testleri 1 mm/dk hızla yapıldı. Veriler one-way ANOVA ve Tukey HSD testi kullanılarak istatistiksel olarak analiz edildi.

Bulgular: Gruplar arasında anlamlı bir fark vardır (p<0,05). Tüm rezin siman grupları için kumlanmış ve bonding ajan uygulanmış gruplar, yüzey işlemsiz gruplara göre daha yüksek bağlanma dayanımı göstermiştir (p<0,05). Kumlama + Clearfil SE Bond (Kuraray) + Panavia SA Cement (yaşlandırma uygulanmamış) grup en yüksek bağlantı değerini (182,761±41,55) gösterirken,

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(15.32±7.38) non-treated (thermocycled) groups had the lowest values.

Conclusion: Sandblasting and bonding agents can improve bond strength. The thermocycling period decreased the bond strength values for all groups.

Keywords: Titanium, resin cement, thermocycle, bond strength, semipermanent cements, methacryloyloxydecyl dihydrogen phosphate

MIS Cement (17,681±9,33) ve Panavia SA Cement (15,32±7,38) yüzey işlemsiz (yaşlandırma uygulanmış) gruplar en düşük değerleri göstermiştir.

Sonuç: Kumlama ve bonding ajan uygulaması bağlanma dayanımı arttırmıştır. Isıl döngü ile yaşlandırma tüm gruplar için bağlantı değerlerini düşürmüştür.

Anahtar Sözcükler: Titanyum, rezin siman, termal siklus, bağlantı dayanımı, semipermanent simanlar, MDP

Introduction

The durability of the connection between the prosthetic superstructure and implant is an essential factor for the longevity of implant-supported fixed denture prostheses (FDPs). This integrity is achieved by either cement or screw retention, which has advantages and disadvantages compared with one another (1). In today's dental practice, the choice of cement versus screw retention of implant-supported FDPs mostly depends on the clinicians' experience and preference with respect to the clinical situation (2).

One advantage of cement-retained implant-supported FDPs is the compensation of improperly placed implants. Especially in the anterior region, it is often impossible to manage aesthetics due to the visibility of the screw access hole (3). Moreover, it is observed that clinicians prefer cemented implant restorations because of their lower complication rate and higher fracture resistance of veneering porcelain (4,5). The situation becomes more challenging if the basic mechanical parameters are not optimum as well, such as reduced abutment/restoration interface and over tapered abutment due to the interarch tooth relations, especially for a single implant-retained crown, which might probably result in decementation, although permanently cemented. However, it is often preferred to make temporary cementation for cement-retained FDPs to maintain retrievability without damage to abutment or implant. However, temporary cementation might result in debonding, especially for restoring reduced abutments because of poor physical properties such as decreased tensile strength and increased solubility (6,7). According to this problem, using permanent cements, including polycarboxylate cement and self-adhesive resins, seems appropriate for cementing fixed implant-supported prostheses due to their high retentive values and lower retention loss risk (8,9). Several studies have also reported the unexpectedly high bonding values of polycarboxylate cement with Titanium (Ti) structures and indicated that some dental cements, including glass ionomer and polycarboxylate, alter the protective Ti oxide layer, resulting in colour changes (10). Even the instructions for using one polycarboxylate cement state that a discoloration effect may result when used with Ti. Thus, it is necessary to use resin cement (RC) for Ti cementation, especially when applying Ti to new areas for aesthetics, such as two-component abutments.

To get the advantages of both cementation types, semipermanent cements that provide the reduction of retention by using

petroleum jelly or acrylic with polyurethane resin are advised by various manufacturers, particularly for cementing implant-supported crowns for adequate retention and easy restoration removal (11,12). For both semipermanent and permanent cement types, surface treatments are an important effect for clinical longevity. Several studies about the bond strength of Ti and the effect of different surface treatments were published in the literature (13-15). Micromechanical and chemical bonding effects were compared with these studies (14). Sandblasting with Al₂O₃ particles is the most commonly used method for micromechanical retention promotion and chemical bondings, and it can be achieved by both bonding mechanisms, such as silica coating systems (coJet and Rocatec) and metal primers (16,17). Several surface treatment combinations are possible, so studies investigate the effects of different cementation type protocols. But there are no guidelines on the most appropriate luting procedure between the Ti-RC bond strengths.

