

---

[All ETDs from UAB](#)

[UAB Theses & Dissertations](#)

---

2015

## Bond Of Dual Cure Resin Cements To Enamel And Dentin In Self Cure Mode

Rashmi Radhakrishnan  
*University of Alabama at Birmingham*

Follow this and additional works at: <https://digitalcommons.library.uab.edu/etd-collection>

---

### Recommended Citation

Radhakrishnan, Rashmi, "Bond Of Dual Cure Resin Cements To Enamel And Dentin In Self Cure Mode" (2015). *All ETDs from UAB*. 2769.  
<https://digitalcommons.library.uab.edu/etd-collection/2769>

This content has been accepted for inclusion by an authorized administrator of the UAB Digital Commons, and is provided as a free open access item. All inquiries regarding this item or the UAB Digital Commons should be directed to the [UAB Libraries Office of Scholarly Communication](#).

BOND OF DUAL CURE RESIN CEMENTS TO ENAMEL AND DENTIN IN  
SELF-CURE MODE.

by

RASHMI RADHAKRISHNAN

DR. JOHN.O.BURGESS, COMMITTEE CHAIR  
DR. AMJAD JAVED  
DR. NATHANIEL.C.LAWSON  
DR. JACK.E.LEMONS  
DR. LANCE.C.RAMP

A THESIS

Submitted to the graduate faculty of The University of Alabama at Birmingham  
in partial fulfillment of the requirements for the degree of  
Master of Science

BIRMINGHAM, ALABAMA

2014

Copyright by  
Rashmi Radhakrishnan  
2015

BOND OF DUAL CURE RESIN CEMENTS TO ENAMEL AND DENTIN IN  
SELF-CURE MODE.

RASHMI RADHAKRISHNAN  
MASTER'S IN CLINICAL DENTISTRY

**ABSTRACT**

Dual cured resin cements are frequently used to cement all ceramic restorations. The increased thickness and /or opacity of restorations block light transmission. In these situations, where little or no light can be transmitted through the restorations, the polymerization of the cement is in the self-cure mode.

**Purpose:** This study measures the bond strength and mode of failure of dual cure resin cements to enamel and dentin in a self-cure mode using e.max rods.

**Methods:** 160 extracted human molars were ground to mid-coronal dentin (N=80) or superficial enamel (N=80) and finished to 320 grit disks. Four adhesive/cement combinations - Multilink Primer A & B/Multilink Automix (MA, Ivoclar), Panavia V5 Tooth Primer/Panavia V5 Paste (PanV5, Kuraray Noritake Dental), Optibond XTR/NX3 (NX3,Kerr) and Scotchbond Universal/RelyX Ultimate (RXU,3M); were applied to enamel and dentin using a self-etch, self-cure mode. A 1mm×1mm block of e.max (Ivoclar) was etched with 5% HF (20sec), rinsed, dried, silanated, coated with each cement and seated onto enamel or dentin. 100g of force was applied to the tooth/e.max assembly for 8 minutes. To more closely replicate the oral cavity, the experiment was repeated but specimens were fabricated by applying pressure to the e.max/tooth assembly for 10 minutes in an incubator (37°C). After 24hrs storage (at 37°C), samples were

debonded at 1mm/min crosshead speed in a universal testing device. A 2-way ANOVA and Tukey post hoc analysis was performed for each material ( $\alpha < 0.05$ ).

**Results:** Panavia V5 showed significantly higher bond strengths to dentin compared to RelyX Ultimate and NX3 at both room temperature and elevated temperature. RelyX Ultimate and NX3 showed a significant increase in bond strength to dentin at elevated temperature. Bond strengths of resin cements were significantly increased at elevated temperature compared to room temperature. **Conclusions:** Panavia V5 showed high bond strengths to both enamel and dentin in a self-cure mode. An increase in temperature significantly affects the polymerization of dual cure resin cements in the self-cure mode. Hence this study further emphasizes the importance of performing tests that can closely replicate clinical situations.

## **ACKNOWLEDGEMENTS**

I would like to express my deepest appreciation and thanks to my committee chair, Dr. John.O.Burgess for being a great teacher and mentor and guiding me through this project.

I would also like to thank the rest of my committee, Drs. Jack Lemons, Nathaniel Lawson, Lance Ramp and Amjad Javed for their time, immense support and participation in this project. Special thanks to Mr. Preston Beck for all the technical support required for this project.

I will be forever grateful to my beloved husband and daughter for their selfless and relentless support. Lastly, I would like to thank my parents whose prayers have helped me get to where I am today. Thank you all.

## TABLE OF CONTENTS

ABSTRACT .....	iii
ACKNOWLEDGEMENTS .....	v
TABLE OF CONTENTS .....	vi
LIST OF FIGURES .....	viii
LIST OF TABLES .....	ix
INTRODUCTION .....	1
Background .....	2
Light Transmission Through Indirect Restorations .....	2
Formulation Of Resin cements .....	3
Type Of Light Curing Unit .....	3
Dentin Bonding Systems .....	4
Resin Cements And Dentin Bonding Agents: Incompatibility .....	5
Testing Methodology .....	9
Purpose .....	9
Pilot Study .....	9
STUDY, HYPOTHESES AND AIMS .....	10
MATERIALS AND METHODS .....	11
Specimen Preparation (Enamel and Dentin) .....	11

e.max Rod Preparation.....	11
Cementation .....	12
RESULTS .....	14
Shear Bond Strength Test .....	14
Mode Of Failure.....	15
DISCUSSION .....	15
SUMMARY .....	19
CONCLUSIONS.....	19
REFERENCES .....	29



## LIST OF FIGURES

Figure	Page
1. Grinding of teeth.....	11
2. Polishing with abrasive disks.....	11
3. Sectioning of e.max cylinders.....	12
4. Polishing of e.max rods.....	12
5. Gilmore needle.....	13
6. Instron Universal Testing Machine.....	13
7. Shear bond strength of dual cure resin cements for enamel .....	27
8. Shear bond strength of dual cure resin cements for dentin.....	28

## LIST OF TABLES

Table	Page
1. Cements and adhesives used in this study .....	20
2. e.max Pretreatment .....	21
3. Application of adhesives for dual cure resin cements .....	22
4. Shear bond strength to enamel at room temperature .....	23
5. Shear bond strength to enamel at elevated temperature.....	24
6. Shear bond strength to dentin at room temperature .....	25
7. Shear bond strength to dentin at elevated temperature .....	26

## INTRODUCTION

Cementation is an important procedure that ensures the success and longevity of indirect restorations. Resin cements have become the material of choice in the cementation of all ceramic restorations; tooth colored inlays, onlays, veneers, and crowns. These resin luting agents have the ability to bond both to the tooth and the restoration and are also known to reduce microleakage, decrease post-operative sensitivivity, recurrent caries and marginal staining<sup>1</sup>.

