EVALUATION OF SHEAR BOND STRENGTH OF FOUR COMMERCIALLY AVAILABLE RESIN CEMENTS -IN VITRO STUDY

Dissertation submitted to



TAMIL NADU DR.M.G.R. MEDICAL UNIVERSITY

In the partial fulfillment of the requirement for the degree of

MASTER OF DENTAL SURGERY (PART II – BRANCH I) PROSTHODONTICS AND CROWN & BRIDGE APRIL 2011

CERTIFICATE

This is to certify that this dissertation titled "Evaluation Of Shear Bond strength of four commercially available resin cements-In vitro study" is a bonafide record of work done by Dr. PIYUSH JAVIYA under my guidance during his postgraduate period between 2008-2011. This Dissertation is submitted to TAMILNADU Dr. M.G.R. MEDICAL UNIVERSITY, in Partial fulfilment of requirements for the Degree of Master of Dental Surgery in Prosthodontics and Crown & Bridge (Branch I). It has not been submitted (partial or full) for the award of any other degree or diploma.

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DECLARATION

I, Dr. PIYUSH JAVIYA,, do hereby declare that the dissertation titled "Evaluation Of Shear Bond strength of four commercially available resin cements-In vitro study" was done in the Department Of Prosthodontics, TamilNadu Government Dental College & Hospital, Chennai 600 003. I have utilized the facilities provided in the Government Dental College for the study in partial fulfilment of the requirements for the degree of Master of Dental Surgery in the speciality of Prosthodontics and Crown & Bridge (Branch I) during the course period 2008-2011 under the conceptualization and guidance of my dissertation guide, Dr. SABARIGIRINATHAN, MDS.

I declare that no part of the dissertation will be utilized for gaining financial assistance for research or other promotions without obtaining prior permission from the TamilNadu Government Dental College & Hospital.

I also declare that no part of this work will be published either in the print or electronic media except with those who have been actively involved in this dissertation work and I firmly affirm that the right to preserve or publish this work rests solely with the prior permission of the Principal, TamilNadu Government Dental College & Hospital, Chennai 600 003, but with the vested right that I shall be cited as the author(s).

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ACKNOWLEDGEMENT

I consider it my utmost privilege and honour to express my most sincere and heartfelt gratitude to my esteemed Chief *Dr*. *C.THULASINGAM, M.D.S.*, Professor and Head, Department of Prosthodontics, Tamilnadu Government Dental College and Hospital for his wholehearted support, guidance, help, encouragement and never ending patience without which this study would not have ever been possible and also for constant inspiration throughout the period of my post graduate course.

My sincere thanks to *Dr. K.S.G.A. NASSER*, M.D.S., Principal, Tamil Nadu Government Dental College and Hospital, for his kind help, constant inspiration, encouragement and for permitting me to use the facilities in the institution.

I am extremely thankful to my guide *Dr. C .SABARIGIRINATHAN* **M.D.S.**, Associate Professor, Department of Prosthodontics, Tamil Nadu Government Dental College and Hospital for the invaluable suggestions and support that he has rendered at various stages of my dissertation. Without his help this dissertation would not have come out in a befitting manner. I would like to express my thanks to *Dr. A. MEENAKSHI*, M.D.S., Additional Professor, Department of Prosthodontics, Tamil Nadu Government Dental College and Hospital, for all the inspiration and guidance she has provided throughout my post graduation.

I am thankful to my Assistant Professors, *DR.K.VINAYAGAVEL M.D.S.*, *DR. RUPKUMAR*, *M.D.S. DR. G SRIRAMPRABHU M.D.S*, *DR. T. JEYANTHIKUMARI M.D.S. DR. G.GOMATHI M.D.S.*, *DR K. RAMKUMAR M.D.S*, *DR.HARISHNATH M.D.S.*, *DR. M. KANMANI M.D.S.*, for guiding and helping me at different stages of this study.

I thank *Dr. RAVANAN*, Reader, Department of Statistics, Presidency College, Chennai for helping me with the statistical analysis for this study.

I am much thankful to my co-postgraduates for their timely help, constant support and encouragement.

I am highly indebted to my family and friends for their blessings, love, motivation, encouragement and support.

I thank Almighty God for all the blessings; He has showered upon us for the completion of this venture.

INTRODUCTION

Dentists have searched for ideal restorative material for many years, although direct restorative materials such as amalgam, cements and composites have been used with reasonable good success during past several decades, but they are not ideal for large restorations or for fixed partial dentures¹.

When a restoration is placed in aesthetic zone, the surface quality and aesthetic potential over a period of time becomes very crucial to provide a life like restoration to the patients.

The end of twentieth century saw vast development in all-ceramic dental restorations because of increased popularity of all-ceramic materials².

Dental ceramics are attractive dental restorations, because of their biocompatibility, long term color stability, wear-resistance, and their ability to be formed into desired shapes¹.

Dental restorations using all-ceramic materials in association with adhesive cements have become popular in the last decade, primarily because of esthetic properties such as translucence, fluorescence, and opalescence that better simulate the appearance of natural dentition³.

The cementation process is vital for the clinical success of all-ceramic restorations. It has been purported that some all-ceramic restorations may be cemented with zinc phosphate, glass ionomer, or resin composite cements. Therefore, the success of the cementation process may depend on the composition of the ceramic material. When zinc phosphate or glass- ionomer cements are used, adequate retention form of the preparation is necessary. When this is compromised, adhesive luting systems are recommended. The bond of the resin luting agent to the tooth structure is enhanced by acid etching the tooth structure and by the use of a dental adhesive³.

