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치의학석사 학위논문

Orthodontic bonding technique involving
10-Methacryloyloxydecyle dihydrogen phosphate
for gold alloy surfaces

10-Methacryloyloxydecyle dihydrogen phosphate
를 이용한 금합금 표면상의 접착 시
교정용 장치의 전단접착강도

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서울대학교 대학원
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조 주 영

**Orthodontic bonding technique involving
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지도교수 양 일 형

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논문제목 : **Orthodontic bonding technique involving
10-Methacryloyloxydecyle dihydrogen phosphate for gold alloy surfaces**

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-ABSTRACT-

Orthodontic bonding technique involving 10-Methacryloyloxydecyle dihydrogen phosphate for gold alloy surfaces

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*(Directed by Professor **Il-Hyung Yang**, DDS, MSD, PhD)*

The increase in the demand for adult orthodontics made the bonding of orthodontic appliances to prosthetic surfaces a necessity. The objective of this study was to evaluate the shear bond strength of metal orthodontic tubes bonded to gold alloy surfaces with different surface preparations and different adhesive systems, and to compare the performance of 10-methacryloyloxydecyle dihydrogen phosphate (10-MDP) with conventional methods.

The sample specimens consisted of sixty Type III gold alloy plates (Au 50%, Pd 5%, Ag 32.5%, Cu 11.45%) of a standardized size embedded in cylinders of self-curing acrylic resin. The plates were allocated into four groups, fifteen each, according to different combinations of bonding techniques. The metal orthodontic tube used in this study was a mandibular right first molar direct bond tube with a base area of 20.08 mm². The preparation for each group is as follows:

Group 1, the gold alloy plates were sandblasted with 50µm aluminum trioxide using intraoral sandblaster. Metal primer (Reliance, Reliance Orthodontic Products, IL, USA) was applied. The tubes were bonded with Transbond XT (3M Unitek, CA, USA) and light-cured with a LED curing light;

Group 2, sandblasting was done with 30µm silicon dioxide for silica coating of the gold plates. Silane (Porcelain Primer, Bisco, Schaumburg, IL, USA) was applied. Bonding was carried out in the same manner as in Group 1;

Group 3, the gold plates were sandblasted in the same way as Group 1. Z-Prime Plus (Bisco, Schaumburg, IL, USA) was applied. Bonding of the tube was performed in the same manner as

Groups 1 and 2;

Group 4, the gold plates were sandblasted with aluminum trioxide in the same way as in Groups 1 and 3. Panavia F2.0 (Kuraray Medical Inc, Japan) was used to bond the tubes. Light curing was done in the same manner as in Groups 1, 2 and 3.

Shear bond strengths of each group were evaluated with universal testing machine (Instron 4466, Instron Corporation, Canton, Mass, USA) after 1 hour of storage in room temperature. Following the debonding of the tubes, the adhesive remnant index (ARI) was assessed. Data were analyzed by using language R program. Test of normality and homoscedasticity were performed using Shapiro test and Levene test. Comparison of shear bond strengths among groups was performed with one-way analysis of variance (ANOVA) followed by the Tukey's multiple comparison test, with $P < .05$ considered statistically significant. The differences in ARI scores were evaluated, and the pattern of adhesive remnants was observed.

Group 1 showed the lowest mean SBS value of 6.70 MPa, and Group 3 showed the highest mean SBS value of 9.81 MPa. Statistically significant differences existed between Groups 1 and 2, and between Groups 2, and 4 ($P < 0.001$). There were no statistically significant differences between Groups 4 and 3. The ARI scores showed that significant differences were observed between Groups 3 and 4, and between Groups 3 and 2.

The results revealed that 10-MDP showed promising performance in bonding to gold alloy surfaces. The SBS values of tubes bonded with 10-MDP containing adhesive systems such as Panavia F2.0 and Z-Prime Plus were found to be better than bonding systems using 4-META or silicoating.

Key words : 10-MDP, Shear bond strength, Gold alloy surface, Orthodontic bonding adhesive

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10-Methacryloyloxydecyle dihydrogen phosphate를 이용한 금합금 표면상의 접착 시 교정용 장치의 전단접착강도

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I. Introduction

The demand for adult orthodontics has been consistently increasing for many years. The elderly adults began to consider orthodontic treatments for several reasons such as decrowding of lower anterior teeth for better cleansing, or molar uprighting for a better prosthesis. Among numerous factors to consider for adult patients, a secure bonding of orthodontic appliance on prosthetic surfaces calls for much need and attention. Although orthodontic band procedure can prevent bond failure on prosthetic surfaces, recent studies revealed many shortcomings of orthodontic band cementation in adults such as plaque accumulation, gingival inflammation, and the development of periodontitis due to inadequate subgingival band margins.¹ Therefore, direct bonding of orthodontic appliances has been preferred not only to natural tooth surfaces but also for artificial surfaces including gold crown.

Reynolds² have suggested optimal shear bond strength for natural tooth surface to be between 5.9 and 7.8 MPa. According to many other previous studies, bond strengths ranging from 2.8 MPa to 10 MPa are considered to be adequate for clinical situations.³⁻⁵ These ranges were recommended not only for a secure bonding of the orthodontic appliances, but also for preventing enamel fracture during the orthodontic debonding procedure. However, bonding to gold is a different situation, in the aspect that there cannot be any fractures on the surface during the debonding procedure. Therefore, securing strong bond strength can be a priority when bonding to gold surfaces.