Resin based cements are low viscosity composite materials formulated from Bis-GMA or urethane dimethacrylate resins with filler content between 30% and 66%<sup>2</sup>. They have been classified based on the mode of polymerization into chemically cured/self-cured, light cured and dual cured resin cements (ISO specification 4049 – 2009). Chemically cured resins polymerize by chemical reaction. These cements are especially useful in areas where light curing is difficult which include cementation of metallic restorations, endodontic posts and ceramic restorations with increased thickness, which limits the curing light from adequately polymerizing the cement<sup>3</sup>. However, these have limited working time and prolonged setting time. Light cured resin cements used commonly for cementation of veneers and inlays, have decreased curing depth with increasing thickness of the ceramic due to the attenuation of light<sup>4, 5</sup>. Dual cure resin cements are the most widely used resin cements due to their ability to polymerize by both the chemical reaction of the components as well as by light activation.

## *Background*

### *Light Transmission Through Indirect Restorations.*

A study by Chan and Boyer showed that the hardening of light activated resin cements is affected by thickness and shade of the porcelain<sup>6</sup>. With increasing thickness of the translucent material through which light passes, exposure time needs to be longer for hardening of light activated resins. Generally, the thicker the restoration or the darker its shade, the more critical is the intensity of the incident light to achieve optimal photopolymerization of the material<sup>7-9</sup>. The amount of light transmission through the ceramic restoration affects the potential for polymerization of resin cements<sup>4, 6, 10</sup>. Attenuation of light through composite resins and dentin limiting the polymerization of resin cement have also been well-documented<sup>11, 12</sup>. Further a study on the hardness of three dual cure resin cements reported that chemical curing did not completely harden the cement when light was attenuated by tooth and restoration<sup>13</sup>.

Optical behavior of the restorative material is another factor that affects the transmission of light through the restorative material, which in turn affects the polymerization of the resin cement<sup>14-16</sup>. When visible light reaches the restorative material, part of the light is transmitted through it, part is absorbed, and part is reflected on the surface. The level of opacity of a given material depends on its internal reflectance and transmittance. Opacity is the result of high light scattering with a very low fraction of the incident beam being transmitted through the material. In biphasic materials such as composites and ceramics, light scattering occurs at interfaces with different refraction indexes<sup>17</sup>. The optical behavior of indirect aesthetic materials also depends on their inorganic content, matrix

composition, and particle size as well as the presence of pores incorporated during the processing of the material<sup>17-19</sup>.

#### *Formulation Of Resin Cements*

In light activated resin cements, the initiation system is composed of tertiary amine and one or more photoinitiators such as camphoroquinone<sup>20</sup>. In dual cure systems, a catalyst paste containing a chemical activator (benzoyl peroxide) can be mixed with the light cured resin cement to increase free radical concentration even under insufficient light. When the two pastes are mixed together and exposed to light, free radicals are formed both by photo and chemical activation. It is expected that in areas where light is partially or totally attenuated, the free radicals formed by amine/benzoyl peroxide interaction would compensate for the lack of those that result from amine/camphoroquinone interaction<sup>21</sup>.

#### *Type Of Light Curing Unit*

Light – emitting diode (LED) and quartz – tungsten – halogen (QTH) curing units can provide irradiances from 300 to 1200mW/cm<sup>2</sup> with the former providing a much narrower spectral emission that better matches the absorption band of camphoroquinone<sup>22, 23</sup>. The effects of irradiance, light curing time and type of curing unit (QTH and LED) on the hardening of various resin cements have been determined. High intensity light curing and longer curing times increase cement microhardness. The use of LED units resulted in resin cement microhardness similar to that achieved with conventional QTH<sup>24</sup>.

### *Dentin Bonding Systems*

Enamel bonding introduced by Buonocore<sup>25</sup> was a major breakthrough in adhesive dentistry, which provided an alternative means of achieving adhesion. However, the same technique was not successful when tried on dentin since it is a different substrate compared to enamel. The wetness of dentin had to be compensated for to achieve optimal adhesion<sup>26</sup>. Etching of dentin with 37% phosphoric acid removes the smear layer and mineral phase exposing collagen fibrils. This permeable layer facilitates the infiltration of the resin monomers into the collagen fibril and dentinal tubules forming the resin – infiltrated zone called the hybrid layer. This phenomenon was described by Nakabayashi et.al<sup>27</sup>.

The introduction of hydrophilic agents improved bonding to dentin. This involves the use of an acid, a primer and a bonding agent and is called a three step total – etch bonding technique<sup>28, 29</sup>. However, with the three step total-etch technique, maintaining the wetness of dentin was an issue<sup>30, 31</sup>. The hybrid layer is the weakest link in the adhesive dentin interface<sup>32, 33</sup>. Incomplete resin infiltration within the demineralized intertubular matrix was demonstrated by Tay et.al, resulting in a weak, collagen-rich zone susceptible to hydrolysis and microleakage<sup>34</sup> and is known to be responsible for tooth sensitivity in clinical situations<sup>35</sup>. In addition, the three step total – etch bonding system is technique sensitive and has been known to affect the wetness of dentin. This issue persisted with the two bottle total – etch method developed to simplify the three step total – etch technique. The self – etch system was later developed to overcome this problem and also to reduce the clinician time constraints.

The self – etch systems were made available in two modes namely the two bottle and one bottle self – etch bonding systems. The two bottle self – etch system includes the etchant and primer in one bottle and the bonding agent in a separate bottle, while the one bottle system involves the etchant, primer and bonding agent all in one bottle<sup>36</sup>. The one bottle self – etch system uses a more acidic primer (pH<1) compared to the two bottle self – etch system (pH 1.9 – 2.4). The one bottle system is composed of both hydrophilic and hydrophobic monomers in a relatively high concentration of solvent. Water, as an ionization medium is also essential in this system for the self – etching activity to occur. However, when the solvent evaporates from the adhesive surface, monomer – solvent phase separation occurs resulting in water droplets in the adhesive after the polymerization of the adhesives<sup>37, 38</sup>. The integrity of the resin – dentin bond is maintained by hybrid layer formation. The main difference between the total – etch and self – etch bonding techniques is that the smear layer is removed in the former along with the demineralization of dentin, while the latter incorporates the smear layer into the hybrid layer. The quality of this hybrid layer is crucial and accounts for 40% of the bond strength<sup>39</sup>.