The applications of dual-polymerizing resin cements for all-ceramic restorations have considerably increased due to the ability of these cements to polymerize completely and their greater resistance to occlusal loading. Since the use of all ceramic restorations requires considerable support from the underlying composite resin cement and dentin

for a successful clinical outcome, the luting agent should have high bond strength, not only to the ceramic surface, but also to the tooth surface⁴.

The long-term success of resin bonded all-ceramic restorations depends in part on a durable bond being created between the hard tissues of the tooth and the adhesive cement⁴. A durable bond between the adhesive cement and the restoration is also critical, throughout the lifetime of a

restoration. There is agreement that a stable bond increases both the retention and the fracture resistance of the abutment and the restoration and that it reduces the incidence of micro leakage⁵.

The occlusal forces applied to a restoration are complex and made up of a combination of forces such as shear, tension, compression, and flexure^{6,7}. Accordingly, it follows that no single test can satisfactorily predict the intraoral behavior of an adhesive system⁸. The tests most widely used to examine the bond strength of composite resin to dentin are shear and tensile tests.

The various in-vitro models do not allow perfect imitation of clinical conditions. However, it is permissible to make a relative internal comparison of in-vitro measurements obtained under identical conditions in order to identify one among several materials for a specific purpose⁹. Despite The standard ISO/TS 11405:2003 "Dental Materials – Testing of adhesion to tooth", the direct comparison of several studies with regard to their assessment of dentin bonding is not always possible, as their results may be influenced by additional parameters not covered by that ISO standard.

In addition to the specific in vitro examination parameters, the chemical composition of the selected cementing agent and the related adhesive system influence dentin bonding. A great number of studies on the bond strengths between adhesives per se and the hard tissues of the tooth have been published^{10,11,12}. By contrast, the bond strength between various commercially available cementing agents in combination with an adhesive and the hard tissue of the tooth has been addressed by only a handful of authors.

So an attempt was made to compare and evaluate shear bond strength of four commercially available resin cements in combination with an adhesive and the hard tissue of the tooth.

AIM AND OBJECTIVES OF THE STUDY:

The **aim** oif this study was to compare the shear bond strength of four commercially available resin cements with their respective bonding system to human teeth.

The **objectives** of this in vitro study are:

- 1. To compare the shear bond strength of four commercially available resin cements with their respective bonding system to human teeth.
- 2. To conduct failure mode analysis of resin cements using microscopy.

THE NULL / WORKING HYPOTHESIS:

(1) Differences exist in long-term durability to human dentin between the cementing agents with their respective bonding system.

(2) Simplifying the application procedures of the corresponding adhesives following either three step total-etch, two step total-etch, one-step self-etch, or no use of adhesives,

affect the effectiveness of the bond to human dentin.

REVIEW OF LITERATURE

J.J. LINDEN et al (1991)¹³ determine the effects of porcelain opacity, chemical catalyst, and exposure time on polymerization of light-activated resin-composite cements. Samples of microfill and hybrid composites, with and without catalyst (i.e., dual-cure and visible-light-activated), were polymerized by exposure to visible light through porcelain discs of different opacities. Micro hardness testing (KHN) was used to compare degree of cure for each material at various exposure times. Porcelain opacity did not significantly affect hardness. However, the results indicated that a chemical catalyst and prolonged curing times might be essential for clinical success.

A. DELLA BONA AND R. VAN NOORT (1995)¹⁴ concluded that the tensile bond strength test is more appropriate for evaluating the adhesive capabilities of resin composites to ceramics. As Results from the shear bond strength tests and FEA showed that this particular test has as its inherent feature the measurement of the strength of the base material rather than the strength of the adhesive interface.

MATTHIAS KERN, VAN P. THOMPSON, (1995)¹⁵ evaluated the durability of alternative methods of adhesive bonding to In-Ceram ceramic.

Sandblasting alone or additional use of a silane did not result in a durable bond of a conventional BIS-GMA composite resin to In-Ceram ceramic. A delayed degradation in bond strength was recorded for the combination of thermal silica coating and a conventional BIS-GMA composite resin; no reduction was found after 30 days, but there was a pronounced decrease after 150 days. This degradation indicated that extended storage in a wet environment was needed in laboratory tests.

K. YOSHIDA AND M. ATSUTA (1997)¹⁶ evaluate the durability and shear bond strengths of the different combinations of two adhesive primers and three resin cements to two types of noble metal alloys(Silverpalladium-copper-gold and type IV gold alloys). They concluded that application of Metal Primer was effective for improving the shear bond strengths between each of the three resin cements and both noble metal alloys compared with nonprimed specimens.

PAULO E.C. CARDOSO, et al (1998)⁸ determines bond strength between dentin and three adhesive systems, by means of micro-tensile, shear and tensile tests. They showed one-bottle adhesive system obtained higher bond strength values than the self-etching adhesive upon shear and tensile strength tests.

KOHJI KAMADA et al (1998)⁷ evaluated the effect of various ceramic surface treatments on the shear bond strengths four resin luting agents (Super-Bond C&B, Panavia 21, Clapearl, and Vita Cerec Duo Cement) to Cerec 2 ceramic material. When the ceramic material was treated with the silane coupler or the silane coupling agent after etching with phosphoric acid gel, no significant differences in bond strength were noted between water storage and 20,000 thermal cycles for any of the four resin luting agents. They concluded that combined surface treatment of etching with phosphoric acid and application of silane coupling agent provides the highest bond strengths of resin luting agents to Cerec 2 ceramic material after thermal cycling.

