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UNIVERSITY OF GAZIANTEP
GRADUATE SCHOOL OF HEALTH SCIENCES

EVALUATION OF BONDING STRENGTH OF METAL BRACKETS TO AMALGAM RESTORATIONS USING DIFFERENT SURFACE CONDITIONING METHODS

Dler MOURAD

MASTER OF SCIENCE THESIS

DEPARTMENT OF ORTHODONTIC

THESIS ADVISOR
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DECLARATION

I hereby declare that this thesis is my own work and effort, has been generated by me as the result of my own original research.

I confirm that:

1. Where I have consulted the published work of others, this is always clearly attributed;
2. Where I have quoted from the work of others, the source is always given. With the exception of such quotations, this thesis is entirely my own work;
3. I have acknowledged all main sources of help.

27.6.2014

Dler MOURAD

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ABBREVIATIONS

mm	Millimeter
Psi	Pound per inch
kg/cm^2	Kilogram/centimeter square
Min	Minute
E-GF	E-glass fiber
KAP	Kuraray Alloy primer
MPa	Megapascal
ARI	Adhesive remnants index
SBS	Shear bonding strength
ADA	American Dental Association
µm	Micrometer
PMMA	Poly(methylmethacrylate)
Bis-GMA	Bisphenol-A Glycidyl Dimethamethacrylate
4META	4-Methacryloxy-ethyl trimellitate anhydride
10 MDP	10-Methacryloyloxydecyl dihydrogen phosphate
VBATDT	6-(4-Vinylbenzyl-N-propyl)amino-1,3,5-triazine-2,4-dithione
OSHA	Occupational Safety & Health Administration
FRC	Fiber reinforced composite

TEGDMA	Triethylene Glycol Dimethaacrylate
MMA	Methyl methacrylate
HEMA	Hydroxyethyl methacrylate
EDX	Energy-descriptive X-ray
kN	Kilonewton
ISO	International Organization for Standarization
°C	Celsius

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ÖZET

Ortodontik Metal Braketlerin Farklı Yüzey Pürüzlendirme Yöntemleri Uygulanan Amalgam Yüzeylerine Olan Bağlanma Dayanıklılığının Değerlendirilmesi

Dler MOURAD

Yüksek Lisans Tezi, Ortodonti Anabilim Dalı

Tez Danışmanı: Yard.Doç.Dr.N.Eren İŞMAN

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Bukkal yüzeydeki geniş amalgam dolgular braket tutuculuğu açısından ortodontistler için klinikte zorluk oluşturmaktadırlar. Bu çalışmanın amacı iki farklı kumlama sistemi ve adezivlerinin karşılaştırılması ve E-cam fiber (E-GF) in bağlanma dayanımına (SBS) olan etkisinin araştırılmasıdır. 12 çekilmiş insan molar dişi çalışmanın kontrol grubunun oluşturulması amacıyla kullanıldı. 72 adet amalgam örneği 6 gruba bölündü. Grup 2 ve Grup 3 te amalgam yüzeyi 50 µm alüminyum oksit ile pürüzlendirildi. Grup 2 de amalgam yüzeyine, Grup 3 te amalgam yüzeyine ve braket tabanına Kuraray metal primeri (KAP) uygulaması yapıldı. Grup 4 ve 6 silika tabaka sistemi ile pürüzlendirildi ardından Grup 4 te Rely-X uygulaması, Grup 6 da Rely-X uygulaması ve fiber ilavesi yapıldı. Grup 7 de , Grup 6 ya ilave olarak braket tabanına KAP uygulaması yapıldı. Grup 5 te ise parlatılmış amalgam yüzeyine fiber ilavesi yapıldı. Bütün gruplarda adeziv materyali olarak Transbond XT uygulandı. Çalışma sonuçlarına göre amalgam yüzeyinin SBS değerleri mine yüzeyine göre daha düşüktü, sadece grup 4 teki değerler ile mine yüzeyi arasındaki fark istatistiksel olarak anlamlı değildi. E-GF uygulaması yapılan gruplarında bağlanma başarısızlığı amalgam adeziv ara yüzeyinde gözlendi. Sonuç olarak amalgam silikatizasyon işlemi ortodontik braketlerin bağlanma dayanımlılığının artırılmasında faydalı bir yöntemdir ve E-GF kullanımı koheziv başarısızlığının ve debonding sırasındaki amalgam kırılma riskinin azaltılmasında alternatif bir yöntem olarak kullanılabilir.

Anahtar Sözcükler: Amalgam**,** metal braket, shear bond, orthodontic braketler, silan tabakalama

ABSTRACT

EVALUATION OF BONDING STRENGTH OF METAL BRACKETS TO AMALGAM RESTORATIONS USING DIFFERENT SURFACE CONDITIONING METHODS

Dler MOURAD

Master of Science Thesis, Orthodontic Department

Thesis Advisor: Assis. Prof. Dr. Nurettin Eren İŞMAN

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Bonding orthodontic brackets to extensive amalgam restorations on buccal aspect of molar teeth is still a challenge for orthodontists. This study aims to compare two systems of sand blasting with their adhesive and to investigate the effects of E-glass fiber (E-GF) on shear bonding strength (SBS). 12 extracted molar teeth enamel serves as control group, 72 amalgam samples divided into 6 groups. Groups 2 and 3 were blasted with aluminum oxide 50 μm and treated with Kuraray alloy primer (KAP) on the amalgam surface in group2 and on amalgam surface and bracket base in group 3.

Samples in groups 4, 6 were blasted by silane coating system with application of Relyx silane coupling agent in group 4 and addition of piece of (E-GF) in group 6, for group 7 samples were treated like group 6 with the application (KAP) on the bracket base, only group 5 was not sandblasted and the (E-GF) are applied directly on the polished amalgam surface. All groups were bonded with Transbons XT adhesive system.

The results reported that SBS to amalgam samples were lower than the enamel samples in all groups, only group 4 revealed SBS value not significantly different from control group, regarding the ARI scores the results show bond failure occur at amalgam adhesive interface with the (E-GF) groups, Conclusion: None of the amalgam groups record SBS higher than enamel, Silicatization of amalgam is a useful method for bonding orthodontic brackets to amalgam, and using of (E-GF) decrease the cohesive failure and amalgam damage during debonding.

Key Words: Amalgam, Metal brackets, Shear bond strength, Orthodontic bracket, Silane coating.

1. INTRODUCTION

Adequate bonding strength is essential to accomplish the aim of orthodontic treatment because during the treatment there are usually different types of debonding forces applied to the tooth.

Many studies have be done to increase the value of shear bonding strength of metal brackets to the different intraoral surfaces such as enamel, dentin and restorations like restorative resins, porcelain and amalgam.

The percentage of adult patient who look for orthodontic treatment has been increased in the last decades and extensive amalgam restorations are frequently found in those patients on the buccal or lingual surface of molars, therefore, the ability to bond orthodontic attachments to these restorations will be of clinical importance (1).

Of course banding will be the choice for most practitioners because banding of those extremely restorated teeth can withstand the force of debonding, but according to some literature banding lead to diminished gingival and periodontal health (2, 3). In addition to the time consumed and pain that occur during placing bands.

Bonding of the brackets to the amalgam is still one of the difficult clinical situations manifested in orthodontic practice; new materials have been used in many published studies to gain an acceptable bonding strength that can satisfy the standard requirement of orthodontic treatment.

The bonding strength may depend on the type of the amalgam (4) or the ability of intermediate adhesive to bond to metal (1, 5, 6) or on the different conditioning methods of the amalgam surface (7).

Silicatization of the alloy surface is a system used by many literature in restorative dentistry for intraoral repair of the fractured amalgam restoration by increasing alloy resin bonding (8) and in prosthetic dentistry for repair of fractured porcelain restorations and it was used also in orthodontic to increase the bonding strength of metal bracket to different types of alloy and to improve the adhesion of metal bracket base and resin composite used for bonding (7, 9).

Reinforcing the composite bonded to amalgam by using of supporting fibers have been reported in previous literature (8).

But evaluation of the bonding strength of metal brackets to amalgam after *Silcatization* of amalgam surface and strengthening the adhesive by reinforced fibers was not informed by published studies until now.
In vitro studies are always conducted before clinical application of materials to evaluate the quality of the material and its ability to provide adequate bond strength clinically.

So the purpose of the present study is to compare the in vitro shear bonding strength of metal bracket to *silicatized* amalgam surface with and without fiber reinforcement to the conventional methods of bonding to amalgam using alloy primer and to the standard values of shear bonding strength that obtained after bonding of bracket to the enamel using acid etching which is considered as a gold standard for comparison of any shear bonding strength.

2. LITERATURE REVIEW

2.1. Evaluation of Bonding

Bonding or what is called Adhesion is a word that was used commonly in many branches of our life in industries and everyday life but in dentistry it is the core of modern dental practice.

Adhesion according to concise Oxford English dictionary means sticking together particles of different substances (10).

After the development of acrylic resin and using it as an esthetic dental restoration by addition of pigment to the formula, the problem was how the bond strength of these materials to the enamel can be increased.

The experiment started in England 1950 by Dr. Oskar Hagger who works on the development of Sevitron Adhesive (11), then During the 32nd annual meeting of the international association for Dental Research in 1954, Dr. Michael Buonocore suggested that using 85% phosphoric acid solution resulted in an adhesion of acrylic resin to enamel that lasted 1070 hours when stored in water (12).

After the Bounocore suggestions, the alterations of the surface of substrate before bonding to increase the micromechanical retention become a goal for all practitioners when bonding the adhesive materials to any surface in the dental practice either mineralized tooth surfaces (enamel, dentin, cementum) or restorative materials (amalgam, composite, porcelain, alloys).

The bonding with acid etching of enamel is considered the gold standard for bond strength and longevity to which all other technique are compared (13).

Later three different methods of adhesion have been distinguished (1) micromechanical adhesion in which retention of the material to be bonded is obtained by producing micro holes; which could be seen in acid etching Bounocore technique. (2) Chemical adhesion between molecules of adhesive and molecules of substrate which was invented in the 1970s by Dennis Smith who develop polycarboxylate cement, glass ionomer cement is another good example of chemical bonding that developed later by Wilson, Crisp, and McLean (13), in addition to that chemical bonding can be seen in modern dentistry by

using coupling agent (3). A complex mechanism of wetting and penetration then the formation of a layer of bound material between the restorative and the substrate (14).

Some new methods also incorporate two of these previously mentioned methods like abrasion the substrate surface to create micromechanical retention pits and application of chemical adhesion material to the substrate to mediate the chemical reactions between adhesive and substrate.

Alteration of the surface of substrate can be done by acid etching (12), or by roughing surface by air abrasion using different abrasive material (15, 16), or by ablation using laser.

Bonding is essential part of orthodontic practice and the most important step in fixed appliance therapy, so the maintenance of good bonding during the treatment will reduce the time consumed by the clinician, decrease the total treatment duration, and give better results. Therefore many orthodontic studies have been related to the efficiency of adhesive in bonding brackets to the various type of substrate that encountered inside the oral cavity especially dental enamel which is the surface that more commonly used for bonding orthodontic brackets.

2.1.1. Bonding to enamel

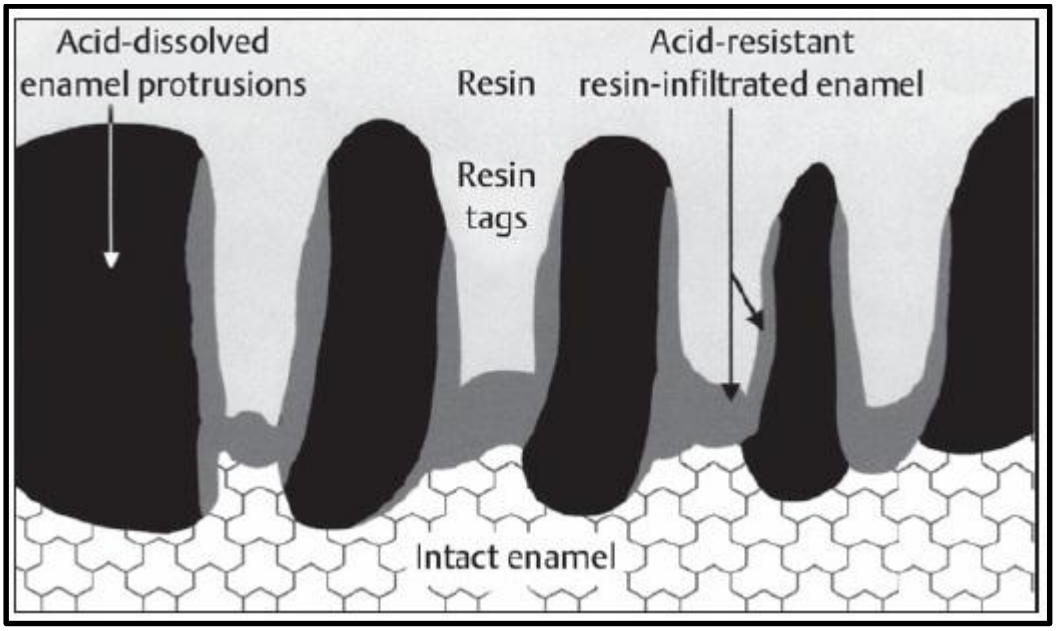
Developing of phosphoric acid etching was a revolution in resin bonding technique because of the good results of bonding strength when used for pits and fissure sealants it is logically to go to the next step and using it with cut enamel surface (17).