#### *Resin Cements And Dentin Bonding Agents: Incompatibility*

Resin cements have been classified based on the mode of polymerization as previously mentioned. Another system of classification of resin cements is based on the type of bonding system used in combination with the resin cement, which includes total-etch cements (use of phosphoric acid and adhesive to bond cement to tooth), self –etch cements (self –etching primer is applied to the tooth prior to cementation) and self –

adhesive cements that do not require pre-treatment of the tooth substrate and involves direct application of the cement to the tooth surface<sup>40</sup>.

Most of the resin luting agents available today is in the dual - cure initiating mode.

However, there has been growing concern regarding the incompatibility of chemical and dual – cure resin luting agents when used in conjunction with simplified adhesive systems<sup>41, 42</sup>.

The understanding of the chemical composition of the adhesive systems is essential to comprehend the incompatibility of the adhesive systems with dual – cured and chemical cured resin systems. Dental adhesives establish a bond to the substrates enamel and dentin and also to the overlying composite resin. Irrespective of the type of adhesive system (total – etch or self – etch) used, they all contain similar ingredients. Nevertheless, there is a difference in the proportional composition among the adhesive systems<sup>43</sup>. The adhesive systems consist of acrylic resin oligomers similar to those in composite restorative materials and luting agents, along with initiators, inhibitors, fillers and organic solvents. The resin oligomer is composed of a polymerizable group, a spacer molecule and a functional group. The polymerizable group reacts with the monomers of the adhesive and the restorative material by copolymerization and is hydrophobic. The functional group exhibits hydrophilic properties. The monomers in the adhesives and composite resin materials polymerize via free radical polymerization. Initiators added in these systems initiate the polymerization reaction. These initiators may be benzoyl peroxide/tertiary amine in the chemical cured composites or photo-initiators like camphoroquinone/tertiary amine in light cured composites. Dual – cured systems are comprised of both chemical and light cured initiator systems. Optimal copolymerization



between the adhesive and the lining composite is imperative for the formation of a successful adhesive bond. Incompatibility between the resin luting agents and adhesives may affect the copolymerization and result in failure of the restorations<sup>43</sup>.

Simplified or self – etch adhesive systems in general are somewhat acidic and have a hydrophilic layer susceptible to hydrolytic degradation. The acidic groups from the oxygen – inhibited layer of the adhesive compete with the peroxides for the aromatic tertiary amines of the overlying resin during the cementation of indirect restorations. This results in an acid – base reaction between the adhesive and the resin luting agent<sup>44</sup>. Tay et.al has documented the adverse interaction between single bottle adhesives and chemically cured composites<sup>45</sup>. The charge – transfer complexes formed between acidic monomers and aromatic tertiary amines prevent the latter from participating in the redox reaction and impede free radical generation resulting in incomplete polymerization. Light activated polymerization proceeds via generation of free radicals from the activation of a photoinitiator to its excited triplet stage. An intermediate excited complex, which releases free radicals on dissociation, is formed by the reduction of the activated photoinitiator by a less nucleophilic amine accelerator<sup>45</sup>. The incompatibility between acidic resin monomers and chemical cured composites was thought to be due to a slower rate of polymerization and should not occur when light cured resins are used. However, incompatibilities between light cured resins and self – etch or simplified adhesives have also been reported. In dual cured systems, which can polymerize by both the chemical and light cured modes, the polymerization of the cement is mostly in the chemical cured mode especially in clinical situations where the passage of light is limited<sup>46, 47</sup>. Therefore

regardless of the mode of polymerization, the concentration of amine has to be adjusted in self – etching systems<sup>48</sup>. The substitution of tertiary amines with photo – accelerators has not eliminated this incompatibility issue, which has led researchers to believe that there may be other reasons for these problems to occur.

Hydrolytic degeneration may be one of the reasons for this incompatibility<sup>42, 45</sup>. Resin degradation is related to water sorption within the hybrid layer. Water ab- and ad-sorption of simplified adhesive systems have been studied by many researchers<sup>49-51</sup>. They have reported that hydrophilic resins have higher water sorption than hydrophobic resins. This water sorption lowers the modulus of elasticity of resins and was thought to contribute to reduction in bond strength. Regardless of the bonding technique, water is drawn from dentinal tubules to the adhesive resin interface. This phenomenon has been described by Tay<sup>52</sup>. Water movement across the cured adhesive layer may occur in the regions of increased concentrations of dissolved inorganic ions; uncured, water soluble, hydrophilic resin monomers; and dissolved collagen/proteoglycan components within the air-inhibited layer of the adhesive. These water-soluble agents may lower the local water concentrations and thereby establish an osmotic gradient causing water to move from a region of low solute concentration (the dentinal tubules) to a region of higher solute concentration (the air inhibited layer of the adhesive uncured composite interface). This may also give rise to osmotic blistering. As this blistering was evident in the chemical-cured composites, the water permeation may be partially responsible for the ineffectiveness of simplified adhesives and chemical- cured resins.

### *Testing Methodology*

Clinical trials are the ultimate test for dental materials. However, it is difficult to differentiate the reasons for failure of a material due to the simultaneous impact of diverse variables on restorations within the oral cavity. Laboratory testing provides a narrow means of evaluation. It is possible to test one variable while keeping a few others constant<sup>53</sup>, though a direct correlation to the clinical situation may not be possible. Laboratory testing does provide a faster and more convenient alternative for screening dental materials, and bond-strength tests are often used to test adhesive systems. The rationale behind this methodology is that the stronger the adhesion between the tooth and biomaterial, the better it will resist stress imposed by resin polymerization and oral function. Many different types of bond strength tests exist, but the shear and microtensile are the most commonly reported.

### *Purpose*

The main purpose of this study was to evaluate the shear bond strength of three commercially available dual cured resin cements and a newly introduced non amine containing resin cement and also to measure the bond strength of these cements to both enamel and dentin using e.max rods in the self-etch and self cure mode.

### *Pilot Study*

A pilot study of the materials resulted in the failure of bond to dentin of two of the dual cure resin cements namely RelyX Ultimate and Nexus 3 used in combination with their respective bonding agents in the self-etch and self-cure mode. This information provided

a direct input into subsequent investigations.

