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ISTANBUL YENİ YÜZYIL UNIVERSITY

HEALTH SCIENCES INSTITUTE

DEPARTMENT OF ORHODONTHICS

MASTER THESIS

ABDULBASET NAGI ABUHRES

Supervisor

Prof. Dr. İlter Uzel

ISTANBUL

July 2018

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COMPARSION OF SHEAR BOND STRENGTH AND MICROLEAKAGE OF CERAMIC FLASH-FREE ORTHODONTIC BRACKETS

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ACCEPTANCE AND APPROVAL

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Abstract

The aim of this study are: to determine *in vitro* bond strength of ceramic APC orthodontic brackets when bonded using a self-etching primer (SEP) after Thermo-cycling, and to assess Microleakage following Thermo-cycling under orthodontic brackets.

Materials and Method:

Sixty previously extracted intact human premolar teeth (N =60, n = 20 per group) were randomly allocated to one of three test groups. The brackets used in this study were 3M Unitek, include Clarity Advanced Ceramic for the control group, APC Flash-free Adhesive-coated Advanced Ceramic and APC Plus Adhesive-coated Advanced Ceramic for the two study group. After brackets were bonded to teeth, specimens were thermocycled between
5 C^o to 55 C^o for 5000 cycles. All specimens were subjected to bracket removal using the Instron Universal testing machine to measure shear bond strength (SBS) and Microleakage for each sample.
The shear bond strength, and microleakage of each group were statistically compared using t-test $p<0.05$.

Results: The mean shear bond strength of Clarity Advanced Ceramic bracket group had significantly higher mean shear bond strength value (**16.4350**) when compared with APC Flash-free (**11.6885**) and APC Plus (**12.0995**) bracket group (**p=0.00 , p=0.00** respectively).
The microleakage is higher at gingival sides in all groups compared with those observed in occlusal sides for both adhesive interfaces. Enamel-adhesive interfaces exhibited more microleakage than did the adhesive-bracket interfaces. Also, brackets bonded with self-etching primer system showed significantly higher microleakage at the enamel-adhesive interface of the gingival side.

Conclusions: Clarity Advanced Ceramic brackets bonded with 37 % phosphoric acid gel showed higher SBS and less microleakage than Ceramic APC flash-free and Ceramic APC Plus bonded with self-etching primer system after Thermo-cycling.

Dedication

This thesis is dedicated to:

My Country

Libya

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I thank you

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Abbreviations

GIC: Glass ionomer cement.

RBC: Resin bonded cement.

SBS: Shear Bond Strength.

RBCs: Resin based composites.

APC: Adhesive pre-coated.

MPa: Mega Pascal.

N : Newton.

μM: Micrometer

mm: Millimeter.

Sec: Second.

n : Nnmber.

1. Literature review

1.1. Introduction

The acid-etching technique was first introduced to dentistry in 1955 by Buonocore [1]. There has been some concern about the possible damage by acid-etching to enamel. In In 1955 Buonocore started using 85 % ortho-phosphoric acid for 30 seconds and found that the bond strength of acrylic restorations was increased by etching the enamel surface [1]. The improvement of the bond strength is derived by dissolution of hydroxyapatite crystals in the surface to create micro-porosities into where the fluid adhesive can flow (Retief, 1978) [4].

1.2. Enamel structure, composition and characteristics

1.2.1. Enamel formation

Enamel formation (amylogenesis), is a two stage process. The first stage produces a partially mineralized (approximately 30 %) enamel. As soon as the full width of enamel has been deposited, the second stage involves influx of additional material and water to achieve more than 96 % mineral content. The mineral influx makes the crystals form, and during the first stage grow thicker and wider. This is a complicated process under the cellular control, and the associated cells will show significant morphological changes throughout amylogenesis, which reflecting their evolving physiological activity.

1.2.2. Enamel structure

The major organizational units of mammalian enamel are rods (prisms) and inter-rod enamel which called (inter-prismatic substance). The enamel built from long and closely packed, ribbon-like carbonato-apatite crystals measuring from 60 to 70 nm in width and 25 to 30 nm in thickness. The calcium phosphate unit cell has a hexagonal symmetry shape and stacks up to create a hexagonal outline to the crystal. However, a fully mature enamel crystal are not perfectly hexagonal but exhibit an irregular outline because they press against each other during the final stage of their growth.

The rod is cylinder in shape and consisting of crystals with long axes which run parallel to the longitudinal axis of the rod. The inter-rod region surrounds each rod and its crystals are orientated in a different direction from those making up the rods. The space between rod and inter-rod enamel in this region is defined by a narrow space which contain organic material known as the rod sheath. The major organizational pattern of mammalian enamel is explained as cylindrical rods embedded in the inter-rod enamel.

1.2.3. Enamel composition and morphology

The completely shaped finish is an exceptionally mineralized extracellular matrix which comorising of 4 % organic matrix and water and 96 % mineral. The inorganic contents of enamel is mainly crystalline calcium phosphate that called hydroxyapatite.
The high content of mineral in the enamel makes it extremely hard, which enabling it to withstand the occlusal forces applied during function. Despite of the hardness is similar to that of mild steel, enamel is brittle and the underlying layer of more resilient dentine is substantial to maintain its integrity.
The enamel is a translucent and it varies in color from light-yellow to grey-white. Also It varies in thickness from approximately 2.5 mm over working surfaces to a feather-edge at the cervical margin. This variation in thickness influences the color of enamel as the underlying yellow dentine shines through the thinner parts.
A number of specific structures characterizes the enamel surface. The striae of Retzius frequently extend from the dentin-enamel junction to the outer surface of enamel, where they end in a shallow furrows known as perikymata, that run in circumferential horizontal lines across the face of the crown. Lamellae or (cracks) in the enamel appear as jagged lines. The enamel surface in unerupted teeth consists of a structureless surface layer that is lost rapidly by attrition, abrasion and erosion in erupted teeth. Striae of Retzius appear as a series of dark lines that extending from the dentino-enamel junction toward the tooth surface, if viewed in transverse section.

1.3. Preparation of tooth surface for clinical procedures

1.3.1. Prophylaxis

The tooth surface is covered by a pellicle consisting of a protein thin layer that forms on the surface of enamel by selective binding of glycoproteins from saliva. It is preferable to clean the tooth surface before using a chemical etching in order to allow ease of access of chemical etchant to the enamel surface.
One of the most commonly used methods of cleaning is a brush or rubber cup, used in a slow-speed handpiece, accompanied by an abrasive agent. The prophylaxis paste should be abrasive enough to remove all types of accumulations from the enamel surface without causing abrasion. The most commonly used abrasives are silica, pumice, and zirconium

silicate, which are harder than enamel. In fact, It is expected that there will be some enamel loss when those materials are used. It has been reported that initial prophylaxis by using a bristle brush for 10 to 15 sec can abrade as much as 10 µm from the enamel, whereas only 5µm might be lost when a rubber cup is used.

1.3.2. Acid etch technique

In 1955, Buonocore's study on acid etching of enamel surfaces originated from industry, where phosphoric acid preparations were used to obtain better adhesion of paint and resin coatings to treated metal surfaces. It was found that treating the enamel with phosphoric acid increased the retention of acrylic filling materials (1). Supported by investigations done by Silverstone (1974) and Retief (1974), acid solutions in concentrations of 20 to 50 % applied for 1 to 2 minutes were found to make the most retentive conditions, and advised for clinical use (3,4). In 1967, Gwinnett and Matsui described the ability of dental adhesive resins to penetrate the subsurface microporosities that created in etched ground enamel (5). In 1968, Buonocore suggested the concept of micro-mechanical retention, recognizing resin tag formation created by the principal adhesion of resins to acid etched enamel (6). The clinical technique of bonding to enamel did not become recognized until the early 1970s, since up to that time restorative materials were inacceptable.

When we etching the enamel with phosphoric acid, changes the surface of the enamel in two ways:

- Etching dissolves a thin layer of enamel
- Etching produces the enamel porous by partially dissolving the ends of the enamel prisms.

In 1955, Buonocore suggested etching the tooth surfaces by using 85 % phosphoric acid for 30 sec (1). In 1971 the enamel was etched with 50 % phosphoric acid for 2 minutes and approximated the loss of enamel tissue to be between 5 and 25 µm in depth. Etching for 90 sec with 30 % phosphoric acid Fitzpatrick and Way in 1977 found an usual enamel loss of 9.9 µm (7).

In 1980, Pus and Way were treated 50 human premolars by using 43 % phosphoric acid gel and another group of 50 with 37 % liquid ortho-phosphoric acid, and all for 90 sec. They found the average losses of enamel tissue were 7.5 and 6.5 µm respectively. Wickwire and Rentz (1973) submitted premolars to variable etch times and determined that enamel dissolution increased with time (8).

1.3.3. Etchant concentration

In 1978, the tensile bond strengths was calculated after etching with different 2-60 % phosphoric acid liquids. Acid with 16 % produced the highest bond strength, but the results for 2 % acid were similar to those for 40 %. In 1986, also found no statistical difference in tensile bond strength after 1 minute application of 2 %, 5 % and 35 % phosphoric acid while the loss of enamel was considerably greater with 35 % acid than with 2 % acid.

The most consistently suitable and uniform etching pattern was described after using of 30 to 40 % phosphoric acid .

In 1974, Rock reported extensively higher bond strengths for teeth treated with 30 % phosphoric acid than with 50 % phosphoric acid (9). Phosphoric acid with 37 % is most commonly used clinically as it offers similar bond strengths to higher concentrations, with less damage to the enamel surface.

1.3.4. Duration of etching

The influences of using different etching time on bond strength is still controversial, particularly with respect to ceramic brackets. In 1990, increasing the bond strengths after reducing etching time when premolars were etched for 15 and 60 sec by using 37 % phosphoric acid. In 1985, Barkmeier *et al.* in 1985, used 15 and 60 sec etching time, while in 1980 and Beech and Jalaly evaluated 5, 15, 60 and 120 sec intervals (10, 11).

All of them reported no decrease in bond strength as a result of reduced etch times. In 1999, Osorio *et al* reported increase in shear bond strength when enamel was etched for 60 sec and the amount of adhesive remaining on the tooth was greater as well (12). Though a 15 sec etch still produced a bond stronger than that required for successful orthodontic bonding. They also reported that 15 sec etch time produced a clean etch site after debonding.

In 1996, examination of shear bond strength after 15, 30 and 60 sec etch times, using 37 % phosphoric acid. Debondıng of the brackets was performed at 5 minutes, 15 minutes or 24 hours. The 15 sec etch / 5 minute debonding time specimens had a lower shear bond strengths than other groups. After 60 sec of etch teeth showed a sign of tooth surface damage after debonding.

In 1986, Carstensen studied the clinical failure rate of mesh-based metal brackets bonded to 1134 anterior teeth, after etching for 30-35 sec with 37 % phosphoric acid. Just 10 brackets were lost during the 16 month of study period. In a second study a comparison was made between the effects of etching for 15-20 and 30-35 sec. It was concluded that 15 sec etch was enough for bracket bonding on anterior teeth (13).

