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Araştırma/ Research Article

THE CHALLENGE OF MDP MONOMER CONTAINING ADHESIVE SYSTEMS: COMPARISON OF SHEAR BOND STRENGTHS

MDP MONOMER İÇEREN ADEZİV SİSTEMLERİN REKABETİ: MAKASLAMA BAĞ DAYANIMLARININ KARŞILAŞTIRILMASI

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ABSTRACT

Aim: The aim of this in vitro study was to evaluate the shear bond strengths' of three MDP monomer containing self-etch adhesive systems.

Material and method: Sixty human third molars were used for the study. The teeth were sectioned with a low-speed diamond disk saw under water coolant to expose mid-coronal dentin. The teeth were randomly divided into three groups(n=20). The restorations (2.30mm diameter and 3mm heigh) builded with using three different MDP containing adhesive systems by aid of Ultradent Bonding Jig. After that, the specimens were placed in a universal testing machine and the shear bond strength was measured at a crosshead speed of 1 mm/min.

Result: Group 1 and 2 showed significantly higher bond strength than group 3($p<0.05$). There were no significant difference in bond strength values between group 1 and 2($p>0.05$) in spite of group 1 showed slightly higher bond strength values than group 2.

Conclusion: Within the limitations of this in-vitro study, all groups showed optimal results but first and second adhesives showed significantly higher values than third group.

Key Words: MDP monomer, Shear bond strength, Adhesive systems

ÖZET

Amaç: Bu in-vitro çalışmanın amacı MDP monomeri içeren üç farklı self-etch adeziv sistemin makaslama bağ dayanımınlarını değerlendirmektir.

Materyal ve Metod: Bu çalışma için atmış adet çekilmiş insan üçüncü molar dişleri kullanlmıştır. Dişler bir elmas kesme diski yardımıyla ve su soğutması altında kron boylarının orta üçlülerine kadar kesilmiştir. Kesilen dişler rastgele üç gruba ayrılmıştır (n=20). Ultradent Bonding Jig yardımıyla ve üç farklı MDP içeren self-etch adeziv sistem kullanılarak restorasyonlar yapılmıştır (2,30 mm çap ve 3mm yükseklik). Sonrasında örnekler test cihazına alınmış ve makaslama bağlanma değerleri ölçülmüştür.

Bulgular: 1. Ve 2. Grup restorasyonlar, 3. Gruba göre anlamlı derecede daha yüksek makaslama bağ değeri gösterdi. ($p>0.05$). Bunun yanında 1. Grubun 2. Gruba göre nispeten daha yüksek bağ değeri gösterdiği saptandı.

Sonuç: Bu in-vitro çalışmanın sonuçlarına göre, bütün grupların optimal bağ değerlerigösterdiği saptandı. Ancak 1. ve 2. Gruplar 3. Gruba oranla anlamlı derecede daha iyi bağlanma değerleri gösterdi.

Anahtar Kelimeler: MDP monomer, Makaslama bağ dayanımı, Adeziv sistemler

INTRODUCTION

For many years, the dental researchs in operative dentistry field striving to achieve better adhesion of dental composites to dental hard tissues, because strong adhesion between the tooth and restorative material would not only protect the sound tooth structure from unnecessary expansion for retention but also prevent the marginal gap formations occuring because of the polymerization stresses. And also consequently, less micro-leakage and restoration stability may be obtained.[1-7]

In 1955, Buonocore introduced the concept of acid etching which chemically treating the enamel to

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alter its suface characteristics to allow for adhesion of acrylic resins to the enamel. And on the basis of this idea total etch systems were developed in which both the enamel and dentin surfaces are acid conditioned to allow for resin adherence to tissues. Also in 1962 Ray L. Bowen advented the resin composite and brought a significant revolution in the field of esthetic restorative dentistry. Although dental composite resins have a lot of advantages there are still a challenge for clinicians to find a perfect bonding system and technique.