## STUDY, HYPOTHESES AND AIMS

This study measured the shear bond strength of 4 dual cure resin cements to enamel and dentin using e.max rods in the self-etch and self-cure mode at room (25°C) and elevated temperatures (37°C).

### *Hypothesis 1: (null)*

There will be no difference in shear bond strength of e.max to either enamel or dentin among the 4 dual cure resin cements.

Specific Aim 1: The bond strength of e.max rods to superficial enamel and midcoronal dentin will be measured using 4 dual cure resin cements.

### *Hypothesis 2: (null)*

There will be no effect of temperature on the shear bond strengths of the 4 dual cure resin cements.

*Specific Aim 2:* The e.max rods will be bonded to the tooth substrates using the 4 dual cure resin cements and allowed to self-cure at room temperature (25°C) and at elevated temperature (37°C).

## MATERIALS AND METHODS

The four dual cure resin cements and their corresponding adhesive agents are listed in Table 1.

*Specimen Preparation (Enamel and Dentin):* 150 freshly extracted human teeth were ground to expose a flat bonding surface using a model trimmer shown in Figure 1. This flattened enamel (superficial) and dentin were ground with a series of abrasive SiC discs ending with 320 grit to establish a standardized surface shown in Figure 2. Teeth were abraded with the paper for 30 seconds, rotated 90 degrees and abraded with the disks for another 30 seconds to minimize scratches in the dentin and enamel. Prepared teeth were sonicated in water to remove finishing debris.



Figure 1: Grinding of teeth



Figure 2: Polishing with abrasive disks

*e.max Rod Preparation:* Rectangular rods of e.max were fabricated by milling blocks, sectioning (Isomet, Buehler Ltd, Lake Bluff, IL, USA) shown in Figure 3, into 3mm in height and a square bonding area of 1mm×1mm. These were then sintered according to

recommended protocol. To establish a uniform surface, each specimen was abraded with 320 grit abrasive discs for 15 seconds. All specimens will be subjected to ultrasonic cleaning in distilled water for 5 minutes. The polished surfaces of e.max were then acid etched with 5% HF acid gel. The gel was applied evenly over the bonding surface for 20 seconds with a microbrush. The surface was cleaned with water for 10 seconds. Each surface treatment agent was applied on the e. max surface following each cement's instruction as given in Table 2.



Figure 3: Sectioning of e.max cylinders



Figure 4: Polishing of e.max rods

*Cementation:* The adhesive agents corresponding to each dual cure resin cement was first applied to the tooth substrate either enamel or dentin according to the manufacturer's instructions which is given in Table 3. The e.max rods were then cemented to the prepared tooth with the cements listed in Table 1 according to the manufacturer's instructions using a Gilmore needle with a constant load of 110gms shown in Figure 5. Excess cement was removed using a small microbrush. 10 specimens of each cement/adhesive combination were self-cured at room temperature and the experiment was repeated by applying pressure to the e.max/tooth assembly for 10minutes in the incubator at 37°C for self curing of the resin cements at elevated temperature (37°C). All

samples (self-cured) were then stored in an incubator at 37°C in a zip lock bag with a wet paper towel for 24 hours. The specimens were then mounted on a steel fixture and subjected to shear loading to failure using a universal testing machine (Instron 5565, MA, USA), shown in Figure 6 at a crosshead speed of 1 mm/min 24 hours after fabrication.

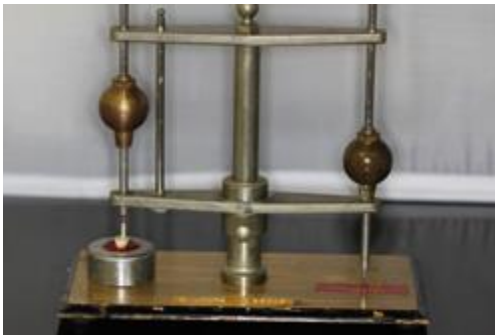


Figure 5: Gilmore needle



Figure 6: Instron Universal Testing Machine

## RESULTS

### *Shear Bond Strength Test*

The mean shear bond strength values of the four dual cure resin cements to enamel and dentin at room temperature (25°C) and elevated temperature (37°C) are shown in graphs in Figure 7 and Figure 8 respectively. The values of the bond strength tests for enamel and dentin at room temperature (25°C) and elevated temperature (37°C) are listed in Tables 4,5 and 6 and 7.

A qualitative examination of Figure 7 showed that Panavia V5 exhibited higher shear bond strengths to enamel compared to the three other dual cure resin cements. Figure 7 showed that Panavia V5 exhibited higher shear bond strengths to dentin compared to the other three dual cure resin cements. In addition the increase in bond strength of RelyX Ultimate and Nexus 3 at elevated temperature is also demonstrated. The significant differences were analyzed quantitatively by ANOVA and Tukey's post hoc tests.

The ANOVA -showed that there were significant differences in shear bond strength among materials at elevated temperature for dentin but not for enamel. Significant differences in shear bond strength among materials were also found at room temperature for dentin. At room temperature, the bond strength of Panavia V5 was significantly higher than that of RelyX Ultimate and Nexus 3 for enamel.

The Tukey's multiple pairwise comparisons revealed that Panavia V5 had the highest bond strength compared to RelyX Ultimate and Nexus 3 to enamel and also Panavia had



the highest bond strength to dentin compared to Multilink Automix, RelyX Ultimate and Nexus 3. There was a significant increase in bond strength with the increase in temperature for Nexus 3 and RelyX Ultimate. However, with enamel, the effect of elevated temperature was significant only for RelyX Ultimate.

### *Mode Of Failure*

Keyence Microscope images of the debonded surfaces of the enamel and dentin substrates at a magnification of 30X revealed that the predominant mode of failure (95%) was at the tooth adhesive interface. The remaining 5% constituted the specimens of RelyX Ultimate and Nexus 3 that failed to bond to dentin at room temperature.

## DISCUSSION

The results of the study revealed that Panavia V5, which is a dual cure resin cement, showed the highest bond strengths to both enamel and dentin in a self-etch and self-cure mode.

It was interesting to note that RelyX Ultimate and Nexus 3 failed to bond to dentin at room temperature (25°C) and that there was subsequent improvement in bond strength to dentin at elevated temperature. This shows that heat transfer resulting in the temperature change may accelerate the process of self-curing in these dual cure resin cements.