The concentration of acid that was used by Buonocore was decreased to overcome the effect of high concentration on the oral environment (18), and the duration of placing the acid etch on enamel also reduced (19). Phosphoric acid is so commonly used in dental practice although there are many acids that can be used as an etchant like pyruvic, citric, oxalic, nitric or maleic acid.

The acid etch remove the organic pellicle on the surface of intact enamel to allow better bonding with composite by forming micro depressions in the enamel surface then the resin fill this depression and replace the dissolved hydroxyapatite crystals and forms resin tags (Pattern 2.1).

In bur cut enamel a smear layer form a barrier between resin adhesive and enamel (20), so acid etch will clean this layer then demineralize the hydroxyapatite crystals to create

tiny retentive holes in which the resin materials take place and this enhance the mechanical adhesion of resin material (21).



Pattern 2-1 Resin Tags in intact enamel [22]

2.1.2. Bond strength of orthodontic brackets

Many studies have been done to measure the bond strength of various dental materials (6, 22, 23). In orthodontic, bond strength is a unique term which can be explained as the value that reflect the force needed for deboning to occur in Newton divided by the surface area of the bracket base in mm^2 which is equal to one megapascal (24), one megapascal is equal to 145 pounds per square inch (psi) which also can be converted into kg/cm^2.

The force value is frequently used in literature to compare quality of the adhesion, and the validity of bonding material for different situation.

The easiest method to measure the bond strength of orthodontic brackets is to simulate the intraoral conditions in the laboratory and then application of this force on the samples of brackets to measure the strength. The in vivo studies are giving more reliable values of bonding strength but it is more difficult and need cooperation from patient (25-27); until now there is no standard technique for in vitro bond strength testing, there are many factors affecting the test: (a) the direction of the force applied, (b) the speed and configuration of the cross head of testing machine, although slaw speed will allow

better control of sample position and the force applied but it is believed that debonding occur in the oral cavity is more complex mechanism and it happens in more rapid rate, (c) the brackets design, (d) the statistical methods used for comparison of raw data (24), a study done by Eliades and Brantley reported different bond strength with different bracket design (28), these variation may be due to (a) various shape of bracket base, (b) stress concentration is changed from group to group because of the morphology of the bracket, (c) difference in thickness of the bonding material, (d) different rheological properties of adhesive .

2.1.2.1. Types of bond strength testing:

Understanding the basic principles in mechanic is important for the researchers in dental material field to be able of applying the same principles for biologic studies.

The properties of orthodontic materials should be tested in the biomaterial laboratories before clinical application to test its efficiency and ability to produce the desired effects.

The direction of the force applied will determine which type of test will be done, testing of orthodontic bond strength can be:

Shear /peel testing:

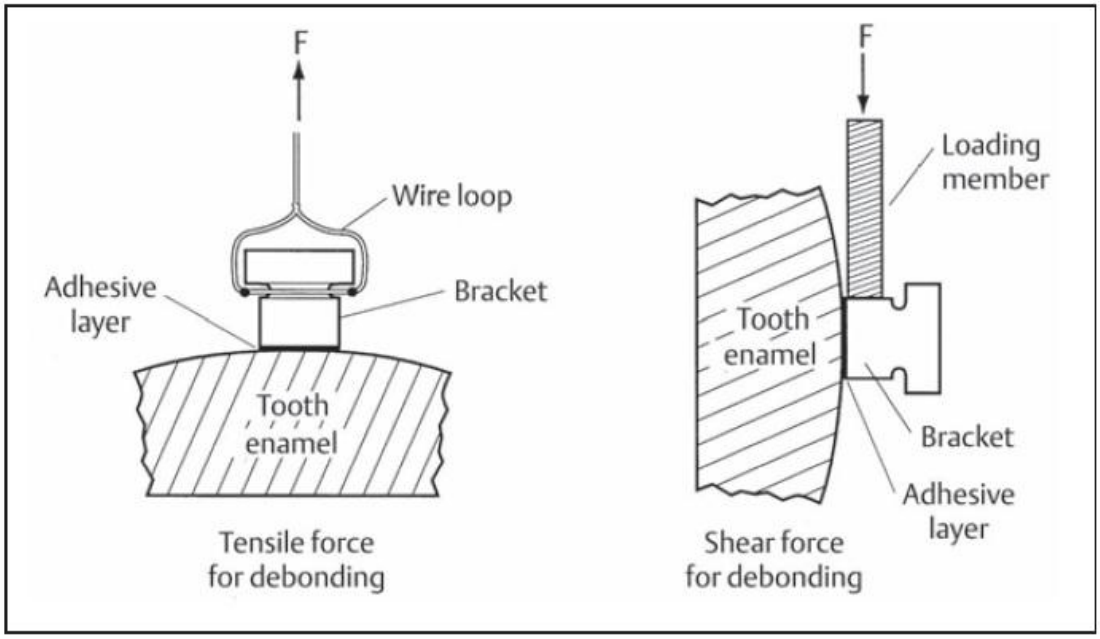
In mechanical engineering the shear stress is the stress that applied on an object in parallel direction to the object so that the shear force cause one side to move along the other side, in orthodontic the shear test is usually done by positioning the debonding blade parallel to the surface and bonded bracket (29), (Pattern 2.2).

Tensile testing

Tensile strength is the maximum strength that a material can tolerate when pulling action is applied to it (30). In orthodontic tensile strength is done by application of the force perpendicular to the surface and bonded bracket but in pulling manner (31), (Pattern 2.2).

Torsional testing:

Torsion is bend that occur in the material when torque is applied to it perpendicular to long axis, according to Katona orthodontic testing of torsional strength can be done by twisting the bracket until debonding (31).



Pattern 2-2 Shear & Tensile bond strength test [22]

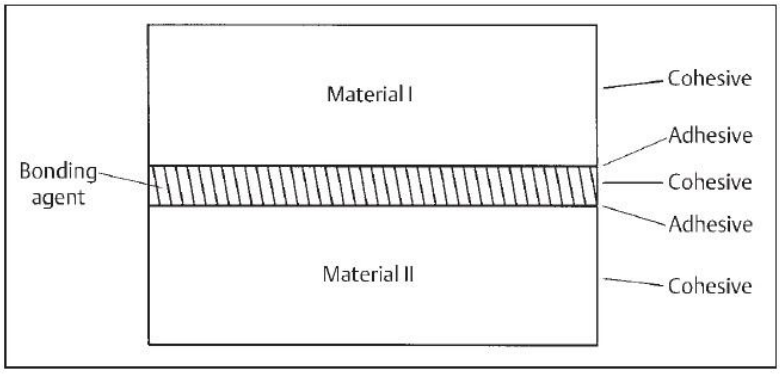
2.1.2.2. Bond failure

For long clinical success of any adhesive the bond strength of 20 to 25 MPa (2,900–3,400 psi) is essential in the stress bearing area (13), but this high force is not necessary for orthodontic bonding.

Bond Failure means breaking the bond between the adhesive and the substrate by application of force, this failure may occur in two areas so it can be classified according to the area of failure into:

Adhesive failure when the failure occurred in surface between adhesive and substrate

Cohesive failure if failure occurred inside the adhesive material (11), (Pattern 2.3).



Pattern 2-3 Sites of bond failure

Some times during the in vitro testing procedure the teeth to tested are fractured, this also considered a type of cohesive failure and it means that the bonding strength is more than the teeth strength and this is not an advantage because it will cause loss of the substrate structure, so an optimal bonding strength is necessary for dental applications (13).

In most of instances failure of bonding occur as both adhesive and cohesive failure so to determine the percentage of each of them there is a scale that determine the amount of adhesive remain on the substrate and on the restoration or brackets (11).

This scale is called adhesive remnants index (ARI) which was used for the first time by Artun and Bergland to determine the percentage of adhesive on the tooth, the index was a result of pilot study that have been done on 20 extracted teeth.

ARI criteria:

Score 0: no adhesive left on the tooth;

Score 1: less than half of the adhesive left on the tooth;

Score 2: more than half of the adhesive left on the tooth; and

Score 3: all adhesive left on the tooth with a distinct impression of the bracket mesh (32).

ARI became one of the most frequent and usual methods of testing the quality of adhesive strength in orthodontic practice, there was many studies tried to modify and improve the index , a study was conducted by of O'Brien *et al.* (33) try to develop a new method of evaluating the remnants by digitizing the magnified enamel picture and the amount of remnants is determined from the brackets base .

Some of studies state ARI under a magnification of 10X, other used 16X and 20X but according to Montasser & Drummond there is a significant difference in ARI scores

according to magnification and 20X decrease the sample with score 0 and increase the sample of score 2 (34), which mean to some extent the remnants are more obvious in 20X.

2.1.2.3. Testing machine

Testing the force of debonding is done by universal mechanical instruments worked with screw driven or servohydraulic that differentiate by the methods of cross head movement and application of force to the specimen (24). In the screw driven machine the cross head moves by two screws attached to it but in the servohydraulic machine the head moves by oil pressure pump. Because both types of testing machine can be used to measure the debonding force applied in different direction either shear, tensile or torsion bonding strength they are called universal testing machine in the literature (24).

2.1.2.4. Accuracy of bond strength tests

There is a wide conflict about the reliability of bond strength test in laboratory environment and the application of the values obtained from in vitro studies to the clinical practice.

There are many factors affecting the bond strength intra orally and cannot be simulated very well in laboratory, in addition to that the validity of the equation (force divided by surface area) that is used in most mechanical tests is still not approved by many researches because this equation assume that stress is evenly distributed over the surface area but a research has been done by Van Noort *et al.*. depending on finite element analysis reported different stress pattern through the surface area (35). Another factor which affects the certainty of in vitro tests is the repeated cohesive failure in the substrate (36).

The solution to found a way out of these problems was with invention of micro tensile bond tester which reduce the rate of substrate failure by making the samples more small (37). According to many studies bond strength test is used only for comparison of different system quality by testing them under the same conditions and by the same testing machine with changing some variables (38), therefore its recommended to make short term clinical studies to support the laboratory findings (24).

2.2. Cast Alloys

That cast alloys was divided by American dental association in 1984 into high nobel alloys, nobel alloys, and predominantly base metal alloys, according to ADA (gold, platinum, ruthenium, palladium, iridium, rhodium and osmium) are noble metals. although silver is nobel metal but it was not considerd a nobel by ADA because of liabelity to corrosion, there were so many methods to increase the bonding of resin to metal alloys varying between roughning of the surface to enhance adhesion by using differnt particles for sandblasting, diamond burs, or by using slicone carbid grits, and other methods like tinplating and Adlloy technique by adding gallium and tin solution electrically (24). With these different methods the bonding of orthodontic bracket could be more easier to metallic restoraed teeth especially amalgam which is the most common.

2.2.1. Amalgam

Amalgam is a popular word in all languages which means a mixture of two or more metals one of them is mercury (14).

The Word Amalgam originated in late 15th century: from French *amalgame*, Medieval Latin *amalgama* or from Greek *malagma* and all of them mean an emollient (10).

The tin mercury filling was reported to be used in China in A.D 600 (39), this material was first produced in western society in France in 1800 then found a way to USA in 1830 (40).

In 1850 one of the dentist who used amalgam in United State of America was at risk of malpractice and here what is called amalgam war was started (41).

Until now the war is still in progress, but amalgam is still used by many practinioner over the world especially in load bearing area.

The alloy powder according to the ISO Standard for dental amalgam alloy (ISO 1559) is composed of silver, tin and copper. Small quantities of zinc, mercury and other metals such as indium or palladium may be included in some alloys (14).

The high temperature at which components of alloy are melted will cause also oxidation that will affect the physical properties of amalgam therefore addition of zinc will be of great benefit to react with the oxides and forming a layer of zinc oxide which is easily

removed. Commercially there are many types of amalgam alloys depending on the composition as we previously said, or depending on the shape and size of the particles. The particles could be cut from ingot of alloy to form a lathecut particles, the size of theses particles my be fine or coarse, the other methods is called *atomization* by spraying the melted alloy in an inert gas so that the droplets of alloy will direclty solid to spherical shaped particles .

Many alloy powders are formulated by mixing particles of varying size or even shape in order to increase the packing efficiency of the alloy and reduce the amount of mercury required to produce a workable mix (14).

2.2.2. Bonding resin to cast alloys and amalgam

It is clear that conventional bonding with acid etch and diffusion of resin molecules in the amalgam cannot be done in the same way as enamel and the standard value of shear bonding strength that required to withstand the various forces applied in oral cavity is difficult to be obtained with amalgam.

In specific situations we need to bond resin materials to amalgam restorations to repair fractured old amalgam without replacing the amalgam or to bond orthodontic brackets to teeth with extensive buccal amalgam restorations.

From the theoretical point of view improving resin bonding to amalgam can be done by three ways (a) alteration of the surface of amalgam restoration to establish mechanical retention for the resin like sand blasting (4), or laser ablation (42), (b) modification of resin material to enhance bonding to metal molecules (6), (c) Using of an adhesive material that can promote the bonding between resin and amalgam (8). Most of these methods were evaluated by several studies and the highest shear bonding strength obtained was recorded in (Table 2.1).

2.2.2.1. Modification of amalgam surface

Previously the roughening of metal surface was done by diamond bur that produce macro mechanical large retention pits in the restoration but the development of intra oral abrasion methods using air borne particles lead to more successful bonding of resin to various alloy used in the oral cavity without removing the restoration.