Adhesive systems are currently available as three-step, two-step and single step systems, depending on how the three cardinal steps of etching, priming and bonding to tooth substrate are accomplished. And there are various results about their bonding successes in literature.[1-16] Adhesion of resin materials to enamel has become a routine and reliable aspect of contemporary clinical dentistry because of enamel's highly inorganic substrate but adhesion to dentin has proved to be more difficult and less predictable due to the complex structure of dentin with a low inorganic content, heterogenous nature, presence of dentinal fluid and smear layer.[11-15,17,18.] Formerly there were seperate chemical components and also several application steps were needed for priming and bonding. Now current advances have focused on the development of delivery systems that simplify the steps involved. The newer concept of self etching systems have proven to be good both scientifically and clinically. They reduce the clinical steps, provide adequate bonding to enamel and dentin, prevents dentin from over-drying or releasing wet and ensure post operative comfort for patients.[10,11]

In self etching adhesive systems the functional acidic monomers are responsible from the etching process. There are a few functional monomers like, 4-methacryloyloxyethyl trimellitic anhydride (4-META), 2- methacryloyloxyethyl phenyl hydrogen phosphate (Phenyl-P) and 10-methacryolyloxydecyl dihydrogen phosphate (MDP)[19,20] These monomers serve various functions like etching tooth substrates, enhancing monomer penetration and also provide that adhesives chemically react with dental substrates.[21,22] In various studies researchers tried to understand the adhesion mechanisms of self-etch adhesives to dental hard tissues which including acidic monomers like 10-methacryloyloxydecyl dihydrogen phosphate (MDP), 4-methacryloyloxyethyl trimellitic acid (4-MET) or 2-methacryloyloxyethyl phenyl hydrogen phosphate (Phenyl-P).[23-27] However first Yohsida et al.[28,29] introduced the electrostatically bonding of these monomers to hydroxyapatite (HAp) and the producing of calcium salts which called "adhesion-decalcification concept" and also it has been shown in several studies that MDP monomer have better properties like lower water solubility, higher dentin bonding durability and more readily and intensively ionic binding capability to HAp than others.[23-31] Due to all these outstanding features MDP or MDP like monomers are becoming the most commonly preferred functional monomer in commercial self- etching adhesives. There is several in-vitro testing methods to evaluate the success of dental restorative products by mimicking the conditions of oral environment. In this context the mastication process which is one of indentation, basically related to shearing phenomenon the true nature of adhesive strength of the materials at the interface is depicted by the shear bond strength. This testing method is also very effective for assessing the bonding success of the products because the quality and efficacy of bonding of these adhesive materials is reflected in their mode of failure-either cohesive, adhesive or mixed. The number of cohesive failures within the dentinal substrates increases with increasing bond strengths.[32]

In this study we aimed to evaluate and compare the shear bond strengths and failure modes of different MDP monomer containing adhesive systems which one of them is the inventor of this product. One of these adhesives was two bottle self-etch system (inventor) and the others were one bottle self-etch system and our null hypotheses were that
(1) the shear bond strength of the two bottle self-etch system is significantly higher than the other two groups and, (2) the one bottle self-etch systems have the similar shear bond strength values.

MATERIALS AND METHODS

The Kırıkkale University Committee on Investigations Involving Human Subjects reviewed and approved the protocol and consent form used for this study.

Sixty human third molars, free of cracks, caries, and restorations on visual inspection, were used for the study. The teeth were scraped of any residual tissue tags, kept in a 2.6% sodium hypochlorite solution and rinsed under running water for 15 minutes each. Later, they were cleaned with pumice and stored in normal saline at 4°C until use.

The teeth were sectioned with a low-speed diamond disk saw (Markus Inc., Michigan, USA) under water coolant to expose mid-coronal dentin. The sections of the teeth including the roots were embedded in autopolymerizing acrylic resin to form cylinders of 2.5 cm in diameter and 5 cm high. Dentin surfaces were flattened using 600, 800 and 1200 grit waterproof polishing papers. The teeth were randomly divided into three groups (n=20).

