CHAPTER II

LITERATURE REVIEW

In 1955, Buonocore introduced tooth surface treatment with phosphoric acid to improve adhesion between acrylic resin and enamel.[6] Subsequently, in 1965, Newman recommended direct bonding in orthodontic treatment.[7] This development has improved the treatment result as it has no need of tooth separation for banding, therefore it reduces patient's discomfort and pain from tooth separation. The technique also provides more acceptable esthetics. It causes less plaque accumulation and decreases the amount of decalcified enamel surfaces as it is easier for patient to clean. Moreover it requires less chair-time to apply and remove the bands.[8,9] Consequently, this orthodontic bonding system is widely accepted and has had the effect of increasing the types of adhesives in the marketplace.

Nevertheless, orthodontists should beware of bond failures, which lead to unfavorable effects such as prolonged treatment time, increased cost of materials and need for extra dental visits. Several factors affecting the success of bonding were taken into account in this *in vitro* study, such as 1) the type of tooth, 2) the fluoride content of the teeth, 3) disinfection of the teeth and storage media before bonding, 4) the elapsed time of storage following bonding, 5) the type of loading, 6) the configuration of the specimen testing jig, 7) the crosshead speed of the mechanical testing machine, and 8) the bond area of the bracket.[10] Only some clinical factors influencing the success of orthodontic bonding systems are discussed, for instance,

I) the tooth surface and its preparation, II) the etching and priming techniques, III) the light curing unit and IV) the adhesive systems.[11-13]

I. Tooth surface and its preparation

A previous study revealed that the chemical composition of the enamel surface, such as calcium, phosphorus, oxygen, nitrogen and carbon, has no effect on shear bond strength.[14] But when comparing human enamel to bovine enamel, the investigation demonstrated the development of bovine enamel and dentin is more rapid during tooth formation, resulting in large crystal grain and more lactic defect than in the development of human enamel. And after etching, human enamel has arcade-shaped prisms, whereas bovine enamel reveals a small oval shape, which makes the bond strength 21-44% weaker with bovine enamel than with human enamel.[15] Therefore, to study orthodontic bracket bond strength, human enamel is more appropriate.[1,3,5,16-22]

Considering tooth position, bond failures are predominantly found in posterior mandibular areas, particularly in partially erupted mandibular premolars that are the most common teeth exhibiting bond failures.[23] An unerupted ectopic tooth that needs surgical exposure for bracket placement is an additional, extreme example. The major difficulty is in obtaining and maintaining a completely dry operating field throughout the process. Fluid contamination at the time of surgical operation leads to the increased probability of bond failure.[24]

II. Etching and priming techniques

1. Conventional etching (total-etching)

In the etching step with phosphoric acid, the most common etchant for dental bonding, calcium is selectively dissolved from the enamel structure and washed from the tooth surface during rinsing. This situation leads to an exposed honeycomb structure, as it produces microporosities on the enamel surface.[25] Such created pits enhance the ability of the polymerizing resin to penetrate the etched enamel creating micromechanical retention.[26]

A previous investigation of the enamel surface with scanning electron microscopy, identified five etching patterns on the buccal surfaces.[27] Figure 1 shows a typical Type 1 etching pattern, where the prism cores were preferentially removed, leaving the prism peripheries intact. The Type 2 etching pattern is shown in Figure 2, in which the prism cores remain relatively intact while the prism peripheries are selectively demineralized. Figure 3 exhibits a Type 3 etching pattern, which is a mixture of Type 1 and Type 2 configurations. The Type 4 pattern demonstrates a pitted enamel surface, as shown in Figure 4. The Type 5 pattern is characterized in Figure 5, demonstrating a flat, smooth surface after etching. The etching patterns were observed in all acid groups after either 60 or 90 seconds of acid application. Moreover, the study also indicated that Type 1 and Type 2 etching patterns predominantly appeared on the coronal areas of the buccal surfaces. The Type 3 etching pattern, a combination of Types 1 and 2, was primarily located at the middle third. The Types 4 and 5 etching patterns, which were less well defined patterns, were observed mostly in the cervical regions of the buccal surfaces.[27]
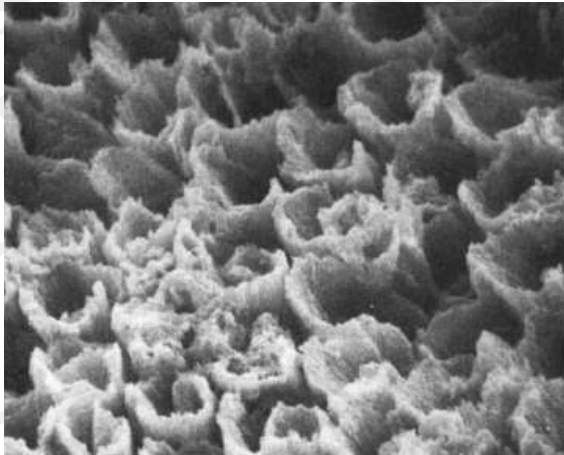
Figure 1 Scanning electron micrograph showing the Type 1 etching pattern after