The air particles that were used for roughening of metal alloy varies from aluminum oxide particles to cubic boron nitride and synthetic diamond particles (43) or even silica coating particles which usually came with porcelain repairing kits (8).

Table 2-1 Published studies with SBS and test parameters of resin composite bonding to amalgam

Study	Alloy Type	Surface Conditioning	Adhesive Primer	Resin Composite	Thermocycles	Type of Bracket	The Highest Bond Strength MPa	Material used
Sperber ***et al.(5)***	Admixed non-gamma 2 amalgam	50 µm[Al2O3] 5% Sodium Sulfide	N.A.	Phase II resin Panavia EX C&B Metabond	10.000 cycle	Maxillary central incisor metal brackets	18.67	50 µm [Al2O3] Panavia EX
Nergiz ***et al.(7)***	Gold-silver, Palladium-silver, Nickel-chromium, Cobalt-chromium, and titanium	30&125 µm Diamond bur 50&110 µm [Al2O3] 30 µm Silica	(ESPE-Sil)	Concise Self-curing composite resin	5000	Maxillary central incisor metal brackets	19	30µm Silica ESPE-Sil Concise
Oskoee ***et al.(42)***	Admixed non-gamma 2 amalgam	Er,Cr:YSGG laser 50 µm [Al2O3]	Kuraray Alloy Primer	Panavia F resin	N.A	Premolar Metal brackets	6.30	Er,Cr:YSGG laser Panavia F
Zachrrison ***et al. (1)***	Non-gamma 2 Lathecaut amalgam	50 µm [Al2O3]	All bond II Scotch bond Concsie	Panavia Ex SuperbondC&B Geristore Concise	N.A	Mandibular incisor metal bracket	6.4	50 µm [Al2O3] Superbond C&B
Buyukyilmaz ***et al. (4)***	Sphereical Admixed Lathcut	50&90 µm [Al2O3]	Reliance metalbond Amalgambond plus Allbond II	Modified Concise composite resin	1000 cycles	Mandibular incisor metal brackets	11	Spherical amalgam 90 µm [Al2O3] Reliance metabond
Germec ***et al.(6)***	Lathecut non-gamma 2 amalgam	50 µm [Al2O3]	Reliance Metal Primer Power Bond™ OLC One-Step Plus	Unite Resinomer	1000 cycles	Mandibular incisor metal brackets	7.15	50 µm [Al2O3] Reliance Metabond Unite
Ozcan ***et al.(8)***	Lathecut non-gamma 2 amalgam	30 µm Silica 50 µm [Al2O3]	Kuraray Alloy Primer Espe sil Schotchbond Multipurpose Adhesive	Tetric Ceram	6000 cycles	No bracket only composite	23.6.	30 µm Silica Espe sil Everstick Tetric Ceram

2.2.2.2. Modification of resin materials

The ability of resin to achieve a chemical bond with metal may improve the dental practice and reduce the time consumed to alter the surface of metal for retention.

The resin material was introduced into prosthetic dentistry before 1900 but the poly-methylmethacrylate (PMMA) was used as a denture base for the first time in 1936 (44), this material was heat processed so it couldn't be used intraorally. Depending on the same principle resin composite Bisphenol-A Glycidyl methamethacrylate (Bis-GMA), or Bowen's resin was produced and employed for different restorative procedure (44). Changing the chemical composition of conventional resin contributed to the invention of new resin material with improved bonding properties to metal and various substrates. Some of these chemically modified resins are used more commonly over the recent years. They can be supplied in powder- liquid system such as self-curing composite in which quartz filler powder is mixed with a liquid of dimethacrylate monomers and a methacrylate–phosphate, others are applied in one bottle system that contain

- (4-META) resin (4-methacryloxy-ethyl trimellitate anhydride)
- (10 MDP) resin (10-Methacryloyloxydecyl dihydrogen phosphate)
- (VBATDT) resin (6-(4-Vinylbenzyl-N-propyl)amino-1,3,5-triazine-2,4-dithione) (45, 46), those materials are called *Metal Adhesives.*

The adhesion of the resin materials to the metal framework and the adhesion of metal restoration to tooth structure have been mentioned in many papers of prosthetic dentistry. In the early of 1970 retention to metal was depending on macromechanical concepts, but later because of the need to develop more precise restoration in prosthodontics the concept of micromechanical retention was employed (47).

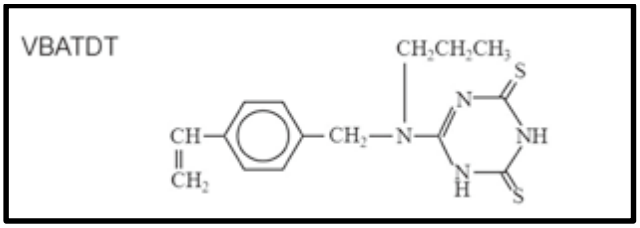
Micromechanical retention is considered the main bonding technique used to adhere the resin composite to enamel and dentin (48), and it depend on the process of hybridization which means the dispersal of resin molecules in the micro holes created by acid etching (49).

The first generations of metal adhesives containing (4-META) were produced by *Tanaka et al.* and it gave bond strength of 250 kg/cm^2 even after 500 thermocycling.

The two carboxylic groups of (4-META) attached to the aromatic group and responsible of the acidic nature of the adhesive but the hydrophobicity of aromatic group moderate the acidity of carboxyl groups (50) so the best solvent for this material is acetone (51).
The (10-MDP) was developed on the basis of (4-META) monomer by Kuraray (Osaka, Japan), it was first used as etching monomer, because of the dihydrogenphosphate group.
Chemically, this adhesive is very hydrophobic because of the long carbonyl group; Kuraray used acetone because again it is the best solvents for this monomer. This monomer was considered as the most promising for bonding to enamel and dentin by Yoshida *et al.* (52).

These two adhesive were used firstly with non-noble alloys (45), and gave a good result for repairing fractured amalgam restoration with resin (46) .
One of specific characteristic of metal surface is formation of a natural oxide layer that play an important role in bonding mechanism (14) but formation of this layer is limited to non-noble alloys.
New attempts were done to produce oxidizing layers on the noble metal alloys to improve the bonding of (4-META) to this alloys, this layer was produced by tinplating technique (53) which was very complicated and expensive method to produce oxide layer (47), another method was to increase the temperature of some metals into 400 °C in air for 10 minutes which will produce a copper oxide layer that aid in retention of modified resin (14)..
To eliminate the complexity of tinplating and the time consuming of heating method, a new material called (VBATDT) was synthetized to improve the bonding to noble metal alloys (54, 55), the invention of this material which is considered very cost effective for bonding to precious metal alloys defines the beginning of the second generation of metal adhesive (47). This monomer contains sulfur which inhibits the polymerization of resins initiated by the (BPO-amineredox) system. Therefore using of sulfur containing materials is preferred with partially oxidized tri-n-butylborane (TBBO) as an initiator (47).
In the present study Kuraray alloy primer was used which (VBATDT), (10 MDP) solved in acetone, because of that (ALLOY PRIMER) improve the bond strength to both precious and also nonprecious metals, and amalgam contain a mixed of noble and non-noble alloys so it is indicated as substrate for bonding by alloy primer.

Phosphoric acid group of (MDP) is responsible for bonding to the nonprecious contents of an alloy, while the sulfur atoms in (VBATDT) bonds to the precious contents of the alloy the double bonds on the other end of the molecule copolymerize with the resin monomers (Pattern 2.4).



Pattern 2-4 VBATDT chemical Composition

2.2.2.3. Bonding to metal using adhesive material

Another technique to improve bonding to metal was by sandblasting the surface of metal alloy in tribomechanical approach by using silica coated corundum particles of a 30 μm size, the corundum particles produce roughening of surface and transfer the silica particles to the substrate. This surface called *silicatized* surface to which a Silane coupling agent is applied to promote bonding to resin material (14).

2.3. Air Abrasion

Air abrasion introduced for the first time by Dr.Robert Black for Roughening of the cavity wall and non-mechanical preparation of cavity form (56).

But the suggestion of Black to use air abrasion for cavity preparation did not gain the support required to continue the researches in air abrasion technology. This was because the popular restorations at that time were amalgam and gold and cavity preparation by air abrasion need high suction of powder which was not available that time and the invention of air turbine hand piece eliminate any chance for air abrasion technology to get popular (57).

Then in 1995 a new research was done for using of aluminum oxide particles for abrasion of enamel and dentin surface with and without using of acid etch.

After that many researches had been done in field of air abrasion, some of them used enamel or dentin as a substrate (15, 16, 58), and some of them used different type of restorations, even the orthodontic bracket base had been sand blasted by air abrasion technology in some literatures (8, 9, 59).

Air abrasion also called kinetic cavity preparation works by generating of aluminum oxide particles of specific size, silica oxide particles or other particles with high air pressure and this particles will hit the substrate structure with high velocity and alternate the surface or remove the substrate depending on the size of particles, amount of air pressure, the type of the particles, the nozzle diameter of the handpiece, angulation of nozzle of the handpiece, distance from, and duration of exposure.

For etching the surface of most substrate air pressure of 80 PSI with particle size between 27 to 50 μm and distance of about 2 mm will be enough (8, 43).

The system of air abrasion may be mechanical control of air flow or digital that allows interrupted pulsed mode of action, the system must be used always with rubber dam, high suction and proper evacuation and care must be taken in case of respiratory diseases (57).

Also one of the important things is that air abrasion of amalgam will cause vaporization of mercury according to some studies, air abrasion of amalgam for 1 min releases mercury vapor four times in excess of the OSHA standard (60) so the duration of action for etching should be so short.

2.4. Silane Concept in Bonding Resin Composite to Metal

Silane coupling agent is a material that is frequently used in prosthetic dentistry usually for repairing of porcelain fixed partial dentures. Silanes materials constitute a large group of organic compounds in which (S) Silicon Atom binds with two functional groups the organic functional groups like:

(vinyl $–CH=CH_2$, allyl $–CH_2CH=CH_2$, amino $–NH_2$, isocyanato $–N=C=O$) which usually react with the organic materials like resin composite material, and the inorganic functional group or alkoxy group (e.g.methoxy O CH_3, ethoxy$–O–CH_2CH_3$) which combine with the inorganic surfaces like metals (61).

According to this the silane general formula is R-Y-SiX3, where R is an organofunctional group, Y a linker part, and X are hydrolyzable alkoxy groups (62).

So Silanes has the ability to make a bridge between two absolutely different materials because of the two functional groups. And sometimes it may contain a reactive group such as chloride (-Cl) or may be propylene (-$CH_2CH_2CH_2$–) is added between silicon atom and organic functional group especially when one of the substrate to be bonded is metal.

Silanes may be divided according to the number of silane atoms in the molecules into Monofunctional, Bifunctional, or Trisfunctional.

2.4.1. Application of silane in dentistry

Silanes are useful materials for various applications in dental science. At the beginning silane was used for bonding of filler and matrix in the new composite formula.

Dr. Bowen insert a strong ceramic filler particle into composite material to form a much stronger dental restorative material but the bonding between the ceramic material which constitute the filler and the matrix of resin composite need to be enhanced by coating the filler particles of ceramic with dual functional Silanes (13), also Silanes can be used in impression material but one of the most effective application of silane is in dental adhesive for repairing fractured crown and bridge with resin composite,

In1989 (3M ESPE, SEEFELD, GERMANY) company introduce a product named (ROCATEC) for extra oral conditioning and tribochemical silica coating, the particles size was 110 µm of silica particles (63).

This technique of conditioning and application of silane primer improve the bond strength of resin composite to metal, porcelain and even to composite itself (64), after (ROCATEC) the company reduced the size of silica particles to 30µm for more efficient use for intraoral application and introduce (COJET) silane system for repair of fractured porcelain restorations (8).

Another silane product produced by (3M ESPE, SEEFELD, GERMANY) is (RelyX ARC) system for cementing crown and bridge more efficiently by using silane.

2.4.2. Chemical reactions of silane

Reaction of silane with water which is called hydrolyzation of silane is essential for forming silanol groups that bind together to form dimers and then dimers form a bigger silioxane oligomer. The PH [4-5] is needed for hydrolyzation to occur, and the simplified chemical reactions are described as:

$R´–Si(OR)_3 + 3H_2O \rightarrow R´–Si(OH)_3 + 3R–OH$

During the condensation reactions:

$R´–Si(OH)_3 + R´–Si(OH)_3 \rightarrow R´–Si(OH)_2–O–Si–(R´)(OH)_2 + H_2O$

Hydrogen bonding between the siloxane monomers and oligomers occurs in the solution. Silane oligomers react with each other, forming branched hydrophobic siloxane bonds (–Si–O–Si–), and with an inorganic matrix (e.g. silica, metal oxides that contain hydroxyl –OH groups) forming (–Si–O–M–) bonds (M = metal).

The mechanism of binding the silanol group with metal is depending to a large extent on the type of metal and ability of metal surface to oxidize and give hydroxyl groups, the acidic silanol groups then can react with the (OH–) groups on the metal and form siloxane bonds of both types (–Si–O–M–), and above and between them (–Si–O–Si). Recent studies about the chemical reactions of silane reported a formation of a film, a hydrophobic and branched polysiloxane layer that may also contain free hydrogen-bonded oligomer, and as result of condensation reaction free water molecules may appear (61, 62, 65).