From the study above 15 sec of etching became routinely recommended for bonding orthodontic brackets. However if a bracket is to be ligated within 5 minutes after bonding, 30 sec of acid etching is recommended. Using 60 sec etch is considered too severe and should not be used.

1.3.5. Etch pattern

Most of bond strength experiments has been performed on extracted premolar teeth. Considerable differences were found for SBS between different tooth types and opposing dental arches. The upper anterior teeth gave higher shear bond strengths than upper posterior teeth and lower posterior teeth showed higher bond strengths than lower anterior teeth.

Additional evidence has suggested that different types of teeth show biological differences in etch patterns after acid priming and this may affect bond strengths. In 1974, Marshall *et al.* examined the etched surfaces under the scanning electron microscope and reported a high degree of variation in etching pattern from tooth to tooth and in different parts of the same tooth, following the same process of etching (14). Certain parts of the enamel appeared to have a thicker, denser or more tightly adhering prismless layer that impeded dissolution of the underlying enamel prisms and this was more pronounced on premolars than molars. This led the authors to assume that molars required a longer etching time than premolars. In 2001, examining the existence and pattern of differences in SBS between teeth types when bonding the orthodontic brackets. There were statistically significant differences in the mean SBS were found, with canine and pre-molar teeth showed higher strength than incisors.

1.4. Adhesion and Adhesives

1.4.1. Resin based composites (RBCs)

Composite materials consist of two or more components. the resin based composite usually contains organic binder and inorganic filler, whose particles are typically encapsulated with a coupling agent to bind them to the resin matrix.

The monomer is often used for bis-glycidyl methacrylate (bis-GMA) or Bowen's resin is often used. Bowen's resin, like methyl-methacrylate, is subjected to free-radical addition re-polymerization. Its larger molecular structure with side chains capable of bearing cross-correlation means that it has a lower polymerization shrinkage and coefficient of thermal expansion than methyl-methacrylate-based adhesives. However, the larger molecular structure also means that it is very viscous and in order to make it clinically usable it cab be used with low viscosity dimethacrylate monomers, such as diethylene glycol dimethacrylate or tri-ethylene glycol methacrylate. In recent years other dimethacrylate monomers, such as urethane dimethacrylate (UDMA), have been used to replace all or some of the bis-GMA. The least favorable feature of RBC is volumetric contraction during the conversion of monomer to polymer. The resin matrix of all resin bond cement shrinks by about 10% which causes stresses at the bonded interface with the adjacent tooth surface.

The other component of the RBC is a dilute monomer that reduces viscosity, allowing proper mixing with inorganic components to help manipulate. The greater the concentration of the diluted monomer in the formula the less viscosity and the greater the effect on the polymerization contraction. Some manufacturers prefer a combination of UDMA and dimisharylate diethyl phenylethyl glycol de ether (DIA-EMA). This produces a less viscous mixture of bis-GMA and eliminates the need for reduced resins and thus reduces the amount of shrinkage that can otherwise occur.

Inorganic filler particles consist of glass beads or rods, aluminium silicate, barium, strontium and borosilicate glasses. The filler reduce the shrinkage of polymerization and coefficient of thermal expansion of the material as well as improving the abrasion resistance. The first RBC contained pure silica particles with an average of 20 μm in diameter. This particle size limits the finish because relatively large particles lose easily, which produce a rough surface with poor lustre. Poor wear resistance and the surface is prone to staining. Smaller filler molecules increase the surface area of the filler and improve polishability. The inclusion of aluminum, lithium or crystalline quartz has improved the properties of the material. Whether these factors are important in the thin section found under orthodontic brackets is unclear. However, all properties such as

Young's modulus, tensile force and compressive strengths, and wear resistance are all increased when the filler molecules are able to bind to the resin matrix.
The resin based composites can be classified according to particle filler size to:

- Macro-filled 10 -100 μm
- Mid-sized 1 – 10 μm
- Mini-filled 0.1 – 1 μm
- Micro-filler <0.1 μm

Latest RBCs are filled with particles averaging from 0.1 to 1 μm. Fumed colloidal silica filler particles of size 0.04 μm are combined to produce hybrid resin bonded cement with improved handling properties. Fumed silica increases the ratio of filler that can be introduced into the resin. A hybrid resin is a composite in which at least 7-15 % of the filler is fumed silica. A micro-filled RBC is exclusively composed of micro-filled particles. Thus the total surface area of filler is maximized but heavy loads can not be resisted.
Resin bonded cement may be either chemically cured, light cured or dual cured. Chemically activated RBCs are used as benzoyl peroxide, tertiary amine or sulphinic acid initiators have. Visible light cure composites contain alpha-diketone such as camphorquinone and an amine. On the application of visible light of wavelength 460-485 nm free radicals are created.

1.4.2. Glass ionomer cements (GIC)

The earliest glass ionomer cements contained an ion-leachable glass that reacts with a water-soluble polymer acid to yield a cement. The glass powder was prepared from a calcium alumino-silicate glass containing fluoride, whereas the set cement comprises a higher molecular weight organic-inorganic complex. A typical glass has 6 elements, fused together at 1000-1300 C^o. The ratio of Al_2O_3 to SiO_2 is important for correct reactivity. Fluoride is an important component in GIC as it contributes to the therapeutic value of the cement, helps in the glass industry by reducing fusion temperature and enhances the working properties and mechanical properties of the cement. In certain conditions, fluoride content gives more translucent glass. The sources of fluoride ions in GIC are: Calcium Fluoride, Strontium fluoride, Lanthanum fluoride, Sodium hexa-fluoroaluminat and Aluminium trifluoride. Radio-opaque glasses are used in some materials: in this calcium may be replaced by barium or strontium.

The first type of GICs are luting cements, used in orthodontic bonding. It is a fine-grain materials with low thickness when set. The most common ones are used for cementing bands but have also been used to bond orthodontic brackets. As restorative materials, type II GICs are used, and Type III are lining materials and fissure sealants.

Glass ionomer cements sets on mixing the basic glass with an aqueous poly (alkenoic) acid to produce an acid-base reaction. This setting reaction will release hydrogen ions that penetrate the fluro-aluminosilicate glass surface and release calcium, strontium and aluminum. The last setting reaction may continue for weeks or months. The values of compressive strength have been shown to increase over a year and the bond strength increases for a month after which the rate slows.

The material subjected to hygroscopic influences during setting. Too small amount of water causes dehydration and increases the excitability of the reaction, and damages the cement surface. Wilson and McLean (1988) reported potential problems in testing the bond strength by placing new mixed conventional GIC in water for 24 hours before the test, causing elution the ions required theoretically for the formation of the cross linked polyacrylate chains (15).

The glass ionomer cements adhere directly to the enamel without the necessity to additional bonding agents and surface treatments. The basic adhesion mechanism is derived from the acid's ability to clean, penetrate and roughen the tooth surface that decreases the surface energy and facilitating both mechanical and chemical bonding. The carboxylic group in the polymer acid forms ionic bonds with metal ions.

GIC exposure to neutral aqueous solutions leads to water absorption and release of ions such as sodium, silica, calcium and fluoride. Fluoride in the matrix is available for disposal under neutral conditions, although this equivalent to a very small percentage of fluoride ions in the total set cement. Release mechanisms are either short or long term. The short-term reaction releases relatively high levels of fluoride during post-setting maturation process. Long-term fluoride releases are low, due to the diffusion between the material and the environment that may cause the absorbed or released of the fluoride.

In 50-75 % of orthodontic patients, the demineralization is happens on no less than one labial tooth surface at debonding. Indication of demineralization was accounted for as an imperative clinical issue following five long periods of dynamic orthodontic treatment at the point when GICs discharges fluoride, this will impact the lessening of demineralization and back off progression of caries, however the protection from caries just fractional. The counter microbial impact of the fluoride is affirmed. Notwithstanding, in Mill operator's examination 1999 made sense of that there is no distinction in decalcification esteems between patients encountering settled machine treatment with apparatuses that were reinforced with each glass-ionomer or composite adhesive.

1.4.3. Resin modified glass ionomer cements

GICs arranged to be blend with water-solvent resin monomers and watery poly-acrylic corrosive. Called resin modified glass ionomer cement (RMGICs), which known as materials that subjected to polymerization and acid-base response, which support the setting response in the dark.

Acid-base response is starts when powder and fluid are blended just like with any ordinary GICs. The response continues moderately gradually and produces a low pH (1.5). Cross-interface is started by an oxidation response or by free radicals discharged by the photograph polymerization impetus. A hard blend frames inside which the corrosive base response proceeds. The advantage of RMGIC is that the photograph polymerizer improves the setting all the more rapidly and diminishes the affectability of the materials to water. The concoction response proceeds after the light response that has started is finished. Extra favorable position of RMGICs over customary GICs is the near speed in the improvement of mechanical quality. The hydrogel stage isn't generally seen with regular concrete after light treatment on the grounds that the polymerization response in the monomer gives incredible quality to the material.the polymerization reaction in the monomer gives great strength to the material.

1.4.4. Self etch primer

The SEPs created from the chemistry under dentine bonding which contain methacrylic acid would bond to dentine. Buonocore (1955) demonstrated that glycerophosphoric acid-methacrylate containing resin would bond to acid etching dentine (1).

As other options to phosphoric acid, polyacrylic acids have been utilized as a part of a push to lessen the prospected harm caused by demineralization of the outer layer of enamel resulting to etching with phosphoric acid. In any case, the subsequent bond qualities were low.

Some new bonding techniques join the priming and conditioning in one material to use on both enamel and dentin which improve as well the cost effectiveness.

The active element of (SEP) is methacrylate phosphoric acid. Phosphate Group on Phosphoric Acid Ester Methacrylate breaks down calcium and expels it from the hydroxyapatite lattice. Rather than rinsing calcium, it is complex with the phosphate and incorporated into the network when polymerization is polymerized. The profiling procedure and the monomer in uncovered enamel bars are synchronized.

Transbond Plus© 3M Unitek is an primer utilized for bonding in orthodontic. Makers assert this joined etch and primer can limit the time required for the bonding of fixed appliances and White in (2001) approximated the general time that we can save during bracket bonding to be 65 % (16). Nonetheless, the saved time ıs questionable, where pumicing and priming stages are erased and SEPs must be blended on the tooth surface for 3 and 20 seconds. The makers likewise guarantee that the item works adequately in a humid situation. In this way, the disengagement of the enamel surface to anticipate salivary contamination may not be unequivocal when utilizing SEP. SEP is less sensitive system on the grounds that the material endure the moister contamination. Fundamentally lower SBS quality in the test group that did not utilize air drying after applying SEP than the other two test groups, SEP with air drying and regular two-organize cement framework.

Utilizing of SEP relies upon compelling cleaning of the enamel surface with pumice prophylaxis before to bonding. This stage can be erased in conventional bonding.

There are clashing reports on the clinical execution of SEPs and traditional etch and bond procedures.

In case of bond-failure when we use one-stage system is similar or higher than the conventional two-stage technique and the bonding time is reduced, it would be better to use a one-stage adhesive system in orthodontic daily treatment. Manning *et al.* (2005) planned a prospective clinical trial to evaluate the bond failure rates of brackets bonded with a SEP (Transbond Plus©) and a traditional acid-etched technique with control adhesive (Transbond XP©). No statistical difference was found between clinical bond failure rates of the brackets bonded using SEP or a traditional acid-etch and resin technique respectively. In 6 months, the bond failure rate for both groups (1.8 %) was low compared with other published data (17).