Although the bonding strength for natural tooth surface has been well established by many studies,⁶⁻⁸ there have been several studies to increase bonding strength for gold surface,⁹⁻¹² which need much to be discussed. However, the studies about the bonding strength to gold surfaces were mainly on the surface preparations and application of additional materials to an existing bonding system in the orthodontic field. Roughening with a diamond bur⁹ and silica or aluminum oxide sandblasting¹⁰ were usually studied for the surface preparation studies. Coupling agents such as primers containing 4-methacryloyloxyethyl trimellitate anhydride (4-META) and silane were used as an additional bonding agent to enhance chemical bonding between the bracket base and gold surface.^{9, 12, 13}

Orthodontic adhesives such as Transbond XT (3M Unitek, CA, United States), a light-cured adhesive¹¹, or Fuji Ortho LC (GC, Tokyo, Japan), a resin-modified glass-ionomer cement¹⁴ have been routinely used in orthodontic practice. Because a huge leap of development has been

obtained in the field of dental materials throughout the last decade, studies involving the usage of unconventional adhesives in the orthodontic field are worth the attention. Such trials have been made using prosthetic band cement materials. Fundamentally, cementation materials differ from bonding materials in aspects of viscosity, workability, and methods of curing. Several cement materials with medium to high viscosity have been introduced, as well as dual-curing properties, which might be favorable in the orthodontic bonding procedure.

Trials of using dental cements which are traditionally categorized into areas of prosthetic dentistry for orthodontic bonding have been attempted, such as SuperBond C&B (SunMedical, Japan).^{9, 12, 15} More recently, bonding orthodontic brackets to porcelain surfaces has been attempted, with success, using Panavia F2.0 (Kuraray Dental, Japan), a dual-cure resin containing a phosphate monomer, namely 10-methacryloyloxydecyle dihydrogen phosphate (10-MDP).¹⁶ The performance of 10-MDP is also assumed to show promising results in bonding to gold alloys, as it was previously shown in porcelain bonding. However, there are no studies of the usage of 10-MDP based adhesive system in orthodontic bonding to gold alloy.

The aim of this study was to evaluate the shear bond strength of metal orthodontic tubes bonded to gold surfaces with different surface preparations and different adhesive systems comparing the typical bonding system and prosthetic cementation system. The null hypothesis was that there is no difference in SBS on the gold surfaces according to the adhesive systems.

II. Review of Literature

Bonding to gold surfaces

Orthodontic bonding to artificial surfaces such as porcelain or gold has been an issue of interest for many years. Bonding instead of banding can provide many advantages, such as less plaque accumulation, gingival inflammation, and loss of gingival attachment during orthodontic treatment in patients of all ages.^{1, 17} Also, bonding can be more esthetic in visible areas, and orthodontic attachments can be bonded in areas where placing bands are difficult or physically impossible.

Previous studies performed since decades ago have recommended a variety of methods which

secure strong bond strengths between metal surfaces and orthodontic appliances.^{9-12, 18, 19}

Improvement of bond strength to gold can be made by different surface preparations, through usage of different primers and adhesives, and various combinations of these methods.

Various surface preparations

For bonding to natural tooth surfaces, surface preparation using phosphoric acid etching technique has been known as the method of choice.²⁰⁻²⁶ Trials of different surface preparations on gold alloy have been performed for the purpose of enhancing bond strength, and to match the acceptable bonding strength shown in acid etched natural tooth surfaces. Roughening with a diamond bur^{9, 10, 18} was a simple way of increasing the surface area, which allowed for a better mechanical bonding. It has been proven to increase bonding strength between cast gold crowns and resin when using highly filled resin adhesive.¹⁸ However, the bond strength when using lightly filled resin did not show such good results, and it was much weaker than acid etching of natural teeth.

The introduction of intraoral sandblasting made it possible for the clinicians to roughen the gold surfaces in a more effective way than using a diamond bur. The irregularities created by abrasion with sandblasting have been known to be much greater than those with a diamond bur or a green stone.^{9, 10} The most commonly used abrasive in orthodontic field is aluminum oxide particles of various sizes ranging from 50 to 110 micron. Silicon oxide particles have also been used for silicoating purposes. Although this method allowed for a better surface preparation by increasing the surface area effectively, many studies have shown that sandblasting alone cannot enhance bond strength significantly. Application of additional primers or adhesives was needed after the sandblasting procedure.

Tin plating has been suggested for bonding to noble metal alloys.²⁷⁻²⁹ It creates a thin layer of tin on the gold surface, and allows for a chemical and mechanical bonding between the gold surface and resin. Surface treatment of noble alloys with a solution of gallium and tin has been shown to increase the bond strength of composite to gold alloys³⁰. However, its intraoral use is not approved by the U.S. Food and Drug Administration (FDA), for some organic tin compounds may be toxic. Therefore, tin plating is not currently used in routine clinical practice.