applying phosphoric acid for 1 minute. (Magnification X 1750) (Galil,1979)

Figure 2 Scanning electron micrograph showing the Type 2 etching pattern after

applying phosphoric acid for 1 minute. (Magnification X 1750) (Galil,1979)

Figure 3 Scanning electron micrograph showing the Type 3 etching pattern after

applying phosphoric acid for 1 minute. (Magnification X 1750) (Galil,1979)

Figure 4 Scanning electron micrograph showing the Type 4 etching pattern after

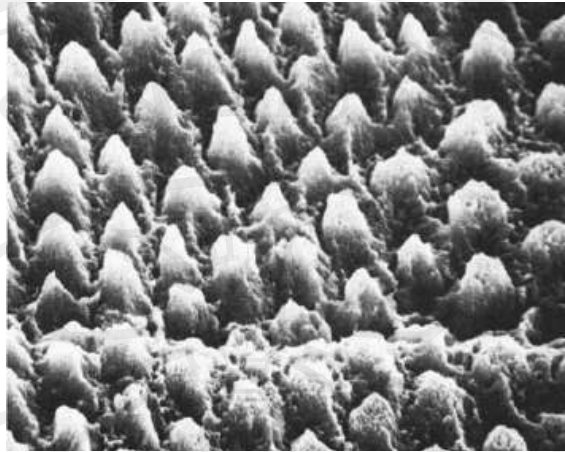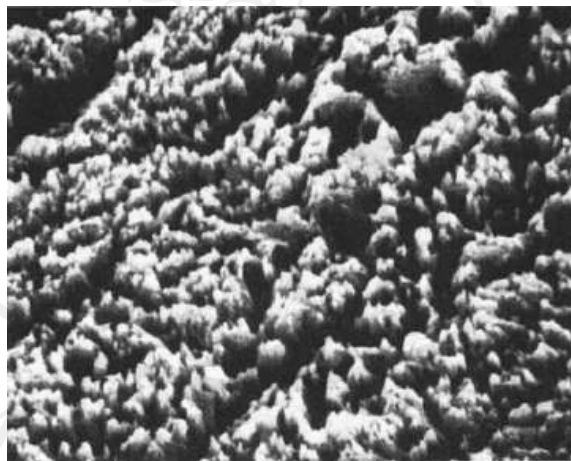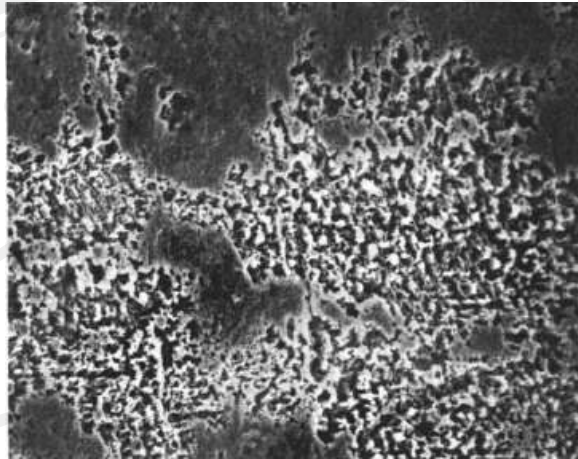applying phosphoric acid for 1 minute. (Magnification X 1750) (Galil,1979)