The shear bond strength of all the dual cure resin cements to enamel was higher than that

to dentin. This may be attributed to the increased mineralized content of enamel compared to dentin.

Several in vitro studies in the past have measured the bond strength of dual cure resin cements in different polymerization modes. A study by Braga, et al<sup>54</sup> on the shear bond strength of porcelain to dentin where conical ceramic specimens of porcelain were bonded to 60 human dentin surfaces using two dual cure resin cements and one chemically activated resin cements revealed that light activated dual cure resin cements showed higher bond strengths compared to the chemically activated resin cement. The specimens were debonded at time intervals of 10 minutes, 30 minutes, 90 minutes and 7 days. However, the chemical curing ability of dual cure cements was not tested in this study. This is of particular importance as in situations where the restorations are thick or opaque light may not be entirely transmitted through the restoration and the polymerization of the luting agent will depend heavily on its chemical curing ability.

A study by Stewart et.al<sup>55</sup> measured the bond strengths of three light polymerized, two dual polymerized and one auto polymerized resin cements. The cements were bonded to 480 ceramic discs following different preconditioning treatments and to 60 dentin surfaces following adhesive application in the total – etch mode. While two of the dual cure cements and the adhesive agents were light activated, the adhesive agent for Nexus, dual cure resin cement was not light activated but the cement was light polymerized.

This study revealed that bond strengths of cements were better with auto polymerized and light polymerized adhesives compared to dual polymerized adhesives. However, due to the inconsistencies in the curing mode of the adhesives and the cements, the bond strengths thus measured are not comparable.

The comparison of seven dual polymerizing resin cements in the chemically cured and light activated mode revealed that the bond strength were higher when dual polymerizing cements were light activated. The dual polymerizing cements were applied to 480 dentin surfaces following the application of the adhesives in a total etch mode and light cured<sup>56</sup>.

Holderegger et.al<sup>57</sup> compared the shear bond strength of 4 dual cure resin cements in the self - cure mode. 160 dentin surfaces were treated with two total etch, one self-etch cement system and one self-adhesive cement. All the dual cure resin cements were polymerized in the self-cure mode. The self- adhesive cement showed the lowest bond strength compared to the cements used in the total-etch and self – etch mode. In this study one of the total-etch cements was applied to the dentin surface after light curing of the adhesive.

The comparison of bond strength of three dual cure resin cements and one self-cure resin cement to 120 dentin surfaces showed that light activated dual cure resin cements had higher bond strengths compared to chemically cured resin cements<sup>58</sup>.

Most studies measuring the bond strength of dual cure resin cements used them mostly in the light activated mode. Very few studies have reported the bond strength of commercially available dual cure resin cements in the self cure mode and used along with self-etch adhesives.

In this thesis the bond strength of three commercially available dual cure resin cements, Multilink, RelyX Ultimate and Nexus 3 and one experimental dual cure resin cement, Panavia V5 were compared on enamel and dentin by testing the self-etch adhesives and allowing them to polymerize in the self- cure mode. The effect of temperature difference

on the bond strength of resin cements to dentin has been shown clearly demonstrated. A significant increase in bond strength of RelyX Ultimate and Nexus 3 to dentin was determined. This, also, further emphasizes the need to conduct experiments that more closely simulate oral conditions. In addition, the effect of chemistry on the bond strength of resin cements could also have been significant, in that the Panavia V5 with a non amine initiator showed the highest bond strength.

## SUMMARY

Studies in the past have shown that light attenuates as it passes through dentin, composite resination and porcelain<sup>6, 12</sup>. The thickness of a restoration, opacity and the type of light curing unit used can affect the transmittance of light. When the light that reaches a dual cure resin cement is insufficient this may lead to inadequate polymerization of the luting agent thereby affecting the bond of the restoration to the prepared tooth surface. This condition may compromise the retention and subsequent survival of a restoration. In addition, inadequate polymerization of the luting agent may affect the mechanical and physical properties of the cement thus leading to loss of marginal integrity and microleakage. Further, the strength of the restoration may be compromised and can result in bulk fracture and failure of the restoration. Hence, it is important to evaluate the chemical curing ability of dual cured resin cements. In addition, it is important to perform experiments closely replicating clinical situations.

## CONCLUSIONS

1. The shear bond strength of e.max to enamel and denting using the four dual cure resin cements was significantly different. *Hypothesis 1(null) was rejected.*
2. The effect of temperature on the bond strengths of the dual cure resin cements in the self-cure mode was significant for two dual cure resin cements namely RelyX Ultimate and Nexus 3. *Hypothesis 2 (null) partially was rejected.*

Table 1: Cements and adhesives used in this study.

<i>Adhesive/Cement</i>	<i>Manufacturer</i>	<i>Lot No.</i>	<i>Expiration Date</i>
Multilink Automix resin cement	Ivoclar Vivadent	T15098	2016-02
Multilink Primer A		T16980	2015-11
Multilink Primer B		T11725	2015-10
Monobond Plus – Primer		T07775	2016-02
RelyX Ultimate Resin Cement	3M ESPE	552721	2015-10
Scotchbond Universal NX3 Resin Cement	Kerr	550034	2016-04
		5106937	2016-03
		5139874	2016-05
		5130012	2016-03
Optibond XTR Primer		5181917	2016-04
Optibond XTR Adhesive		5175678	2016-04
Panavia V5	Kuraray Noritake Dental	131114-U	Expiration not mentioned
Panavia V5 Tooth Primer		131015-P	Expiration not mentioned
Clearfil Ceramic Primer Plus		570006	2016-04

Table 2: e.max Pretreatment

<i>e.max</i>	<i>Pretreatment</i>	<i>Cement</i>	
Etched with 5%	Monobond Plus	Multilink Automix	
Hydroflouric acid	Scotchbond	RelyX Ultimate	Air died
	Universal		
	Optibond XTR	Nexus 3	
	Primer		
	Clearfil Ceramic	Panavia V5	
	Primer		

Table 3: Application of adhesives for dual cure resin cements

Dual cure resin cement	Adhesive	Adhesive application to tooth substrate
Multilink Automix	Multilink Primer A	Mix both primers in 1:1 ratio and apply with agitation to the surface for 20 secs and air thin for 5 secs
	Multilink Primer B	
RelyX Ultimate	Scotchbond Universal	Apply adhesive with agitation to the surface for 20 secs and air thin for 5 secs
Nexus 3	Optibond XTR Primer	Apply primer to the surface with agitation for 20 secs and air thin for 5 secs followed light brushing application of adhesive for 15 secs
	Optibond XTR Adhesive	
Panavia V5	Panavia V5 Primer	Apply primer with agitation for 20 secs, air thin for 5secs