JEFFREY C. CHANG et al (1998)¹⁷ compared the tensile bond strengths between Dicor castable ceramics and enamel of four dual-cure cements: Twinlook, Optec Dual-Cure Luting Cement, Clearfil CR Inlay, and Dual Cement. They concluded All four dual-cured cements formed strong bonds between enamel and Dicor cement, ranging from 14.90 MPa to 18.35 MPa, and there was no statistically significant difference.

R. R. BRAGA et al (1999)¹⁸ evaluated the early shear strength of bonding between porcelain and dentin, using dual-cure cements (Porcelite and Dual) and chemically activated cement was also tested (C&B luting

composite). They concluded both dual-cure cements tested presented similar results. The bond strength of dual-cure cements to dentin was higher at all time intervals than that obtained for chemically activated material. The high values for the coefficient of variation confirmed the technique-sensitive nature of the porcelain/dentin bonding procedure. Although dual-cure cements reach higher bonding strength values faster than the chemically activated material, it is not recommended to stress the bonding until 90 minutes after cementation, because the strength at that time is much lower than the maximum.

R.R. BRAGA, R.Y. BALLESTER, M. DARONCH (2000)¹⁹ evaluated the extrusion shear strength of the bond between feldsphatic porcelain and bovine dentin at different time intervals, using three adhesive systems based on dual-cure cements and one based on self-cure cement. The adhesive systems evaluated included: C&B/One-Step, Enforce/Prime&Bond NT Dual-Cure, RelyX ARC/Single Bond and Variolink II/Syntac SC. They proved that High characteristic strengths were observed after 15 min when dual-cure cements were used. In general, the values found at 24 h or 7 days were higher than at 15 min. However, there was always a considerable probability of bonding failure at low stress levels for all the systems tested.

Y. KITASAKO et al (2000)²⁰ determined the influence of storage solution on the bond durability of three resin cements (Panavia 21, Kuraray Co.; BISTITE, Tokuyama Co; MASA Bond, Sun Medical Co.) to bovine dentin over the period of 1 year. Four storage environments were studied as follows: water changed every day for 1 year; water unchanged for 1 year; Phosphate Buffered Saline (PBS) changed every week over 1 year; PBS unchanged for 1 year. Ten teeth were also tested for each material at 1 day as a control. There was no statistical difference in the mean bond strengths between the water and PBS storage solutions in All cements, the results for the shear bond strengths in the changed storage solution groups were significantly lower than those where the storage solution remained unchanged. There were statistical differences between the 1 day results and the changed water groups among all cements. Thus concluded that storage condition influenced the long-term durability of dentin bonding with resin cements.

Y. KITASAKO et al(2001)²¹ evaluate the bond durability of three resin cements viz. Panavia 21, BISTITE resin cement, and MASA Bond (experimental resin cement) bonded to bovine dentine over a period of 3 years at 1 day, 6 months, 1 and 3 years after cementation of a composite rod. Panavia 21 and BISTITE strengths were significantly lower (P<0.05) at all times compared with MASA Bond, and 1 day strengths for all three materials were significantly higher (P<0.05) than 3 year strengths. And concluded that the type of resin cement seemed to have an influence on the long-term durability of bonding to dentine.

GREGORY P. STEWART, et al (2002)⁶ evaluated immediate and 6 month shear bond strengths between a feldspathic ceramic and 4 different resin cements(Nexus, Panavia 21, RelyX ARC, and Calibra) with the use of 6 different surface-conditioning treatment(sanding with 600-grit silicon carbide paper, micro etching with aluminum oxide, sanding followed by silane application, micro etching followed by silane application, hydrofluoric acid– etching, and hydrofluoric acid– etching followed by silane application). They conclude within the limitations of study, hydrofluoric acid– etching followed by silane application produced the best bonds at 24 hours and 6 months with all 4 cements. Auto- and lightpolymerized adhesives were associated with higher bond strengths to dentin than dual-polymerized adhesives.

R. JANDA et al (2003)²² evaluated a new surface treatment method to obtain good bond strength between a luting composite and several ceramics. The surfaces of Empress II, InCeram-Alumina, InCeram-Zirconia and Frialit (ZrO2) were ground under water-cooling with 400 grit grinding

paper, afterwards polished with 800 grit and air-dried. After the flame treatment, a methacryl silane was applied followed by a luting composite. Prior to measuring shear bond strength, the specimens were thermo cycled 5000 times in a water-bath between 5 and 55 ^oC. Shear bond strength measurements indicated that the optimal treatment time was 5 s/cm² and concluded The PyrosilPen-Technology is an easy and effective method for surface-treating silicate, aluminum oxide and zirconium oxide ceramics to obtain good bonding to luting composites.

CRISTIANE SOARES MOTA et al (2003)²³ evaluate the tensile bond strength of 4 resin luting agents (Resin Cement, Rely X ARC, Nexus, and Enforce) to bovine enamel and dentin. After 7 days of storage in distilled water at 37°C, specimens were subjected to tensile forces in a universal testing machine at a crosshead speed of 0.5 mm/min until fracture. The bond strengths obtained for Resin Cement, Rely X ARC, Nexus, and Enforce were statistically the same for enamel. For dentin, bond strengths for Rely X ARC, Resin Cement, and Enforce were significantly higher than for Nexus. Significantly higher bond strengths were also observed for enamel than dentin.