2.5. Fiber Reinforced Materials (FRC)

Even that the composite material was used for a long time in dental application but one of the problems manifested by the clinician was the mechanical properties of composite especially in situation of long intra oral treatment therefore in the last two decades, there were many literatures and researches on the reinforcement of theses material to improve their mechanical and physical properties.

Combination of fibers bundle into a matrix of composite to increase the ability to withstand the load and intraoral environment without affecting the chemical properties of the matrix was introduced in the many fields before dentistry.

Fiber reinforcement composite was used in space technology, in the marine and industries, building construction, inner house decoration and furniture, medical applications (e.g. bone plates for fracture fixation, implants, and prosthetics), sports equipment, electronics (e.g. Printed circuit boards) and other industrial products (e.g. step ladders, oxygen tanks, and power transmission shafts) (66). The properties of this new materials can be summarized in more strength more resistance to fatigue, better tensile strength (67). The fibers which are the main components in these new materials give various characteristic depending on the fiber quantity and quality, fiber length, and fiber orientation. The fiber was applied in various lengths to meet the different demands in dentistry. The orientation of these fiber could be unidirectional (all fibers in one direction), bidirectional (fibers in two directions, usually normal to each other), or the multidirectional orientation of fibers which is obtained by weaving.

The strength of the FRC depends on the quantity of fibers and directions of construction so the difference between unidirectional and bidirectional is that the first give strength in one direction only but in the bidirectional the material has a good strength in both vertical and horizontal and this strength may differ according to the number of the fibers in each direction.

The arrangement of the fibers is also important; fibers can be arranged discontinued or continuous.

The short discontinues has less strength, so the types of fibers can be summarized as:

(a) Unidirectional continuous fibers

(b) Bidirectional continuous fibers

(c) Multidirectional continuous fibers

(d) Unidirectional discontinuous fibers

(e) Random discontinuous fibers

Fibers can be made of glass or carbon, aramoid, polyethylene, natural, boron and ceramic (66).

Glass fibers are made of thin strands of silica based material and other material with different ratios but the most commonly used for reinforcing are E-glass fibers and S-glass fibers.

E-glass fibers are used usually for electrical application from which the letter E came from. A typical nominal chemical composition of E-glass fibers is SiO_2 54 wt%, Al_2O_3 14 wt%, CaO + MgO 22 wt%, B_2O_3 10 wt% and $Na_2O + K_2O$ less than 2 wt%

The oral environment which is rich of chemical and physical loads like temperature change, saliva effects, occlusal forces, PH changes require a material that can withstand all of these conditions without degradation.

The second main components of FRC is the matrix polymers which can be cross linked like Bis-GMA composite, Triethylene glycol dimethacrylate (TEGDMA), Ethylene glycol dimethacrylate (EGDMA), or linear monomer like Methyl methacrylate (MMA) and Hydroxyethyl methacrylate (HEMA).

The applications FRC in clinical dentistry were usually in directly placed restoration like short span bridge, for reinforcing post in endodontic (carbon FRC) (14), and for reinforcing removable dentures in dental laboratory. Most of fibers applied for dentistry are preimpregnated or semi preimpregnated in resin (PMMA and Bis-GMA).

2.6. Thermocycling

Simulating of intraoral environment during in vitro studies is important to test the adhesive material ability to withstand the oral conditions. The temperature of the oral cavity is normally 37° C but it is not stable especially during eating, drinking and breathing because the temperature of the hot and cold foods and drinks will change the normal temperature of the oral cavity which in turn cause contraction or expansion of restorative and adhesive material (68). The air humidity, temperature and velocity have to some extent effects on the temperature of intraoral environment (69).

Thermocycling process means changing the temperature of in vitro samples in a manner similar to oral cavity and for a specific number of cycles in unit of time. So we have four variables for thermal cycling: the temperature, number of cycles per unit time, dwell time and duration of the process.

The normal tooth sample will differ from restoration sample because of different thermal conductivity.

There is no specificity in thermal cycling for in vitro studies of bond strength, studies used different number of cycles per unit time ranging from (1-1000000) at temperature

ranges from 0-36° C for low temperature and from 40-100° C for high temperature, the dwell time range from 4 s to 20 min (70).

The reality of the effects of thermal change on bonding strength was reported by some studies to be true (71, 72), but always determination of regime is difficult to do .

Various study have been done to investigate the effects of thermocycling on shear bonding strength using different bonding material, different light sources, direct or indirect bonding.

Bishara *et al.* reported a significant difference in shear bonding strength of cyanoacrylate after thermocycling at 5 °C to 55 °C for 500 times (71).

Daub *et al.* also used the same regimen of Bishara 5° C to 55° C for 500 times for direct and indirect bonding and also found specific difference when compared to previous study using the same technique but without thermocycling.

Arici *et al.* used two regimens 5 °C to 55 °C for 200 and 20000 and also found specific decrease in shear bonding strength of two material before and after 200 cycles then after 20000 cycles.

AL-Moaleem found that thermocycling of restorative material (amalgam, composite, compomer) of one week for 70 cycles, one month for 300 cycles and three months for 900 cycles cause specific difference between the SBS in the same group especially for amalgam.

Sokucu *et al.* found a specific difference between shear bonding strength of metal brackets to extracted teeth at two regimes of thermocycling 500 and 10000 using different types of light source.

However different standards of thermocycling were studies, exposing the samples to the same conditions of oral cavity is useful logically.

2.7. Storage Medium:

Absolute reproduction of the oral conditions in laboratory setting is impossible because replication of the normal bacterial flora exists in the oral cavity and simulation of the salivary pH, temperature variation, and the muscular activity in the same sample is a mission that cannot be done easily.

The conventional storage medium that used regularly for in vitro studies was water, but artificial saliva has been introduced and recommended to be employed as a storage medium by Finnema *et al.* (73).

3. THE MARTERIAL AND METHOD

The study had been done in Gaziantep university/college of dentistry/Orthodontic department, electron microscope images and analysis had been done in Gaziantep university/engineering faculty/ electron microscope laboratory.

Ethics approval of this study had been acquired from Gaziantep University ethical Committee with approval document number 209.

The thesis is based on a comparison study consists of 84 samples divided into 7 groups.

The purpose of the study is to compare different methods and systems to bond metal brackets to amalgam restorations.

3.1. Materials Used in the Study

All the materials, laboratory equipment and computer programs that are used in this study are listed here in sections according to their usage with their manufacturers details:

3.1.1. Sample preparation

- Dental stone (Elite Model, 165744, Zhermack, Rovigo/Italy)
- Silicon Impression material (Silibest, BMS Dental, Capannoli/Italy)
- Silicon activation gel (Catalyst gel, BMS Dental, Capannoli/Italy)
- Self-cure acrylic (Meliodent, Bayer Dental, Leverkusen/Germany)
- Amalgam (Cavex Avalloy III spill, Cavex Holland BV, Haarlem/The Netherlands)
- Amalgam instruments (153 Condenser, Hu-Friedy Mfg. Chicago/USA)
- Amalgamator (TAC-400M, Kinea TAC, Montegrosso/Italy)
- Metallographic Polishing machine(Minitech 233,Presi UK Ltd, United Kingdom)
- Silicon carbide grinding paper (BuehlerMet II, Illinois/USA)
- Ultrasound bath (easyclean230V, Renfert, Hilzingen/Germany)

3.1.2. Surface treating

- Intraoral air powered abrasion device (Prophy-mate neo, Model PMNG-M4-P,NSK, Tochigi/Japan)
- Fumed silica granules (Aeroperl 300 pharma, Evonik Industries AG, Hanau/Germany)
- Aluminm oxide (Korox_R , Bego, Bremen/Germany)
- Chronometer

3.1.3. Bonding materials

- Acid etch (phosphoric acid 37%, Gel Etch, 3M Unitek, Monrovia, CA/USA)
- Metal primer (Alloy PrimerTM, Kuraray Medical, Tokyo/Japan)
- Silane based primer (RelyX, 3M ESPE, USA)
- polymer-monomer gel pre-impregnated photopolymerizable bidirectional E-glass-fiber sheets (Ever stick net, StickTech, Turku/Finland)
- Adhesive composite (Transbond XT adhesive paste, 3M Unitek, Monrovia-CA/USA)
- Bonding primer (Transbond XT adhesive primer , 3M Unitek, Monrovia-CA/USA)
- Bracket (Master Series, American Orthodontics, Sheboygan, WI/USA)
- LED (Valo, Ultradent, UT/USA)

3.1.4. Debonding

- Thermocycler (THE-1100, SD Mechatronic GMBH, Feldkirchen-Westelham / Germany)
- Universal Testing Machine (AGS-X, Shimadzu, Kyoto/Japan)
- Material testing software (TRAPEZIUM X Ver 1.1.2, Shimadzu, Kyoto/Japan)

3.1.5. Microscopical Analysis equipment

- Stereomicroscope (Leica DM5000 B, Wetzlar, Germany)
- Quantitative image analysis program (Leica Qwin.Plus v3.6, Cambridge, United Kingdom)
- Image processing & analyzing program (image J 1.48v, Wayne rasband, National institute of health, USA)
- Electronic microscope (JEOL 6390LV scanning electron microscope, JEOL USA, Massachusetts/USA)
- EDX analysis detector
- Camera photography (Eos 550D, Canon, Japan)

3.1.6. Statistical program

SPSS version 21 for Macintosh (SPSS Inc., Chicago, IL, USA)

3.2. Material ingredients

3.2.1. Adhesive materials

3.2.1.1. Transbond XT light cure adhesive system

This system was introduced by 3M unitek company as an adhesive for bonding orthodontic metal and ceramic brackets to tooth surfaces, it is supplied in 6 ml adhesive primer and 4g adhesive paste syringe (Figure 3.1), According to the manufacturer safety data sheet MSDS in 2014 the primer consist of Triethylene Glycol Dimethaacrylate (TEGDMA) and BIS-GMA in a ratio that not mentioned accurately because of trade secret but it is near to 45-50% for both ingredients.

The adhesive paste consist of silane treated quartz 70-80%, Bis-GMA 10-20 %, Bisphenol A bis (2-Hydroxyethyl ether) Dimethaacrylate 5-10%, and Silane Treated silica 2%.

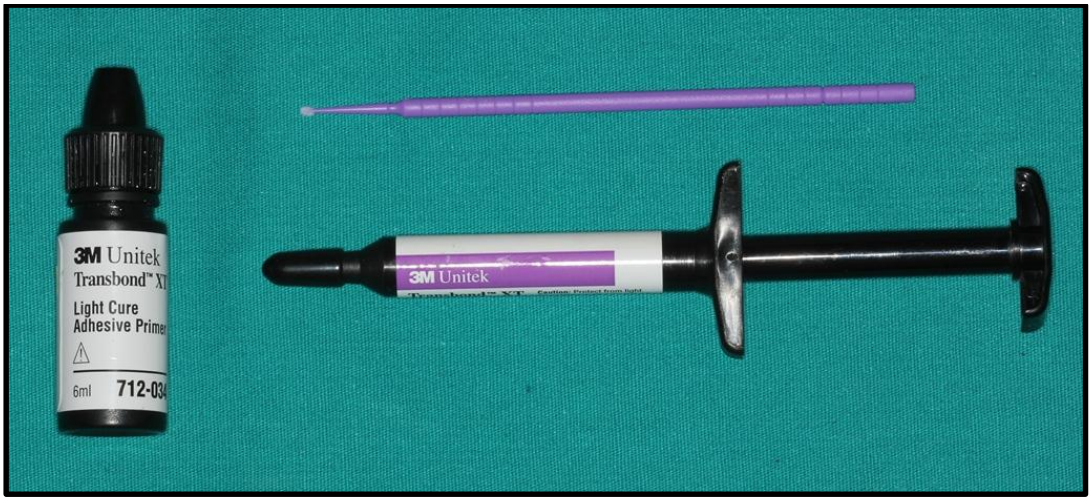


Figure 3.1 Transbond XT adhesive system

3.2.1.2. *Kuraray* alloy primer

It is a metal primer to improve the bond strength of composite to dental alloys; it is supplied as a transparent liquid in 5ml bottle (Figure 3.2). The liquid consists of acetone solvent more than 90% and 6-(4-Vinylbenzyl-N-propyl)amino-1,3,5-triazine-2,4-dithione and 10-Methacryloyloxydecyl dihydrogen phosphate.

3.2.1.3. Rely X ceramic primer

It is a vial of 5 ml of transparent prehydrolized silane based primer (Figure3.3) that act as a bonding primer to porcelain, ceramic, procured composite and silicatized metal surface. It consists basically of less than 2% of Methacryloxypropyl-trimethoxysilane solved in a solvent of ethyl alcohol 70-80% and water 20-30 %.

Figure 3.2 Kuraray Alloy Primer

Figure 3.3 RelyX Ceramic Primer(Silane Based Primer)

3.2.1.4. EverStick NET

It is 30 cm^2 of bidirectional mesh fiber consist of silanated E-glass fiber impregnated with BIS-GMA and PMMA with a thickness of 0.06 mm. It is used usually for aesthetic labial splinting of movable teeth (Figure 3.4).

Figure 3.4 everStick Net (E-glass fiber Sheet)

3.2.1.5. Air Abrasion Materials

3.2.1.6. AEROPERL 300 Pharma

Is a granulated high purity colloidal silicon dioxide for using with pharmaceutical product, the average particles size is 30 µm and with PH from 3.5 to 5.5 and consists of SiO_2 in a percentage of 99% and heavy metal less than 25 ppm and chloride lest than 250 ppm (Figure3.5).

