Influence of MDP Content in Different Resin Cements and Zirconia Primers on Ceramic-Resin Cement Bond Strength

Farklı Reçine Simanlardaki ve Zirkonya Primerlerindeki MDP İçeriğinin Seramik-Reçine Siman Bağlantı Kuvvetine Etkisi

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ABSTRACT Objective: Evaluating the influence of MDP in different primers and resin cements on microtensile bond strength to zirconia ceramic after thermal cycling. **Material and Methods:** 6 zirconia blocks (23x20x10 mm) were prepared and divided into 3 groups (n=2) according to the following surface treatments: (1) air-particle abrasion with 50 µm Al2O3 particles, (2) air-particle abrasion and zirconia primer application, (3) air-particle abrasion and MDP-containing primer mixture application. Composite resin blocks were bonded to the pretreated zirconia surfaces using 2 different resin cements. Zirconia-composite blocks were cut to microbars with a cross-section of 1.0 ± 0.2 mm. The samples were thermocycled and microtensile bond strength tests were performed. Samples were evaluated under optical microscope. Data were analyzed with 1- and 2-way ANOVA and Tukey multiple comparison tests (α=.05). **Results:** MDP based resin cements provided similar bond strengths (p>0.05) to air-particle particle abraded and primer pretreated zirconia surfaces. Bis-GMA based resin cements provided statistically higher bond strengths (p<0.05) to primer pretreated zirconia surfaces than air-particle particle abrasion alone. No statistically significant differences (p>0.05) were detected between the zirconia primers used. **Conclusion:** MDP containing resin cements or primers increase the bonding strength of zirconia. When using fixed partial dentures with zirconia substructures, clinicians are advised to use either primers or resin cements with MDP, if not both.

Key Words: Zirconium oxide; resin cements

ÖZET Amaç: Bu çalışmada, MDP içeren primerlerin ve reçine simanların zirkonya seramiğine bağlantısının termal siklüs sonrasında incelenmesi amaçlanmıştır. **Gereç ve Yöntemler:** 6 adet zirkonya blok (23x20x10 mm) hazırlanmış ve aşağıdaki işlemlere göre 3 gruba (n=2) ayrılmıştır: (1) 50 µm Al2O3 partikülleri ile kumlama, (2) kumlama ve zirkonya primer uygulaması, (3) kumlama ve MDP-primer karışımı uygulaması. İşlem yapılan zirkonya yüzeylerine kompozit reçine bloklar iki farklı reçine siman kullanılarak simante edilmiştir. Zirkonya-kompozit bileşke yapı kesilerek 1,0 ± 0,2 mm kalınlığında mikro-çubuklar elde edilmiştir. Örnekler termak siklüs kullanılarak yaşlandırılmış ve bağlantı kuvveti test edilmiştir. Örnekler ışık mikroskobu altında incelenmiştir. Veriler 1 ve 2 yönlü varyans analizi ve Tukey çoklu karşılaştırma testleri ile istatistiksel olarak değerlendirilmiştir (α=.05). **Bulgular:** MDP esaslı reçine siman, kumlanmış ve primer uygulanmış zirkonya yüzeylerine benzer bağlantı kuvvetleri göstermiştir (p>0,05). Bis-GMA esaslı reçine siman primer uygulanmış zirkonya yüzeyine, yalnızca kumlanan örneklere kıyasla daha yüksek bağlantı kuvveti göstermiştir (p<0,05). Kullanılan zirkonya primerleri arasında bağlantı kuvveti açısından istatistiksel olarak anlamlı bir fark saptanmamıştır (p>0,05). **Sonuç:** MDP içeren reçine simanlar veya primerler zirkonyaya olan bağlantıyı kuvvetlendirmektedir. Zirkonya altyapılı köprü protezlerinde klinisyenlerin her ikisini olmasa da, MDP içeren reçine simanları veya primerleri kullanmaları önerilmektedir.

Anahtar Kelimeler: Zirkonyum oksit; rezin simanları

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INTRODUCTION

All-ceramic systems gained favor in the recent years due to high esthetic demands of the patients and biocompatibility requirements. For the lithia based ceramics, a major problem was low fracture resistance. In order to increase the mechanical properties, zirconia-based materials are employed as core materials for crowns and bridges in prosthodontics, due to their high strength and toughness. For contemporary fixed prosthodontic applications, fabrication of zirconia-based frameworks using the CAD/CAM system is a standard routine.[1]

Zirconia restorations can be cemented by conventional methods or using adhesive cements.[1] For the adhesive cementation of zirconia, both light curing and self curing materials are reported to be used in literature.[2-7] Although in a recent meta-analysis reported majority of the evaluated studies (85%) used a light-curing protocol to polymerize the composite cement, the cement must also adequately self-cure in the case of zirconia restorations, since its opacity will impair the photo-polymerization of the cement.