Figure 5 Scanning electron micrograph showing the Type 5 etching pattern after

applying phosphoric acid for 1 minute. (Magnification X 1750) (Galil,1979)

2. Self-etching

According to the conventional total-etch procedure with phosphoric acid, the
bonding process is a chair-time-consuming task and is prone to moisture
contamination, as it needs rinsing and drying before primer application. Self-etching
primers have been developed by combining etching and priming steps simultaneously,
resulting in a time reduction of approximately 1.97 seconds per tooth, and a reduction
in procedural errors.[28] The lower acidity of self-etching primers than of phosphoric
acid produces a lower etching ability of self-etching systems. However, it causes less
damage to enamel and also less irritation to soft tissue.[29,30]

Historically, self-etching primers were originally introduced for bonding to
dentin. Subsequently, it has been proposed to be effective on enamel, even though
enamel is composed of less organic content than dentin. Self-etching primer contains
methacrylated phosphoric acid ester, which contains a phosphate group, as the active
acid ingredient that dissolves and removes calcium from hydroxyapatite, similarly to

phosphoric acid. However, the calcium forms a complex with the phosphate group and is incorporated into the network when the primer polymerizes. In this manner the acid is neutralized. Agitating the primer on the tooth surface serves to ensure that fresh primer is transported to the tooth surface. The etching process and penetration of the exposed enamel rods occur simultaneously.[31] The penetration of primer into the depth of the etched surfaces ensures a good microscopic mechanical interlock between the enamel and resin. Resin infiltrates into enamel to a depth of 0.6-0.7 μm, which is less than the depth of phosphoric acid infiltration, which allows 1.0 μm resin tags to produce an enamel hybrid layer.[32] Nevertheless, a recent study claimed that a self-etching adhesive system provided adequate superficial resin penetration to produce acceptable resin-enamel bond strength.[32] However, the greater the adhesive penetration, the greater the risk of enamel damage in the de-bonding process.

Some researchers have proposed three mechanisms to stop the etching process.[33] First, the acid groups attached to the etching monomer are neutralized in a similar manner to with phosphoric acid, by forming a complex with the calcium from the hydroxyapatite. Second, as the solvent is driven from the primer during the air burst step, the viscosity rises, slowing the transport of acid groups to the enamel interface. Finally, as the primer is light cured and the primer monomers polymerized, transport of acid groups to the interface is stopped.[33]

Contamination of etched enamel interferes with the resin penetration, as most of the porosities become plugged, resulting in inadequate amounts and lengths of resin tags to provide retention.[34] Manufacturers have developed new self-etch systems containing hydrophilic primer components, which include an acetone-soluble ester, systems that are efficient even in the presence of moisture.[1]

III. Light curing unit

As the metal bracket is opaque, a light curing unit must scatter the light as indirect irradiation to the enamel.[35] The light from enamel trans-illumination reaches the tooth/adhesive interface easier than the bracket/adhesive interface, causing inadequate polymerization of adhesives in the bracket mesh base and lower bond strength. The efficient polymerization of all of the adhesive in the bracket mesh base provides stronger interlocking of composite resin in the bracket base. Therefore, cohesive failure within the adhesive is more likely than adhesive failure at either the tooth/adhesive or bracket/adhesive interface.[36]

The efficiency of polymerization of light-curing adhesives depends on many factors, such as the matching of wavelength and curing time. Light-curing adhesives contain a photo-initiator, which accelerates the curing process when activated by the optimum wavelength of the light-curing unit. The most common photo-initiator for polymerization is camphorquinone, which absorbs light of 400 to 500 nm wavelength (blue light in the visible light spectrum). Camphorquinone has a peak absorption of 465 nm, which is close to the peak wavelength of light from the light-emitting diode (LED) in the light-curing unit (460 nm).[37]

The curing time can be reduced if a higher intensity light is applied. Reduction of curing time improves the result of bracket placement by decreasing the risks of saliva contamination.[38]

IV. Adhesive systems

Adhesion is the attachment of one substance to another whenever they come into close contact with each other. The most important requirement for adhesion is

that the two materials to be bonded to each other must be in sufficiently close and intimate contact. The intimate contact between the bonded substrates depends on the surface tension of the substrate and the viscosity of the adhesive.[39]