Usage of different primers and adhesives

Following the surface preparation of gold surfaces, trials of using various types of primers and adhesives have been performed through many previous studies. Metal primers containing 4-META have been used to obtain a chemical bond between gold surfaces and resin, with acceptable results.^{9,10, 12} For gold surfaces which were silicoated by sandblasting with silicon oxide particles, silane was applied to increase bond strength.^{11, 13, 31}

Some studies advocated the usage of intermediate resins when bonding orthodontic appliances to metal surfaces. Intermediate resins such as All-Bond 2 (Bisco, Illinois, United States), a third-generation dentin bonding agent, have been used in previous studies.^{9, 32} Primer B in All-Bond 2 is claimed to be an effective metal primer when repairing a porcelain-metal crown, or bonding a metal-based restoration. However, there are no independent reports of the efficiency of these intermediate resins when bonding to gold or other metal surfaces, especially in the orthodontic field.

Various types of adhesives have also been used to improve bonding to metals. Adhesives containing acidic monomers such as 4-META and 10-MDP have been known to provide chemical bonding with metal surfaces. Super-Bond C&B, an adhesive containing 4-META, was used for bonding of orthodontic appliances with successful outcome.^{9, 15} 10-MDP containing adhesive, Panavia Ex (Kuraray Dental, Japan), has also been used for orthodontic bonding in previous studies. Its chemical bonding ability has been shown to be promising,^{15, 33, 34} but some studies did not advocate its routine use for orthodontic purposes for the inconvenience of its manipulation in clinical situations.^{34, 35} Panavia Ex is widely used in the field of prosthodontics, and in order to initiate its setting, an anaerobic environment is needed.³⁶ Therefore, an oxygen barrier (Oxyguard) was needed for its use. However, with the development of a dual-cure adhesive, Panavia F2.0, the need for this barrier was eliminated, and its manipulation was made easier.

Some studies compared the performance of light-cured glass ionomer cements to composite adhesives when bonding ceramic brackets to metal and porcelain.¹⁴ It was shown that light-cured glass ionomer cements provide sufficient strength for bonding ceramic brackets, but in terms of bond failure site and bracket fracture, they provided no advantage over composite adhesives.

For bonding resin to noble alloys, there have been many recent studies in the field of prosthodontics and dental materials suggesting the usage of primers containing thione monomers³⁷⁻⁴¹. These studies suggest that acidic monomers (4-META and MDP) are appropriate

for base metal alloys, including Co-Cr alloy and titanium alloy, while thione monomers such as 6-methacryloyloxyhexyl 2-thiouracil-5-carboxylate (MTU-6), or 6-(4-vinylbenzyl-*n*-propyl) amino-1,3,5-triazine-2,4-dithione, -dithiol tautomer (VBATDT) are suitable for noble metal alloys such as gold alloy and silver-palladium-copper-gold (Ag-Pd-Cu-Au) alloy. Examples of metal primers currently available in the market containing these thione monomers are Alloy Primer (Kuraray Dental, Japan), V-Primer (Sun Medical, Japan), or Metaltite (Tokuyama Dental, Japan). Many studies have performed trials of bonding resin to noble alloy surfaces using these primers, and acquired successful results.^{40, 42-44} However, there are no existing studies in the orthodontic field involving the usage of these primers when bonding appliances to noble alloy surfaces.

Many efforts for further enhancement of bonding strength to gold surfaces were made in previous studies through various combinations of these surface preparation methods and usage of different primers and adhesives.^{9, 11-15, 18, 31, 40, 45}

III. Materials and Methods

Sixty gold alloy plate specimen made of Type III gold alloy (Au 50%, Pd 5%, Ag 32.5%, Cu 11.45%, Argen Co. San Diego, CA, USA) were fabricated into a flat, square shape of a standardized size (10 x 10 x 2 mm: length x width x height). Each gold plate was embedded in a cylinder of self-curing acrylic resin (Leocryl, Leone, Sesto Fiorentino, Italy) so that the plate is parallel to the direction of shear force exerted by the blade of universal testing machine. A mandibular right first molar tube (Low-Profile Non-Convertible Tubes, American Orthodontics, WI, USA) with a base area of 20.08 mm² was then bonded at the center of the plates (Figure 1). The details of experimental materials used for the bonding procedures are shown in Table 1.

The gold plates were allocated into four groups, fifteen in each group, according to different combinations of bonding techniques which are as follows (Table 2):

Group 1: The gold plates were sandblasted with 50µm aluminum trioxide (GAC, Bohemia, NY, USA) using intraoral sandblaster (Air-Flow Handy II, EMS Corp USA, Dallas, TX, USA) under the

pressure of 2.5 bar, at a distance of 10 mm for 15 seconds. After sandblasting, the plates were cleaned and air-dried thoroughly. Metal primer (Reliance, Reliance Orthodontic Products, IL, USA) was applied and air-dried for 30 seconds. The tubes were bonded with Transbond XT (3M Unitek, CA, USA) according to the manufacturer's instructions. Excess adhesive was removed with a sharp instrument. The gold plates were light-cured with a LED curing light (Ortholux LED curing light, 3M Unitek) for 40 seconds, 10 seconds each from mesial, distal, gingival, and occlusal margins according to the recommendation of a previous study.¹¹

Group 2: Sandblasting was done with 30 μ m silicon dioxide (Cojet-Sand, 3M ESPE, Seefeld, Germany) for silica coating of the gold alloy surface. After gentle removal of excessive particles with air, silane (Porcelain Primer Single Component, Bisco, Schaumburg, IL, USA) was applied and allowed to air-dry for 30 seconds. Transbond XT was used for bonding adhesive in the same manner as in Group 1.