Adhesive cementation of zirconia requires surface treatments prior to cementation. Several surface conditioning procedures are used for zirconia ceramics such as: Al_2O_3 air-particle abrasion, ceramic coating, tribochemical silica coating, chemical etching and laser irradiation.[2] It has been reported that sufficient bond strength between the adhesive resin cements and zirconia ceramics is achieved after air-particle abrasion of the zirconia cementation surface.[3,4] The air-particle abrasion basically applies Al_2O_3 particles between 30 to 250 μm at high pressure (between 2-3 bars). This process removes the uppermost layers of the zirconia and the roughened surface provides micromechanical retention with the adhesive resin cement.

In addition to surface treatments of zirconia material, the resin cement can also be enchanced in order to increase the adhesion. Several adhesive systems contain specific functional monomers which improve the performance of adhesion. The functional monomers have the advantages of conditioning dental tissues, increasing the monomer penetration, and improving the chemical adhesion to dental tissues.[4]

Methacryloyloxydecyldihydrogenphosphate (10-MDP) is one such material; it is a hydrophilic phosphate monomer which causes acidic decalcification of dental hard tissues and binds to calcium ions or amino groups of tooth structure; resulting in the increase of diffusion and adhesion of resin cement. Several studies reported it to be one of the most successful materials in the market for chemical bonding.[8,9] It should be noted that some relatively new materials in the market such as self-adhesive cements include multifunctional phosphoric acid dimethacrylate modified monomers and therefore require conditioning the dentin or ceramic surfaces before cementation.[10-15] For most zirconia-bonding techniques, the use of an 'MDP-containing primer' reported to have a positive effect. Some commercial MDP-containing primers, also contain a silane bifunctional molecule. Whether the MDP and silane combination has a synergistic or antagonistic effect on bonding is currently not known.[2]

The bond strength evaluation is easily and effectively done by destructively testing the adhesive/cohesive joints.[16] Both tensile and shear tests are documented for evaluation of bond strength in the studies.

The present study aimed to evaluate and compare the influence of 2 different primers on microtensile bond strength of resin cements to zirconia ceramic after artificial aging (thermocycling) and air-particle abrasion. The null hypothesis tested was that there is no difference between MDP containing and non-MDP containing adhesive groups.

MATERIAL AND METHODS

The materials used in the study and application procedures are displayed in Table 1.

SAMPLE PREPARATION

6 zirconia blocks (23x20x10 mm) were prepared by cutting a presintered CAD/CAM milling block (Kavo Everest ZS-Blank, Kavo, Biberach, Ger-

TABLE 1: Brands, manufacturers, chemical compositions and application protocols of the materials used in the study.

Material	Manufacturer (Lot number)	Composition	Application
Filtek Ultimate	3M ESPE, St. Paul, MN, USA (N441873)	bis-GMA, UDMA, TEGDMA, bis-EMA, fillers (72.5% by weight)	Apply onto prepared zirconia block samples Light polymerize for 20 s
Z-Prime Plus	Bisco Inc, Schaumburg, IL, USA (1200012922)	MDP, Acetone	Apply onto sample surface for 20 s Dry gently with air Light polymerize for 10 s
Clearfil Porcelain Bond Activator Silane Coupling Agent	Kuraray Co, Osaka, Japan (17BBA)	3--trimethoxysilylpropyl methacrylate, Hydrophobic aromatic dimethacrylate, others	Mix with “Clearfil SE Bond” Apply onto sample surface for 20 s Dry gently with air Light polymerize for 10 s
Clearfil SE Bond	Kuraray Co, Osaka, Japan (000093)	*Primer:* 10-MDP; Hydrophilic Dimetacrilate; di-camphorquinone; N,N-diethanol-p-toludine; water *Adhesive:* 10-MDP; bis-GMA; HEMA; hydrophobic dimetacrylate; dl-camphorquinone; N,N-diethanol-p-toludine; silanated colloidal silica	Mix with “Clearfil Porcelain Bond Activator Silane Coupling Agent” Apply onto sample surface for 20 s Dry gently with air Light polymerize for 10 s
Panavia F 2.0	Kuraray Co, Osaka, Japan (41174)	Paste A: 10-Methacryloyloxydecyl dihydrogen phosphate; Hydrophobic aromatic dimethacrylate; Hydrophobic aliphatic dimethacrylate; Hydrophilic aliphatic dimethacrylate; Silanated silica filler; Silanated colloidal silica; dl-Camphorquinone; Catalysts; Initiators *Paste B:* sodium fluoride; Hydrophobic aromatic dimethacrylate; Hydrophobic aliphatic dimethacrylate; Hydrophilic aliphatic dimethacrylate; Silanated barium glass filler; Catalysts; Accelerators; Pigments; Others	Mix equal amounts of Paste A and B for 20 s.; apply onto the sample surface; light polymerize for 40 s
Duolink	Bisco Inc, Schaumburg, IL, USA (1400006040)	bisphenol A glycidylmethacrylate, Triethylene glycol dimethacrylate	Automix base and catalyst, apply onto the sample surface; light polymerize for 40 s