3.2.1.7. KOROX 50

It is a very common blasting material consists of 99.6% of (50 µm) alpha corundum particles of angular shape and great hardness.

It is the aluminum oxide product of choice in studies for abrasion of metal and porcelain (Figure3.5).

Figure 3.5 Blasting Materials (on the left side Silicon dioxide, on the right side Aluminum oxide)

3.2.1.8. The silica coating sand

The sand that is used in this study for blasting with silane coupling agent was a mix of the previous products in a ratio resembling to a large extent the COJET Sand produced by 3M ESPE company with 97% by weight aluminum oxide particles 50μm and 3% of silicon dioxide particles 30μm.

After mixing the particles was examined under light microscope to evaluate the presence of two types of particles in the mix (Figure 3.6).

The efficacy of the sand was confirmed by pilot study of three samples on amalgam then evaluation of the surface of amalgam under light microscope.

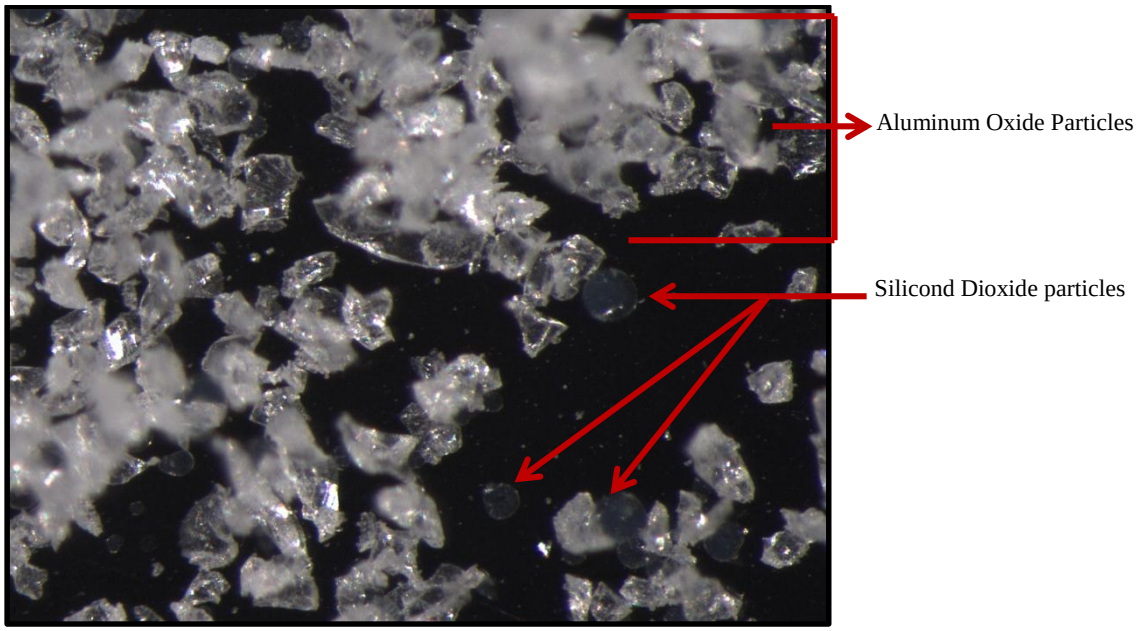


Figure 3.6 Silicon dioxide & aluminum oxide powder (100X)

3.2.1.9. Amalgam

The amalgam used in this study is Cavex Avalloy which is non-Gamma II lathe-cut high copper amalgam packed as predosed capsules.

Chemically it consists of : 45% silver, 30.5% tin,24% copper and 0.5% zinc and with a mixing ratio of 10 parts of alloy to 10.3 parts of mercury.

3.3. Experimental Methods

3.3.1. Sample preparation

Two types of samples were used in this study: 1) enamel samples consisting of 12 newly extracted lower molar teeth and 2) amalgam sample consisting of 72 amalgam packed in acrylic blocks randomly divided into 6 groups of 12 specimens per group.

3.3.1.1. Enamel sample

Lower Molars teeth were collected over one month from oral surgery department in Gaziantep University and stored in buffered saline, the saline was changed every 5 days to prevent bacterial growth, and then 12 molars with no caries, no restorations and intact buccal surface were selected randomly.

The teeth are cleaned by periodontal curette to remove any attached soft tissue, and every tooth was treated on the buccal surface with pumice and rubber cup then rinsed

with tap water, air dried and embedded in acrylic blocks with buccal enamel surface exposed for bonding. Each enamel surface was polished with silicon carbide grinding paper with polishing machine using 600, 800 and 1000 grit grinding paper respectively to create a relatively flat enamel surface on the buccal aspect of molar for bonding of central incisor bracket.

3.3.1.2. Amalgam sample

A cuboid block of dental stone was poured and timmed then a cavity (width 6 mm, length 6 mm and depth of 3 mm) was prepared in the stone using fissure bur with converge proximal walls and two auxiliary retentive holes for retention of amalgam, then an impression of this stone model was obtained using polyvinylsiloxane impression material to make five molds of silicone (Figure 3.7). After that self-curing acrylic was poured into these molds to create 72 acrylic blocks with the same standard cavity in each of them (Figure 3.8).

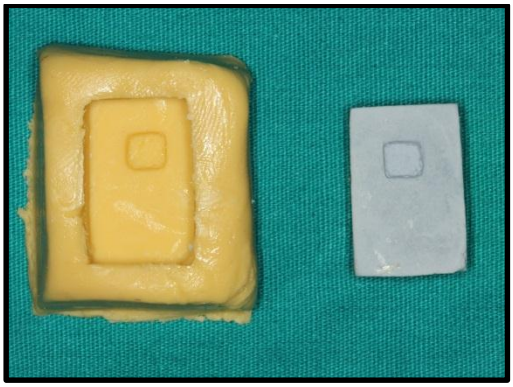


Figure 3.7 impression of the stone block with the amalgam cavity

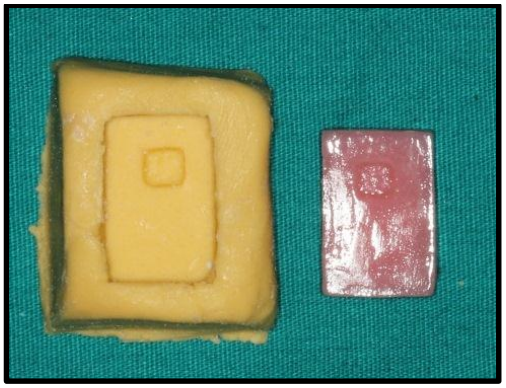


Figure 3.8 Self-curing acrylic resin blocks with amalgam cavity

Each of the non-gamma II amalgam capsule was triturated 18 sec in amalgamator and condensed into the cavity prepared in acrylic block until slightly overfill the cavity (Figure 3.9). And then the amalgam samples were allowed to harden for 24 hours as recommended by manufacturer.

Every amalgam surface was polished with silicone carbide grinding paper of 240, 400 grit for 20 seconds on the polishing machine with a speed of 500 turn per minute and by single operator to ensure that constant pressure 45 to 47 was applied on each sample.

Then polishing was continued by 600, 800 and 1000 grit silicon carbide grinding paper for 10 seconds by each paper with the same previous setting regarding speed and pressure. (Figure3.10)

Figure 3.9 Overfilled amalgam before polishing

Figure 3.10 Amalgam surface after polishing

All the samples were cleaned in ultrasonic bath for 10 min and examined under light microscope at 20X magnification for any scratch remained, samples with any crack in the center of amalgam are excluded and replaced by new amalgam sample.

After that the samples were thermocycled from 5° to 55° C so that they remain 20 sec. in each bath and 5 sec. outside and for 1000 cycles according to the recommendation of the International Organization for Standardization (ISO/TS 11405).

3.3.2. Surface treatment

Air abrasion of the amalgam surface was done using intraoral air abrasion device, and three different blasting particles were used:

First, the powder case was cleaned with air syringe for 20 seconds and then the device was used empty for another 20 seconds to ensure that no remaining particles from other abrasion powders were remained inside the case.

Aluminum oxide powder was loaded inside the powder chamber and 24 samples were abraded from a distance of 10 mm perpendicular to the amalgam surface at 85 psi for 4 seconds (74), (Figure3.11).

Second the silica coating sand was loaded into the device after cleaning the device in the same way previously mentioned then other 36 amalgam samples were abraded at the same conditions regarding pressure, time and distance.

Figure 3.11 Sandblast of the surface of Amalgam

Two additional samples of amalgam were prepared and treated with silica alone then examined under electron microscope to ensure that silica alone is not enough for treating amalgam surface.

The other 12 amalgam samples were left not treated to work as a control for treated groups.

After air abrasion all the samples are placed in ultrasonic bath for 10 min.

Table 3-1 Number of amalgam samples and type of sand blast used

surface treatment method	Alumium oxide	Silicon dioxide	Silica coating powder**	Polished non treated
Number of samples	24	2*	36	12

*additional samples not included in the total number of samples in the study

**97% aluminum oxide +3% silicon dioxide

3.3.3. Electronic microscope analysis

After surface alteration of amalgam surfaces two samples from each sand blasted amalgam group were examined under the electronic microscope to evaluate the surface alterations that occurred because of air abrasion and compared them to the polished control amalgam.

3.3.4. Bonding procedure

The 84 samples prepared for this study were divided into 7 groups listed in (Table 3-2).

3.3.4.1. Enamel samples

Group 1: Each of 12 extracted molar samples was etched with 37% phosphoric acid for 15 sec. and rinsed with water for 15 sec. then air dried with oil free air for 20 sec.

The Transbond XT primer was applied to the etched enamel surface using micro applicator brush and the bracket was bonded to enamel surface using Transbond XT adhesive and polymerized with light emitting diode LED light curing unit for 20 sec. on the mesial side and 20 sec. on the distal side as recommended by the manufacturer.

All the procedures in bonding had been done by one operator to ensure standard outcome.

3.3.4.2. Amalgam samples

All the layers applied to the amalgam surface were shown in (Pattern 3.1).

Group2: Each of the 12 amalgam samples sand blasted by aluminum oxide was bonded by metal primer.

Kuraray Alloy primer was applied on the amalgam surface using micro applicator brush and left to dry then Transbond XT primer and Transbond XT adhesive was used to bond

the bracket to the amalgam surface and polymerized by LED light curing unit for 20sec. on the mesial side and 20 sec. on the distal side as recommended by the manufacturer.

Group3: 12 number of amalgam samples sand blasted by aluminum oxide were bonded using metal primer on the amalgam surface and bracket base.

Kuraray alloy primer was applied on the amalgam surface and on the bracket base using micro applicator brush and left to dry then Transbond XT primer and Transbond XT adhesive was used to bond the bracket to the amalgam surface and polymerized by LED light curing unit for 20 sec. on the mesial side and 20 sec. on the distal side as recommended by the manufacturer.

Group4: 12 number silica coating sand blasted amalgam were bonded by silane coupling agent.

RelyX silane coupling agent was applied on the amalgam surface using micro applicator brush and left to dry for 5 min. as directed by the manufacturer then Transbond XT primer and Transbond XT adhesive was used to bond the bracket to the amalgam surface and polymerized by LED light curing unit for 20 sec. on the mesial side and 20 sec. on the distal side as recommended by the manufacturer.

Group5: 12 number polished amalgam samples without sand blasting were bonded using adhesive composite reinforced with bidirectional preimpregnated E-glass fiber.

One piece of fiber sheet (width 3, length3.5, area 10.5) were cut and placed on the amalgam surface (Figure 3.12), then Transbond XT primer and Transbond XT adhesive was used to bond the bracket to the amalgam surface and polymerized by LED light curing unit for 20 sec. on the mesial side and 20 sec. on the distal side as recommended by the manufacturer.

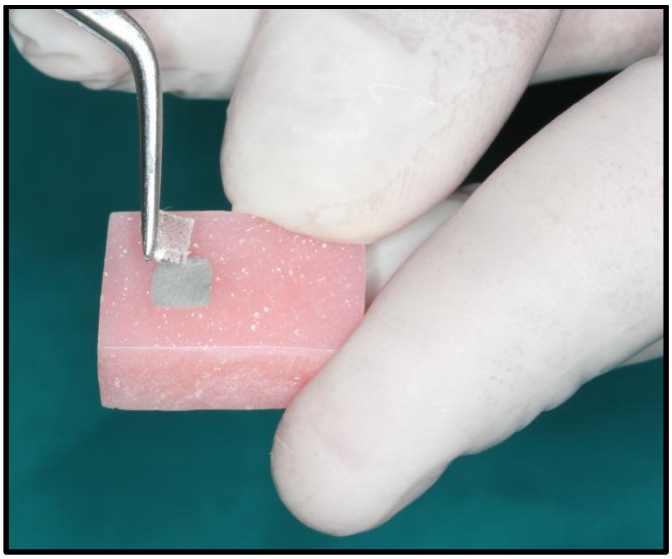
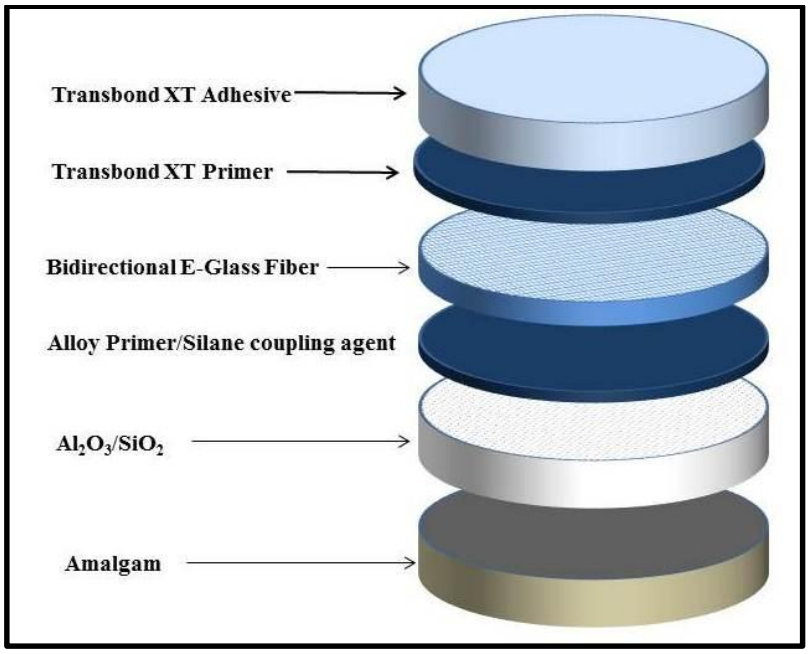


Figure 3.12 A piece of Bidirectional Preimpregnated E-glass fiber

Group6:12 number of amalgam samples sandblasted with silica coating sand were bonded using silane coupling agent and composite reinforced with bidirectional preimpregnated E-glass fiber.