GILBERTO ANTONIO BORGES et al (2003)²⁴ assess the surface topography of 6 different ceramics (IPS Empress, IPS Empress 2, In-Ceram

Alumina, In-Ceram Zirconia, and Procera) after treatment with either 10% hydrofluoric acid etching (20 seconds for IPS Empress 2; 60 seconds for IPS Empress and Cergogold; and 2 minutes for In-Ceram Alumina, In-Ceram Zirconia, and Procera). or airborne 50 micron aluminum oxide particle abrasion. They concluded that Hydrofluoric acid etching and airborne particle abrasion with 50 micron aluminum oxide increased the irregularities on the surface of IPS Empress, IPS Empress 2, and Cergogold ceramics. Similar treatment of In-Ceram Alumina, In-Ceram Zirconia, and Procera did not change their morphologic microstructure.

MARKUS B. BLATZ et al $(2003)^{25}$ evaluated the bond strength of a phosphate-modified resin luting agent with and without silanization to an air particle–abraded Procera All Ceram intaglio surface compared with a conventional resin bonding system before and after artificial aging. Composite cylinders were fabricated with Z-250 composite and bonded to the ceramic specimens with either Panavia 21 TC or Rely X ARC (control) and their corresponding bonding/silane coupling agents. In addition, Panavia was used without silanization as suggested in similar studies. Bond strength with Rely X ARC and its silane coupling agent decreased significantly (P<.000) after artificial aging. Panavia 21 after silanization revealed significantly different (P<.003) early and late bond strengths but achieved

the highest bond strength after artificial aging. Bond strengths of Panavia without silanization both early and late were not significantly different. Thus concluded that Panavia 21 in combination with its corresponding bonding/silane coupling agent can achieve an acceptable resin bond to the air particle–abraded intaglio surface of Procera AllCeram restorations after artificial aging, which had mixed effects on the other investigated groups. The conventional resin luting agent revealed the most dramatic decrease in bond strength.

CATHERINE C. BEGAZO et al (2004)²⁶ find the optimal choice of luting cement to Synthoceram, an aluminum oxide-reinforced glass ceramic material. The bond strength of five different commercial luting cements (Ketac Cem, Rely XLuting, Fuji Plus, Panavia F & Xeno Cem.) to the ceramic material was evaluated. The effect of surface treatments, etching, sandblasting, silanizing, and A combination of these treatments was also investigated. Based on the results of this study, they concluded the use of resin composite based cements is preferred for cementation of an all-ceramic restoration with an aluminum oxide-reinforced glass ceramic base. Surface treatments of etching and/or sandblasting followed by silanization provide the highest bond strength values.

FLAVIO H. RASETTO et al (2004)²⁷ evaluate the degree of polymerization. The power outputs from a conventional halogen (3M Unitek), a plasma arc (Apollo 95E), and a high-intensity halogen (Kreativ Kuring Light Model 2000) light were measured by a radiometer. The light intensity (mW/cm2) from these units was also measured after transmission through 0.25-, 0.40-, and 0.60-mm-thick Procera copings and through 1mm-thick disks of feldspathic porcelain (Ceramco II), aluminous porcelain (Vitadur Alpha), and a castable pressed ceramic (IPS Empress). Intensities of light from 3 polymerization units, conventional halogen light, highintensity halogen light, and plasma arc, were 660, 1050, and 2475 mW/cm² respectively, and these together with the ceramic veneer thickness dictated the light transmission through veneers. They found only the plasma arc and the high-intensity halogen polymerization units emitted light of sufficient energy to effect polymerization of a resin luting agent. With conventional halogen polymerization units, there may be insufficient light transmission through thicker veneers or all-ceramic crowns for adequate light polymerization.

HEINZ LUTHY (2006)²⁸ evaluate the shear bond strength of different cements(Ketac-Cem, Nexus, Rely X Unicem, Superbond C&B, Panavia F, and Panavia 21.) to densely sintered zirconia ceramic after aging

by thermocycling. They found none of the fractures occurred at the interface of the metallic rods. The assemblies failed either at the interface between the ceramic surface and the cements or within the cements. Thermo cycling affected the bond strength of all luting cements studied except for both Panavia materials and Rely X Unicem. After thermo cycling—bond strengths for Ketac-Cem and Nexus were quite low. Nexus in combination with tribochemical silica-coating of ceramic surface produced higher bond strength. The four adhesive resin cements (Rely X Unicem, Superbond C&B, Panavia F, and Panavia 21) gave superior results. The strongest bond to zirconia was obtained with Panavia 21.

EBRU CAL et al $(2006)^{29}$ studied Effect of a dentin adhesive system containing antibacterial monomer-MDPB (Clearfil Protect Bond) on the shear bond strength of all-ceramic-IPS Empress 2 restorations luted with three different dual-polymerizing systems (Variolink 2, RelyX ARC and Panavia F 2.0). They conclude Application of the antibacterial adhesive increased the shear bond strengths of all three dual-polymerizing systems to dentin (p<0.00). The antibacterial adhesive system Clearfil Protect Bond can be safely used to prevent the potential risk of complications resulting from bacterial activity regardless of affecting the bond strength of IPS Empress 2 restorations luted with the dual-polymerizing systems used in this study. **G.J.P. FLEMING et al (2006)**³⁰ test the validity of the proposed resin-strengthening mechanisms to facilitate an improved understanding of the possible mechanisms involved. The results show that a layer of well-bonded cement does substantially increase the fracture resistance of an aluminous core porcelain.

A. DELLA BONA et al $(2006)^{31}$ use fracture mechanics and fractography to determine the K_A(the apparent interfacial fracture toughness) of the adhesion zone of resin/ceramic systems, testing the hypothesis that K_A is affected by ceramic microstructure and ceramic surface treatments. The results of this study show that there is a synergistic effect of HF etching and Silane coupling on K_A for the systems studied and KA are affected by the ceramic microstructure and ceramic surface treatments, confirming the study. They concluded all fractures occurring within the adhesion zone originated from the Vickers indentation; this study suggests that the micro tensile test may be preferable to conventional shear or flexural tests as an indicator of composite-ceramic bond quality.