10-MDP= 10-methacryloxydecyl dihydrogen phosphate; HEMA= 2-hydroxyethyl methacrylate; Bis-GMA= bis-phenol A diglycidylmethacrylate

many). The blocks were sintered to the final dimensions of 18x15x6 mm and then duplicated in composite resin (Filtek Ultimate 3M ESPE, St. Paul, MN, USA) blocks using teflon molds (Figure 1). Zirconia blocks were equally divided into 3 groups (n=2) according to the following surface treatments: (1) air-particle abrasion with 50 µm Al_2O_3 particles for 15 and 30 seconds at 2 bar pressure from a distance of approximately 10 mm (Easyblast, BEGO, Bremen, Germany), (2) air-particle abrasion (Same procedure as above) and zirconia primer (Z-Prime Plus; Bisco) application, (3) air-particle abrasion (Same procedure as above) and MDP-containing primer (Clearfil SE Bond Primer) /silane coupling agent (Clearfil Porcelain Bond Activator) mixture application. Each surface treatment group was further divided into 2 subgroups based on cement type and composite resin blocks were bonded to the pretreated zirconia surfaces using 2 different resin cements (Panavia F 2.0; Duolink). Each zirconia-composite block was cut into microbars using a microtome (ISOMET low speed saw, Buehler Ltd., Lake Bluff, IL, USA) with a cross-section of 1.0 ± 0.2 mm and 10 intact microbars were selected from each sliced block to form final subgroups in microtensile testing (n=10); a total of 60 zirconia/composite microbars were used for testing (Figure 2). The samples were thermocycled for 10,000 cycles between 5 and 55°C (Nova Inc. Konya, Turkey).

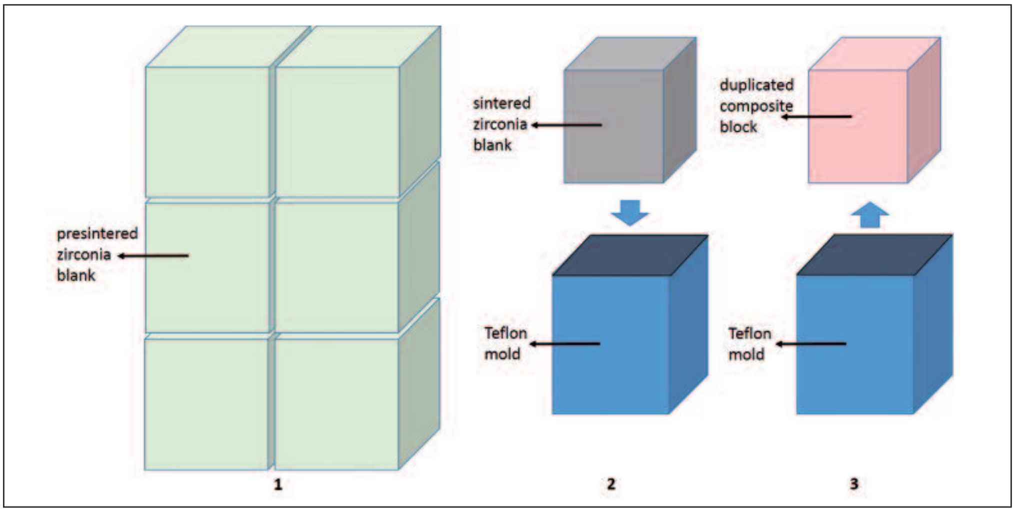

FIGURE 1: Preparation of test samples. (1) 6 presintered zirconia blanks are milled. (2) Teflon mold is fabricated using a sintered zirconia blank. (3) Composite blocks in the dimensions of sintered zirconia blanks are duplicated using the teflon mold.