The use of SEP significantly reduced but was clinically acceptable shear bond strength compared to conventional etch and prime. Significantly, more residual adhesive was found on tooth surfaces following SEP. Also, bond strength was greater in two-step adhesive than the one-step self-etching system. However both adhesives performed clinically well, suggesting that differences in bond strengths may be clinically insignificant.

Hirani and Sherriff in (2006) examined bond strengths, rebond strengths and failure sites of adhesive pre-coated brackets and conventional brackets when bonded with a SEP (Transbond Plus©) and conventional acid etching and conditioning. No difference was found in shear bond strength between APC and conventional brackets with SEP. Most of bond failures occurred at the adhesive-enamel interface (18).

1.5. Orthodontic bracket design

1.5.1. Metal brackets

The earliest metal brackets were milled from cold drawn stainless steel and had perforated bases into which adhesive could flow. Stainless steel brackets do not form a chemical union with an adhesive but they retained at the base-adhesive interface by mechanical interlocking.

The original metal pads contained just one row of perforations along the outer margins and the relatively larger inner smooth surface was incapable of contributing to retention. This base design was changed to foil-mesh bracket bases, which provide better bond strength and less plaque retention. The foil mesh was welded to a solid metal backing. Weld points called gobbets, are un-retentive and microscopy reveals that the adhesive is susceptible to fracture in regions adjacent to these sites.

Figure 1.1 3M Unitek Victory© series orthodontic bracket

1.5.2. Plastic brackets

These brackets were first announced in the early of 1970s. Firstly they were made from acrylic or polycarbonate and were tolerated by orthodontists as an alternative to metal brackets for short time. The basic problems were quickly identified, including color and odor change, but more importantly their lack of strength and rigidity resulted in problems of bonding, tie wing fractures and permanent deformation. Polycarbonate bracket slots distort with time under constant stress, making them insufficiently strong to withstand longer treatment times or to express torque.

To overcome the lack of strength and stiffness of the original polycarbonate brackets, high quality medical polyurethane brackets and reinforced polycarbonate with ceramic or fiberglass fillers and/or metal slots have been introduced. Polycarbonate brackets with reinforced metal slots show much lower creep than conventional polycarbonate brackets despite torque problems.

1.5.3. Ceramic brackets

These brackets were introduced in 1980s. They offer advantages over steel brackets in terms of appearance. Ceramic brackets provide higher strength, greater resistance to wear and deformation, better colour stability and, superior aesthetics. All currently available ceramic brackets are made of aluminium oxide in either polycrystalline or monocrystalline form, depending on the method of fabrication.

From a single crystal of sapphire, the first brackets were each milled using diamond tools. The latest monocrystalline alumina (MCA) brackets are formed from extrusions of synthetic sapphire. Ceramic brackets can not chemically bond with acrylic and diacrylate bonding adhesives due to their inert aluminium oxide composition. As a result, the early ceramic brackets used a silane coupling agent to act as a chemical intermediary between the ceramic bracket base and the adhesive resins. This chemical retention resulted in a very high bond strengths that caused the enamel / adhesive interface to be stressed during debonding, resulting in irreversible damage to the enamel.

In 1988, The American Association of Orthodontists conducted a survey of members' experiences with chemically-bonded ceramic brackets. The results have led the association to advise its members on potential health concerns with ceramic arches and recommended practitioners to discuss possible risks with their patients as part of the informed consent process. Most of the currently available ceramic brackets rely on mechanical retention only using standard adhesives or chemically treated materials, without the need for additional special bonding agents.

The polycrystalline alumina brackets (PCA) are made by injecting molding of submicron-sized particles of alumina suspended in the resin and fusing them to combine the alumina to produce a bracket that is finally machined to shape. Polycrystalline ceramics have a high coefficient of friction due to their rough and more porous surface. Machined ceramic brackets produce far greater frictional forces than stainless steel brackets.

In 1995, Birnie did not encourage the use of sliding mechanics with ceramic brackets, and suggested bonding metal brackets to the premolars if this movement is required (19).

The fragility of ceramic brackets can cause problems in debond. Its hard nature can cause tooth abrasion to the dentition and a clear enamel wear has been found on the palatal surfaces of upper incisors from contact with the lower labially placed ceramic brackets after only six weeks of using them.

Figure 1.2 3M Unitek Transcend© ceramic orthodontic bracket

1.5.4. Metal-reinforced ceramic brackets

In an effort to improve the frictional characteristics of polycrystalline ceramic brackets, the manufacturers have introduced reinforced metal slots in order to provide smooth sliding mechanics and additional strength (Clarity© brackets, 3M Unitek). Different metal lined polycrystalline brackets are now available with 18 carat of gold inserts; these are reported to be superior to stainless steel in relation to frictional resistance.

Figure 1.3 3M Unitek Clarity© series orthodontic bracket

1.5.5. Bracket base morphology

The design and shape of the base of the orthodontic bracket is believed to affect the mode of bond failure and to have an effect on the damage of enamel surface during removal of the bracket. Dents or undercuts in the bracket base can provide mechanical retention. Most commonly used in metal brackets, a mesh is welded to the base of the bracket to form a mechanical retention structure. The welded mesh bracket bases are not without defects; clinically the pads are flexible, especially those with the thinnest mesh size. These easily deformed and bended away from the surface of the tooth, resulting in soft tissue trauma and adversely affect mechanical retention. On debonding the components of mesh-based brackets tend to separate, leaving the wire mesh attached to the tooth surface. Brazing replaced welding as the technique for connect mesh foil to the bracket base, thus preventing the mesh strands from being flattened during assembly. Brackets manufactured as a separate components have a weak level between the bracket and base.

The bond strength of foil-mesh brackets is affected by the diameter of the wire mesh and the number and size of openings per unit area. The free size available affects the penetration of the resin, which also depends on the filler size.

Microscopy reveals the air voids at the adhesive / base interface, possibly because of polymerization contraction or by air retention during bracket fixing. Knox *et al.* (2000) investigated the effect of bracket base morphology and orthodontic bonding agent on adhesion strength and concluded that adhesive had a significant impact on bond strength and that special base designs improved adhesive penetration or improved penetration of a curing light (20).

The literature presents conflicting reports on the effect of thermocycling using different ceramic bracket types on SBS. The SBS of two metallic brackets was tested with a single mesh bracket base and the other with a double-mesh bracket base using Transbond XT© adhesive. The shear bond strengths of each test groups was similar and the Adhesive Remnant Index (ARI) comparisons indicated that both types of bracket had the same bracket failure patterns. These results indicated that the single and double mesh bracket bases have similar SBS and bracket failure modes.

Although ceramic brackets offer better aesthetics, concerns have been raised regarding the increased risk of enamel surface damage on debonding and found there are no statistical difference in bond strengths between ceramic and metal brackets. Enamel separation was found only when there was a chemically coated base on the ceramic bracket and thus higher bond strength. Some ceramic brackets use a silane coupler as a chemical intermediary between the bracket base and the adhesive resin. Silane treatment for the ceramic bracket base unites the silica component of the bracket with the composite resin to produce a chemical bond. Silane-treated ceramic brackets show unpredictable and extremely high bond strengths that increase the risk of tooth damage.

Manufacturers sometimes apply a serrated base to ceramic brackets e.g. Transcend© 1000 (3M Unitek). Bond strength is lower than silane treated brackets but higher than stainless steel. It has been suggested that the microcrystalline retentive material of ceramic brackets provides opportunities for stronger bonding between bracket and adhesive than the metal brackets. Habibi *et al.* (2007) compared the characteristics of debonding of metal and ceramic brackets and concluded that the risk of enamel damage when debonding mechanically retained ceramic brackets was not greater than the risk when debonding metal brackets (21).

The ongoing challenge is to develop a bond between orthodontic attachments and enamel that is strong enough to survive the course of treatment but can be broken to get rid of damage without damage to the enamel surface.

1.5.6. Adhesive pre-coated brackets

In order to standardize the amount of composite on the bracket base the use of adhesive pre-coated brackets (APC) will be used in the present study. Both metal and ceramic brackets have been available since 1991 as adhesive pre-coated versions. The pre-coated composite used is a copy of Transbond© XT (3M Unitek), modified to give an increase in viscosity. It can be used in conjunction with Transbon© Plus Self-Etching Primer (TPSEP). APC brackets are originally designed to attempt to save time on the chair by allowing faster and easier bonding procedures.

The advantages of APC over conventional light-cured systems:

- Consistent quality and quantity of light-cured adhesive
- Easier clean-up following bonding,
- Reduced waste,
- Improved infection-control,
- Better inventory control.

Moreover, better control of both the bracket and adhesive with the use of APC is claimed to improve bond strength and thereby reduce the clinical failure rate. The advantage of light-cured adhesives is that they provide the orthodontist with enough time to place the bracket on the enamel surface accurately before polymerization. One of the disadvantages of the light-cured approach is the time it takes to expose each bonded bracket to light.

Increasing the viscosity of the adhesive used on APC brackets, combined with the retention of the mesh imbedded in the base of metal bracket, may significantly reduce the SBS. In response to this data, the manufacturer modified the adhesive used in the pre-coating (APC1 to APC2).

It seems that the duration and intensity of exposure to light is critical to the SBS of APC brackets. APC brackets cured by light for 40 seconds with the halogen unit had a bond strengths similar to uncoated brackets bonded with Transbond© XT. Using of plasma light provides worthwhile time savings when bonding orthodontic brackets, while producing equal bond strength with those in quartz halogen lights.

Reynolds (1975) proposed the clinically accepted SBS of 6-8 MPa (22). Sfondrini *et al.* (2002) reported greater bond strengths than this regardless of the type of light or bracket used (23). Even light-curing for 2 seconds with the micro-xenon light produced clinically acceptable bond strengths for both uncoated and pre-coated brackets. The reduced curing time attained by the micro-xenon light represents a great advantage for both the patient and the doctor.

1.6. Bracket removal

1.6.1. Bond strength testing

The literature contains a large number of publications on the testing of bond strength of materials, the results of which were coded by manufacturers to support their products. However, little attention has been paid to the details of the test procedures used and we need to standardize testing procedures to measure bond strengths, to allow for valid comparisons between different bonding agents.

Hobson and McCabe (2002) investigated the relationship between the characteristics of enamel etch and resin-enamel bond strength. Twenty-eight patients had the buccal surfaces of teeth etched and replicated for examination under the scanning electron microscope. No statistical difference was found in etch patterns between upper and lower teeth. However, the median bond strength varied significantly between different tooth types, with less bond strength found on the upper first molar and the highest on the lower first molar. An ideal etch pattern was not essential in order to produce a strong bond (24).

SBS with SEP compared with conventional two stage bonding systems in laboratory studies. Brackets bonded with the SEP were found to have a significantly lower mean shear bond strength compared with those bonded with a conventional two-stage adhesive system. However, following the application of mechanical stress, the mean survival times for brackets bonded with the SEP or the conventional two-stage bonding systems were similar.