Surface wetting by a liquid is commonly characterized by the contact angle of a droplet on the solid surface (Figure 6). The better the liquid spreads over the surface, indicated by a contact angle close to 0 degree, the better the wetting that takes place. The wetting phenomenon depends on the difference between the surface tension of the adhesive and of the adherend. The surface tension of the liquid is taken approximately as the free surface energy of the substrate. A liquid will wet completely when the free surface energy is higher than surface tension, but when the free surface energy is lower, the wetting will be incomplete.[40]

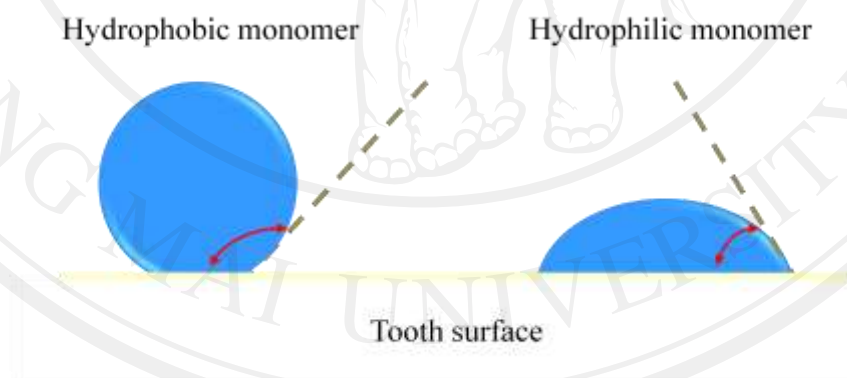Hydrophobic monomer   Hydrophilic monomer

Tooth surface

Figure 6 Contact angle between adhesive and tooth surface. **Left:** high contact angle; **right:** low contact angle. (Modified from Ibsen and Neville, 1974)

Viscosity is a term describing consistency. A thick, pasty material cannot be expected to readily flow over surfaces. The viscosity interferes with the

establishment of maximum wetting in spite of the fact that strong attraction may exist between molecules of a viscous adhesive and those of the adherend's surface.[41]

It can be concluded that whenever the substrate has high surface energy and the adhesive has low viscosity, the microporosities and irregularities of a surface can be filled to achieve micromechanical retention.[42]

In orthodontics, commercially available adhesives contain different organic matrix monomer components, for example, BisGMA (Bisphenol-A-glycidyl dimethacrylate) (Figure 7), HEMA (2-hydroxyethyl methacrylate) (Figure 8), PEGDMA (Polyethylene glycol dimethacrylate) (Figure 9) and TEGDMA (Triethylene glycol dimethacrylate) (Figure 10).[43]
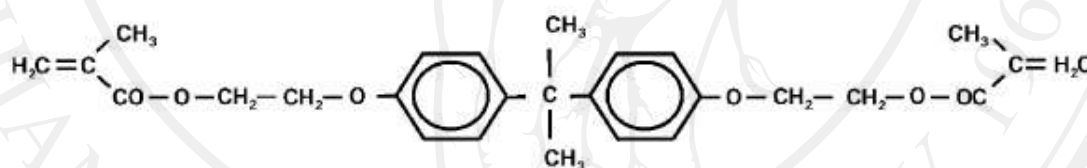

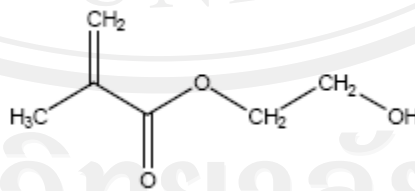**Figure 7** BisGMA (Bisphenol-A-glycidyl dimethacrylate) (Van Landuyt,et al.,2007)

Figure 8 HEMA (2-hydroxyethyl methacrylate) (Van Landuyt,et al.,2007)
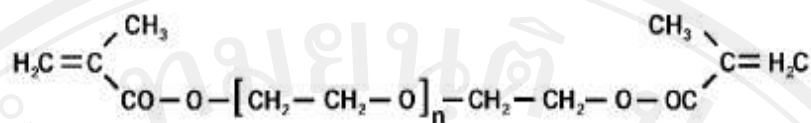
Figure 9 PEGDMA (Polyethylene glycol dimethacrylate) (Van Landuyt,et al.,2007)