In group 1, Clearfil SE Bond system (Clearfil SE Bond, Kuraray Co Ltd, Osaka, Japan) was used, first primer was applied to the dentin surface using microbrush by scrubbing for 20 seconds. The dentin surface was then dried with oil-free light pressured air. Than the SE bonding agent was applied to dentin surfaces and light cured for 10 seconds (XL3000 Curing Light, 3M-ESPE, Grafenau, Germany).

In group 2, Scoth Bond Universal (3M ESPE, St. Paul, MN, USA) was used. The one bottle self-etch adhesive was applied to the dentin surface using microbrush by scrubbing for 20 seconds. The dentin surface was then dried with oil-free light pressured air and light cured for 10 seconds.

In group 3, ALL Bond Universal (BISCO Inc., Schaumburg, USA) was used. The one bottle self-etch adhesive applied on dentin by two separate coats using a microbrush by scrubbing for 10-15 seconds per coat. The dentin surface was then dried with oil-free light pressured air and light cured for 10 seconds.

In all groups after adhesive application, the specimens were clamped in the Ultradent Bonding Jig (Ultradent Products; South Jordan, UT, USA), and respectively according to the groups; Photo Posterior (Kuraray Co Ltd, Osaka, Japan), Filtek P60 (3M ESPE, St. Paul, MN, USA), Aelite LS Posterior (BISCO Inc., Schaumburg, USA). The posterior restorative composites were carefully inserted into the surface by packing the material into cylindrical-shaped plastic matrices with an internal diameter of 2.30 mm and a height of 3 mm. Excess composite was carefully removed from the periphery of the matrix with an explorer. The composite was cured with a quartz halogen curing light (XL3000 Curing Light, 3M-ESPE, Grafenau, Germany) for 40 seconds. Materials used in the study were shown in Table 1.

Table 1. Materials used in the study.

Material	Composition
Clearfil SE Bond (Kuraray Noritake Dental Inc, Okayama, Japan)	*Primer* 10-Methacryloyloxydecyl dihydrogen phosphate (MDP), HEMA, hydrophilic aliphatic dimethacrylate,dicamphoroquinone, N-diethyl-p-toluidine, and water *Bond* 10-Methacryloyloxydecyldihy drogenphosphate (MDP), bisphenolA-glycidyl methacrylate (bis-GMA), HEMA, hydrophobic aliphatic dimethacrylate, dicamphoroquinone, N-diethyl-p-toluidine, and colloidal silica
Single Bond Universal (3M ESPE, St. Paul, MN, USA)	MDP Phosphate Monomer, Dimethacrylate resins, HEMA, Vitrebond^{TM} Copolymer, Filler, Ethanol, Water, Initiators, Silane
ALL Bond Universal (BISCO Inc., Schaumburg, USA)	MDP, Bis-GMA, Ethanol, Water, HEMA, Initiators
Photo Posterior (Kuraray Co Ltd, Osaka, Japan)	Silanated silica filler, silanated barium glass filler, silanated colloidal silica, TEGDMA, Bis-GMA, Urethane tetramethacrylate, di-Camphorquinone, Initiators, Accelarators, Pigments
Filtek P60 (3M ESPE, St. Paul, MN, USA)	Bis-GMA, UDMA, Bis-EMA, zirconia/silica fillers
Aelite LS Posterior (BISCO Inc., Schaumburg, USA)	Ethoxylated Bis-GMA, Bis-EMA, TEGDMA, Glass fiber, Amorphous silica

After storing in an incubator at 37°C in 100% humidity for 24 hours, the specimens were placed in a universal testing machine (Instron 8500, Instron Corporation, Canton, USA) and the shear bond strength was measured at a crosshead speed of 1 mm/min. The shear bond strength of composite resin to dentin was recorded in Newtons (N) and calculated in MPa taking into account the cross-sectional area of the composite buildup.

After the testing procedure, the fractured surfaces were observed with a stereo-microscope (SZ-

TP Olympus; Tokyo, Japan) at a magnification of 20× to determine failure modes and classified as adhesive failures (occuring purely within the restoration-dentin interface), cohesive failures within the composite or cohesive failures within the tooth and mixtural failures (combination of the adhesive or any of the cohesive modes).