1.6.2. Unit of measurement of bond strength

There has been confusion in the literature over the unit of measurement most appropriate for describing bond strength. Units such as Pascal, Mega Pascal, Newton per millimeter squared or Mega Newton per meter squared have been used. These units provide an indication of the force per unit area required to dislodge the bracket. The use of force as an indicator of bond strength is only appropriate where the area is well controlled, but difficult to measure. As long as the dimensions of the bracket base are quoted, the use of Newton or Mega Pascal is appropriate in quoting bond strength.

1.6.3. Direction and method of debonding

In 58 out of 66 papers examined by Fox and McCabe (1994), an Instron or similar testing machine was used. Other devices used included a pair of specially designed opening pliers and various other testing machines. Forty four of the papers examined tested the specimens in shear mode, 16 in tensile and six used a combination of directions.

Further examination of the papers that reported using an Instron testing machine reveals further differences in the method and direction of debond. A problem arises with the precise relationship of the bracket and its link with the testing machine. The majority of studies use a wire loop around the bracket to connect it to the machine.

The majority of research into SBS with a universal testing machine has applied unilateral forces to the test specimen. The results can not be applied to clinical debonding. Debonding with sharp-edged pliers that apply a bilateral force at the bracket base-adhesive interface has been found to be an effective method of debonding ceramic brackets and its use *in vitro* simulates more closely the debonding forces applied in actual clinical situations.

2. Materials and Methods

Laboratory testing was divided into two distinct phases:

1. Measurement of SBS of three orthodontic bracket types bonded with two adhesive technique. SBS was measured using an Instron universal testing machine
2. Assessment of microleakage of each tooth sample following bracket removal.

2.1. Tooth specimens

In this study we used 60 extracted teeth. Specimens were prepared from maxillary and mandibular premolar teeth, previously extracted for orthodontic purposes. The teeth had been extracted from patients attending T.C Istanbul Yeni Yüzyıl University Dental Hospital. It is likely that all or the vast majority of the extracted teeth were taken from patients living within this area.

The Human Tissue Act (2004) provides a legislative framework for matters relating to body donation and the removal, use and storage of human organs and tissue. The storage and use of extracted teeth for research comes under these guidelines. In accordance with the Human Tissue Authority (HTA) guidelines, consent is not required from donors when anonymized tissue is used for research. This research was registered and approved by the University of T.C Istanbul Yeni Yüzyıl Research and Ethics Committee.

Following extraction, the teeth were stored in specimen jars containing distilled water and thymol crystals (0.1 % weight / volume) to inhibit bacterial growth. Specimens were subsequently stored in the dark at 10° +/- 5°. Time lapse from extraction to testing ranged up to 12 months.

Inclusion criteria for tooth specimens were as follows:

- Intact labial enamel surface,
- Specimen correctly stored following extraction.

Exclusion criteria were:

- Caries,

- Restorations in the tooth,
- Gross enamel hypoplasia,
- Enamel defects,
- Cracking of labial enamel surface,
- Specimen stored incorrectly following extraction.

All teeth were examined under normal surgery light conditions to assess suitability for inclusion. Pronounced cracking was designated as those teeth with cracks detectable by direct visual inspection.

Jars containing the stored teeth were placed into a box and allocated in rotation to each of the three test groups by a process of physical randomization.

2.2. Tooth preparation

The teeth were mounted horizontally in a self-curing orthodontic acrylic resin contained within brass cylinders (8 mm radius, 30 mm length) at cemento-enamel junction. The teeth were embedded so the labial surface of crowns were projected above the surface of the acryl. Specimens were then stored at room temperature in distilled water, again to prevent dehydration of the enamel.

2.3. Enamel surface preparation

Enamel preparation and bracket bonding was standardized in the following way:

1. Labial enamel surfaces were polished with fluoride free pumice slurry using a rubber prophylaxis cup attached to a slow handpiece for 10 sec.
2. Rinsed with air / water spray for 15 sec and dried with a stream of oil-free compressed air for 10 sec.
3. Three test groups, each containing 20 teeth, were prepared.

2.4. Power calculation

In our study, power analysis was performed to determine the number of samples. The sample size was found to be 17 for each group in the analysis of the power analysis performed with G * power 3.1 program and in the sample width analysis performed by taking 0.80 power value in 2 study groups (alpha error probability = 0.05). A total of 60 samples was included in this study and divided in to 3 groups each group 20 samples.

2.5. Bracket selection

Group 1 were bonded with Clarity™ / Advanced Ceramic brackets (3M Unitek, Monrovia, California). This is a ceramic twin bracket designed to blend with one's natural tooth color, and they resist and discoloration over the course of the patient's treatment. The base matching the curvature of the tooth for maximum contact and strong, consistent bond strength.

Group 2 were bonded with Clarity™/ Advanced APC Flash-Free brackets (3M Unitek, Monrovia, California). These brackets are made from a polycrystalline ceramic. APC™ Flash-Free Adhesive is a unique composition of compressible nonwoven mat (a 3M core technology), soaked with a relatively low viscosity adhesive resin (another 3M core technology). When an APC Flash-Free adhesive coated appliance is seated in place on a tooth, the compressible mat lets the resin seep out to fill the space between the appliance bonding base and the tooth.

The bonding base is geometrically designed for optimal tooth fit with a nonwoven material. The smooth molded features facilitate direct and indirect bonding techniques. The bracket base is designed with a V-shaped groove running occluso-gingivally. With the recommended debonding pliers (3MUnitek, Monrovia, California), internal collapse of the bracket occurs on debonding and consequently removal from the tooth with minimal surface damage. These brackets are not moisture tolerant adhesive.

Group 3 were bonded with Clarity™ / Advanced APC™ Plus brackets (3M Unitek, Monrovia, California). APC™ Plus series are an all-ceramic conventionally ligated, twin bracket with metal slot. The manufacturers recommend use of a specifically designed debonding tool to minimize enamel surface damage when removing brackets. Again the plier encourages the bracket to collapse internally and peel away from the tooth surface. The filler material in APC plus Adhesive is mixture of quartz, fumed silica and glass. Also
APC adhesive contains hydrophilic monomers that convert it to moisture tolerant.

Table 2.1: Test groups

GROUP	Number of samples	DESIGNATED ETCH	PRIMER	ADHESIVE	BRACKET TYPE
1	20	Phosphoric acid (37%)	Transbond™ XT Primer	Transbond™ XT Light Cure Adhesive	Clarity / Advanced Ceramic
2	20	Nil	Transbond™ Plus Self Etching Primer	Transbond™ XT with low viscosity and less filler materials	Clarity / Advanced APC™ Flash-Free
3	20	Nil	Transbond™ Plus Self Etching Primer	Transbond™ XT with high viscosity and more filler materials	Clarity / Advanced APC™ Plus

Group 1 – Phosphoric acid etch, Transbond™ XT Primer, Clarity/ Advanced Ceramic brackets (control group)

We will use this group as a control group during this study.

The exposed enamel surface was etched with 37 % ortho-phosphoric acid for 30 sec. Etch was applied to the tooth surface with a brush and agitated during the etching period. The etched surface was then washed with water for 15 sec and dried with oil-free compressed air until the surface of the etched enamel had a frosty appearance.

Transbond™ XT primer (3M Unitek, Monrovia, California) was applied to the etched surface and the tooth lightly blown with a stream of oil-free compressed air to ensure that a thin layer of primer remained before light curing for 10 sec.

An upper premolar Clarity / Advanced ceramic bracket was applied directly to the etched and primed tooth surface using Transbond™ XT Adhesive. Each bracket was firmly pressed at the middle of the clinical crown.

Excess adhesive was cleared from around the bracket periphery with a sharp explorer, and the bonding material polymerized by exposure to light intensity of 1600 mW / cm^2 for 3 sec through the bracket.

Figure 2.1: Polishing buccal surface of tooth.

Figure 2.2: Etching of prepared tooth surfaces with 37 % phosphoric acid.

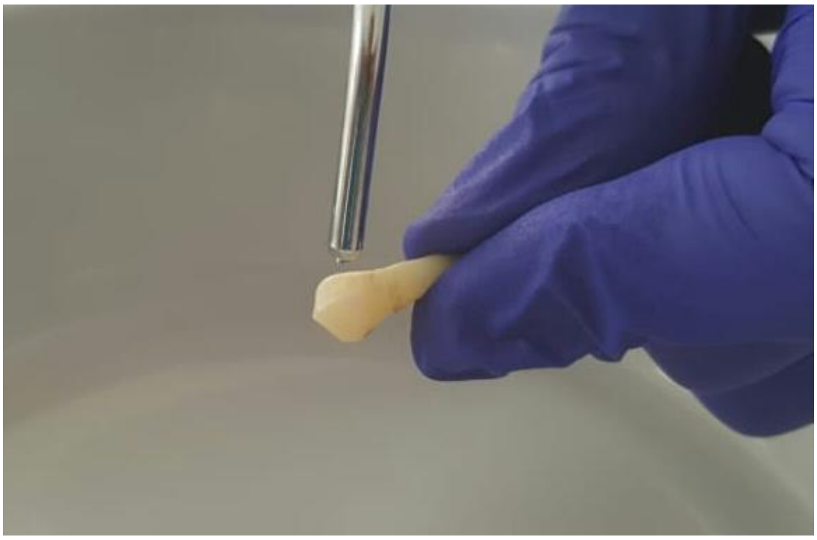

Figure 2.3: Washing surface of tooth from acid and dry it.

Figure 2.4: Apply Transbond XT primer on etched surface.

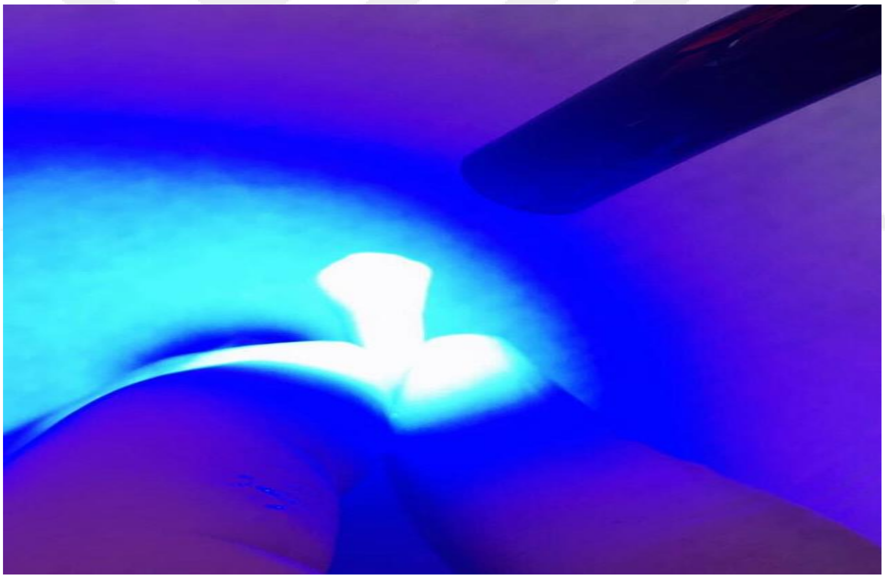

Figure 2.5: Curing primer for 3 sec.