Table 4: Shear bond strength to enamel at room temperature

Sr.No	Multilink	Panavia V5	RelyX Ultimate	Nexus 3
1	10.78	21.14	7.28	3.42
2	12.64	33.75	8.32	12.84
3	14.00	19.27	6.12	11.47
4	18.36	17.65	4.54	9.66
5	13.70	15.08	1.90	5.85
6	17.88	12.59	9.91	2.47
7	12.15	11.43	7.54	4.91
8	13.63	15.43	8.75	6.07
9	7.38	25.61	11.45	5.74
10	23.87	8.34	5.64	11.00
Mean	14.32	18.03	7.15	7.34
Standard Deviation	4.4	7.4	2.7	3.6

Table 5: Shear bond strength to enamel at elevated temperature

Sr.No	Multilink	Panavia V5	RelyX Ultimate	Nexus 3
1	12.14	10.98	6.14	14.52
2	15.54	5.67	14.04	18.06
3	13.93	24.98	1.38	3.96
4	8.71	18.98	10.89	14.61
5	17.97	26.75	15.75	5.20
6	6.57	20.79	18.35	4.26
7	13.55	13.59	10.60	2.54
8	7.73	18.73	22.40	5.11
9	28	25.83	12.42	3.04
10	8.07	12.69	18.14	5.95
Mean	13.22	17.90	13.01	7.72
Standard Deviation	6.4	7.0	6.2	5.7

Table 6: Shear bond strength to dentin at room temperature.

Sr.No	Multilink	Panavia V5	RelyX Ultimate	Nexus 3
1	9.52	15.83	0.00	0.00
2	17.83	20.79	0.00	0.00
3	6.35	22.63	0.00	0.00
4	21.21	16.85	0.00	0.00
5	7.75	9.28	0.00	0.00
6	14.28	18.55	0.00	0.00
7	1.06	11.05	0.00	0.00
8	3.58	11.83	0.00	0.00
9	1.60	5.10	0.00	0.00
10	4.32	11.83	0.00	0.00
Mean	13.22	17.90	0.00	0.00
Standard Deviation	8.75	14.37	0.00	0.00

Table 7: Shear bond strength for dentin at elevated temperature

Sr.No	Multilink	Panavia V5	RelyX Ultimate	Nexus 3
1	4.94	26.65	5.63	1.65
2	20.39	22.08	9.42	6.32
3	17.43	13.69	1.64	2.12
4	3.63	22.30	4.56	4.35
5	12.12	15.21	4.24	4.38
6	1.50	22.46	0.80	2.57
7	2.8	11.6	2.3	0.9
8	5.10	6.74	0.88	3.57
9	7.48	29.22	6.70	1.30
10	7.69	20.86	4.02	2.38
Mean	8.31	19.08	4.02	2.96
Standard Deviation	6.37	7.04	2.92	1.68

Figure 7: Shear bond strength of dual cure resin cements for enamel

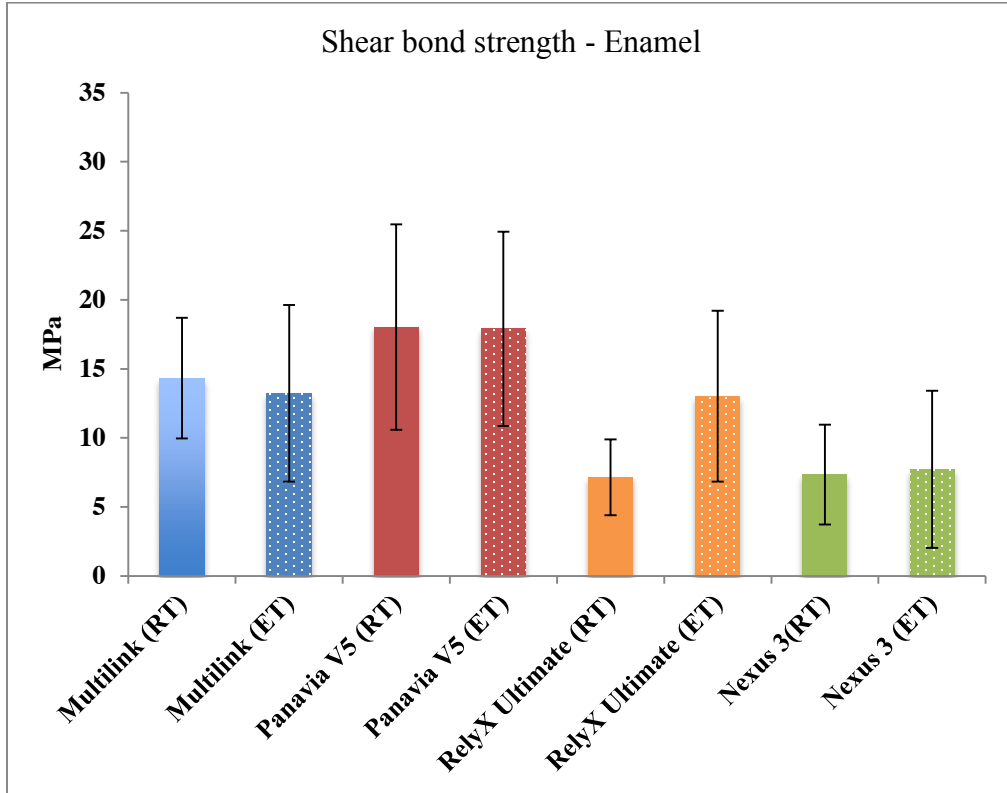
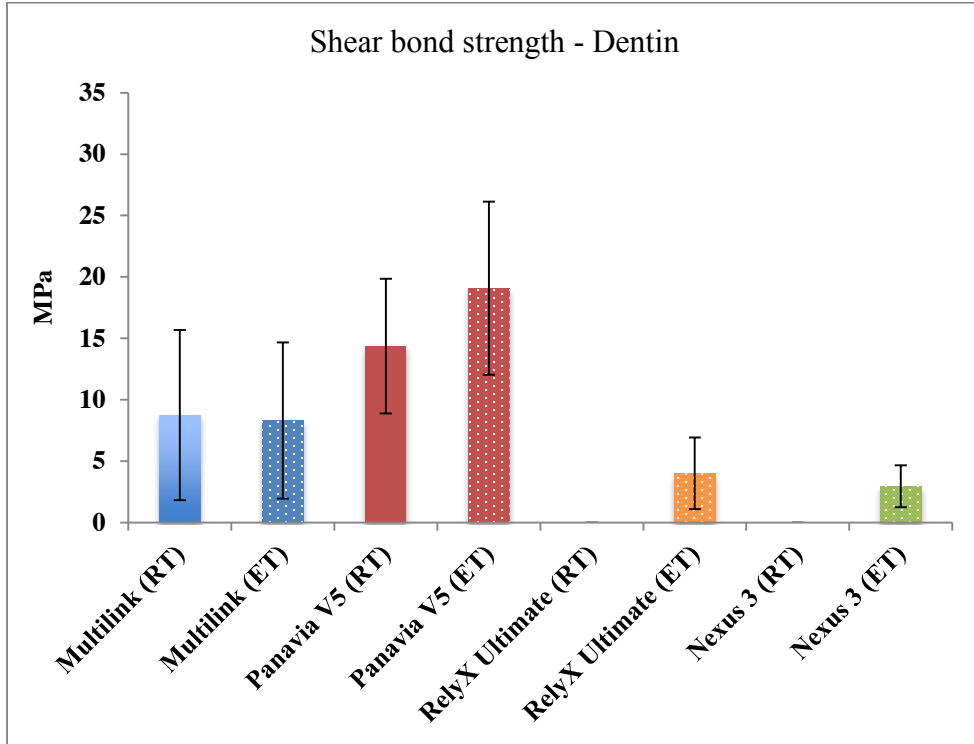