Thermocycling can simulate the effect of destructive oral conditions with temperature changes and masticatory forces (18). Therefore, this study aimed to evaluate and compare the effect of surface treatments on shear bond strength (SBS) of two self-adhesive RCs and one semipermanent cement to Ti surfaces before and after thermocycling. The null hypothesis tested was that the RCs tested provide similar bond strength to the non-treated Ti surfaces and the surface modification of Ti enhanced the bond strength values for all types of RCs.

Methods

A total of 240 Ti discs (10 mm in diameter and 3 mm in height) were fabricated and embedded in acrylic resin blocks. A total of 20 groups were planned for this study, with n=12. Firstly, 240 Ti discs were randomly divided into two groups; half of the specimens were sandblasted with 110 µm Al₂O₃ particles, and the other half had no surface treatments (non-treated). Both sandblasted and non-treated specimens were divided into five subgroups, which received one of the following surface conditions and self-adhesive RCs: (a) Panavia SA Cement (Kuraray Noritake Dental Inc., Okayama, Japan), (b) Clearfil SE Bond (Kuraray Noritake Dental Inc.) + Panavia SA Cement, (c) RelyX U200 (3M-Espe, MN, USA), (d) Single Bond Universal + RelyX U200 and (e) MIS Crown Set Cement (MIS, Israel). The materials used in this study are listed in Table 1.

Bonding and Testing Procedures

The Ti discs were ultrasonically cleaned in 96% isopropanol for 3 min, followed by air drying. A mould with a 4 mm diameter and 2 mm thickness was placed on the central area of each of the Ti surfaces. RCs were applied into the moulds with or without using the relevant bonding agents for the determined subgroups, and the cementation procedures were completed according to the manufacturer’s instructions (Figure 1). For the bonding groups, a bonding agent was applied for 10 s, air-dried for 5 s and light-cured for 20 s at a 5 mm distance from the sample’s surface and at a 1,200 mW/cm intensity (BluePhase curing light, Ivoclar Vivadent, Liechtenstein) for polymerisation. The RC application procedure was the same for all bonding and non-bonding groups, and RC was applied with the same Teflon mould. The samples were light-cured for 5 s with the same distance and light source for initial polymerisation. After gently removing the Teflon mould, each side of the RC cylinders was light-cured for 20 s. The bonding process was performed as recommended by the manufacturers. The bonded specimens were stored in distilled water at 37 °C for 24 h, and subsequently, each group was again divided into two subgroups, according to whether performing thermocycling or not. The thermocycling procedure was set as 5,000 cycles between 5 °C and 55 °C (Thermal Cycler Tester, Dental Teknik, Konya, Turkey); the dwell and transfer times were 30 and 10 s, respectively. The SBS tests were performed using a universal testing machine (TSTM 02500, Elista Ltd., Şti., Istanbul, Turkey) at a 1 mm/min crosshead speed via a knife-edge rod. The failure loads were in N. The failure modes were

analysed under a stereomicroscope (Olympus SZ40, Olympus Optical Co., Tokyo, Japan) at 40x magnification. The failures were classified as adhesive, cohesive or mixed failure.

Statistical Analysis

Statistical package SPSS software (version 21.0, SPSS Inc., Chicago, IL, USA) was used at a significance level of $\alpha=0.05$. The Kolmogorov-Smirnov statistical test was performed on the SBS values to evaluate the normal distribution and homogeneity of variances. One-way analysis of variance and Tukey’s honestly significant difference test were conducted to determine statistical differences in the SBS values between subgroups, and an independent paired t-test was applied to determine the effect of thermocycling on SBS values.

Results

Table 2 lists the mean, median and standard deviation of the SBS values and summarises the results of the statistical tests that indicated significant differences between groups ($p<0.05$). The differences between the groups are shown in Table 1. Sandblasted and sandblasted + bonding agents provided significantly higher SBS compared with the non-treated and non-treated + bonding agent groups for all RC types ($p<0.05$), except for MIS Crown Set Cement ($p>0.05$). In both non-thermocycled and thermocycled specimens, the sandblasted Clearfil SE Bond + Panavia SA Cement group showed the highest values, whereas the non-treated Panavia SA Cement thermocycled (15.32 ± 7.38) and non-treated MIS Cement thermocycled (17.681 ± 9.33) groups showed the lowest values.