JOANNE NGO UY et al (2006)³² investigated the load-fatigue performance of complete gold crowns cemented with 4 types of resin cement(C & B Opaque [CBO], Calibra Esthetic [CE], RelyX Unicem[RU], and Panavia F [PF]) and a control, zinc phosphate cement (HY-Bond). Fatigue load of 73.5 N was applied at an angle of 135 degrees to the long axis of each tooth-crown specimen. Preliminary failure was defined as the propagation of a crack in or around the crown luting cement layer. The number of cycles to preliminary failure and the cement failure location were determined. Group CE had the highest rank of cycles to preliminary failure, while HBZPC had the lowest cycles to preliminary failure. Group CE had a significantly higher failure cycle count compared to PF (P=.016), RU (P=.001), and HBZPC (P..001), but was not significantly different from CBO (P=.112). There was no significant difference in the failure cycle count between RU and HBZPC (P=.070). Of the 4 resin cement groups, Groups CE, CBO, and PF were significantly superior to HBZPC.

JATYR PISANI-PROENCA et al $(2006)^{33}$ evaluate the micro tensile bond strength (mTBS) of 3 resin cements(1 self-adhesive universal resin cement (RelyX Unicem) or 1 of 2 resin-based luting agents (Multilink or Panavia F), to a lithia disilicate–based ceramic submitted to 2 surface conditioning treatments(no conditioning (no-conditioning/control), or 5% hydrofluoric acid etching for 20 seconds and silanization for 1 minute (HF+SIL). Specimens were thermal cycled (5000 cycles, 58C-558C) and tested in tension at 1 mm/min. The surface conditioning factor was significant (HF+SIL>no-conditioning) (P<0.0001). Considering the unconditioned groups, the mTBS of RelyX Unicem was significantly higher than that of Multilink and Panavia F. Previous etching and silanization yielded statistically higher mTBS values for RelyX Unicem and Multilink when compared to Panavia F. Spontaneous debonding after thermal cycling was detected when luting agents were applied to untreated ceramic surfaces.

K. HIKITA et al $(2007)^{34}$ studied The bonding effectiveness of five adhesive luting agents to enamel and dentin using different application procedures was using a micro-tensile bond strength protocol (TBS). Composite resin blocks (Paradigm, 3M ESPE) were luted using Linkmax (LM; GC), Nexus 2 (NX; Kerr), Panavia F (PN; Kuraray), RelyX Unicem (UN; 3M ESPE) or Variolink II (VL; Ivoclar-Vivadent) instructions. For some luting agents, modified application procedures were also tested, resulting in four other experimental groups: Prompt L-Pop + RelyX Unicem (PLP + UN; 3M ESPE), Scotchbond Etchant + RelyX Unicem (SE + UN; 3M ESPE), Optibond Solo plus Activator +Nexus 2 (ACT+ NX; Kerr) and KEtchant gel + Panavia-F (KE + P; Kuraray). The experimental groups were classified according to the adhesive approach in self-adhesive (UN), etchand-rinse (ACT+ NX, NX, KE + P, SE +UN and VL when bonded to enamel) and self-etch adhesive luting agents (LM, PLP +UN, PN and VL when bonded to dentin). The specimens were stored for 24h in distilled water at 37 °C prior to micro TBS testing. They concluded following a correct application procedure, the etch-and-rinse, self-etch and self adhesive luting agents are equally effective in bonding to enamel and dentin.

ANDREE PIWOWARCZYKA, et al (2007)⁹ examined the longterm adhesion of seven dual-polymerizing cementing agents one compomer cement (PermaCem), five resin cements (RelyX ARC, Panavia F, Variolink II, Nexus 2, Calibra) and one self-adhesive universal resin cement (RelyX Unicem) to human dentin in vitro. One subgroup (n = 10) was tested after 150 days of storage in water at 37 °C (time t1), the other subgroup (n = 10) was tested after 150 days of storage plus 37,500 thermal cycles (time t2). Values were

slightly higher at t1 (5.9 \pm 4.7MPa) than at t2 (4.9 \pm 4.2MPa) (p = 0.0044). Polymerization

with light activation $(6.5\pm5.1$ MPa) yielded higher strengths than polymerization without

 $(4.3\pm3.3$ MPa) (p < 0.0001). Thus concluded that Cementing agents/adhesive systems and the polymerization method influence the long-term bond to hard dental tissues.

GILBERTO ANTONIO BORGES et al (2007)³ evaluate the bond strength between a densely sintered alumina ceramic and bovine dentin with

2 adhesive resin cements and a resin-modified glass ionomer cement(Panavia F, RelyX ARC, or RelyX Luting.) using an extrusion shear strength test. After 24 hours of storage at 37°C, an extrusion shear test was performed in a universal testing machine at 0.5 mm/min until bonding failure. The highest strength values were obtained with Panavia F, and they were significantly higher (P < .05) than each of the other 2 cements, which were not significantly different from each other and they concluded an MDP-containing adhesive system (Panavia F) provides better extrusion bond strength to a high-density alumina ceramic than a Bis-GMA resin luting agent system (RelyX ARC) or a resin-modified glass ionomer cement system (RelyX Luting).