Figure 8: Shear bond strength of dual cure resin cements for dentin



## REFERENCES

1. Peumans M, Hikita K, De Munck J, Van Landuyt K, Poitevin A, Lambrechts P & Van Meerbeek B (2007) Bond durability of composite luting agents to ceramic when exposed to long-term thermocycling *Oper Dent* 32(4) 372-379.
2. R.G C (1993) *Restorative Dental Materials* CV Mosby 199-203.
3. Simon JF & Darnell LA (2012) Considerations for proper selection of dental cements *Compend Contin Educ Dent* 33(1) 28-30, 32, 34-25; quiz 36, 38.
4. O'Keefe KL, Pease PL & Herrin HK (1991) Variables affecting the spectral transmittance of light through porcelain veneer samples *J Prosthet Dent United States* 434-438.
5. Breeding LC, Dixon DL & Caughman WF (1991) The curing potential of light-activated composite resin luting agents *J Prosthet Dent* 65(4) 512-518.
6. Chan KC & Boyer DB (1989) Curing light-activated composite cement through porcelain *J Dent Res* 68(3) 476-480.
7. Myers ML, Caughman WF & Rueggeberg FA (1994) Effect of restoration composition, shade, and thickness on the cure of a photoactivated resin cement *J Prosthodont* 3(3) 149-157.
8. el-Badrawy WA & el-Mowafy OM (1995) Chemical versus dual curing of resin inlay cements *J Prosthet Dent* 73(6) 515-524.
9. Akgungor G, Akkayan B & Gaucher H (2005) Influence of ceramic thickness and polymerization mode of a resin luting agent on early bond strength and durability with a lithium disilicate-based ceramic system *J Prosthet Dent United States* 234-241.
10. Rueggeberg FA & Craig RG (1988) Correlation of parameters used to estimate monomer conversion in a light-cured composite *J Dent Res* 67(6) 932-937.
11. Council on Dental Materials IaE (1985) Visible light-cured composites and activating units. Council on Dental Materials, Instruments, and Equipment *J Am Dent Assoc* 110(1) 100-102.
12. Chan KC & Boyer DB (1985) Curing light-activated composite resins through dentin *J Prosthet Dent* 54(5) 643-645.
13. Hasegawa EA, Boyer DB & Chan DC (1991) Hardening of dual-cured cements under composite resin inlays *J Prosthet Dent United States* 187-192.
14. Cardash HS, Baharav H, Pilo R & Ben-Amar A (1993) The effect of porcelain color on the hardness of luting composite resin cement *J Prosthet Dent United States* 620-623.
15. Rasetto FH, Driscoll CF, Prestipino V, Masri R & von Fraunhofer JA (2004) Light transmission through all-ceramic dental materials: a pilot study *J Prosthet Dent United States* 441-446.
16. Soares CJ, da Silva NR & Fonseca RB (2006) Influence of the feldspathic ceramic thickness and shade on the microhardness of dual resin cement *Oper Dent* 31(3) 384-389.
17. William D. Callister DGR (2013) *Materials Science and Engineering: An Introduction*, 9th Edition.

18. Heffernan MJ, Aquilino SA, Diaz-Arnold AM, Haselton DR, Stanford CM & Vargas MA (2002) Relative translucency of six all-ceramic systems. Part II: core and veneer materials *J Prosthet Dent United States* 10-15.
19. Heffernan MJ, Aquilino SA, Diaz-Arnold AM, Haselton DR, Stanford CM & Vargas MA (2002) Relative translucency of six all-ceramic systems. Part I: core materials *J Prosthet Dent United States* 4-9.
20. Neumann MG, Schmitt CC, Ferreira GC & Correa IC (2006) The initiating radical yields and the efficiency of polymerization for various dental photoinitiators excited by different light curing units *Dent Mater England* 576-584.
21. Hofmann N, Papsthart G, Hugo B & Klaiber B (2001) Comparison of photo-activation versus chemical or dual-curing of resin-based luting cements regarding flexural strength, modulus and surface hardness *J Oral Rehabil England* 1022-1028.
22. Price RB, Felix CA & Andreou P (2005) Evaluation of a dual peak third generation LED curing light *Compend Contin Educ Dent* 26(5) 331-332, 334, 336-338 passim; quiz 348.
23. Santos GB, Medeiros IS, Fellows CE, Muench A & Braga RR (2007) Composite depth of cure obtained with QTH and LED units assessed by microhardness and micro-Raman spectroscopy *Oper Dent* 32(1) 79-83.
24. Santos GC, Jr., El-Mowafy O, Rubo JH & Santos MJ (2004) Hardening of dual-cure resin cements and a resin composite restorative cured with QTH and LED curing units *J Can Dent Assoc* 70(5) 323-328.
25. Buonocore MG (1955) A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces *J Dent Res* 34(6) 849-853.
26. Pashley DH (1989) Dentin: a dynamic substrate--a review *Scanning Microsc* 3(1) 161-174; discussion 174-166.
27. Nakabayashi N, Kojima K & Masuhara E (1982) The promotion of adhesion by the infiltration of monomers into tooth substrates *J Biomed Mater Res* 16(3) 265-273.
28. Nakabayashi N (1992) The hybrid layer: a resin-dentin composite *Proc Finn Dent Soc* 88 Suppl 1 321-329.
29. Nakabayashi N (1992) Adhesive bonding with 4-META *Oper Dent Suppl* 5 125-130.
30. Kanca J, 3rd (1992) Resin bonding to wet substrate. 1. Bonding to dentin *Quintessence Int* 23(1) 39-41.
31. Kanca J, 3rd (1992) Improving bond strength through acid etching of dentin and bonding to wet dentin surfaces *J Am Dent Assoc* 123(9) 35-43.
32. Wang T & Nakabayashi N (1991) Effect of 2-(methacryloxy)ethyl phenyl hydrogen phosphate on adhesion to dentin *J Dent Res* 70(1) 59-66.
33. Burrow MF, Tagami J & Hosoda H (1993) The long term durability of bond strengths to dentin *Bull Tokyo Med Dent Univ* 40(4) 173-191.
34. Tay FR, Gwinnett AJ, Pang KM & Wei SH (1996) Resin permeation into acid-conditioned, moist, and dry dentin: a paradigm using water-free adhesive primers *J Dent Res* 75(4) 1034-1044.