The highest SBS values were recorded for the Panavia sandblasted + bonding agents than the other groups after thermal cycling ($p<0.05$). However, there was a significant effect on applying bonding agents on sandblasted or non-treated surfaces for only Panavia cement for both thermal and non-thermal conditions ($p<0.05$). Additionally, sandblasting alone created higher SBS values for Panavia and RelyX cements compared with applying bonding agents only and non-treated surfaces ($p<0.05$). The SBS values of the sandblasted and sandblasted + bonding agent Panavia and RelyX groups were significantly different from those in the non-treated and non-treated + bonded groups ($p<0.05$).

Also, for the thermocycled groups, the Panavia SA Cement non-treated group showed the lowest SBS values, followed by the MIS Crown Set Cement non-treated, MIS Crown Set Cement sandblasted and RelyX Cement non-treated groups. But there

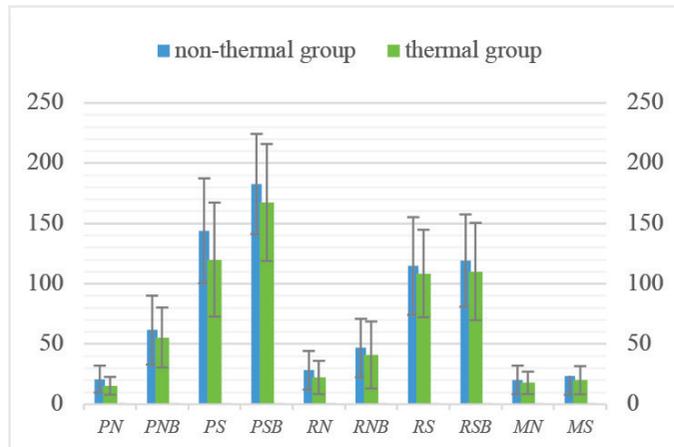


Figure 1. Comparison of the shear bod strength of the groups

Table 1. Materials and surface treatments used in this study

Cementing procedure	Surface treatments, code	
	Non-treated, N	110 µm Al ₂ O ₃ Sandblasting, S
	Panavia SA Cement, P	Panavia SA Cement, P
	Clearfil SE Bond (Kuraray) + Panavia SA Cement (Kuraray), PB	Clearfil SE Bond (Kuraray) + Panavia SA Cement (Kuraray), PB
	RelyX U200 (3M-Espe), R	RelyX U200 (3M-Espe), R
	Single Bond Universal + RelyX U200 (3M-Espe), RB	Single Bond Universal + RelyX U200 (3M-Espe), RB
	MK Crown Set Cement (Moredent), M	MK Crown Set Cement (Moredent), M

were no statistically significant differences between these four groups ($p > 0.05$).

The thermal cycling effect was evaluated and showed that the thermocycling period decreased bond strength values for all groups, but there were no statistically significant differences ($p > 0.05$).

Failure Mode Analysis

All failure types of the sequential sandblasted and sandblasted + bonding agents were seen as adhesive. Adhesive failures were also the predominant failure types in all RC types and both thermal and non-thermal conditions. Mixed and cohesive failure modes of RC were evident in the sandblasted and sandblasted + bonding agent groups of Panavia and RelyX cements for both thermal and

non-thermal conditions (Table 3). Microscopic image samples for each failure mode are displayed in Figure 2.

Discussion

The results of this study indicate that RCs that bonded to non-treated Ti surfaces yielded similar SBS values regardless of the cement type and thermocycling procedure. Nevertheless, although the bond strengths of two self-adhesive RCs tested were significantly increased by sandblasting with or without the application of bonding agents, sandblasting did not have any favourable effect on the bond strength of semipermanent cement. Therefore, the null hypothesis was partially accepted. As for the groups with bonding agents solely, no statistical differences were found compared with the non-treated groups

Table 2. Mean and standard deviation of the tested groups

Non-thermocycle.		Sig.		Thermocycle.		Sig.		T-test sig.
Group	Mean	SD		Mean	SD			
PN	20.9 ^a	11.2		15.3 ^a	7.4			0.117
PNB	61.5 ^b	28.6		55.4 ^b	24.9			0.554
PS	143.9 ^{c,d}	43.5		120.01 ^c	47.3			0.211
PSB	182.8 ^e	41.5		167.4 ^d	48.5			0.414
RN	28.2 ^a	16.03		22.2 ^a	13.8			0.294
RNB	46.7 ^{a,b}	24.2		40.9 ^{a,b}	27.8			0.576
RS	114.7 ^c	40.5		108.5 ^c	36.3			0.603
RSB	119.2 ^c	38.3	0.000	110.1 ^c	40.4	0.000		0.568
MN	20.2 ^a	11.9		17.7 ^a	9.3			0.621
MS	23.6 ^a	15.7		19.9 ^a	11.7			0.500

The same superscripts indicate statistically insignificant difference.