MICROTENSILE TESTING

Prior to microtensile testing, the samples were stored in dry containers at room temperature. The microtensile bond strength tests were performed with a universal testing machine at a crosshead speed of 0.5 mm/min (Shimadzu AG-IS, Shimadzu, Kyoto, Japan) (Table 2).

FAILURE ANALYSIS

The mode of failure were investigated using an optical microscope (Leica M80, Leica Microsystems, Wetzlar, Germany) at ×40 magnification. The failure types were categorized as: adhesive failure between zirconia block and resin cement (A), cohesive failure of composite (C-C), cohesive failure of zirconia (C-Z), mixed failure between resin cement and composite (M-C) and mixed failure between resin cement and zirconia (M-Z). The number distribution of failing types were converted into percentages (Table 3).

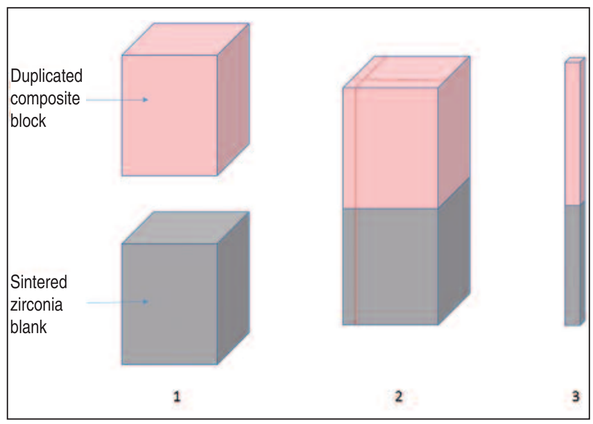

FIGURE 2: Preparation of test samples. (1) Duplicated composite blocks are bonded to sintered zirconia blanks after surface treatments. (2) Zirconia-composite blocks are cut into microbars. (3) Samples are ready for thermocycling and microtensile testing.

TABLE 2: Mean microtensile bond strengths (MPa) for each group. Groups labelled with different letters are significantly different ($p<.05$).

Groups	n	Mean Microtensile bond strength (MPa)	Standard deviation	Significance
Air-particle abrasion + Panavia	10	19.2^{A}	2.8	0.282
Air-particle abrasion + Z prime plus + Panavia	10	23.5^{A}	5.8	0.237
Air-particle abrasion + Clearfil primer + Panavia	10	25.2^{A}	5.3	0.266
Air-particle abrasion + Duolink	10	11.8^{B}	2.7	0.048
Air-particle abrasion + Z prime plus + Duolink	10	20.7^{A}	4.4	0.126
Air-particle abrasion + Clearfil primer + Duolink	10	21.6^{A}	3.9	0.208

TABLE 3: Percentage distribution of failure modes per group.

Groups	A	C-C	C-Z	M-C	M-Z
Air-particle abrasion + Panavia	90	0	0	10	0
Air-particle abrasion + Z Prime plus + Panavia	100	0	0	0	0
Air-particle abrasion + Clearfil primer + Panavia	100	0	0	0	0
Air-particle abrasion + Duolink	80	0	0	20	0
Air-particle abrasion + Z Prime plus + Duolink	90	0	0	10	0
Air-particle abrasion + Clearfil primer + Duolink	90	0	0	10	0

A: Adhesive failure between zirconia block and resin cement, C-C: Cohesive failure of composite, C-Z: Cohesive failure of zirconia, M-C: Mixed failure between resin cement and composite, M-Z: Mixed failure between resin cement and zirconia.

STATISTICAL ANALYSIS

The dependent factor of microtensile bond strength and the independent factors of air-particle abrasion and primer application were analyzed with two-way ANOVA with significance level at $p < 0.05$, followed by Tukey multiple comparison tests, with a $p < 0.05$ significance level using statistical software (SPSS 12.0; SAS, Chicago, Ill, USA).

RESULTS

The statistical evaluation of microtensile test concluded following results (Table 2): Using the resin cement with or without any one of the the silane coupling agents (Z-Prime Plus and Clearfil mixture) did not display significant difference for Panavia (p>0.05). The highest bond strength was reported in Panavia group with air-particle abrasion and MDP containing Clearfil primer, however no statistical significance was present compared to other Panavia groups (p>0.05). For the Duolink resin cement, the air-particle abrasion group displayed lowest mean bond strength, which was significantly lower compared to other tested groups (p<0.05). Using Duolink with primers significantly increased the bond strength, however there was no statistically significant difference between 2 primers (p>0.05). When the zirconia samples were only sandblasted and no primers were used, the bond strength of Panavia was significantly higher compared to Duolink (p<0.05). There was no statistically significant difference between bond strength of resin cements to zirconia when any one of the primers were used after air-particle abrasion (p>0.05).