35. Perdigao J, Geraldeli S & Hodges JS (2003) Total-etch versus self-etch adhesive: effect on postoperative sensitivity *J Am Dent Assoc* 134(12) 1621-1629.
36. Ernst CP (2006) Options for dentin bonding *J Esthet Restor Dent* 18(2) 61-67.
37. Van Landuyt KL, De Munck J, Snauwaert J, Coutinho E, Poitevin A, Yoshida Y, Inoue S, Peumans M, Suzuki K, Lambrechts P & Van Meerbeek B (2005) Monomer-solvent phase separation in one-step self-etch adhesives *J Dent Res* 84(2) 183-188.
38. Tay FR & Pashley DH (2003) Have dentin adhesives become too hydrophilic? *J Can Dent Assoc* 69(11) 726-731.
39. Pashley DH & Carvalho RM (1997) Dentine permeability and dentine adhesion *J Dent* 25(5) 355-372.
40. Burgess JO, Ghuman T & Cakir D (2010) Self-adhesive resin cements *J Esthet Restor Dent* 22(6) 412-419.
41. Tay FR, Suh BI, Pashley DH, Prati C, Chuang SF & Li F (2003) Factors contributing to the incompatibility between simplified-step adhesives and self-cured or dual-cured composites. Part II. Single-bottle, total-etch adhesive *J Adhes Dent* 5(2) 91-105.
42. Suh BI, Feng L, Pashley DH & Tay FR (2003) Factors contributing to the incompatibility between simplified-step adhesives and chemically-cured or dual-cured composites. Part III. Effect of acidic resin monomers *J Adhes Dent* 5(4) 267-282.
43. Van Landuyt KL, Snauwaert J, De Munck J, Peumans M, Yoshida Y, Poitevin A, Coutinho E, Suzuki K, Lambrechts P & Van Meerbeek B (2007) Systematic review of the chemical composition of contemporary dental adhesives *Biomaterials* 28(26) 3757-3785.
44. Pegoraro TA, da Silva NR & Carvalho RM (2007) Cements for use in esthetic dentistry *Dent Clin North Am* 51(2) 453-471, x.
45. Tay FR, Pashley DH, Suh B, Carvalho R & Miller M (2004) Single-step, self-etch adhesives behave as permeable membranes after polymerization. Part I. Bond strength and morphologic evidence *Am J Dent* 17(4) 271-278.
46. Cekic-Nagas I, Egilmez F & Ergun G (2012) Comparison of light transmittance in different thicknesses of zirconia under various light curing units *J Adv Prosthodont* 4(2) 93-96.
47. Kim MJ, Kim KH, Kim YK & Kwon TY (2013) Degree of conversion of two dual-cured resin cements light-irradiated through zirconia ceramic disks *J Adv Prosthodont* 5(4) 464-470.
48. Moszner N, Salz U & Zimmermann J (2005) Chemical aspects of self-etching enamel-dentin adhesives: a systematic review *Dent Mater* 21(10) 895-910.
49. Burrow MF, Satoh M & Tagami J (1996) Dentin bond durability after three years using a dentin bonding agent with and without priming *Dent Mater* 12(5) 302-307.
50. Ito S, Hashimoto M, Wadgaonkar B, Svizero N, Carvalho RM, Yiu C, Rueggeberg FA, Foulger S, Saito T, Nishitani Y, Yoshiyama M, Tay FR & Pashley DH (2005) Effects of resin hydrophilicity on water sorption and changes in modulus of elasticity *Biomaterials* 26(33) 6449-6459.

51. Malacarne J, Carvalho RM, de Goes MF, Svizero N, Pashley DH, Tay FR, Yiu CK & Carrilho MR (2006) Water sorption/solubility of dental adhesive resins *Dent Mater* 22(10) 973-980.
52. Tay FR & Pashley DH (2003) Water treeing--a potential mechanism for degradation of dentin adhesives *Am J Dent* 16(1) 6-12.
53. Van Meerbeek B, Perdigao J, Lambrechts P & Vanherle G (1998) The clinical performance of adhesives *J Dent* 26(1) 1-20.
54. Braga RR, Ballester RY & Carrilho MR (1999) Pilot study on the early shear strength of porcelain-dentin bonding using dual-cure cements *J Prosthet Dent* 81(3) 285-289.
55. Stewart GP, Jain P & Hodges J (2002) Shear bond strength of resin cements to both ceramic and dentin *J Prosthet Dent* 88(3) 277-284.
56. Piwowarczyk A, Bender R, Ottl P & Lauer HC (2007) Long-term bond between dual-polymerizing cementing agents and human hard dental tissue *Dent Mater* 23(2) 211-217.
57. Holderegger C, Sailer I, Schuhmacher C, Schlapfer R, Hammerle C & Fischer J (2008) Shear bond strength of resin cements to human dentin *Dent Mater* 24(7) 944-950.
58. Altintas S, Eldeniz AU & Usumez A (2008) Shear bond strength of four resin cements used to lute ceramic core material to human dentin *J Prosthodont* 17(8) 634-640.