*One-way analysis of variance ($p < 0.05$).

**Tukey honestly significant difference ($p < 0.05$).

PN, Panavia SA Cement non-treated; PNB, Clearfil SE Bond + Panavia SA Cement non-treated; PS, Panavia SA Cement sandblasting; PSB, Clearfil SE Bond + Panavia SA Cement sandblasting; RN, RelyX U200 non-treated; RNB, Single Bond Universal + RelyX U200 non-treated; RS, RelyX U200 sandblasting; RSB, Single Bond Universal + RelyX U200 sandblasting; MN, MK Crown Set Cement non-treated; MS, MK Crown Set Cement sandblasting.

Table 3. Types of bonding fracture failures for each group

Failure types	Non-thermocycle			Thermocycle		
	Adhesive	Mixed	Cohesive	Adhesive	Mixed	Cohesive
PN	10	2	-	12	-	-
PNB	8	3	1	9	3	-
PS	7	4	1	8	4	-
PSB	5	5	2	6	5	1
RN	12	-	-	12	-	-
RNB	8	4	-	9	3	-
RS	8	4	-	8	4	-
RSB	6	5	1	9	3	-
MN	12	-	-	12	-	-
MS	12	-	-	12	-	-

PN, Panavia SA Cement non-treated; PNB, Clearfil SE Bond + Panavia SA Cement non-treated; PS, Panavia SA Cement sandblasting; PSB, Clearfil SE Bond + Panavia SA Cement sandblasting; RN, RelyX U200 non-treated; RNB, Single Bond Universal + RelyX U200 non-treated; RS, RelyX U200 sandblasting; RSB, Single Bond Universal + RelyX U200 sandblasting; MN, MK Crown Set Cement non-treated; MS, MK Crown Set Cement sandblasting.

for RelyX ($p>0.05$). Meanwhile, the bond strength values of the sandblasted + bonding agent groups were significantly higher than the bonding agent and non-treated groups ($p<0.05$).

The permanent RCs used in this study (Panavia SA Cement and RelyX U200) are dual-cured RCs applied solely and applied with a universal adhesive that belongs to their manufacturers (Clearfil SE Bond and Single Bond, respectively). This bonding agent and Panavia SA Cement contain the monomer 10-methacryloyloxydecyl dihydrogen phosphate (MDP), which was originally designed to bond to metal oxides, although its use has been extended to oxide ceramics (19). It has been stated that MDP-containing RCs are preferable to obtain a chemical bond between the hydroxyl groups of the passive surface and the phosphate ester group of the MDP (19,20). The other cement used in this study is a semipermanent cement, MIS Crown Set Cement, which is advised by the manufacturer due to its high retention properties and easy restoration removal ability. In our study, the bond strength of MIS Crown Set Cement provided the lowest values even for the sandblasted Ti surfaces.

On the other hand, sandblasting of Ti significantly enhanced the bond strength of Panavia SA Cement and RelyX U200 than that of MIS Cement. Therefore, it is sensible to prefer sandblasting of Ti abutments and using permanent cements with bonding agents including MDP for reduced abutment/restoration interface and over tapered abutments in the existence of limited interarch space. After sandblasting, both the debris and metal oxide layer are removed from Ti alloys. However, immediately afterwards, a thin, stable oxide layer is reproduced, which interacts with the MDP monomer, forming a chemical bond between the dihydrogen phosphate group and metal oxides (18,21). In a study by Koizumi et al. (22), primers containing MDP were determined to be more effective for treating Ti than other primers without MDP. Di Francescantonio et al. (23) stated that the direct application of Panavia F 2.0 to Ti surface without using an adhesive primer provided a favourable bond strength due to the MDP content. Correspondingly, in our

study, Panavia samples with or without using a bonding agent (Clearfil SE Bond 2) yielded similar bond strength values as well. But although there were no significant differences between all RC types for the thermocycled non-treated groups ($p>0.05$), the non-treated thermocycled Panavia SA Cement group showed the lowest values among all groups in our results. This situation can be explained by the fact that the MDP content in Panavia SA Cement is not as high as the MDP content in Clearfil SE Bond, which is defined as the gold standard for MDP content. Nevertheless, the Clearfil SE Bond + Panavia SA Cement sandblasting group showed the highest bond strength among all the other groups both before and after thermocycling.