The evaluation of distribution of failure types concluded following results (Table 3): In all of the 6 groups highest number of the failures were observed to be adhesive. For the Panavia resin cement, all of the samples in primer applied groups failed adhesively. No cohesive failure of composite or zirconia was observed in any of the samples. A relatively few number of mixed failure were observed between resin cement and composite. The mixed failure between resin cement and zirconia were not observed.

DISCUSSION

The aim of this study was to evaluate the efficiency of the different primers and resin cements with and without the MDP monomer when bonded to zirconia. The null hypothesis was rejected; the presence of MDP in either primer or resin cement increased the bond strength to zirconia. This finding is in accordance with current literature. A recent meta-analysis reported that adhesion of the luting cements is significantly influenced by the surface conditioning method, cement type, test method and aging condition. The study reported that physical conditioning of the zirconia in terms of surface roughening and silica coating results in highest increase in bond strength. After physical conditioning method, MDP monomer based cement presented the highest bond values compared to those of other resin cements.[17]

The main testing methods of bond strength; macroshear, microshear, macrotensile and microtensil are evaluated thoroughly in the literature. The macroshear test is reported to be most frequently used method.[2] The shear bond-strength test has repeatedly been documented to result in inhomogeneous stress distribution along the interface, for instance often leading rather to ‘cohesive’ failures in the substrate than to ‘adhesive’ failures at the actual interface.[18-20] As a result of this, shear and microshear bond strength tests can be expected to be less discriminative in disclosing differences in bonding effectiveness to zirconia than tensile tests.[21] In the current study, microtensile test is applied for a more accurate evaluation for the bond strength.

The microtensile bond strength results displayed that, for the Panavia resin cement (which contains MDP); using a primer with MDP does not affect the bond strength. Therefore, first outcome of the study is that presence of MDP in either primer or resin cement is enough. However, in the Duolink group, which is a cement without MDP, both MDP primer and Z prime plus achieved better results. Second outcome is that using a primer for zirconia restorations increases the bond strength.

In order to achieve the bonding between ceramic restoration and resin cement, chemical bonds and micromechanical interlocking is required. When using conventional silica based ceramics, acid etching and application of silane coupling agent results in successful ceramic-resin bonding. However zirconia does not have a glass phase which can be removed with acid etching to create a rough surface. Also, silica-silane bonds cannot be established in zirconia since silica is not present in the material. This is a clinical challenge. In order to overcome this challenge, several pretreatment procedures are investigated in the literature. These methods are deviations of ceramic coating of zirconia cementation surface, some examples are; coating with fused micro-glass pearls, coating with feldspathic porcelain, coating with glaze ceramics and selective-infiltration etching.[22-25] Using silane coupling agents after these procedures are reported to increase the bond strength, the explanation for this is very likely the presence of glass phase on the coated surface. However, air-particle abrasion still yielded highest bond strength compared to these applications.[2] In the current study the air particle abrasion were applied in order to simulate the clinical applications. It is a procedure which both cleans the surface from impurities and increases the surface area.[26] The increase in the surface area increases surface energy and therefore wettability.[27]

In the present study, one of the resin cements used were Panavia F 2.0, which contains a phosphate monomer investigated thoroughly in the literature. There were studies suggesting using this material without silane or bonding to high strength ceramics, however using a MDP containing bonding/silane coupling agent mixture reported to increase the bond strength. Former studies also reported that; air-particle abraded, silanized and conventionally luted resin cement samples were failed following simulated aging. Only air-particle-particle abrasion and use of a MDP containing luting agent provided a long term bonding between resin cement and zirconia.[28-31] Findings of the current study is partly in accordance with former studies, where application of MDP containing Panavia with or without air particle abrasion or application of primers didn't result in a significant difference in bond strength.[32] The study evaluted the effect of different primers on zirconia-resin cement bonding, however the bond strength of the samples were not tested before the aging procedures; this may be a limitation of the study, which may be investigated in further studies. The important finding of the current study is that the presence of MDP, in any one component of the bonding/silane/resin cement complex significantly increases the bond strength between zirconia and resin cement.

CONCLUSIONS

Within the limitations of this study it can be stated that using MDP containing resin cements or primers increases the bonding strength of zirconia. When using fixed partial dentures with zirconia substructures, clinicians are advised to use either primers or resin cements with MDP, if not both.

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