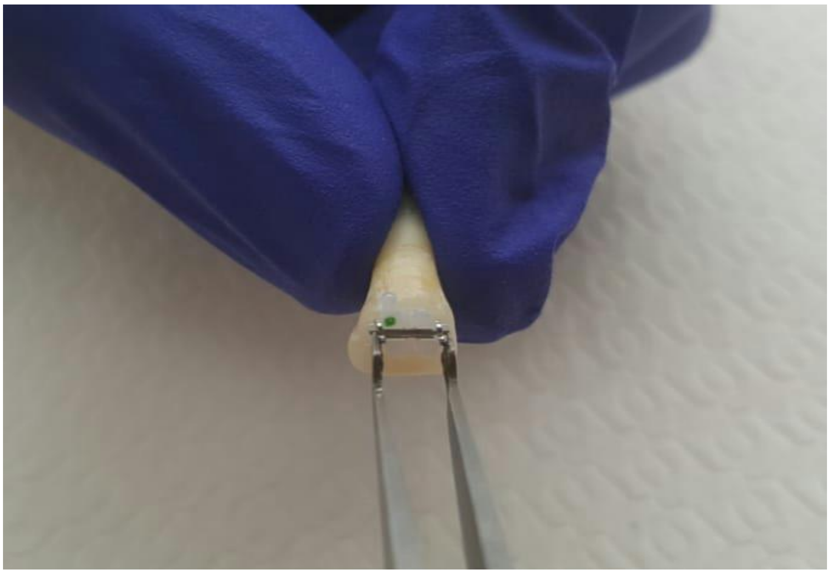

Figure 2.6: Placing the bracket in ideal position after Transbond XT composite was applied on the bracket base and then excessise composite was removed.

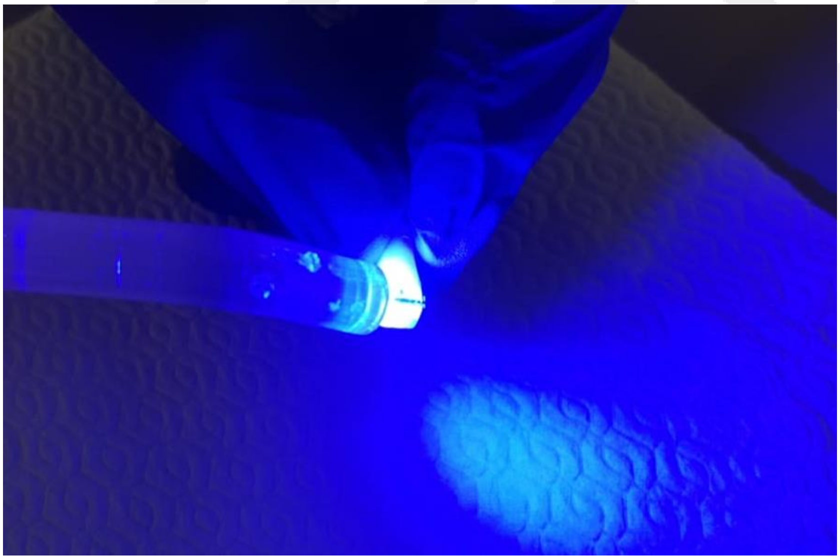

Figure 2.7: Polymerizing adhesive for 3 sec through the brackets

Group 2 – Self etching primer, Clarity / Advanced APC™ Flash-free brackets

Excess water was removed from the labial enamel surface, but the bonding surface kept moist, as per the manufacturers instructions. Transbond™ Plus self-etching primer (SEP) (3M Unitec, Monrovia, California) was activated using thumb pressure against the sequenced pouches in the dispensing sheath. SEP was transferred to the prepared enamel surface using the applicator brush provided. This was rubbed into the exposed enamel surface for 15 sec with the micro brush. The primed surface was then lightly blown with a stream of oil-free compressed air to disperse the solution into a thin film and allow evaporation of the carrier solvent, leaving a glossy enamel surface. A new sheath and applicator was used for each specimen.

An upper premolar Clarity APC™ Flash-Free bracket pre-coated with Transbond™ XT adhesive was applied directly to the prepared tooth surface and firmly pressed at the middle of the clinical crown.

In this type of brackets there is no excess adhesive around the bracket periphery should be removed, and the bonding material polymerized by exposure to light intensity of 1600 mW / cm^2 for 3 sec, and 3 sec for the adhesive composite through the bracket.

Group 3 - Self etching primer, Clarity / Advanced APC™ Plus brackets

The specimens for Group 3 were prepared as for Group 2 except we polymerize the adhesive composite 3 sec mesial and 3 sec distal to the bracket. An upper premolar Clarity / Advanced APC™ Plus bracket, pre-coated with Transbond™ XT Adhesive was applied and bonded directly to the tooth surfaces as per Group 1 except that the excess material should be removed from around the bracket with a sharp explorer.

Figure 2.8: Polishing buccal surface of tooth.

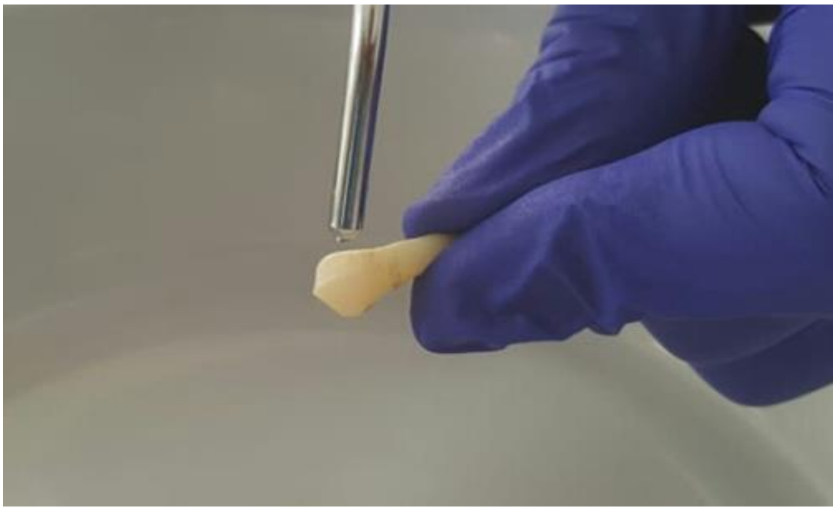

Figure 2.9: Drying enamel surface of tooth.

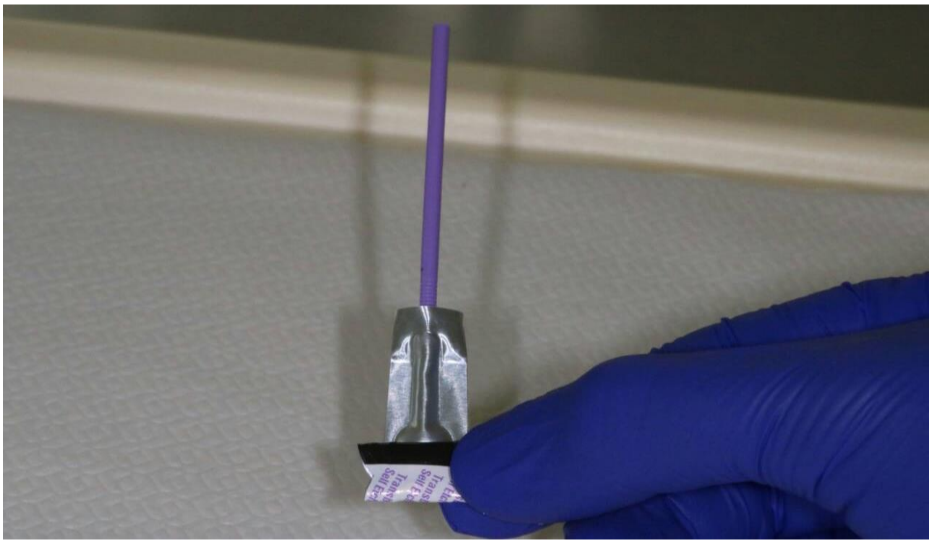

Figure 2.9: Trans bond plus self-etching primer activing by thumb pressure against the sequence punches in the dispending sheath.

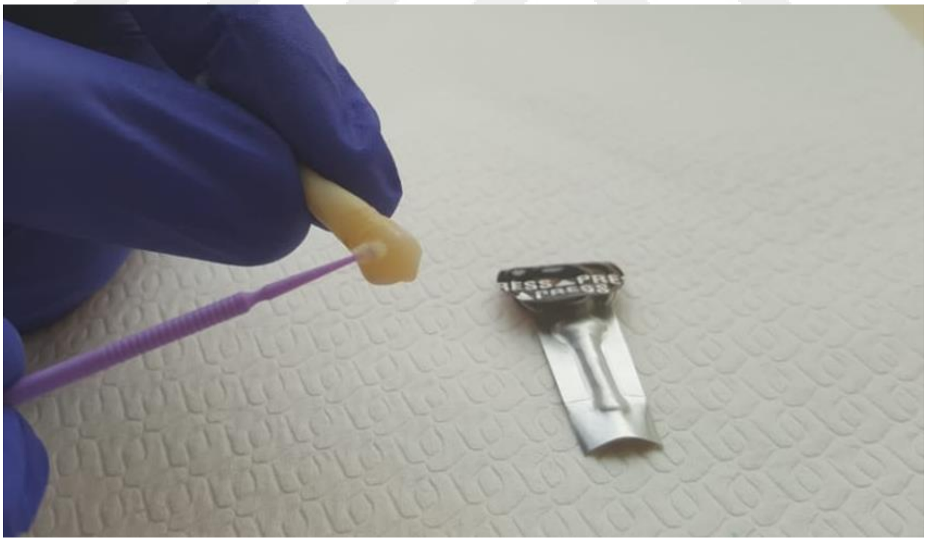

Figure 2.11: SEP was applied to enamel surface for 15 sec.

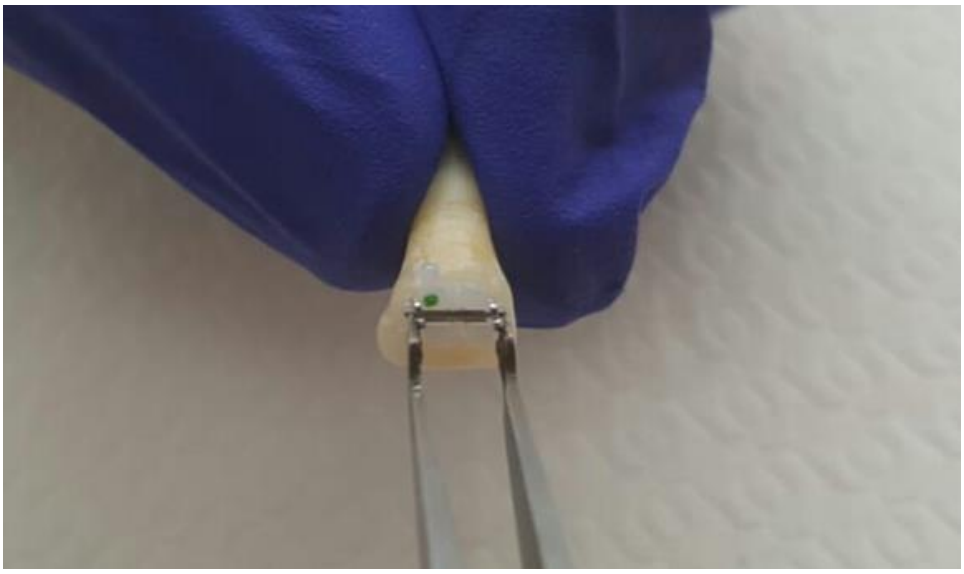

Figure 2.12: Placing APC plus pre-coated bracket in ideal position.