One specimen from each group was sputter coated with gold after fracture and prepared for SEM examination. Coated specimens were then observed under the SEM (JEOL JSM 6400, Tokyo, Japan) with different magnifications.

The mean and standard deviation were calculated for the groups. One way analysis of variance (ANOVA) and Tukey HSD tests (significance level 0.05) was performed to determine significant differences in bond strengths between the groups.

RESULTS

The mean shear bond strengths and standart deviations are shown in Table 2.

There were statistical difference between different letters. Group 1 and 2 showed significantly higher bond strength than group 3 ($p<0.05$). There were no significant difference in bond strength values between group 1 and 2 ($p>0.05$) in spite of group 1 showed slightly higher bond strength values than group 2.

At the evaluation of failure modes by stereomicroscope all groups showed generally mixed failure but:

In group 3, mixed failures were occured mainly in the adhesive layer and less were in the restorative material but there were no dentin cohesive failure in this group. On the other hand in groups 1 and 2, mixed failures were occured like cohesive ones, both in the restorative materials and in the dentin structures (Table 3) (Figures 1-3).

In SEM analysis dentin and composite cohesive failures of groups 1 and 2 were seen more clearly. In these groups adhesive layers were seen with very sharp and smooth margins. In group 3 a thin superficial adhesive layer was seen. And this was seen that there was no breaking of effect in dentin structure in group 3 (Figures 4-9).

Table 2. Mean shear bond strengths and standart deviations (n=20).

Groups	**Mean±SD**	**Significance**
1 (SE Bond)	27,74 ± 7,66	a
2 (Scoth Bond)	25,36 ± 6,61	a
3 (All Bond)	20,3615 ± 3,81	b

Table 3. Failure modes and numbers

		Failure Mode		
Groups	N	Adhesive(N)	Mixed(N)	Cohesive(N)
1	20	0(%0)	18(%0) 6(%30) (Dentine-Composite) 12(%60) (Composite-Adhesive)	2(%10) 1(%5) (Dentin) 1(%5) (Composite)
2	20	0(%0)	13(%65) 5(%25) (Dentine-Composite) 8(%40) (Composite-Adhesive)	7(%35) 4(%20) (Dentin) 3(%15) (Composite)
3	20	3(%15)	17(%85) (Composite-Adhesive)	0(%0)

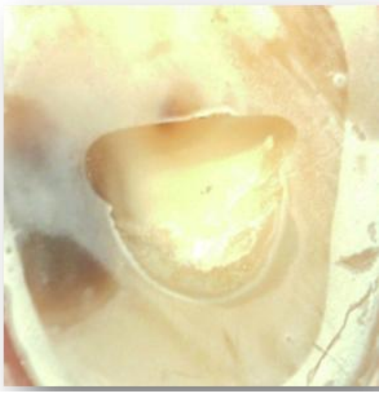

Figure 1. Stereomicroscope image of 1st group

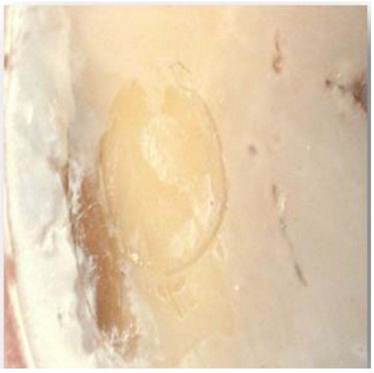

Figure 2. Stereomicroscope image of 2nd group

Figure 3. Stereomicroscope image of 3rd group

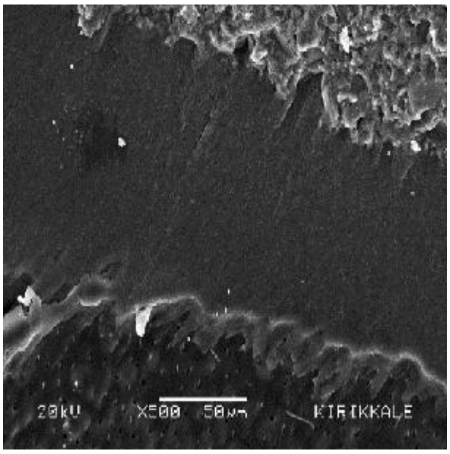

Figure 4. SEM image of 1st group (Magnification of 500×)