Figure 10 TEGDMA (Triethylene glycol dimethacrylate) (Van Landuyt,et al.,2007)

Most of the early adhesive materials were based upon the viscous aromatic dimethacrylate (Bis-GMA) monomer.[39] With a large molecular size and chemical structure, it is superior to other monomers by excellence of lower volatility, lower polymerization shrinkage, more rapid hardening, and production of a stronger and stiffer resin. However, the large molecular mass of Bis-GMA results in its high viscosity. A variety of other monomers has been added to achieve a suitable viscosity of Bis-GMA. Moreover, due to the hydrophobic property of Bis-GMA, moisture contamination interferes with the bonding process.

Transbond™ XT (3M Unitek®, Monrovia, California, USA), is most commonly used as a control system in studies of adhesive systems for orthodontic bracket bonding. According to the manufacturer, Transbond™ XT contains Bis-GMA, which has a hydrophobic property, as a major monomer. Most studies suggest that after saliva contamination, Transbond™ XT has unacceptable bond strength for clinical needs.[1,2,4,5]

Recently, manufacturers have developed moisture-resistant adhesive systems by adding hydrophilic monomers, such as HEMA, PEGDMA and TEGDMA, to allow resin infiltration into etched enamel at the level of the prism.[43,44] This characteristic leads to reduce interfacial porosity and, therefore, results in increased adhesion. In addition, it promotes greater bond strength through polymerization.[45]

HEMA is a small monomer that contains two functional groups, which are hydrophobic and hydrophilic components.[43] This structure allows HEMA-containing adhesive to be efficient in moisture-contaminated conditions as they produce a lower contact angle and an extension of the molecules that bond to the resin composite.[1]

PEGDMA is an cross-linked monomer and water-soluble enhancer of the adhesive.[43] PEGDMA can enhance the solubility and infiltration of Bis-GMA into the wet surface and can promote homogenous distribution of the hydrophobic component throughout the interface.[46]

TEGDMA has low molecular weight with relatively high hydrophilicity that contributes to a greater degree of curing. It demonstrates a superior viscosity and copolymerization behavior. The essential components of this substance are an ester of ethyleneglycol and the α,β-unsaturated methacrylic acid.[47]

Besides organic matrix monomers, silanes have been added into adhesives by some manufacturers. Silanes are recognized as efficient coupling agents, extensively used in composites and adhesive formulations.[48] Coupling agents are used to bond the filler particles to the resin matrix chemically to achieve the optimal properties of the adhesive. Moreover, silanes displace adsorbed water and provide a strong chemical bond between the oxide groups on the glass filler surface and the polymer molecules of the resin.[49]

Several moisture-resistant orthodontic adhesives have been commercially available worldwide. However, only Transbond™ PLUS color change adhesive (3M Unitek®), Beauty Ortho Bond® (Shofu®, Kyoto, Japan) and Assure® (Reliance® Orthodontic Products, Inc., Itasca, Illinois, USA) were selected for this study.

The chemical composition of each adhesive according to the manufacturer is shown in Table 1.

Table 1 The chemical composition of each adhesive according to the manufacturer.