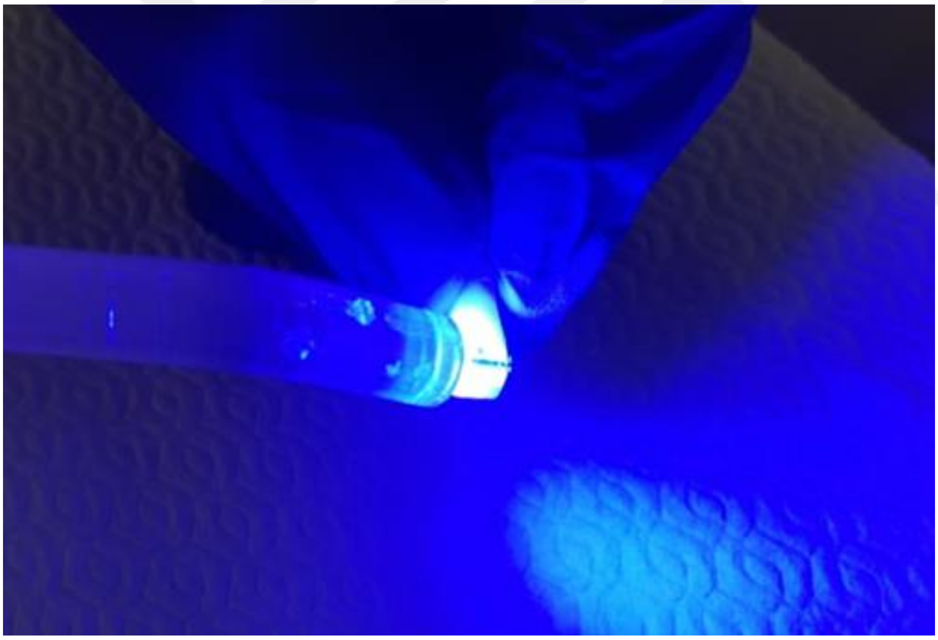

Figure 2.13: Polymerizing adhesive material of APC bracket for 3 sec.

2.6. Bracket placement

Brackets were bonded in all groups to the labial surface at the intersection of the long axis of the clinical crown (LACC) and the clinical crown long axis midpoint (LA point).

Each bracket was seated directly into the correct position and firmly pressed for 5 sec . Peri-bracket excess adhesive was removed using a number 9 sharp probe and pressure reapplied for a further 5 sec.

Each specimen was individually light cured in accordance with the manufacturer's instructions using an Ortholux™ Luminous curing light (3M Unitec, Monrovia, California). A curing time was 3 sec mesial and distal to Flash-Free brackets and only 3 sec through the brackets for both Clarity Advanced and APC Plus brackets according to manufacture of 3M Company. Using an 8 mm diameter curing tip at a light intensity of 1600 mW/cm^2 and wavelength 430 – 480 nm was used. The frequency of the light emitted was calibrated before use with the in-built light intensity meter. The light tip placed on the sensor and an indicator light shows whether the output is adequate. Variation in exposure times would affect the bond strength.

All specimens were allowed to bench cure for 10 min before being placed in a hot water bath (Grant Instruments) filled with distilled water and maintained at 37° +/- 1°C for 24 hours in darkness. Leaving the specimens for 24 hours prior to debonding does not reflect clinical practice. However it does allow adhesive cements to mature to optimal bond strength. The ISO document CD TR 11405 recommends specimens to be placed in water at 37° for short-term storage of 24 hours.

After bonding, the specimens were subjected to a thermocycling process, which was performed between 5 C^o to 55 C^o for 5000 cycles, with dwell time of 30 sec per bath.

2.7. Preparation of specimens for microleakage assessment

Microleakage assessed by the dye penetration technique. For this purpose, the teeth apices sealed with sticky wax and the entire surfaces of the teeth covered by two consecutive layers of nail varnish up to 1 mm around the bracket margins. The specimens then immersed in a solution of 1 % of methylene blue dye for 12 hours at room temperature. After removing from the solution, the teeth thoroughly rinsed and then mounted in metal rings poured with self-curing acrylic resin, so that the buccal surfaces of the teeth were parallel to the direction of the debonding force.

2.8. Bond strength testing

Each brass cylinder with its embedded specimen was assembled in the customized jig in the lower cross head of the Instron Universal testing machine (Model 5544, Instron Inc., Canton, Massachusetts, USA). The jig had a cylindrical hole (8 mm radius) into which each brass cylinder was fitted. The brass cylinder could adjusted in both a rotational and in-out direction, enabling shear forces to be directed at right angles to the long axis of the bracket body by metal rod. Specimens were mounted purposely to direct the applied force occluso-gingivally and parallel to the labial tooth surface.

A shear-peel force was applied through a metal rod engaged between the fixed upper crosshead and the occlusal tie-wings of the bracket. This ensures that the point of application is at the same distance from the bracket / resin interface in all cases, helping to make the method of testing more reproducible. This distance was fixed for each specimen, an increase in distance from the tooth would increase the bond strength.

During testing the Instron had a 2 KN load cell and cross-head speed of 1.0 mm / min.

Bespoke Merlin software electronically connected to the Instron machine recorded the results of the load applied at failure in Kg and Newtons and this data was subsequently converted to MPa.

$$\text{MPa} = \frac{\text{Load (mass) (Kg)}}{\text{Bracket base area}} \times \text{gravitational acceleration constant (9.81)}$$

1 Kg = 9.81 N

1 MPa = N / mm^2

The bracket base size for each bracket type was determined by taking the average sum of the widths and lengths of 10 brackets measured using digital calipers, accurate to 0.01 mm.

Figure 2.14: Laboratory set-up: Instron Universal Testing Machine**.**

2.8. Microleakage measuring following debonding

After debonding, the teeth were examined by one calibrated examiner under stereomicroscope (Dino-Lite Pro, Anmo Electronics Corp, Taiwan) at 20X magnification and the deepest penetration of the dye under the bracket was measured in millimeter perpendicular to the bracket margin.

2.9. Method error calculation

20 % of the specimens were randomly selected and re-examined with an interval of one week. The standard error of method, as calculated using Dahlberg's formula (1940) was below the level of statistical significance, $p > 0.05$.

3. Statistical analysis

Statistical calculations were performed with SPSS (statistical package for the social sciences) (2008) statistical software program for windows. Independent t-test was used to assess for a statistically significant difference in mean values between test groups for SBS and microleakage. Equal variance t-test was performed during the evaluation qualitative data. Statistical significance level was established at $p<0.05$.

Figure 2.16: Used material.

Figure 2.16: Intact premolar teeth.

Figure 2.17: Instruments used during the preparation.

4. The Results

The highest mean of SBS was for Clarity advanced brackets bonded with etch and primer which was 16.4350 MPa. The second highest mean SBS was 12.0995 MPa for APC-Plus ceramic brackets bonded with SEP. The lowest mean of SBS was 11.6885 MPa for Flash free ceramic brackets bonded with SEP. Tables 3.4, 3.5, 3.6 and figure 3.1.

Table 3.1: Mean shear bond strengths (MPa) of the control and experimental groups.

SBS	N	Mean	Std. Deviation	Std. Error Mean
Group 1	20	16.4350	5.19848	1.16242
Group 2	20	11.6885	3.44312	.76990
Group 3	20	12.0995	4.03270	.90174

Table 3.2: Comparison of SBS between groups one and two (MPa)

Group	t-test for Equality of Means							Levene's Test for Equality of Variances	
	95% Confidence Interval of the Difference		Std. Error Difference	Mean Difference		df	t	Sig.	F
	Upper	Lower							
Group 1	7.56903	1.92397	1.39426	4.7465	.002	38	3.404	.032	4.984
Group 2	7.58321	1.90979	1.39426	4.7465	.002	32.980	3.404		

Table 3.3: Comparison of SBS between group one and three (MPa).

Group	t-test for Equality of Means							Levene's Test for Equality of Variances	
	95% Confidence Interval of the Difference		Std. Error Difference	Mean Difference	Sig. (2-tailed)	df	t	Sig.	F
	Upper	Lower							
Group 1	.00415	-.10715	.02749	-.0515		38.069	-1.874	.000	18.383
Group 3	.00471	-.10771	.02749	-.0515	.071	29.144	-1.874		

Table 3.4: Comparison of microleakage from incisal side between group 1 and group 2

Group	t-test for Equality of Means							Levene's Test for Equality of Variances	
	95% Confidence Interval of the Difference		Std. Error Difference	Mean Difference	Sig. (2-tailed)	df	t	Sig.	F
	Upper	Lower							
Group 1	7.31373	1.35727	1.47117	4.3355	.005	38	2.947	.194	1.747
Group 2	7.31979	1.35121	1.47117	4.3355	.006	35.788	2.947		

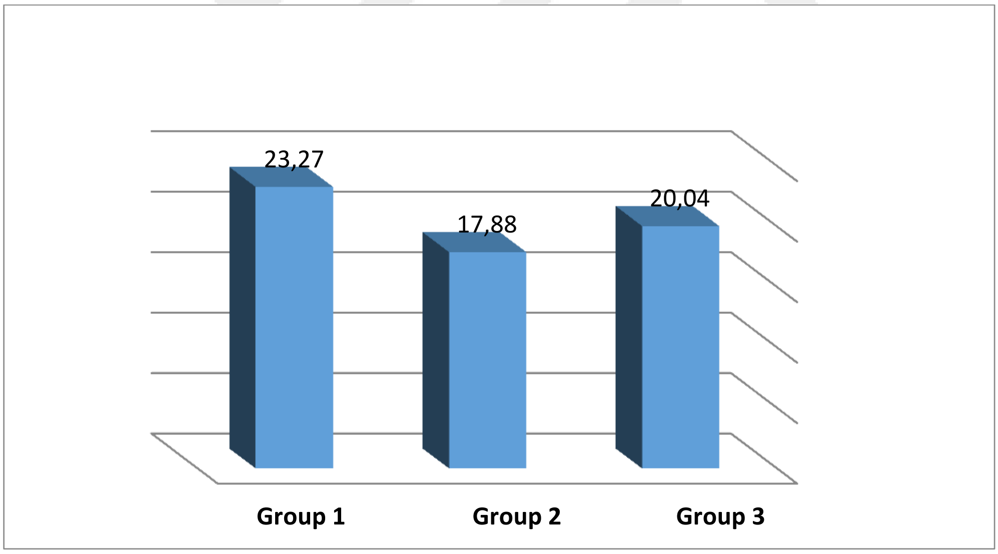

Figure 3.1: Bar-chart of Shear bond strength in MPa between three study groups.