Group 3: The gold plates were sandblasted in the same way as Group 1. Z-Prime Plus (Bisco, Schaumburg, IL, USA) was applied on the gold surface and air-dried for 30 seconds. Transbond XT was used for bonding adhesive in the same manner as Groups 1 and 2.

Group 4: The gold plates were sandblasted with aluminum trioxide in the same way as in Groups 1 and 3. After sandblasting, the plates were cleaned and air-dried thoroughly. Panavia F2.0 (Kuraray Dental, Japan) was used for bonding adhesive following the manufacturer's instructions. Light curing was done in the same manner as in Groups 1, 2 and 3.

After 1 hour of storage in room temperature, the gold alloy plate specimens were mounted in a universal testing machine (Instron 4466, Instron Corporation, Canton, Mass, USA) for debonding (Figure 2). Shear force was applied to the interface between gold and bonding adhesive with a blade attached to the crosshead, which moved in an occlusogingival direction at a speed of 1mm/min (Figure 3). The maximum load needed for debonding was recorded in N, and converted to MPa. Force in MPa equals the force in N divided by the bracket base area in mm². The shear blade and the gold plates were carefully positioned so that they were parallel to each other. The blade tip was made sure to contact the gold-resin adhesive interface, not the orthodontic tube.

Following the debonding of the tubes, the adhesive remnant index (ARI) was assessed⁴⁶ to

determine the location of bond failure. This index consists of scores from 0 to 3: score 0, no adhesive left on the gold surface; score 1, less than half of the adhesive left on the gold surface; score 2, more than half of the adhesive left on the gold surface; and score 3, all adhesive left on the gold surface with a distinct impression of the bracket mesh.

Statistical analysis among the groups was performed using a language R program (Vienna, Austria). Test of normality and homoscedasticity were performed using Shapiro test and Levene test. As for the results, comparison of shear bond strengths (SBS) among groups was performed with one-way analysis of variance (ANOVA) followed by the Tukey's multiple comparison test, with $P < 0.05$ considered statistically significant. The differences in ARI scores were evaluated, and the pattern of adhesive remnants was observed.

IV. Results

Difference of shear bond strength among groups

Mean value, standard deviation, and statistical significance of SBS for each group are given in Table 3. Group 1 showed the lowest mean value of 6.70 MPa and Group 3 showed the highest mean value of 9.81 MPa. Although there was an increasing order of the mean values as Group 1, 2, 4, and 3, the statistically significant differences existed between Groups 1 and 2, between Group 2, and 4 ($P < 0.001$). There was no statistically significant difference between Groups 4 and 3 (Figure 4).

Difference of ARI scores among groups

The distribution of ARI scores is given in Table 4. In Group 2, almost all adhesives were found on the gold surface, whereas in Group 4, all of the adhesives were found on the base of the orthodontic tube. A significant difference was observed between Groups 3 and 4, and between Groups 3 and 2 (Table 4).

V. Discussion

It is known that 4-META (Figure 5) forms a hydrogen bond with hydroxyl groups on the metal surface, and is therefore capable of chemical bonding. 10-MDP monomer (Figure 6) is also found to form chemical bonds with a variety of surfaces, including metallic surfaces. It has a dihydrogen phosphate group which is hydrophilic, and this group is capable of acid demineralization and chemical bonding to tooth structure as well as metallic and nonmetallic surfaces.⁴⁷ There have been previous suggestions of performing comparative studies on the bonding strength of 4-META resin adhesives and 10-MDP Bis-GMA resin adhesives to metal surfaces.⁹ For orthodontic bonding to ceramic surfaces, the performance of 10-MDP has been shown to be promising.¹⁶ However, the effect of 10-MDP on orthodontic bonding to metal surfaces as well as comparison of its performance with 4-META has not been studied yet. This study focused on the effect of 10-MDP on bonding orthodontic tubes to gold alloy surfaces, as well as comparison of its performance to other conventional methods.

In previous studies involving orthodontic bonding strength, orthodontic brackets were mostly used for testing. However, orthodontic molar tubes were used in the present study instead of orthodontic brackets. The reason for the selection of this appliance is that gold crown can usually be seen in the posterior teeth area, where orthodontic tubes are used rather than brackets. Also, the clinical incidence of orthodontic bracket bond failure is higher for posterior teeth.⁴⁸ Therefore, orthodontic molar tubes were chosen for bonding in this study. It also has been found that the size of orthodontic bracket base surface area does not influence the shear bond strength,^{49, 50} so the size of the base surface area of the orthodontic molar tube was assumed to be insignificant to the values of the shear bonding strength. Furthermore, the result of this study can be applied to the case of orthodontic brackets.