F. MONTICELLI et al (2008)³⁵ studied differences in the resin cement diffusion into dentin exist among commercial adhesive cements. Composite cylinders were luted on mid-coronal dentinal surfaces by etchand-rinse cement (Calibra), a self-etching system (Panavia F 2.0), and 4 selfadhesive cements (Multilink Sprint, Rely X Unicem, G-Cem, Bis-Cem). They found that Conventional acid etching resulted in partially infiltrated adhesive interfaces differing from those achieved with the application of self-etching primer. No hybrid layer\ and/or resin tag formation was detectable at the interfaces bonded with self-adhesive cements. Limited decalcification/infiltration was observed for self-adhesive cements into the underlying dentin. Self-adhesive cements were not able to demineralize/dissolve the smear layer completely.

O. ADDISON et al (2008)³⁶ studied the hypothesis that ceramic strength enhancement was conferred by the characteristics of the resin ceramic hybrid layer. Dentin porcelain discs were polished with a P4000grade abrasive paper, and half were centrally indented at 9.8 N. Further discs were alumina-air-abraded. Groups of 30 specimens were coated with resin cement thicknesses varying from 0 to 250 ± 20 micron before bi-axial flexure testing. They concluded resin cement coating significantly increased the mean strength that was attributed to a resin-ceramic hybrid layer sensitive to surface texture.

C.J. SOARES et al (2008)³⁷ hypothesized that stress distribution inside the testing specimen is affected by microtensile specimen shape and attachment method. Rectangular, hourglass, and dumbbell-shaped specimens, all with a 1 mm2 cross-sectional testing region, were modeled as indirect ceramic restorations luted to dentin. Stress analysis showed a direct correlation between attachment modes and stress distribution, with shear stresses observed in models with less surface attachment. Increasing the number of faces for specimen attachment to the metallic gripping device

resulted in a more homogeneous and regular distribution of stress, with tensile stress concentrated at the adhesive interface. Dumbbell-shaped specimens showed improved stress distribution compared with rectangular and hourglass-shaped specimens.

GUREL PEKKAN et al (2009)⁴ examine shear (S) and tensile (T) bond strengths between 2 all-ceramic systems (IPS Empress 2 (E) and Cergo Pressable Ceramic (C)). And human dentin using 3 dual-polymerizing resin cements. (Nexus 2 (N) with Self-Etch Primer, Duo-Link (D), and Variolink II (V), with their respective bonding systems). They found Significant differences were observed in shear and tensile bond strength values of the adhesive systems used (P<.05). Duo-Link showed the highest mean bond strength values, whereas Nexus 2 revealed lower shear and tensile bond strength values. Thus concluded Cementing agents/adhesive systems may influence the bond to dental hard tissues. Dual-polymerizing activators may have a negative effect on polymerization of the bonding agent.

C.W.M. CHUNG et al (2009)³⁸ examined the effect of saliva contamination on the micro tensile bond strength (mTBS) of resin luting cements to dentin. For RelyX ARC (ARC, 3M ESPE), dentin surfaces were etched with 32% phosphoric acid. The subgroups were: ARC-control (uncontaminated), ARC-I (saliva contamination, blot-dried), ARC-II (saliva

contamination, rinse, blot-dried) and ARC-III (saliva contamination, rinse, re-etch, rinse, blot-dried). For Panavia F 2.0 (PF, Kuraray), the subgroups were: PFcontrol (uncontaminated), PF-I (saliva contamination, dried), PF-II (saliva contamination, rinse, dried), PF-III (primer, saliva contamination, dried), PF-IV (primer, saliva contamination, dried, primer re-applied) and PF-V (primer, saliva contamination, rinse, dried, primer re-applied). Composite blocks were luted onto dentin using the two cements. Bonded specimens were sectioned into 0.9 mm*0.9 mm beams for mTBS testing. For ARC, salivary contamination of etched dentin (ARC-I) significantly lowered bond strength (p = 0.001). For PF, salivary contamination of dentin before (PF-I) and after application of primer (PF-III and PF-IV) significantly lowered bond strength (p < 0.001). Thus they concluded that Saliva contamination during luting deteriorated the bond quality of resin cements.

SHUZO KITAYAMA et al (2010)³⁹ evaluate and compare bond strengths of different primers and resin cements to silica-based and zirconia ceramics. Silica-based and zirconia ceramic specimens were ground flat with #600-grit SiC paper. The ceramic surfaces were airborne-particle abraded and then divided into 11 groups of seven each: untreated (control); and conditioned with one of the six primers in combination with a resin cement

from the same manufacturer as follows: Bistite II/Tokuso Ceramic Primer, Linkmax/GC Ceramic Primer, RelyX ARC/RelyX Ceramic Primer, Panavia F 2.0/Clearfil Ceramic Primer, and Resicem/Shofu Porcelain Primer and Resicem/AZ Primer. Stainless steel rods were bonded to the ceramic surfaces using one of the five resin cements. After 24-h water storage, the tensile bond strengths were tested using a universal testing machine. Result shows Conditioning with primers containing a silane coupling agent (all the primers except AZ Primer) significantly enhanced bond strengths of resin cements to silica-based ceramic. For zirconia ceramic, Resicem/AZ Primer exhibited significantly higher bond strength than the other groups except Panavia F 2.0/Clearfil Ceramic Primer. Thus the use of primers containing a silane coupling agent improved resin bonding to silica-based ceramic. On the other hand, the use of primers containing a phosphoric acid monomer or a phosphate ester monomer improved resin bonding to zirconia ceramic.