Table 3.5: Comparison of microleakage from incisal side between group 1 and Group 3

Group Statistics

Group	Number of samples	Std. Error Mean	Std. Deviation	Mean
Group 1	20	.01302	.05823	.0230
Group 3	20	.02261	.10111	.0535

Independent Samples Test

Group	t-test for Equality of Means							Levene's Test for Equality of Variances	
	95% Confidence Interval of the Difference		Std. Error Difference	Mean Difference	Sig. (2-tailed)	df	t	Sig.	F
	Upper	Lower							
Group 1	.02232	-.08332	.02609	-.0305	.250	38	-1.169	.015	6.480
Group 3	.02276	-.08376	.02609	-.0305	.252	30.353	-1.169		

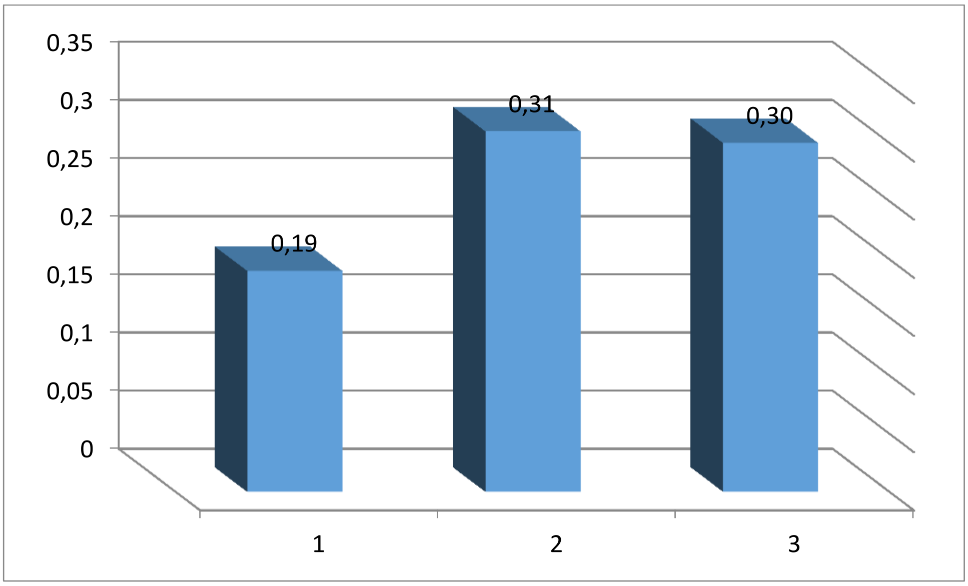

Figure 3.2 : Bar-chart of microleakage from incisal edge between three study groups.

Table 3.6: Comparison of microleakage from gingival side between group 1 and group 2

Group Statistics

Group	Number of samples	Std. Error Mean	Std. Deviation	Mean
Group 1	20	.01575	.07043	.0385
Group 2	20	.03487	.15597	.1390

Independent Samples Test

Group	t-test for Equality of Means							Levene's Test for Equality of Variances	
	95% Confidence Interval of the Difference		Std. Error Difference	Mean Difference	Sig. (2-tailed)	df	t	Sig.	F
	Upper	Lower							
Group 1	-.02303	-.17797	.03827	-.1005	.012	38	-2.626	.000	24.281
Group 2	-.02191	-.17909	.03827	-.1005	.014	26.440	-2.626		

Table 3.7: Comparison of microleakage from gingival side between group 1 and group 3

Group Statistics

Group	Number of samples	Std. Error Mean	Std. Deviation	Mean
Group 1	20	.01575	.07043	.0385
Group 3	20	.02042	.09132	.0565

Independent Samples Test

Group	t-test for Equality of Means							Levene's Test for Equality of Variances	
	95% Confidence Interval of the Difference		Std. Error Difference	Mean Difference	Sig. (2-tailed)	df	t	Sig.	F
	Upper	Lower							
Group 1	.03421	-.07021	.02579	-.0180	.489	38	-.698	.100	2.845
Group 3	.03432	-.07032	.02579	-.0180	.490	35.696	-.698		

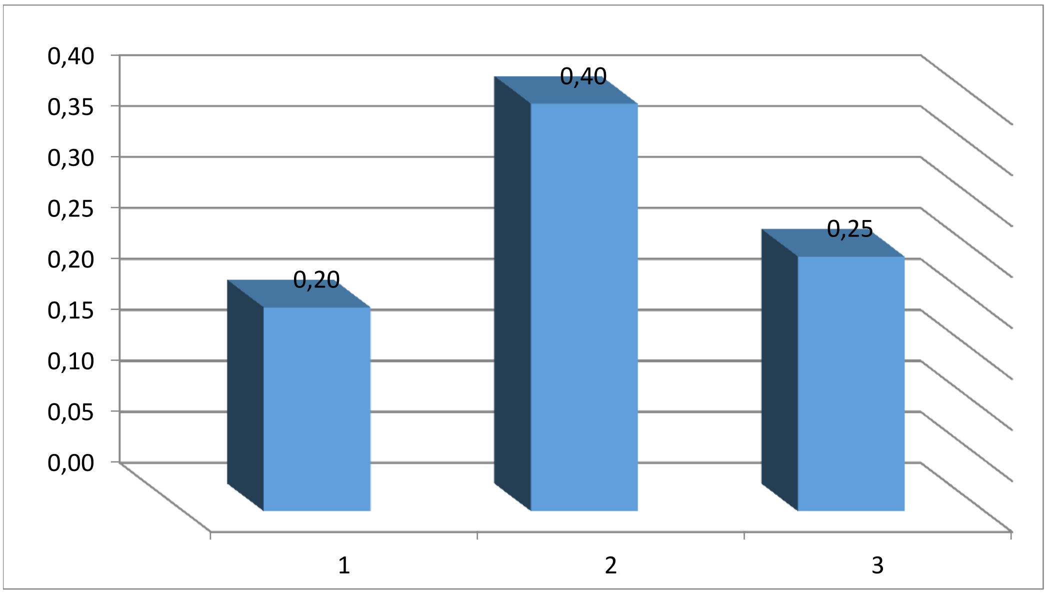

Figure 3.3: Bar-chart of microleakage from gingival side between three study groups.

5. Discussion:

Ceramic brackets are available in many forms. The expensive ceramic brackets which made of monocrystalline which have excellent aesthetics, while the less expensive ceramic brackets are composed of polycrystalline that have comparatively poorer aesthetics. One of the important and challenging requirements of these ceramic brackets is to be able to provide adequate bond strength during the orthodontic treatment along with easy debonding procedure resulting in minimal damage to the enamel surface.

The majority of ceramic brackets depend on the mechanical retention to form an acceptable bond. The advantage of the ceramic bracket over metal bracket is affinity of the ceramic brackets to incident light which allows more transmission of light onto the bracket base resulting in high polymerization of the adhesive and thus, providing a high SBS.

Several factors such as method of enamel conditioning, composition of adhesive, bracket retention mechanism as well as method of debonding influence the forces applied for debonding the brackets. In the present study, similar debonding conditions were performed in all the three groups to minimize the variables influencing the debonding characteristics. The results indicated that different enamel conditioning have different effects on SBS.

In our study, we aimed to evaluate and compare the SBS and microleakage of flash -free and APC-Plus Ceramic orthodontic brackets. We used natural human teeth, so we have increased the variability of the bond strength; in addition, the use of human teeth more closely approximates the clinical situation with respect to tooth morphology. The teeth of similar sizes and shapes were selected to decrease the possible variations and errors. All extracted teeth were stored in storage media until further processing; the storage medium maintains the chemical, physical and mechanical properties of extracted teeth and to prevent dehydration of the teeth. The major storage media used for natural human teeth are formaldehyde, ethanol, chloramine, freezing, water, distilled water, saline solution and thymol are the major storage media use for natural human teeth.

In our study, we used distilled water as a storage media for extracted teeth, which considered as one of the best storage medium capable of reassuring adequate results concerning to the enamel and dentine characteristics. Silva et al. (2006) compared the effect of the storage time and type of storage on bond strength of extracted tooth. They showed that extracted teeth stored in distilled water provided less variation in bond strength values (25).

The enamel surface should be polished, then rinsed with air/water and dried with a steam of oil free compressed air. Kimura et al (2004) had reported that cleaning the tooth surfaces have a higher surface energy that is amenable to bonding (26). In our study, the labial surface of enamel was polished with no fluoride of pumice because the fluoride on the surface can decrease the surface energy of the adherent and decreasing the ability of the adhesive to spread.

Garcia-godoy et al.1991, had reported that the topical application of fluoride can interfere with etching effect of phosphoric acid on enamel surface resulting in reduced bond strength of dental resins (27). Also Aasenden et al (1972) had reported that the fluoride deposits in hydroxyapatite to form fluor-apatite that might affect the bond strength (28).

In this study, we used two different etching protocols for enamel preparation. A conventional etching and self-etching primer technique to know the effect of etching protocol type on the microleakage and bond strength of brackets. The materials used for surface preparation and adhesive were Transbond plus self-etching primer and Transbond XT light cured adhesive and primer. These materials has been widely used in orthodontic clinics.

Bishara et al (2004), had reported that self-etching primer and adhesive systems provide significantly lower but clinically acceptable SBS when compared with a conventional etching and primer technique bonding brackets with Transbond XT adhesive paste (3M Unitek, Monrovia, Calif) (29).

In our study we selected a new type of brackets which are (Clarity Advanced brackets) to evaluate microleakage and bond strength and compare this with both (Flash-free and APC Plus pre-coated ceramic brackets). If the results of SBS in relative to microleakage are clinically acceptable, so we can use the advantage in the clinic to reduce the bonding steps.

The adhesive resin of the samples was polymerized by using an ortholux luminous curing system (3M unitek) with High intensity 1600 mW/cm2 blue LED for 3 sec mesial and 3 sec distal sides of Flash-free brackets and only 3 sec through the bracket for Advanced Ceramic and APC Plus brackets. The ortholux luminous light with a combination of high intensity LED lamp and 8 mm light guide optimized for orthodontic bonding and efficient curing time.

The method used to measure the SBS of orthodontic brackets that bonded to extracted teeth, is the compressive fracture resistance test by using a Universal Testing Machine. There are advantages and disadvantages to such testing and its relevance to clinical practice is questionable. In vitro shear bond strength testing does not exactly replicate the clinical situation; however, it does give an indication of potential or anticipated bond strength in vivo. In reality, potential loading would be complex with the following acting as stresses on the enamel-adhesive and adhesive-bracket interfaces: Multi-directional loading during function such as eating and stress introduced by application of orthodontic force by ligation of an archwire.

The recommendation for standardization of bond strength testing was introduced by Fox et al. in1994 (30). However, the following problem would arise an in vitro investigation: enamel surface structures of extracted teeth may differ from in vivo due to desiccation during storage and bracket removal by using shear force only.