Material	Manufacturer	Components	Composition	%
Transbond ™ XT	3M Unitek®	Etching gel: Primer: Paste:	35% Phosphoric acid Bisphenol A Diglycidyl Ether Dimethacrylate Triethylene Glycol Dimethacrylate 4-(Dimethylamino)-Benzeneethanol DL-Camphorquinone Hydroquinone Silane-treated quartz Bisphenol A Diglycidyl Ether Dimethacrylate Bisphenol A Bis(2-Hydroxyethyl Ether) Dimethacrylate Silane-treated silica Diphenyliodonium Hexafluorophosphate	 45-55 45-55 <0.5 <0.3 <0.03 70-80 10-20 5-10 <2 <0.2
Transbond ™ PLUS Color change	3M Unitek®	Self-etching primer: Paste:	Methacrylate Ester Derivative Water DL-Camphorquinone Dipotassium Hexafluorotitanate Silane-treated quartz Glass reacted with hydrolyzed silane Polyethylene glycol dimethacrylate Citric acid dimethacrylate oligomer Silane-treated silica Bisphenol A diglycidyl ether dimethacrylate 2,6-Di-Tert-Butyl-P-Cresol	75-85 15-25 <1.0 <0.2 35-45 35-45 5-15 1-10 <2 <2 <0.5
Beauty Ortho Bond®	Shofu®	Primer A: Primer B: SALIVATECT Paste:	Water, acetone, initiator Ethanol,phosphoric acid monomer,dye Bis-GMA ,TEGDMA, Glass powder, Silane coupling agent Bis-GMA ,TEGDMA, Glass powder, Silane coupling agent	 N/A
Assure®	Reliance® Orthodontic Products, Inc.	Etching gel: Assure® Primer: Paste:	37% Phosphoric acid Acetone Biphenyl Dimethacrylate Hydroxyethyl methacrylate Hydroxyethyl methacrylate Glass Frit Sodium Fluoride	 >40 >10 >10 8-30 60-99 1-5

Transbond™ PLUS color change adhesive has an initial pink color that expedites the removal of material excess. After photo-activation, it becomes transparent. The adhesive contains fluorosilicate glass as the fluoride source, in which the fluoride release rate drops to half of the initial level after one week and to one-third after four weeks.[50] According to the manufacturer, the adhesive is a moisture-resistant material, resulting from the addition of PEGDMA and hydrolyzed silane, and the reduction of Bis-GMA when compared to Transbond™ XT. Therefore, Transbond™ PLUS can be used under moisture-contaminated conditions with unchanged adhesiveness. The hydrophilic nature of the adhesive allows fluoride diffusion through the cured cross-linked matrix in an aqueous medium. The enamel surface should be specifically prepared for this adhesive with either 37% phosphoric acid and bonding agent or Transbond™ Plus Self-Etching Primer. The pH of Transbond™ Plus Self-Etching Primer works out at 0.4 and increases little within 10 minutes (pH 0.4-0.5).[22]

In previous study comparing color change adhesive systems (Transbond™ PLUS Color Change, Glengloo™, Blugloo™), the shear bond strength of Transbond™ PLUS Color Change adhesive under non-contaminated conditions was not significantly different from that of the others and also was within clinically acceptable limits.[51] Transbond™ PLUS Color Change adhesive, therefore, can be safely used in orthodontic practice since it yields acceptable bond strength (over 8 MPa).[18]

Even in saliva-contaminated conditions, Transbond™ PLUS Color Change adhesive still shows adequate bond strength. Romano and co-workers reported that saliva caused an insignificant decrease in bond strength with Transbond™ PLUS

Color Change adhesive, whereas a conventional total-etching system used with Transbond™ XT (control group) exhibited significantly reduced bond strength.[18] Another study under saliva-contaminated conditions, on the other hand, demonstrated a small increase in bond strength values with Transbond™ PLUS Color Change adhesive, but the increase was not statistically significant[29] (Table 2).

Beauty Ortho Bond® is a new light-cured adhesive system. It is categorized as a self-etching primer system limited to being used with its particular primers (Primer A and B) with a pH of 1.5 which never increases. The adhesive component is composed of TEGDMA, Bis-GMA, and a silane coupling agent. Surface pre-reacted glass ionomer (S-PRG) filler particles are added to ensure fluoride release that promotes re-mineralization of enamel and reduces bacterial colonization, leading to secondary caries prevention.

Since Beauty Ortho Bond® has just been introduced recently, little research has been conducted. Experimental studies of its shear bond strength compared to that of conventional etching systems under non-contaminated conditions have revealed that Beauty Ortho Bond® has significantly lower bond strength, but within clinically acceptable limits [21,52] (Table 2).

Table 2 Previous studies on shear bond strength with orthodontic stainless steel brackets regarding contamination.