RAFAT BAGHERI et al (2010)⁴⁰ measure the shear punch strength of eight resin-containing luting cements(six resin luting cements; Set (SDI), Panavia F (Kuraray), RelyX Veneer (3M/ESPE), VarioloinkII (Ivoclar), Maxcem (Kerr), Nexus2 (Kerr) and two Resin-modified glass ionomer luting cements (RM-GICs); GC Fuji Plus (GC Corporation), RelyX Luting 2 (3 M/ESPE).) before and after immersion in acidic solution and ethanol at

different temperatures (37 °C and 60 °C). For each material a total of 114 disc-shaped specimens were prepared. Six specimens were immersed in distilled water for 24 h at 37 °C, polished and subjected to baseline measurement for shear punch strength. The remaining 108 specimens were randomly divided into 18 groups of six, and immersed in three solutions; distilled water, 0.01 mol/L lactic acid, and 50% ethanol at 37 °C or 60 °C, for 1 week, 1 month or 3 months. Specimens were washed, dried and tested for final shear punch strength. Values were material and solution dependent. Values of Nexus 2 and Rely X Veneer are the highest, and Rely X Luting 2 the lowest. Ethanol and lactic acid specimens showed significantly lower values compared with the distilled water specimens. They concluded that shear punch strengths of the resin-containing luting cements were affected by time and storage solution.

MATERIALS AND METHOD

The materials used for this study are as follows:-

The Cementing agents, Manufacturer, Type of Cements, Batch number and Composition are listed in following table.

| Material | Manufacturer | Туре | Batch no | Chemical composition |
|---|----------------------|----------------|----------|--------------------------|
| Variolink N | Ivoclar Vivadent, | Dual | Catalyst | BisGMA, UDMA, |
| | Schaan,Liechtenstein | curing/light | N01584 | TEGDMA, |
| | | curing resin | Base | barium glass, and silica |
| | | cement | N01552 | fillers, YbF3 |
| Calibra | Dentsply caulk, | Dual- | Base | BisGMA, EBPADM, |
| | Milford, DE 19963, | polymerizing | 1005191 | TEGDMA, |
| | USA | resin | Catalyst | butylhydoxitoluol, |
| | | cement | 100511 | benzoyl |
| | | | | peroxide, barium glass, |
| | | | | silica |
| SeT PP | SDI, Victoria, | Self | 51005112 | Fluoroaluminosilicate, |
| | Australia | etching,self- | | UDMA, |
| | | adhesive resin | | Campheroquinone, |
| | | luting cement | | Acid monomers |
| RelyX | 3M ESPE, Seefeld, | Dual- | 407551 | Phosphoric acid |
| U100 | Germany | polymerizing | | methacrylates, |
| | | self-adhesive | | dimethacrylates, |
| | | universal | | inorganic |
| | | resin cement | | fillers (72 wt.%), |
| | | | | fumed |
| | | | | silica, initiators |
| BisGMA: bisphenol-A diglycidyl ether dimethacrylate; EBPADM: ethoxylated bis-phenol-A- | | | | |
| dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; UDMA: 7,7,9-trimethyl-4,13- | | | | |
| dioxo-3,14-dioxa-5,12-diazahexadecane-1,16-dimethacrylate; YbF3: ytterbium trifluoride; | | | | |

BONDING AGENTS

| Material | Bonding | Manufacturer | Batch | Number of | Chemical |
|---|---|-----------------|-----------|----------------|------------------|
| | system | | number | application | composition/ |
| | | | | steps/approach | solvent |
| Variolink | Excite | Ivoclar | M04952 | Two-step/etch | HEMA, DMA, |
| Ν | DSC | Vivadent, | | and rinse | phosphoric acid |
| | | Schaan, | | | acrylate, highly |
| | | Liechtenstein | | | dispersed |
| | | | | | silicon |
| | | | | | dioxide/ethanol |
| Calibra | Prime & | Dentsply | Prime & | Two-step/etch | R5-62-urethan- |
| | Bond NT | caulk, Milford, | Bond | and rinse | dimethacrylat, |
| | | DE 19963, | NT: | | di- und |
| | | USA | 100607 | | trimethacrylatha |
| | | | Self Cure | | rze, |
| | | | Activator | | Siliciumdioxide, |
| | | | : 100608 | | PENTA/acetone |
| seT PP | No | SDI, Victoria, | | | |
| | bonding | Australia | | | |
| | system | | | | |
| RelyX | No | 3M ESPE, | | | |
| U100 | bonding | Seefeld, | | | |
| | system | Germany | | | |
| HEMA: 2-hydroxyethylmethacrylate; DMA: aliphatic dimethacrylate; PENTA: | | | | | |
| dipentaeryt | dipentaerythritol pentacrylate/phosphoric acid monomer; | | | | |

SILANE COUPLING AGENTS

| Name | Manufacturer | Batch no | Composition |
|------------|-------------------|----------|--------------------|
| Monobond-S | Ivoclar Vivadent, | N01595 | Alcoholic |
| | Schaan, | | solution of silane |
| | Liechtenstein | | methacrylate |
| | | | |
| Calibra | Dentsply caulk, | 100122 | Ethyl Alcohol, |
| | Milford, DE | | Acetone, |
| | 19963, USA | | Benzene |

CERAMIC ETCHANT

| Name of the Etchant | Manufacturer | Batch number | Composition |
|----------------------------|---|--------------|--------------------------|
| IPS Ceramic etching gel | Ivoclar Vivadent, Schaan, Liechtenstein | N39215 | <5% hydrofluoric acid |

BONDING PROCEDURES

| Material | Application steps |
|-------------|--|
| Variolink N | Acid etching (37% phosphoric acid) |
| | for 15 s, rinse, air-dry, apply Excite |
| | DSC for 10 s, air-dry, |
| | light-polymerize for 10 s |
| Calibra | Acid etching (37% phosphoric acid) |
| | for 15 s, rinse, mix Prime & Bond |
| | NT and Self-Cure-Activator |
| | (1:1), surface to remain wet for 20 s, |
| | air-dry for 5 s, light-polymerize for |
| | 10 s |
| SeT PP | NO pretreatment |
| RelyX U100 | NO pretreatment |