In our study, the mounted specimen were placed inside an adjustable vice for SBS testing in a push-pull instron universal testing machine (Model 3382; Instron Corp., Canton, Massachusetts, USA) at a cross head speed of 1 mm/minute connected to a computer that recorded the results of each test. The test was accomplished by using a chisel edge mounted on crosshead of the testing machine. Each tooth was orientated such that the chisel was parallel to the bracket base and equidistant to both incisal tie-wings. The chisel-type working tip was positioned in the occluso-gingival direction in contact with the bracket-enamel junction, producing a shear force at the bracket-tooth interface until the bracket deboned.

In our study, the crosshead moved in a speed of 1 mm/minute, the maximum load necessary to debond or initiate bracket-tooth interface fracture was recorded in Newtons and then converted into Mega-pascals as a ratio of Newton's to surface area of the bracket.

Bond strength in MPa = Force (in Newton)/Surface area of bracket (in 2 mm). After shear mode testing, enamel surfaces of teeth were inspected independently by the same evaluator. An optical microscope (Stereomicroscope) at 20X magnification was used to determine the microleakage.

Axial loading that was performed in the present study may represent occlusal forces with the point of application at the same distance from the bracket resin interface in all cases, helping to make the method of testing more reproducible. Katone et al. (1997), reported that increase in distance from cross head of the instron universal testing machine to occlusal tie wing of bracket would increase the bond strength (31).

SBS should be within an optimum range between 5.8 MPa-13.5 MPa to be supposedly “clinically acceptable” as recommended by Rossouw (Rossouw, 2010) about 10 MPa as mean value (32).

Bracket failure at either of the two interfaces, bracket-adhesive interface or enamel-adhesive interface, has its own advantages and disadvantages (Bishara et al., 2007) (33). Failure at the bracket adhesive interface is advantageous as it indicates good adhesion to the enamel and is safer to deboned (Berk et al., 2008) (34). However, considerable chair time (Khoroushi et al., 2007) (35) is needed to remove the residual adhesive, with the added possibility of damaging the enamel surface during the cleaning process (Justus et al., 2010) (36). Also more enamel loss during cleaning is reported (Bishara et al., 2000) (37). In contrast, when failure occurs at the enamel-adhesive interface, less residual adhesive remains on the enamel and less chair-side time is needed for cleaning. However failure at this interface may cause enamel fracture while de-bonding (Berk et al., 2008) (34).

Before performing mechanical tests, the teeth apices were sealed with sticky wax and the entire surfaces of the teeth were covered by two consecutive layers of nail varnish up to 1 mm around the bracket margins. The specimens were then immersed in a solution of 1 % of methylene blue dye for 12 hours at room temperature. After removing from the solution, the teeth were thoroughly rinsed and embedded in self polymerizing acrylic resin; stainless steel cylinder can be used as model for resin. The teeth have been embedded in acrylic resin blocks to simulate cortical bone and the cemento-enamel junction of teeth should be situated approximately 2 mm above the level acrylic resin to simulate bone crest.

In our study after debonding, the teeth were examined by the same calibrated examiner under a stereomicroscope (Dino-Lite Pro, Anmo Electronics Corp, Taiwan) at 20X magnification and the deepest penetration of the dye under the brackets was measured in millimeter perpendicular to the bracket margin. To examine the measurement error, 20 % of the specimens were randomly selected and re-examined with an interval of five days. The measurement error was determined using the Dahlberg formula and the systemic error of the two measurements was assessed by the Wilcoxon signed rank test.

During function, orthodontic brackets are subjected to either shear, tensile or torsion forces, or even a combination of these factors. The brackets in vivo are also will be under the effect of heat change in the mouth, therefore in our study all teeth bonded with brackets were thermocycled for 5000 cycles between 5 C^o and 50 C^o with a dwell time of 30 sec, which simulated 6 months of intraoral environment.

In the present study, when comparing the mean SBS of different bracket systems, it was observed that the mean SBS of Clarity Advanced brackets was the highest (16.4350 MPa) followed by Clarity APC Plus Adhesive pre-coated brackets (12.0995 MPa), While the bracket system with the least mean SBS was Clarity Advanced APC Flash-free (11.6885 MPa).

These results were in accordance with a number of researches. Bearn et al.1995, Oliver et al.1988, and Cal Neto JP et al.2006, who reported that the mean SBS of ceramic brackets which were manually coated and bonded with Transbond XT was significantly higher than the adhesive pre-coated brackets, and also the rate of bond failure was higher with adhesive precoated brackets as compared to the conventional brackets (38,39,49). Although in contrast to this study in 2016, Lee M and Kanavakis G observed that the SBS of APC Flash–free group was significantly greater than the manually pasted Clarity Advanced ceramic bracket (41).

As regards the different types of enamel conditioning – using the traditional system or a Self-Etching Primer (SEP) – SBS values may vary according to different studies. However, similar mean values of SBS, with no statistical difference between the methods mentioned previously, were observed in some studies. In contrast, several authors have reported greater values of SBS for orthodontic brackets with phosphoric acid conditioning as compared to conditioning with a SEP. On the other hand, examination of the enamel-adhesive interface by scanning electron microscopy has revealed different patterns. Enamel conditioning with Transbond SEP has resulted in a more conservative enamel surface, comparable to that produced by traditional phosphoric acid conditioning. However, the SEP system may be considered for clinical use because its use may entail a decrease in undesirable enamel effects during bonding procedures, and also because it reduces clinical chair time. In addition, it is a less sensitive technique, which can be justified by its one-step procedure.

The use of APC brackets in the present study showed some advantages, such as standard quantity of adhesive, which provide a uniform thickness of the adhesive layer between the bracket and the tooth surface, easy removal of excesses, better asepsis and reduction of occasional loss of material. Although the related literature has revealed greater mean values of SBS when using traditional orthodontic brackets, APC brackets were used instead because they involve the use of a less sensitive technique.

Enamel etching with phosphoric acid created an etch pattern characterized by a deep and uniform demineralization area. These demineralized areas were infiltrated by the resin of the priming solution which produce resin tags penetrating into the demineralized surface. Compared with phosphoric acid, TSEP produced a uniform and more conservative etch pattern with regular adhesive penetration and less aggressive enamel demineralization. When any problem exists during bonding process, it is possible for seeping and leaking of fluids and bacteria to occur between enamel-adhesive interfaces, and we can interpret these as the potential white-spot lesion on the enamel surface. A review of the literature indicated that no studies have compared the microleakage in relation to SBS of ceramic brackets bonded with orthodontic composites to enamel that have been prepared with conventional acid etching and TSEP procedures.

In this study, the dye-penetration method was chosen to determine microleakage of the bonded specimens. This is the most commonly used method to assess microleakage of dental materials. In our study all specimens were evaluated by the two operators at two times to evaluate measurement error.

Microleakage, however, may not be similar on the other sides on a bonded tooth, although studies on restorative dentistry have assumed that one-side assessment is representative of the whole tooth. Airlocks in the marginal gap, leaching of water-soluble tracers during processing, and failure of only a few sections to allow interpretation of the full pattern, limit dye-penetration tests to low reproducibility and precision. It is important to note that the assessments in the present study were made by examine the base of brackets and buccal surfaces of the teeth after debonding of brackets between enamel-adhesive and adhesive-bracket interfaces the four sides (mesial, distal, occlusal, gingival).

In vitro, microleakage is commonly assessed to detect bond failure at the enamel sealant interface through dye penetration. This failure can be due to polymerization shrinkage or different linear coefficients of thermal expansion from tooth hard substances and resin materials. Thermal cycles are widely used to simulate temperature changes in the mouth, generating successive thermal stresses at the tooth-resin interface, therefore thermocycling was performed in this study.

Polymerization shrinkage varies by adhesive composition, percentage of filler, the diluents, or the percentage of the monomer conversion in the specific composite resin and curing type Polymerization shrinkage of the adhesive material may cause microleakage-promoting micro gaps between the adhesive material and the enamel surface, which may initiate microleakage and possible white-spot lesions under the bonding area. However, from an orthodontic perspective this condition is different. Adhesives at the edges of the bracket can compensate some shrinkage and this shrinkage can pull the bracket closer to the enamel by the free floating of the bracket. In contrast to the thick composite resin put in a prepared cavity in restorative dentistry, polymerization shrinkage and the subsequent microleakage is less of a concern in orthodontic adhesives because a thin layer is used.

In 2006, Arhun et al found that microleakage scores obtained from the incisal and gingival margins of the brackets demonstrated significant differences, implying increased microleakage on the gingival side (42). They interpreted these differences as related to the surface curvature anatomy, which may result in relatively thicker adhesive at the gingival margin. However, the authors did not explain which visible light-curing device was used and how. In the present study, the Ortholux Luminous Curing Light with high intensity 1600 mW/cm^2 blue LED.

Results of this study indicated that porcelain brackets showed statistically significantly more microleakage at the gingival side than the occlusal side in the enamel-adhesive interface and no microleakage at mesial and distal margins. We also observed no microleakage in the adhesive-bracket interface. Our findings were similar with Arhun et al, (42) and our interpretation is similar to his.

The presence of resin tags prepared at the enamel surface by acid etching is an important factor to fight leakage. A deeper etching pattern ensures the possibility of better resin penetration, but it does not guarantee a sealant-enamel interface that is free of microleakage or better sealant retention. This is supported by an in vivo study where no differences were found between sealants applied over a self-etching adhesive or H3PO4 etched teeth after 24 months. However, several in vitro studies do not advocate the use of self-etching adhesives on intact enamel because of significantly lower bond strengths, greater microleakage, and an etching pattern that is not deep enough to obtain good penetration of bonding resin. Our results were consistent with the literature for microleakage under brackets at the enamel-adhesive interface (42). Both ceramic brackets bonded with TSEP showed significantly higher microleakage than when the conventional acid-etch method was used.

In previous studies in the literature indicate that ceramic brackets produce stronger bonds than the metallic orthodontic brackets. Arhun et al. (42) reported that the increased strength and difficulty in debonding for ceramic brackets may be attributed to the close adhesion of the ceramic bracket to the adhesive in the absence of microleakage. Similar to the opinion of Arhun et al we thought incomplete polymerization of the adhesives under metallic brackets may explain this difference, because investigators indicated a number of factors that affect the final degree of cure of a resin. These included the chemical structure of the dimethacrylate monomer and the polymerization conditions, that is, atmosphere, temperature, light intensity, photo-initiator concentration, filler type, shade of adhesive resin, and the reflective characteristics of adhesive resin Yoon et al. in 2002, explained this incomplete polymerization of the cure by decreases from the top surface inward (43).

Further studies are necessary in orthodontics to investigate the correlations between microleakage and SBS, different bonding materials, and curing devices. Moreover, a study should be designed to investigate the reason for the difference in the amount of microleakage between the gingival and occlusal sides of the orthodontic brackets.

Conclusions

1. The SBS of Advanced Ceramic Brackets bonded by using conventional acid etching technique is higher than Flash-Free and APC Plus Ceramic brackets bonded with SEP technique.
2. Both Flash-Free and APC Plus brackets have SBS which are clinically acceptable.
3. Microleakage is found to be less under ceramic brackets bonded by using conventional method due to long resin tags.
4. Higher microleakage was observed at the gingival margins compared to occlusal margins which was revealed to the tooth surface anatomy .
5. An inverse relationship was observed between microleakage and SBS using ceramic brackets.

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