ARI scores indicate that Group 4 (Panavia F2.0 group) showed the failure sites at the gold - adhesive interface, and Group 3 (Z-Prime Plus group) showed the sites at bracket - adhesive interface. It has been shown through a previous study that when the bracket base had only mechanical retention with the adhesive, most bond failures occurred at the bracket-adhesive interface whereas the most common site for bond failure of the bracket base which had chemical retention was at the adhesive-surface interface.⁵¹ This suggests that the 10-MDP composition of

Panavia F2.0 had a chemical bonding effect towards the metal bracket base which is non-precious metal for Group 4. As for Group 3, it can be assumed that only mechanical bonding occurred between the bracket base and Transbond XT, which was the adhesive used in this group.

Panavia as an orthodontic adhesive has been used in previous studies to test its shear bond strength and its usefulness.^{15, 33-36, 52-54} Some studies concluded that it is not recommended for routine use as an orthodontic adhesive in its present form,^{15, 35, 36, 52, 53} and others advocated the use of Panavia as an orthodontic adhesive for its acceptable bonding strength.^{33, 34, 54} These previous studies were performed on natural tooth surfaces, amalgam fillings, or ceramic surfaces, but not on precious metallic surfaces. Also in those studies, Panavia Ex and Panavia 21 were used, which are chemically cured in an anaerobic environment. In this study, Panavia F2.0 was used, which has a dual-cure property that is more favorable for orthodontic bonding purposes. The light-curing property of Panavia F2.0 compensated for the relatively low viscosity of this cement, which was considered a weakness for orthodontic bonding purposes.

In previous studies, orthodontic bracket bonding to metal surfaces was found to be best achieved through surface preparation using Cojet, silicoating with silane application, and bonding with Transbond XT adhesive.¹⁰ Its performance was shown to be better than bonding with 4-META primer for gold surfaces.³¹ The SBS values of bonding with silicoating method in previous studies match the values obtained in the present study, which was a mean value of 7.58 MPa.^{11, 31} However, in one previous study, the SBS value of silica coated metal surface was very high with a mean value of 19 MPa.¹⁰ This difference may be due to the usage of different adhesive system, as well as a longer storage period of 1 week. Under the same environment and conditions, the SBS values of bonding to gold after silicoating in previous studies were similar to those obtained in this study.

Since the silicoating surface preparation was known to show an excellent bonding strength when bonding to gold surface, resin adhesive and primer containing 10-MDP in its composition was studied for comparison of their performance. In this study, the SBS values were found to be higher in the groups (Groups 3 and 4) using 10-MDP regardless of its application types in the bonding procedure than in Group 2 (silicoating group). Group 3 which used 10-MDP as a primer type (mean value 9.71 MPa) and Group 4 which used 10-MDP as an adhesive type (mean value 8.95 MPa) showed superior results compared to silicoating group (mean value 7.82 MPa). This

result showed that 10-MDP offers better SBS values than those conventional methods when bonding to gold alloy surfaces.

It is known through previous studies that 10-MDP provide a stable chemical bonding to non-precious metal, by the phosphoric acid group of MDP bonding chemically to the non-precious metal atoms, while the double bonds on the other end of the molecule co-polymerize with the resin monomers.^{16, 55} Also, there is a specific property of 10-MDP which is that it builds a particular nano-sized structure that improves bond stability. It is self-assembled and called 'nano-layering'.^{56, 57} This ability of 10-MDP is thought to additionally explain the strong bond durability well shown in previous studies. However, the ability of 10-MDP to form a chemical bond with precious alloys, including gold-copper-silver (Ag-Cu-Au) alloy is uncertain.

For bonding resin to precious metal alloy, thiol-derivative conditioners, which contain 6-methacryloyloxyhexyl 2-thiouracil-5-carboxylate (MTU-6), or 6-(4-vinylbenzyl-*n*-propyl) amino-1,3,5-triazine-2,4-dithione, -dithiol tautomer (VBATDT) have been recommended for chemical bonding.⁴¹ The reason for good bonding performance of 10-MDP to the precious alloy used in this study can be assumed to be due to the surface preparation of sandblasting with aluminum oxide. Sandblasting with aluminum oxide particles on metal surfaces have been found to increase the aluminum concentration of the metal surface,^{45, 58} and sandblasting of gold alloy surfaces showed statistically significant changes in elemental composition, which were of greater magnitude when 50-micron Al₂O₃ particles were used.⁵⁹ 10-MDP in this study seemed to react with the aluminum oxide particles embedded in the gold alloy surface, therefore enhancing its chemical bonding ability.

Collectively, the results revealed that 10-MDP showed promising performance in bonding to gold alloy surfaces with better SBS values than in the bonding systems using 4-META or silicoating surface treatment. Furthermore, Panavia F2.0 adhesive system can reduce the number of bonding steps because no additional primers are needed. For the conventional Transbond XT adhesive system, a layer of Transbond XT primer needs to be applied after the surface conditioning primer. Without this step, the bonding procedure for gold surfaces can be simplified, which can save chair time and consequently reduce errors in the overall bonding procedure.

Previous bonding studies often included a certain amount of storage period of the orthodontic

appliances, with more favorable results regarding bonding strength after a longer storage time.⁶⁰

⁶¹ In this study, results were obtained immediately after the bonding procedure because it was assumed that the immediate loading of orthodontic appliances is of clinical significance in measuring bond failures. It was shown that shear bond strengths increased as storage time increased in many previous studies. Therefore, values obtained by applying forces immediately after bonding do not seem to be a problem. However, storage in water or thermocycling procedure was not performed in the present study. The moisture conditions during bonding can influence the shear bond strength of orthodontic brackets, and these aspects need to be included in further studies.