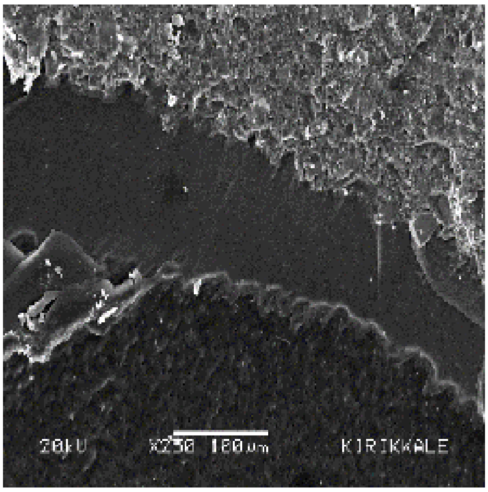

Figure 5. SEM image of 1st group (Magnification of 250×)

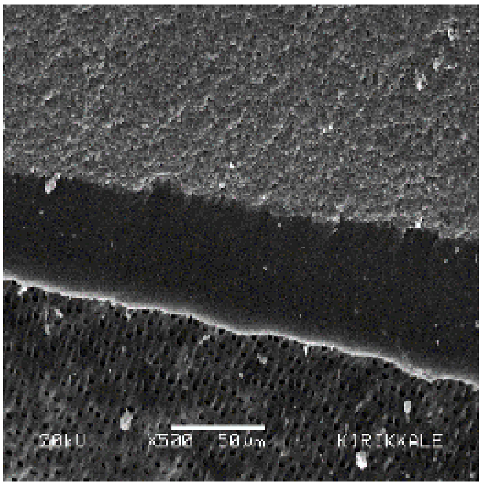

Figure 6. SEM image of 2nd group (Magnification of 500×)

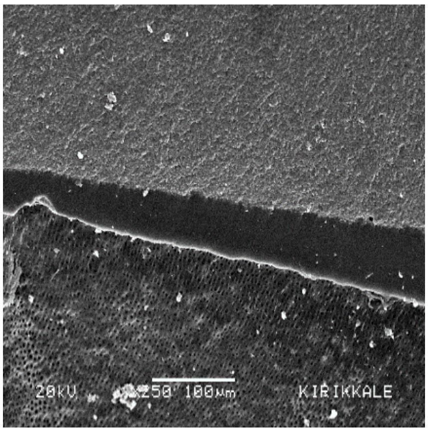

Figure 7. SEM image of 2nd group (Magnification of 250×)

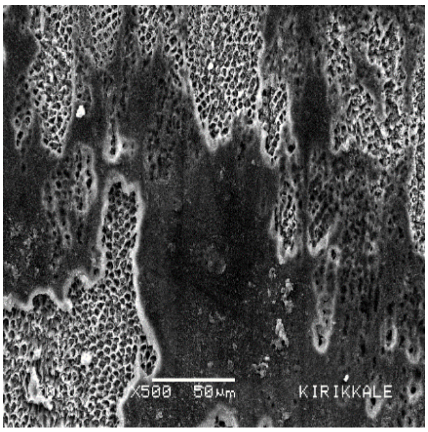

Figure 8. SEM image of 3rd group (Magnification of 500×)

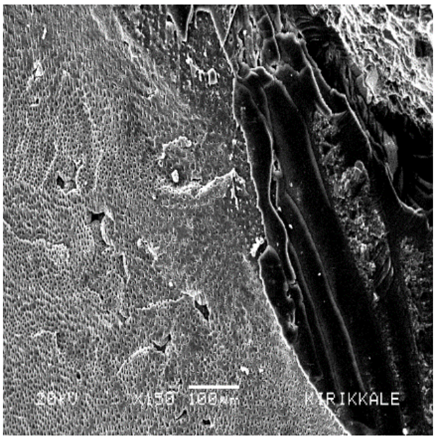

Figure 9. SEM image of 3rd group (Magnification of 150×)