RelyX silane coupling agent was applied on the amalgam surface using micro applicator brush and left to dry for 5 min. as directed by the manufacturer then one piece of fiber sheet (width 3, length3.5, area 10.5) were cut and placed on the amalgam surface, after that Transbond XT primer and Transbond XT adhesive was used to bond the bracket to the amalgam surface and polymerized by LED light curing unit for 20sec. on the mesial side and 20 sec. on the distal side as recommended by the manufacturer.



Pattern 3-1 Schematic Diagram represents all the layers applied to the amalgam surface with the appropriate Sand blast

Group7: 12 number of amalgam samples sandblasted with silica coating sand were bonded using silane coupling agent, alloy primer and composite reinforced with bidirectional preimpregnated E-glass fiber.

Kuraray alloy primer was applied on the bracket base using micro applicator brush and left to dry then Relyx silane coupling agent was applied on the amalgam surface using micro applicator brush and left to dry for 5 min. as directed by the manufacturer, after that one piece of fiber sheet (width 3, length3.5, area 10.5) were cut and placed on the amalgam surface, and as usual Transbond XT primer and Transbond XT adhesive was used to bond the bracket to the amalgam surface and polymerized by LED light curing unit for 20 sec. on the mesial side and 20 sec. on the distal side as recommended by the manufacturer.

Table 3-2 Study groups with experiment design

	Group1	Group2	Group3	Groiup4	Group5	Group6	Group7
Amalgam		+	+	+	+	+	+
Enamel	+						
Aluminum oxide 50µm		+	+				
Silica coating sand 30µm				+		+	+
Alloy primer on amalgam		+	+				
Alloy primer on bracket base			+				+
Silane coupling agent (RelyX)				+		+	+
Preimpregnated bidirectional E glass fiber					+	+	+
Acid etch phosphoric acid 37%	+						
Transbond XT primer	+	+	+	+	+	+	+
Transivesbond XT adh.	+	+	+	+	+	+	+

3.3.4.3. Thermocycling

After bonding the enamel and amalgam samples are thermocyled from 5° C to 55° C with 20 sec. in cold bath, 20sec. in hot bath and 5 sec outside for 1000 cycles.

3.3.5. Brackets surface area

The bracket surface area supplied by the manufacturer was evaluated by using image analyzer software.

Six brackets were randomly selected and examined under the light microscope at 10X magnification then an image had been acquired using the microscope image analysis program.

A scale line of 1 mm had been given by the program which is connected to the microscope (Figure 3.9)

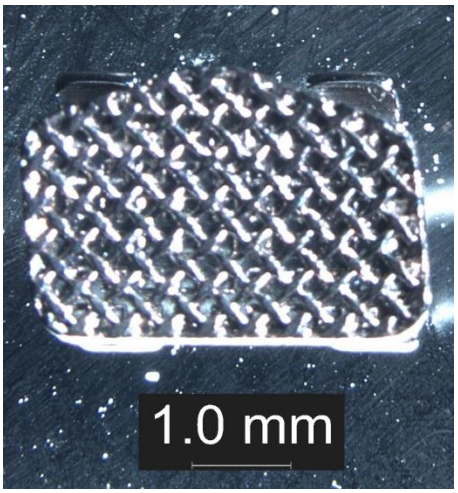


Figure 3.9 bracket image taken by light microscope with the scale line of 1mm

Then all of these images were processed using Image J program after entering the correct scale and make calibration the borders of the bracket base were traced digitally and the surface area was calculated, these steps were repeated 6 six times for each bracket to decrease the bias and the average mean of these measures was calculated to give a value near to accurate.

The surface area obtained by image processing was 10.3 mm2, this surface area was used to determine the shear bonding strength by the formula:

SBS= Force (newton)/(Surface area(mm^2)

Table 3-3 Surface area of the bracket base calculated by Image J software (Sq.mm)

	Bracket1	Bracket2	Bracket3	Bracket4	Bracket5	Bracket6
Area1	10.248	10.178	10.321	10.159	10.256	10.458
Area2	10.357	10.161	10.015	10.546	10.461	10.045
Area3	10.421	10.257	10.256	10.197	10.147	10.149
Area4	10.159	11.019	10.184	10.249	10.573	10.549
Area5	10.015	10.287	10.946	10.115	10.214	10.476
Area6	10.025	10.011	10.64	10.145	10.813	10.354
MEAN	10.20417	10.31883	10.29767	10.23517	10.41067	10.3385
Total Mean 10.30083=10.3						

3.3.6. Debonding procedure

The samples were selected randomly from each group and loaded one by one to the universal testing machine with cross head speed of 0.5 mm/min and 10 kN loading cell.

The steel rod that attached to the cross head move vertically toward the bracket to be tested and debonded it, (Figure 3.13) the force value that needed to debond the bracket was recorded electronically on the computer software trapezium X by newton and then exported as an excel (Microsoft) spreadsheet, the Shear bonding strength was calculated by dividing the force by the surface area (10.3 mm^2) with using Excel (Microsoft 2010) software.

Figure 3.13 Debonding procedure using Universal testing machine

3.3.7. Adhesive remnant index

All the samples were examined under light microscope with a magnification of 20X and an image of the bracket had been acquired and evaluated by two examiners to decrease the error.

The adhesive remnant index was recorded on an Excel (Microsoft 2010) spread sheet according to the scale recommended by Artun and Bergland as follows (32):

0 = No adhesive remained on the tooth (all the adhesive remained on the bracket)

1 = Less than 50 % of the adhesive remained on the tooth (more than 50% of the adhesive remained on the bracket)

2 = More than 50% of the adhesive remained on the tooth (less than 50% of the adhesive remained on the bracket)

3 = All the adhesive remained on the tooth (No adhesive remained on the bracket)

4. RESULTS

Statistical analyzes of the data obtained after debonding was done by SPSS software version 21 (SPSS Inc., Chicago, IL, USA). Shapiro –Wilk test was used to check the normal distribution of the samples and all SBS data were normally distributed in all groups according to this test.

4.1. Shear bonding strength results

One-way ANOVA and Tukey's honest significant difference (HSD) multiple comparison tests were used for independent normally distributed samples to compare quantitative measurements ($p < 0.05$).

During all statistical analyzes $p \leq 0.05$ was accepted as significant difference between groups.

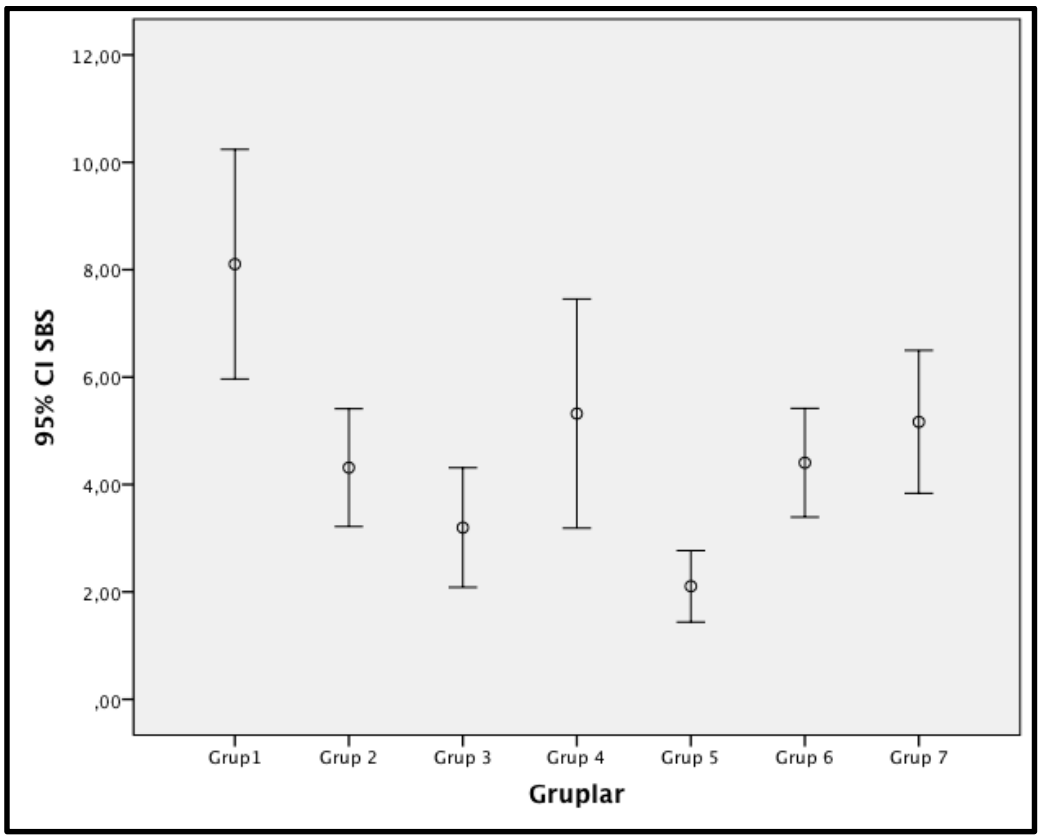
Descriptive data of SBS values including the mean and standard deviations for seven groups are shown in (Table 4.1), according to ANOVA and Tuker's honest significant test all groups are statistically significant varies from the control group, all the methods of bracket bonding to amalgam reported a SBS values lower than the control group value with Transbond XT and enamel (8.1±3.4).

Table 4-1 Descriptive Statistics of shear bonding strength

Group	N	Mean(MPa)	Sdt.Dev.	Min(MPa)	Max(MPa)
Group1	12	8.1	3.4	3.1	13.8
Group2	12	4.3	1.7	1.3	7.2
Group3	12	3.2	1.8	1.1	7.3
Group4	12	5.3	3.4	1.5	11.1
Group5	12	2.1	1.0	0.8	4.4
Group6	12	4.4	4.5	2.6	8.3
Group7	12	5.2	2.1	2.7	8.9
Total	84	4.65	2.8	0.8	13.8

The significance of difference between groups was evaluated by ANOVA results in (Table 4.2).

There was no statistically significant difference between the aluminum oxide and silane coating surface conditioned amalgam groups, but the mean SBS of silicatized amalgam groups (group 4, 6, 7) was (5.0±3.3 MPa) higher than the aluminum oxide treated amalgam group (group 2, 3 3.75± 1.75 MPa).



Pattern 4-1 Statistical analyzes of intergroups SBS Data

Among the amalgam groups the lowest mean SBS was revealed by the polished non treated amalgam group (2.1±1.0 MPa) and the highest mean SBS was manifested by silicatized amalgam surface without fiber reinforcing of adhesive composite (5.3±4.5 MPa).

Table 4-2 ANOVA results of Shear bonding strength (SBS)

	Sum of square	df	Mean square	F	p
Between groups	256,903	6	42,817	8,176	<0.001
Within groups	403,256	77	5,237		
Totals	660,159	83			

The highest SBS value recorded in amalgam groups was (11.1 MPa) of the silicatized amalgam surface without fibers and the lowest value was (0.8 MPa) of polished amalgam surface with fiber application.

Table 4-3 the variance between groups for SBS data using ANOVA

*p<0.001	Group2	Group3	Group4	Group5	Group6	Group7
Group1	0.002 *	<0.001*	0.57	<0.001*	0.003*	0.037*
Group2		0.893	0.933	0.228	1.000	0.970
Group3			0.270	0.904	0.853	0.359
Group4				0.016*	0.957	1.000
Group5					0.188	0.025*
Group6						0.983

4.2. The results of adhesive remnants index

The distribution of adhesive remnants index (ARI) scores in all groups was demonstrated in (Table 4.4), the Mann-Whitney test was used to analyze the data of ARI. For the amalgam groups the highest (0) score was in group five (100%) with polished amalgam and fiber, while the lowest (0) score was in group two (8%) with aluminum oxide treated amalgam and alloy primer.

The highest score (3) was recorded in group four (67%) with silicatized amalgam surface without fiber reinforcing, and the lowest score (3) were among the fiber reinforced groups.