This is an *in vitro* study, performed under a strictly controlled laboratory environment. The results may slightly differ in clinical situations, in which the various forms of the surface of the gold crown, mastication forces, and saliva and moisture contaminations influence the molar tube bonding situation. Further studies need to be done considering the effects of moisture contaminations, longer storage time, and the clinical environment. Additionally, in future studies, it would be interesting to study the usage of thione monomers when bonding orthodontic brackets to gold surfaces.

VI. Conclusion

Panavia F2.0 can be considered to be a positive candidate in situations of bonding orthodontic molar tubes to posterior teeth gold crown.

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VII. Tables

Table 1. Experimental materials used for the bonding procedures

<i>Material</i>	<i>Manufacturer</i>	<i>Composition</i>	<i>Application***</i>
Reliance	Reliance Orthodontic Products, IL, USA	Methyl Methacrylate	Apply a uniform coat. Wait 30 seconds.
Porcelain Primer	Bisco, Schaumburg, IL, USA	Ethanol, Acetone, Silane	Brush on 1-2 thin coats. Wait 30 seconds.
Transbond XT	3M Unitek, CA, USA	Primer: Bisphenol A diglycidyl ether dimethacrylate, triethylene glycol dimethacrylate, 4-(dimethylamino)-benzeneethanol, DL-camphorquinone, hydroquinone Adhesive: Silane treated quartz, Bis-GMA*, Bisphenol-A Bis(2-Hydroxyethyl ether) Dimethacrylate, Silane treated silica, Diphenyliodonium hexafluorophosphate	Apply thin uniform coat of primer. Only a very thin film of primer is necessary. With the syringe apply a small amount of paste onto bracket base. Use sparingly. Light-cure for 20 sec.
Z-Prime Plus	Bisco, Schaumburg, IL, USA	Biphenyl dimethacrylate, MDP**, Ethanol	Apply 1-2 coats uniformly. Dry with an air syringe for

			3-5 seconds.
Panavia F2.0	Kuraray Medical Inc, Japan	Paste A: 10-MDP, Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic methacrylate, Hydrophilic aliphatic dimethacrylate, Silanated silica, filler, Silanated colloidal silica, DL-camphorquinone, Catalysts, Initiators, Others Paste B: Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic methacrylate, Hydrophilic aliphatic dimethacrylate, Silanated barium glass filler, Catalysts, Accelerators, Pigments, Others	Dispense equal amounts of paste A and B. Mix paste A and B for 20 sec. Apply the mixture. Cement and remove the excess paste. Light-cure the margin for 20 sec.

*Bis-GMA: Bisphenol-A diglycidyl methacrylate.

**MDP: methacryloyloxydecyl dihydrogen phosphate

***Information derived from each manufacturer's instructions.

Table 2. Description of different combinations of bonding techniques for each group

<i>Group</i>	<i>Surface Treatment</i>	<i>Primer</i>	<i>Adhesive</i>
1 (n=15)	sandblasted	metal primer + TXT primer	TXT adhesive
2 (n=15)	sandblasted + silica coating	Silane + TXT primer	TXT adhesive
3 (n=15)	sandblasted	Z-Prime Plus + TXT primer	TXT adhesive
4 (n=15)	sandblasted	-	Panavia F2.0

† TXT refers to Transbond.

Table 3. Comparison of shear bond strengths (MPa) among groups

	<i>Mean (SD)</i>				<i>Significant pairs among groups</i>
	<i>Group 1</i>	<i>Group 2</i>	<i>Group 3</i>	<i>Group 4</i>	
<i>Shear Bond Strength</i>	6.70 (1.04)	7.82 (1.12)	9.71 (0.98)	8.95 (1.28)	(1,2) (1,3) (1,4) (2,3) (2,4)

‡ One-way ANOVA with Tukey comparison was used to analyze the difference among groups at a significance level of P = .05.

Table 4. ARI score distribution for each group

<i>Group</i>	<i>N</i>	<u><i>ARI Score</i></u>			
		<i>0</i>	<i>1</i>	<i>2</i>	<i>3</i>
1	15	8	3	-	4
2	15	-	-	1	14
3	15	6	-	-	9
4	15	15	-	-	-

VIII. Figures

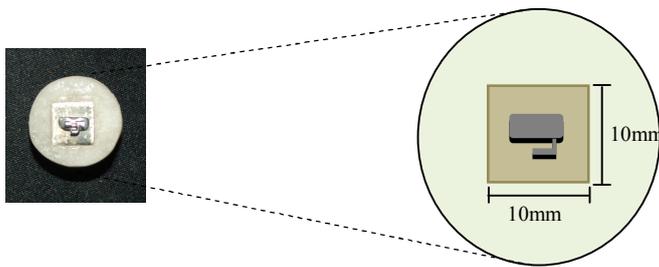


Figure 1. Type III gold alloy plate specimen embedded in an acrylic resin cylinder.