Author	Teeth	Etching system	Bonding System	Agent	Duration	After Con.	Curing time	Storage °C, Days	Thermo-cycling	Mean	SD
Rix,*et al.*, 2001	H, M	37%Ph	Transbond™ XT		-		40 Sec	37°, 30	24 Hr	20.19	
										10.74	
			Assure®	H,Saliva	()	()				10.99	
Webster, *et al*, 2001	B,I	35%Ph	Transbond™ XT	-	10 Sec	5 Sec	50 Sec	37°,2	0	26.88	6.89
				A,Saliva						19.63	4.15
			Assure®	-						20.42	5.06
				A,Saliva						13.72	4.48
Cacciafesta, *et al.*,2003	B,I	37%Ph	Transbond™ XT	-			20 Sec	25°,1	0	11.95	6.20
				Water	()	()				6.85	2.23
				H,Saliva						4.65	1.34
		SEP		-						12.29	1.37
				Water	()	()				5.67	1.55
				H,Saliva						7.25	1.88
Rajagopal,*et al.*,2004	H, P	37%Ph	Transbond™ XT	-			40 Sec	()	0	9.54	3.86
				H,Saliva	()	Leaving surface moist				4.69	3.10
		SEP	Transbond™ PLUS	-						11.10	2.56
				H,Saliva	()	Leaving surface moist				10.79	2.43
Sfondrini,*et al.*,2004	B,I	37%Ph	Transbond™ XT	-			20 Sec	25°,1	0	8.27	1.65
				H,Blood	Not blown off					3.64	1.28
		SEP		-						8.57	2.09
				H,Blood	Not blown off					5.61	1.02
Campoy,*et al.*,2005	H, P	SEP	Transbond™ XT	-			20 Sec	37°,1	0	12.42	3.27
				H,Saliva	Not blown off					11.61	2.74
Romano,*et al.*,2005	H, P	37%Ph	Transbond™ XT	-			40 Sec	37°,1	0	24.60	5.20
		SEP	Transbond™ PLUS							17.50	4.10
				H,Saliva	()	Air stream				14.80	5.30
Kitayama,*et al.*,2007	B,I	SEP	Beauty Ortho Bond®	-			20 Sec	37°,1	0	20.30	4.70
									5000	18.80	4.10
		SEP	Transbond™ XT	-					0	17.60	4.00
									5000	16.40	3.30
Faltermeier, *et al.*,2007	H, M	20%Ph	Transbond™ XT	-			20 Sec	(),1	6000	8.71	1.37
				H,Saliva	15 Sec	()				3.42	0.78
				H,Blood						2.37	1.13
Turk,et al.,2007	H, P	SEP	Transbond™ XT	-			20 Sec	37°,1	0	17.61	4.04
				H,Saliva	Not blown off					10.94	2.05

Table 2 (continued)

Author	Teeth	Etching system	Bonding System	Agent	Duration	After Con.	Curing time	Storage °C, Days	Thermo-cycling	Mean	SD
					Contamination					SBS(MPa)	
*Oztoprak, et al.,*2007	B,I	37%Ph	Transbond™ XT	-			40 Sec	37°,3	0	15.28	1.96
				H,Saliva	()	()				3.79	2.20
				H,Blood						3.08	1.81
		SEP		-						13.76	2.76
				H,Saliva	()	()				13.80	3.96
				H,Blood						5.28	1.47
		37%Ph	Assure®	-						16.40	3.50
				H,Saliva	()	()				10.66	1.67
				H,Blood						6.83	2.88
*Iijima, et al.,*2008	H,M	35%Ph	Transbond™ XT	-			20 Sec	37°,1	0	9.15	1.65
				H,Saliva	Not blown off					1.47	0.93
		SEP	Transbond™ PLUS	-						9.74	1.54
				H,Saliva	Not blown off					7.74	2.35
		SEP	Beauty Ortho Bond®	-						6.47	0.97
				H,Saliva	Not blown off					7.62	2.24
*Paschos, et al.,*2008	H,M	35%Ph	Transbond™ XT	-			20 Sec	37°, 30	1300	13.20	1.60
				A,Saliva	10 Sec	Air-dried 5 Sec				11.10	2.30
		SEP	Transbond™ PLUS	-						10.60	1.60
				A,Saliva	10 Sec	Air-dried 5 Sec				11.80	1.20
Scougall Vilchis,*et al.,*2009	H,P	37%Ph	Transbond™ XT		()		30 Sec	37°,1	0	19.00	6.70
		SEP								16.60	7.30
		SEP	Beauty Ortho Bond®							10.10	3.70
*Vincente, et al.,*2009	B,I	SEP	Transbond™ XT	-			20 Sec	37°,1	0	8.15	4.22
				Water	()	()				6.80	2.91
				H,Saliva						7.50	3.21
			Transbond™ PLUS	-						6.93	3.34
				Water	()	()				6.14	2.40
				H,Saliva						7.78	4.45
*Endo, et al.,*2009	H,P	35%Ph	Transbond™ XT		()		10 Sec	37°,1	0	12.43	2.58
		SEP	Transbond™ PLUS							11.44	2.35
		SEP	Beauty Ortho Bond®							7.47	1.16
*Pseiner, et al.,*2010	B,I	37%Ph	Transbond™ XT	-			40 Sec	37°	0	21.60	5.30
		SEP	Transbond™ PLUS							19.90	4.30