Table 4-4 Distribution of ARI on enamel/amalgam surface

Study Groups	Score0	Score1	Score2	Score3	total
Group1	1(8)	5(42)	3(25)	3(25)	12
Group2	1(8)	2(17	4(33	5(42	12
Group3	2(17)	1(8)	4(33)	5(42)	12
Group4	0(0)	1(8)	3(25)	8(67)	12
Group5	12(100)	0(0)	0(0)	0(0)	12
Group6	9(75)	3(25)	0(0)	0(0)	12
Group7	9(75)	0(0)	3(25)	(0)	12

Table 4-5 The variance between groups for ARI data using Mann-Whitney test

*p<0.001	Group2	Group3	Group4	Group5	Group6	Group7
Group1	0.319	0.410	0.045*	<0.001*	<0.001 *	0.007*
Group2		0.932	0.347	<0.001*	<0.001*	0.002*
Group3			0.347	<0.001*	0.001*	0.007*
Group4				<0.001*	<0.001*	<0.001*
Group5					0.319	0.319
Group6						0.887

The Spearman's rho correlation had been done to evaluate the relation between ARI score and shear bonding strength SBS values and a significant correlation was clarified (correlation coefficient r=227, p=0.038), (Table 4-6).

Table 4-6 the relation between SBS and ARI

		ARI
SBS	Correlation Coefficient (r)	0.227
	p	0.38
	N	84

So there is relation between SBS values and ARI data but because the correlation coefficient is 0.227, this relation is considered weak, (Table 4-7).

Table 4-7 Categorization of correlation strength according to Dancey and Reidy's 2004

Value of the Correlation Coefficient	Strength of Correlation
1	Perfect
0.7 - 0.9	Strong
0.4 - 0.6	Moderate
0.1 - 0.3	Weak
0	Zero

4.3. Results of the electron Microscope

The electron micrograph of the polished amalgam reveals a flat and smooth surface, the silica particles alone cause a little alteration in the surface of amalgam which calculated by image J program and divided over the surface area of the section and report a 0.6% porosity in the amalgam surface, the aluminum oxide alone cause more pronounced pits and scratches in the amalgam surface while reveal a percentage of 3.6% of the surface area of the section while the highest percentage of etching occurs with silica coating powder (5.3%), (Figure 4.2).

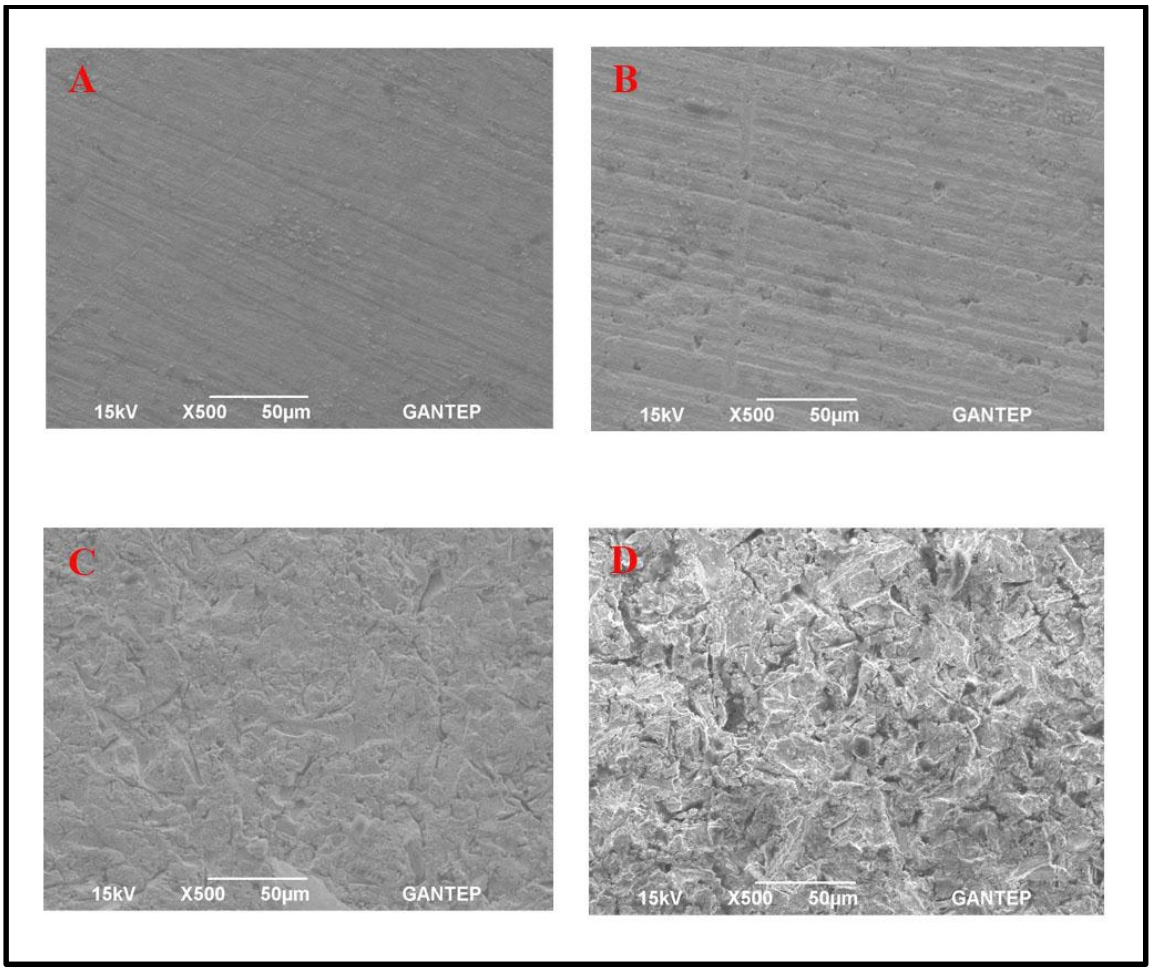


Figure 4.1 500 X Magnification of (A). Polished amalgam , (B). Silicon onlyblasted amalgam, (C). Aluminum oxide blasted amalgam, (D). Silica coating blasted amalgem

4.3.1. Energy-dispersive X-ray (EDX)

The EDX analysis of the atoms in the aluminum oxide group reveals 4.43% of aluminum inside the section under 2000X

Table 4-8 EDX analysis of Aluminum oxide treated amalgam

Element	Concentration (%)
C	31.130%
O	25.937%
Al	4.43%
Cu	5.102%
Zn	0.547%
Ag	12.889%
Hg	14.661%

While the EDX analysis under 2000X magnification (Figure) of the silica coating group illustrate the presence of aluminum (Al) with a percentage (7.547%) and silicon atom (5.570%).

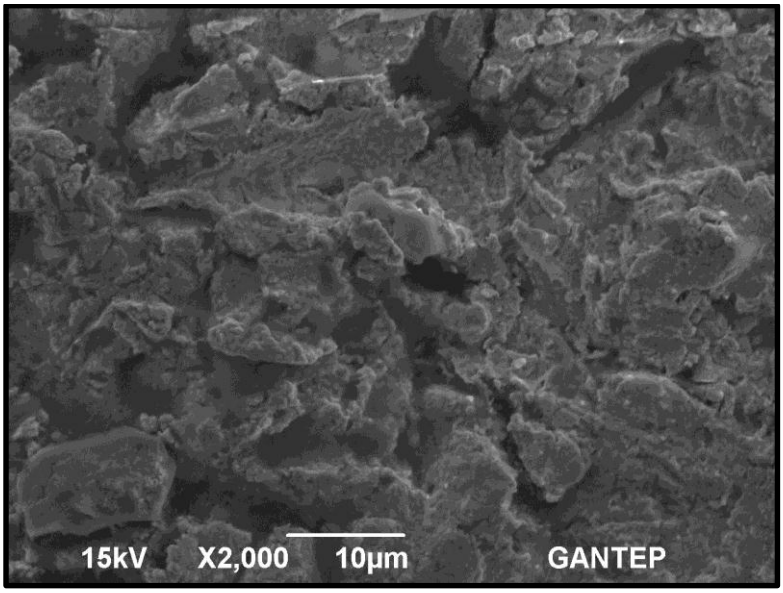
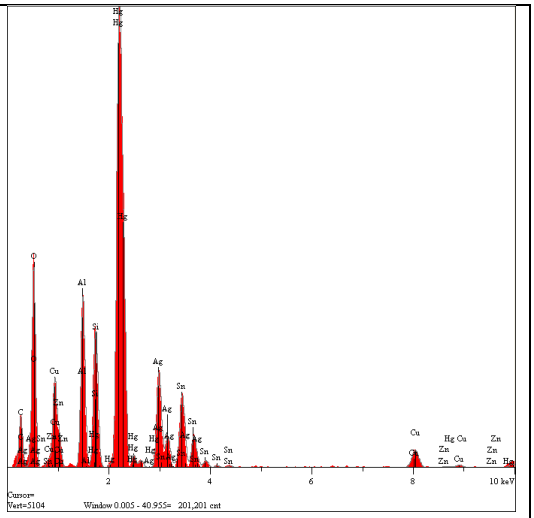


Figure 4.2 Surface of Silicatized amalgam under 2000X magnification

Table 4-9 EDX analysis of Silica coating blast treated amalgam

Element	Concentration (%)
C	7.712%
O	23.408%
Al	7.747%
Si	5.570%
Cu	7.201%
Zn	0.594%
Ag	9.480%
Sn	8.375%
Hg	29.913%



5. DISCUSSION

It is clear for all orthodontists that optimum orthodontic shear bonding strength can withstand the forces and conditions of the oral cavity without damage to the substrate upon debonding.

Amalgam restorations are still common fillings for buccal pits of the molar teeth but the bonding of orthodontic brackets to these restorations is still a dilemma, despite the rapid revolution in field of the dental materials. The present study is planned to evaluate the potency of different methods of surface conditioning with various adhesive materials known of their metal adhesion properties in the bonding of orthodontic attachments to amalgam fillings and comparing the results with the clinically recommended SBS values 6-8 MPa (75).

The values of SBS obtained *in vitro* studies are not applicable directly in clinical situations so these types of studies are still used as a comparison of new materials to some previous adhesive orthodontic materials that are considered gold standard.

Transbond XT adhesive system which is used in this study is regarded the gold standard for orthodontic adhesive in many studies because the high value of shear bonding strength obtained by this adhesive in relation to other adhesives (76).

Creating the *in vitro* conditions that resemble to some extent the intraoral cavity is very useful for testing adhesive materials, the degradation of the adhesive that occur because of the salivary reaction and thermal change is replicated by thermocycling in artificial saliva, moreover the parameters of testing machine are also claimed to be the cause of the variance in results of the same adhesive under same conditions, the configuration of load cell have a great effect on the values obtained in vitro (77). Replication of the SBS test for each sample in all groups in the study is essential for elicitation of logical SBS values.

In this study all samples including the enamel and the amalgam samples were polished under the same conditions on metallographic polishing machine to get good replication of all surfaces, then every sample was sand blasted by the same device with the same parameters, and thermocycled, after that the samples are tested by the same machine with (10 kN) load cell, so the test of this study had been done in the same environment.

Transbond XT was used in many studies in Gaziantep University/Orthodontic department and in present study it was used as control group; the SBS obtained in this study was (8.1±3.4MPa).

In a previous study done by İsman *et al.* (23) in Gaziantep university the SBS with TXT adhesive was (9.9 ±3.2) which is a value near to the present study SBS even that the teeth used in İsman's study were premolars, cross head speed was 1mm/min and without thermocycling while molars were used in this study with 1000 thermocycles and at cross head speed 0.5 mm/min.

Another study by Sokucu *et al.* (78) revealed a SBS value (19.7±5.7) after 500 thermocycles using the same TXT adhesive with premolar teeth, the significant variation between this SBS values is considered to be because of using another model of universal testing machine (LF Plus, LLOYD Instruments, Ametek Inc., England) and under other laboratory environment.

This variety in the SBS among different studies on the same adhesive can be contributed to:

- The type of the teeth: enamel of molar teeth differs in thickness and maturation from enamel of premolar teeth.
- The type of the brackets: different bracket base designs.
- The thermocycling regime.
- The configuration of load cell.
- The duration and the type of storage media used until debonding procedure.

Few studies reported the Bonding strength of metal brackets to the amalgam surface, until 1995 there were no published study evaluating the bonding of orthodontic attachments to amalgam (1), but Zachrisson and Buyukyilmaz place the first block in this field, they used aluminum oxide as a sandblasting powder for the amalgam surface or diamond bur for roughening, in addition to that, many types of metal adhesives were used but most of the previous studies investigated the tensile bond strength not SBS, the highest value of tensile bond strength (6.4MPa) was obtained with aluminum oxide sandblast and super bond C&B which is a (4META) containing adhesive, again Buyukyilmaz & Zachrisson explored more about bonding to amalgam, this time they used three types of amalgam particles (spherical, lathecut and admixed) with various intermediate resin but much higher tensile bond strength was obtained (11 MPa) with reliance metal primer and spherical amalgam. These values cannot be comparable to

present study because different parameters were tested and by completely different laboratory method.