It is well documented in the literature that sandblasting of Ti surfaces enhanced the bond strength of cemented restorations compared with smooth Ti surfaces (18,24-26). Tsuchimoto et al. (27) also reported that sandblasting of Ti increased micromechanical interlocking between resin and Ti, particularly when combined with adhesive primers (27). Yanagida et al. noted that the primers containing MDP monomer improved the bonding durability of composite resins to air-abraded Ti surfaces even after thermocycling (28). Hon et al. (29) compared the effects of five commercially available silane coupling agents for Ti-RC adhesion and reported that conditions, especially thermocycling, significantly affected adhesion, but the five silane coupling agents provided similar and clinically acceptable adhesions. In our study, we used bonding agents that also included silane for Panavia and RelyX. Bonding agents are commonly used in clinical practice as they are easily accessible for clinicians. Because of that, we wanted to compare if there is an adhesive effect of bonding agents, including metal primers or silane coupling agents, for Ti surfaces. Our results are also similar to Hon et al. (29), in which silane content bonding agents provide acceptable adhesion.

Study Limitations

There were some limitations in this study. Firstly, only sandblasting and machined surfaces were used for comparison because sandblasting is clinically the most common surface treatment method for metals, Ti and zirconia. However, various types of surface treatment methods should be investigated for further studies to understand the effects of surface treatments on bond strength to Ti and select a more proper type of luting cement for clinical applications. Secondly, this study only used bonding agents that contain silane coupling agents, and only Ti surfaces were used as substrates. Therefore, further investigations are needed to compare metal primers and silane agents with bonding agents for abutment surfaces. Also, different kinds of abutment materials such as zirconia, lithium disilicate and polyeterkethonekethone can be evaluated for future studies.

Based on these findings, it might be clinically helpful to modify the Ti abutments by sandblasting procedures and pay attention to MDP contents while making the selection of not only the primer or self-etch bonding agent but also the adhesive luting cement itself.

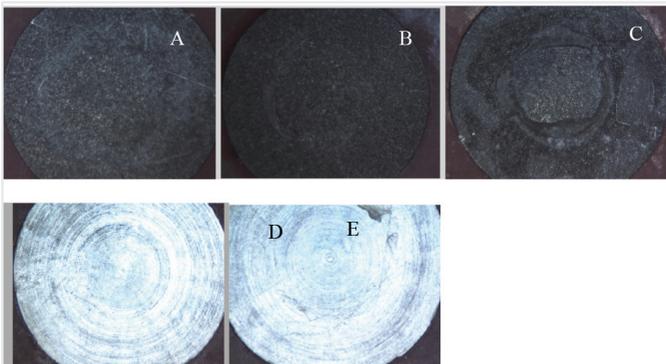


Figure 2. View of failure types; A- Adhesive type for sandblasted surfaces, B-Mixed type for sandblasted surfaces, C- Cohesive type for sandblasted surfaces, D- Adhesive type for non-treated surfaces, E- Mixed type for non-treated surfaces

Conclusion

It can be concluded that there was no effect in respect to bonding to non-treated Ti surfaces among the cements tested before and after thermocycling. Nonetheless, for the sandblasted Ti surfaces, the Panavia SA Cement sandblasting, Clearfil SE Bond + Panavia SA Cement sandblasting, RelyX U200 sandblasting and Single Bond Universal + RelyX U200 sandblasting groups provided higher bond strength than the MIS Crown Set Cement sandblasting group. Therefore, clinicians should prefer a higher retention cement strength with the combination of Ti abutment surface modification for specific cases where weak retention is predicted over single cement-retained crowns.

Ethics

Peer-review: Externally peer reviewed.

Authorship Contributions

Surgical and Medical Practices: C.A., G.S.Ö., Concept: G.S.Ö., C.A., Design: G.S.Ö., C.A., Data Collection or Processing: G.S.Ö., C.A., Analysis or Interpretation: G.S.Ö., B.S.O.A., Literature Search: B.S.O.A., G.S.Ö., Writing: B.S.O.A., G.S.Ö.

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