DISCUSSION

Today, one of the primary objectives of dental researches is to achieve a strong, durable and easily achievable adhesion/bonding of dental materials to tooth tissues which is not only essential for mechanical successes of the restorations but also for aesthetic and biological successes. Especially because of the complete understanding of the nature of tooth substance studies have focused on the chemical structure of dental adhesive materials[(1, 2, 24-33] . In this context, since this concept first inroduced with Scotchbond 2 (3M-ESPE, UK&Ireland) in the early 1990s the developments in self-etch adhesives have been quite intense.

It has been reported that the chemical composition of adhesive systems determines clinical success[11, 33]. Probably the most important part of self-etch adhesives are functional monomers which plays very important role in bonding to tooth tissues by adhesion-decalcification concept. And MDP (10-methacryolyloxydecyl dihydrogen phosphate) is considered to be the most successful one among these monomers.[23,24,26,30,31]

Due to the proven success of MDP monomer (which produced originally by Kuraray Noritake Dental Inc.) some other companies tried to produce similar molecules. The shear bond strengths of three different manufacturer's MDP(or MDP like) containing adhesives were tested in this study. It has been postulated that the minimum bond strength of 17-20 Mpa is needed to resist contraction forces of resin composite materials for tooth hard tissues and also clinical experiences confirm that this bond strength is sufficient for successful retention of resin restoration [4,6,9,11,12,15]. All adhesive systems used in this present study achieved the optimal bond strength values. However, group 3 showed significantly lower values than group 1 and 2 while there were no significance in groups 1 and 2.

In stereo-microscope examinations similar tensile formats were seen at subjects of groups 2 and 3. In both of these groups no adhesive rupture has not occurred, the occured ruptures were mostly mixed which were formed as dentin-adhesive as formed like composite-adhesive. And also only dentine cohesive ruptures were present in groups 2 and 3. This results showed that the bond strengths of both restorative systems are even high enough to break dentin tissue. Besides, the only adhesive ruptures that determined in 3th group were in parallel relationship with the weak values obtained in mechanical tests. SEM images also supports these findings, namely, thin adhesive layer at group 3 seems like weakly spread to the surface, whereas in the other two groups, tight connections seem between dentin, adhesive and restorative composite. Also in SEM images of group 1 and 2 there were no residue seemed on the part of the dentin. This detail may be a proof of the strong adhesive connection in groups 1 and 2 which causes the dentin cohesive rupture. The strong adhesive bonding is important not only to resist contraction and masticatory forces but also for prevention of microleakages[11-15, 23-33]. In this respect, 1th and 2nd groups seem more advantageous according to the shear-bond strength results obtained in our study. However, in secondary caries formation like situations which requires tooth filling removal, group 3 (which have weak but in minimum required strength values of 17-20Mpa) may become advantageous since filler removing may be done with less impact on dental tissues.

Clearfil SE Bond (group 1) two step self etch adhesive system with its original MDP monomer has already proved itself in several studies [24-31,34-36]. Also in recent study the highest results obtained from SE bond group. Scoth Bond Universal (group 2) is a MDP monomer including one step self etch adhesive system recently marketed by a different manufacturer showed as high shear-bond strengts as group 1 therefore the first null hypothesis was rejected. ALL Bond Universal (group 3) is the other recently marketed one step self etch adhesive system showed clinically acceptible but significantly lower results than the other two groups therefore the second null hypothesis was rejected too.

CONCLUSIONS

Within the limitations of this in-vitro study, all groups showed optimal results but first and second adhesives showed significantly higher values than third group. It is a remarkable result that the single bottle Scoth Bond Universal showed similar results with two bottle Clearfil SE Bond. Thus, further studies associated with the other mechanical and physical properties are needed about the MDP functional monomer containing self-etch adhesives. And it is very necessary to examine the possible differences between MDP molecules of different manufacturers. And also it is essential that to investigate the chemical reactions occurring.

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