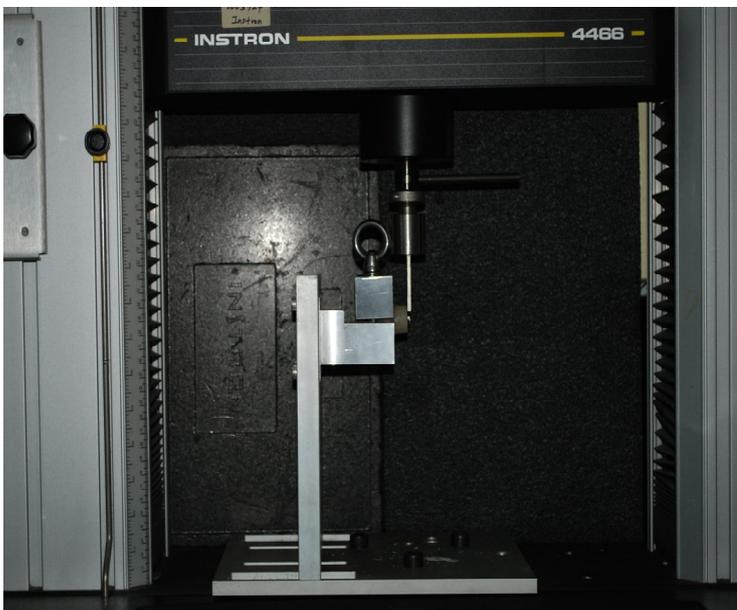


Figure 2. Instron Universal Testing Machine.

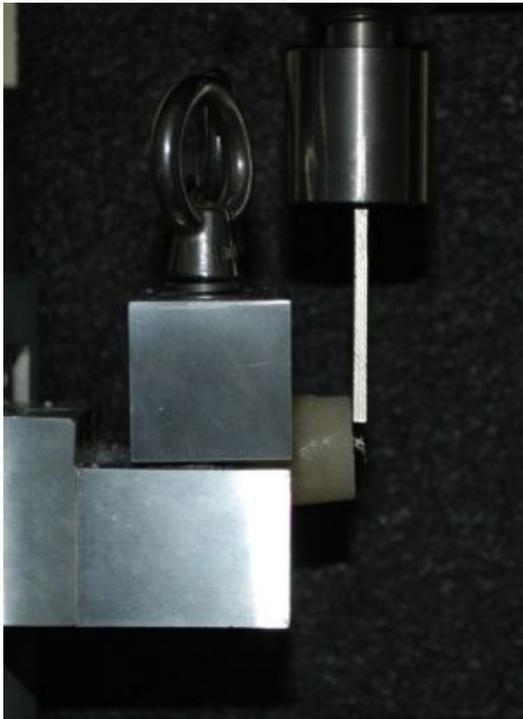


Figure 3. Close-up view of the crosshead blade moving parallel to the specimen.

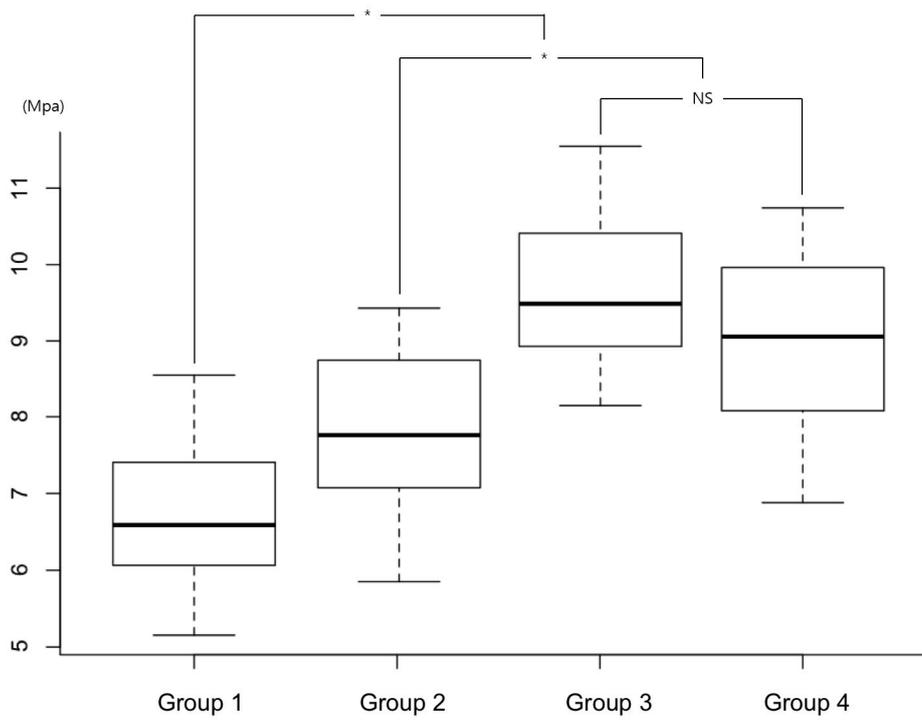


Figure 4. Box plot of one-way ANOVA with Tukey comparison.