After that Sperber *et al.* (5) used also aluminum oxide for sand blasting of amalgam samples with three different adhesives and reported a very high bonding strength (18.67 MPa) with aluminum oxide sand blast and Panavia EX adhesive. The value obtained in the present study with aluminum oxide sand blasting and Alloy primer was much lower than Sperber's findings, this may be due to the Universal testing machine that was used with (50kg) load cell which is equal to (0.49 kN) while in present study the load cell was (10kN), and as stated before in a literature written by Vivik Cheba that decreasing the load cell configuration will increase the SBS values (77), moreover Sperber *et al.* used other adhesive (Panavia EX, C&B Meta bond).

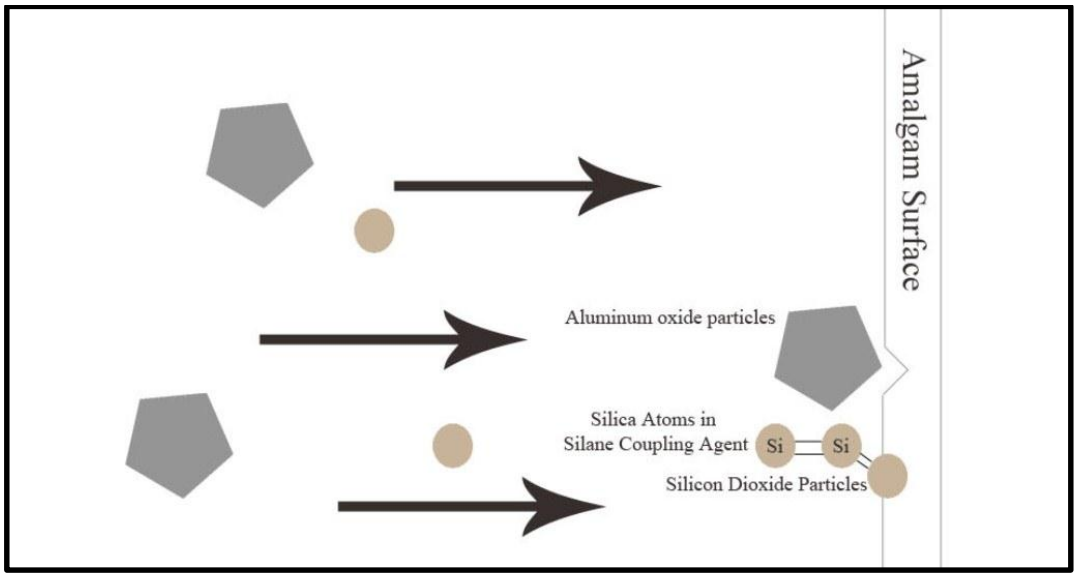
Other study had been done by Germec *et al.* (6) and the highest value obtained was (7.15 MPa) with using reliance metal primer and aluminum oxide sand blasting. Again the value obtained is higher than the SBS of group 2 in this study (4.3MPa), but Germec *et al.* used (50 kg) load cell which is known to reveal higher SBS values.

Oskoee *et al.* (42) apply in his study the same alloy primer of our study (Kuraray) and the same aluminum oxide abrasion particles to amalgam surface but with different loading cell configuration (50kg) and without any report of thermocycling. The Highest SBS was (3.56±1 MPa) which is significantly near to the value obtained in the present study.

This study is the first study that uses Silane coating sand and silane coupling agent for amalgam conditioning before bonding of metal bracket, Atsü *et al.* used silane system in his study on the base of the metal brackets (9) not on the restoration, moreover it is the first study incorporate fiber for reinforcing orthodontic adhesive composite before bonding to amalgam.

Although, bonding to enamel surface with Transbond XT recorded the highest SBS (8.1±3.4 MPa) among all the group in the study but the bonding to the amalgam surface is considered more difficult, statistically the difference between group one (usual bonding to enamel) and group four (bonding to amalgam with silane coating system) is considered not significant ($p<0.05$), this means that The silane coating system reported some values of SBS approximately near to the values obtained by bonding to enamel.

Among the amalgam groups, surface treating using silane coating sand and silane coupling agent in group 4 gave out higher SBS (5.3±3.3 MPa) than aluminum oxide sand blast with alloy primer treated group (4.3±1.7 MPa) this can be explained by that Aluminum oxide particles etch the amalgam surface creating micro pits that increase the surface area for bonding then the silicon dioxide particles are stucked in the pits produced by aluminum oxide because electron microscope images of only silica dioxide blasted surface show micro pits in a percentage much lower than aluminum oxide treated group. After that using of silane coupling agent that contain silicon atoms aids in creating bonds with the silicon atoms stucked inside the amalgam surface (Pattern 5.1).



Pattern 5-1 a Schematic illustration of the Silica coating procedure

In group 6 the silicatized amalgam surface was bonded with Transbond XT after placing a sheet of (~0.06 mm) of fiber sheet to reinforce the orthodontic composite and decrease the cohesive failure, although the SBS values were lower than the mean SBS in group 4 but no significant difference have been found after addition of E-glass fiber to the composite concerning the SBS, but one interesting finding in group 6 and 7 was ARI scores that states the more than 75% of debonded brackets were removed with the entire adhesive and no adhesive remnants remains on the tooth so the failure was adhesive failure between the composite and the amalgam and the fiber success in reinforcing the composite and decreasing the percentage of cohesive failure.

Applying the metal primer to bracket base in group 7 did not give any significant difference when compared with group 4 and 6, the effect of metal primer on the bracket

base was considered significant because the failure occur in most samples in the adhesive amalgam interface.

The application of fibers directly on the polished amalgam surface and bonding with Transbond XT adhesive aid in retention of the brackets on the amalgam because in previous study by Sperber *et al.(5),* all the brackets bonded to the amalgam with Phase II adhesive which has no claim of metal adhesive were debonded during thermocycling. But the SBS on polished amalgam surface is still lower than groups with blasted surface, and the electron microscope image with (500X) magnification show a relatively flat surface without micromechanical interlocking pits, so the bonding strength in this group is completely returned to chemical properties of Transbond XT and may be the fiber reinforcement worked as mesh to increase the surface area of the amalgam surface, even that the bond failure site was 100% at amalgam adhesive interface.

The topography of silicatized amalgam surface under electron microscope show more etched and porous surface than aluminum oxide alone these could be due to the effects of two type of particles on the amalgam surface not only one type. More over the EDX analysis reveal the presence of aluminum atoms on silicatized surface in a percentage more than the aluminum oxide blasted surface which is probably due to the coating effect of silica atoms to adhere to metal.

Regarding the bond strength obtained by using metal primer, the oxide layer produce on the non-precious alloy aids in improving chemical bonding of hydrogen inside the alloy primer to oxygen layer, but because the amalgam samples bonded after only one day of thermocycling, the effect of oxide layer was still minimized so it is expected from alloy primer to work better on old amalgam restorations in the mouth (74).

These retention groove and holes provide a high mechanical retention environment for bonding; in all groups the surface alteration by air abrasion was very useful for increasing the bonding strength when compared with group 5 without sand blast.

It is obvious from the ARI score variance test that The scores in group 5, 6, 7 where E-glass fibers were used are significantly different from other group, most of ARI scores were (0) in those groups, the E-glass fibers reinforced the orthodontic composite to a great value and the mesh like bundles of fibers lead to more structural stability of orthodontic composite and decrease the cohesive failure within the composite to a great extent.

It is obvious from this study that acceptable bonding strength near to the values obtained by enamel can be obtained after surface alteration with Silicatization and

application of silane coupling agent to the amalgam but bonding without surface conditioning is still not considered with amalgam.

6. CONCLUSION

- Bonding strength of metal brackets to amalgam restoration is still lower than the bonding strength obtained with enamel.
- Silicatization of amalgam surface recorded the highest shear bonding strength among all the amalgam groups.
- Application of Kuraray alloy primer on amalgam surface blasted with aluminum oxide 5µm revealed SBS value that is not significantly different from SBS whe amalgam blasted with silane coating sand then application RelyX silane coupling agent
- Application of E-glass fiber does not have any effect on shear bonding strength.
- Application of E-glass fiber reported bond failure at amalgam adhesive interface.

Limitations:

This study used 10 kN load cell so comparing to previous studies using 0.4 kN was not appropriate.

This study did not use saliva as storage media, using of saliva will degrade.

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8. APPENDIX 1: ETHICAL APPROVAL

KLİNİK ARAŞTIRMALAR ETİK KURULU KARAR FORMU

ARAŞTIRMANIN AÇIK ADI	Ortodontik Metal Braketlerin Farklı Yüzey Pürüzlendirme Yöntemleri Uygulanan Amalgam Yüzeylerine Olan Bağlanma Dayanıklılığının Değerlendirilmesi	
ARAŞTIRMANIN PROTOKOL KODU	209	
	GÜVENLİLİK BİLDİRİMLERİ	☐
	DİĞER:	☐
KARAR BİLGİLERİ	**Karar No: 09.06.2014/209**	**Tarih: 09.06./2014**
	Yukarıda bilgileri verilen başvuru dosyası ile ilgili belgeler araştırmanın/çalışmanın gerekçe, amaç, yaklaşım ve yöntemleri dikkate alınarak incelenmiş ve uygun bulunmuş olup araştırmanın/çalışmanın başvuru dosyasında belirtilen merkezlerde gerçekleştirilmesinde etik ve bilimsel sakınca bulunmadığına toplantıya katılan etik kurul üye tam sayısının salt çoğunluğu ile karar verilmiştir. Klinik Araştırmalar Hakkında Yönetmelik kapsamında yer alan araştırmalar/çalışmalar için Türkiye İlaç ve Tıbbi Cihaz Kurumu'ndan izin alınması gerekmektedir.	

KLİNİK ARAŞTIRMALAR ETİK KURULU	
ETİK KURULUN ÇALIŞMA ESASI	Klinik Araştırmalar Hakkında Yönetmelik, İyi Klinik Uygulamaları Kılavuzu
BAŞKANIN UNVANI / ADI / SOYADI:	Prof. Dr.Belgin ALAŞEHİRLİ

Unvanı/Adı/Soyadı	Uzmanlık Alanı	Kurumu	Cinsiyet		Araştırma ile ilişki		Katılım *		İmza
Prof. Dr.Belgin ALAŞEHİRLİ	FARMAKOLOJİ	Gaziantep Üniversitesi Tıp Fakültesi	E ☐	K x ☐	E ☐	H x☐	E ☐	H ☐	
Prof. Dr.Ercan SİVASLI	PEDİATRİ	Gaziantep Üniversitesi Tıp Fakültesi	E x ☐	K ☐	E ☐	Hx ☐	E☐	H ☐	
Prof.Dr. Mehmet KESKİN	PEDİATRİ	Gaziantep Üniversitesi Tıp Fakültesi	E x ☐	K ☐	E ☐	Hx ☐	E ☐	H ☐	
Prof. Dr Feridun IŞIK	GÖĞÜS CERRAHİ	Gaziantep Üniversitesi Tıp Fakültesi	E x ☐	K ☐	E ☐	Hx ☐	E ☐	H ☐	
Prof. .Dr. İlker SEÇKİNER	ÜROLOJİ	Gaziantep Üniversitesi Tıp Fakültesi	E x ☐	K ☐	E ☐	Hx ☐	E ☐	H ☐	
Doç. Dr Bünyamin KISACIK	İÇ HASTALIKLARI	Gaziantep Üniversitesi Tıp Fakültesi	E x ☐	K ☐	E ☐	Hx ☐	E ☐	H ☐	
Doç.Dr.Yasemin ZER	MİKROBİYOLOJİ	Gaziantep Üniversitesi Tıp Fakültesi	E ☐	K x ☐	E ☐	H x☐	E☐	H☐	
Doç.Dr.Beyhan CENGİZ	FİZYOLOJİ	Gaziantep Üniversitesi Tıp Fakültesi	Ex ☐	K ☐	E ☐	Hx ☐	E ☐	H ☐	
Doç. Dr. Kemal ÜSTÜN	DİŞ HEKİMLİĞİ	Gaziantep Üniversitesi Diş Hekimliği Fakültesi	E x ☐	K ☐	E ☐	Hx ☐	E ☐	H ☐	
Doç.Dr.Seval KUL	BİYOİSTATİSTİK	Gaziantep Üniversitesi Tıp Fakültesi	E ☐	K x ☐	E ☐	Hx ☐	E ☐	H ☐	
Uzm.Dr. Cahide Elif ORHAN	FARMAKOLOJİ	Gaziantep İl sağlık Müdürlüğü	E ☐	K x ☐	E ☐	H x☐	E ☐	H ☐	
Eyüp ÇELİK	AVUKAT	Gaziantep Barosu	Ex ☐	K ☐	E ☐	Hx ☐	E ☐	H ☐	
Baha Günhan GÜNGÖRDÜ	İNŞ.MÜH (sivil Üye)	GASKİ	E x ☐	K ☐	E ☐	H x☐	E ☐	H ☐	

*:Toplantıda Bulunma

Etik Kurul Başkanının
Unvanı/Adı/Soyadı: Prof. Dr.Belgin ALAŞEHİRLİ
İmza:

Not: Etik kurul başkanının her sayfada imzasının olması gerekmektedir.

CURRICLUM VITAE

Dler MOURAD was born in Aleppo/Syria 15.01.1985; He finished his primary school in Huda AL-SHARAWI primary school, intermediate and secondary school in Mazen DABAGH secondary school /Aleppo/Syria

He has been awarded a degree of B.D.S in Dentistry from college of dentistry /Hawler medical university /Irbil/Iraq on 27/July/2009, after three years of private practice in Aleppo/Syria; he was accepted in 2012 in Master of Science in orthodontic department of Gaziantep University.