Abbreviations:

B = bovine

H = human

I = incisor

PM = premolar

M = molar

Ph = phosphoric acid

SEP = self-etching primers

A = artificial

Hr = hour

( ) = not indicated

One investigation under saliva-contaminated conditions demonstrated significantly decreased shear bond strength with a conventional etching system which was clinically unacceptable.[2] On the other hand, Beauty Ortho Bond® showed a small increase in bond strength which was, however, not statistically significant. It should be noted that the study was conducted immediately after performing the bonding procedure. The specimens were stored in artificial saliva at 37°C for 24 hours, but not thermocycled, so the ageing of brackets was not allowed to occur[5] (Table 2).

Assure® is marketed as both moisture-resistance and fluoride-releasing. Assure® is a light-cured polyacid modified composite resin or compomer, which is developed to bring the advantages of glass ionomer cements (fluoride release, chemical adhesion, and chelation) to resin based adhesives. Assure® contains a bifunctional monomer, HEMA, which is able to react with the pendant methacrylate

groups of other monomers, as well as with the cations liberated by the glass particles. Nevertheless, the differences between Assure® and glass ionomer cement are the matrix formed mainly during the light-activation and the radical polymerization reaction of monomers.[53]

The results of previous studies of Assure® adhesive under saliva-contaminated conditions are controversial (Table 2). Some studies[4,54] reported that saliva caused a significant decrease in bond strength for Assure® adhesive as for the conventional total etching system used with Transbond™ XT (control group). Another study under saliva-contaminated conditions, on the other hand, demonstrated a small increase in bond strength values for Assure® adhesive, but the increase was not significant.[16]

Besides these clinical factors relating to bond failure, for an *in vitro* study, the bracket should be allowed to stay in a similar condition to being in the oral environment, such as in long-term water storage or thermocycling. Thermocycling is a widely used artificial aging technique, second to long-term water storage, which is the most commonly used method.[55] Thermoclycling is applied to simulate the human oral cavity with routine behaviors that allow temperature change, for example, drinking, eating and breathing, resulting in thermal stress. Thermal stresses can be pathogenic in two ways. Firstly, mechanical stresses induced by differential thermal changes can directly cause crack propagation through bonded interfaces. Secondly, the changing gap dimensions associated with gap volume changes which pump pathogenic oral fluids in and out of the gaps.[56]

The international Organization for Standardization (ISO) TR 11450 Standard (1994) indicates that a thermocycling regimen comprising of 500 cycles in water at 5-55°C is an appropriate artificial aging test. Some studies have followed this standard.

This number of cycles is, nevertheless, probably too few to achieve a realistic aging effect. Recently, various number of thermocycles, 500-6000 cycles, at 5-55°C have been used in orthodontic studies.[52]

Toshihiro and co-workers reported that thermocycling is a proper method for understanding the durability of orthodontic bracket-bonding materials.[52] In addition, another study found that thermal stresses significantly reduced the mean bond strength of the self-etching primer after 2000 thermal cycles.[57] Furthermore, study of self-etching primer used with Transbond™ XT and Beauty Ortho Bond®, demonstrated adequate shear bond strength in both groups even after two years of storage and thermocycling for 6000 cycles at 5-55°C.[52]