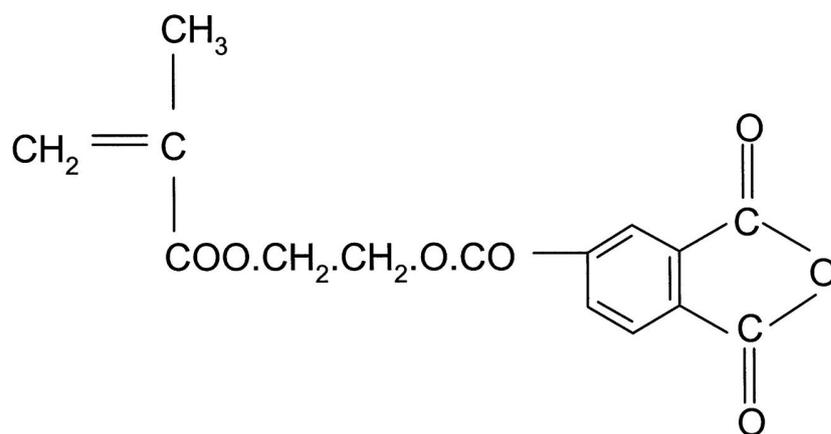


Figure 5. Chemical structure of 4-META.

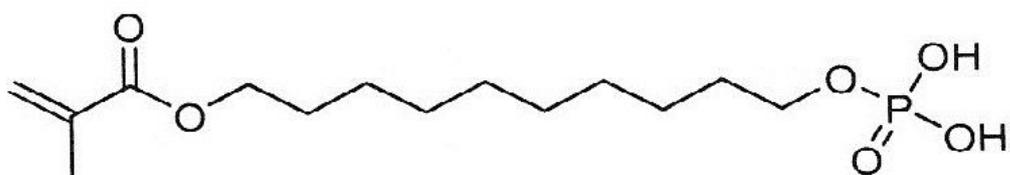


Figure 6. Chemical structure of 10-MDP.

국문초록

10-Methacryloyloxydecyle dihydrogen phosphate를 이용한 금합금 표면상의 접착 시 교정용 장치의 전단접착강도

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조 주 영

최근 성인 교정치료의 수요가 증가함에 따라 치과 보철물에 교정용 장치를 부착해야 하는 빈도가 늘어나고 있다. 본 연구는 금합금 표면에 각기 다른 방법으로 교정용 장치를 부착한 후 이의 전단접착강도를 측정하고, 10-methacryloyloxydecyle dihydrogen phosphate(10-MDP)를 이용한 접착 시의 강도를 기존 방식과 비교하고자 시행되었다.

60개의 일정한 규격의 type III 금속판(Au 50%, Pd 5%, Ag 32.5%, Cu 11.45%)을 자가중합형 아크릴 레진 원통에 매립한 후 다양한 접착방식에 따라 15개씩 총 4개의 군으로 분류하였다. 본 연구에서 사용한 금속 교정용 장치는 하악 우측 제1대구치용 튜브로써, 베이스의 면적은 20.08mm² 이었다.

1군은 금합금판을 50 μ m의 aluminum trioxide입자로 sandblasting한 후 4-META 계열의 metal primer(Reliance, Reliance Orthodontic Products, IL, USA)를 도포하고 Transbond XT(3M Unitek, CA, USA)로 튜브를 접착하였다. 2군은 금합금판을 30 μ m의 silicon dioxide로 sandblasting을 하고 silane(Porcelain Primer, Bisco, Schaumburg, IL, USA)을 도포한 뒤 접착은 1군과 동일하게 하였다. 3군은 1군과 동일하게 금합금판을 sandblasting한 후 Z-Prime Plus(Bisco, Schaumburg, IL, USA)를 도포한 뒤 1,2군과 동일한 방식으로 접착하였고, 4군은 1,3군에서와 동일하게 금합금판을 sandblasting한 후 primer를 도포하지 않고 Panavia F2.0(Kuraray Medical Inc, Japan)으로 튜브를 접착하였다.

실온에서 1시간 보관 후 universal testing machine(Instron 4466, Instron

Corporation, Canton, Mass, USA)을 이용해 각 그룹의 전단접착강도를 측정하였고, 잔존해 있는 접착제 양상을 adhesive remnant index (ARI)로 평가하였다. 정규성과 등분산성 검정을 Shapiro와 Levene test로 시행하고 각 군간 전단접착강도의 비교를 위해 일원분산분석 후 Tukey comparison을 시행하였으며, $P < 0.05$ 를 유의성 있는 결과로 간주하였다.

결과를 보면 전단접착강도는 1군이 6.70 MPa로 가장 낮은 값을 보였고 3군이 9.81 MPa로 가장 높은 값을 보였다. 통계처리 결과 1군과 2군 간에, 그리고 2군과 4군 간에 유의성 있는 차이가 있었다($P < 0.001$). 4군과 3군 간에는 유의성 있는 차이가 없었다. ARI값은 3군과 4군 간, 그리고 3군과 2군 간에 유의성 있는 차이가 있었다.

Panavia F2.0 또는 Z-Prime Plus와 같은 10-MDP를 함유한 접착 시스템을 사용할 경우 교정용 튜브를 금합금 표면에 접착 시 기존의 방법보다 더 나은 전단접착 강도를 보였다. 10-MDP는 금합금 표면에 교정용 장치를 부착할 때 사용하기 적합하다고 볼 수 있다.

주요어 : 10-MDP, 전단접착강도, 금합금 표면, 교정용 접착제

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