METHODOLOGY

- I. Tooth Preparation
- II. Laminate Fabrication
- III. Cementation of Veneers to the tooth
- IV. Experimental design- Randomized four groups of twelve teeth comprising Six Anterior and Six Posterior teeth.
 - Group I Rely X
 - Group II Variolink N
 - Group III Calibra
 - Group IV SeT PP
- V. Measurement of Shear Bond Strength by Universal Testing Machine (UTM)
- VI. Statistical Evaluation

I. <u>TOOTH PREPARATION</u>

Freshly extracted, non-carious permanent human incisors, canines and molars that were not endodontically treated were selected for this study. Calculus and residual periodontal tissue were removed using a surgical knife, scaler, and curette. All teeth were stored in 0.1% thymol solution at room temperature immediately after extraction. None of the extracted teeth had been stored for longer than 6 months.

Preparation of tooth surfaces was carried out by first preparing a flat surface in dentin on stationary disk using SiC sandpaper and water-cooling. The buccal/labial surfaces of teeth were ground to make it parallel to the long axis of the tooth.

After that each tooth was placed in to a silicone mold (2cm*2cm*2cm) and embedded in auto-polymerizing methalmethacrylate resin (DPI, Mumbai, India). After hardening the resin in a pressure pot for 10 min, the specimens were wet-ground sequentially to 600-grit using SiC sandpaper, to obtain a flat surface in superficial dentin. The tooth surfaces were kept moist throughout the procedure of specimen preparation(FIG. 5).
II .LAMINATE FABRICATION

Once the tooth has been prepared Silicone separating media (FIG. 6) is applied on the surface. Then Wax pattern is fabricated with Occlusal wax (FIG. 8) on individual tooth samples so as to closely adapt to the tooth surface.

The fabricated Wax pattern is kept in water for 10 minutes in order to relieve the residual stress. Then the Wax pattern is sprued (FIG. 9) and invested by phosphate bonded investment (FIG. 10) using auto mixer machine (FIG. 11). The investment is allowed to set for 45 minutes before keeping in for burn-out procedure. The Wax burn out (FIG. 16) is done at temperature around 930 degree centigrade.

Once the temperature reaches the above said degree, the pressable ceramic furnace (FIG. 14) is started for pre-heating one hour before scheduled pressing. The preheating temperature in the furnace is about 700 degree centigrade. After burn-out is completed, the ring is immediately placed in the pressing machine with ceramic button and plunger. (FIG. 17) The pressing is started.

The overall time period for the Pressing is 30minutes. In the initial 5minutes there is constant temperature rise of 40 degree centigrade per

minute and once the temperature reaches 920 degree centigrade the pressing is started which takes around 21 minutes.

Once the pressing is over the ring is taken out of the machine, and left for bench cooling. The laminates are then recovered from the investment and cleaned with the sandblasting procedure.

The laminates were then finished and polished (FIG. 20 and FIG.21).

III. <u>CEMENTATION OF VENEERS TO THE TOOTH</u> <u>SAMPLES</u>

When the laminates are ready, the cementation is done according to the manufactures instructions. Before the cementation procedures, the tooth samples are cleaned with pumice flour with the polishing cup with the help of contra angle handpiece in order to remove debris, smear layer etc. All the tooth surfaces are etched with 37% phosphoric acid gel and all the laminates are etched with Hydrofluoric acid gel.

a) <u>CEMENTATION OF LAMINATES WITH RELY X U100</u> (<u>FIG. 23)</u>

Since the cement is Self-Adhesive universal resin cement, there is no pretreatment necessary for bonding. The laminates are etched with IPS Ceramic etching gel (<5%HF) and the tooth is etched with 37% Phosphoric acid gel for 20 seconds. After the stipulated time the laminates and the teeth are rinsed with water and air dried.

The cement is dispensed from the clicker. One click is enough for one laminates on the mixing pad and it is mixed according to the manufactures instructions. The laminated are then loaded with cement and then placed on the tooth surface. It is light cured for 2 seconds and the excess cements is removed from the periphery and then finally light cured for 1 minute.

b) <u>CEMENTATION OF LAMINATES WITH VAIORLINK- N</u> (<u>FIG. 24)</u>

The Excite DSC total etch adhesive is applied on the tooth surface and Monobond-S,silane coupling agent is applied on to the laminates.

Then both are cured according to the manufactures instructions. The base and catalyst are then dispensed with auotmixing pad in the ration of 1:1 ratio. Then it is mixed for 10 seconds and then applied on to the laminates and finally placed on the tooth surface. Then cured for two seconds and excess is removed from the periphery and then final cure is done for 20 seconds after applying Oxygen –blocking gel (glycerine gel)

c) <u>CEMENTATION OF LAMINATES WITH CALIBRA (FIG. 25)</u>

The bonding systems for Calibra are Two-step /Etch and rinse type.

After etching and rinsing thorough with water, Prime and Bond NT and Self- cure Activator are mixed in 1:1 ration and applied on the surface of the tooth which is allowed for 20 seconds. Then it is air-dried for 5 seconds and light polymerized for 10 seconds.

The Calibra silane coupling agent is applied on the ceramic laminates. The base and the catalyst are dispensed on the mixing pad and mixed using a hard plastic spatula, at a base to catalyst ration of 1:1.

After mixing the laminate is luted to the tooth surface light cured for two seconds and excess is removed from the periphery and finally cured for 20 seconds.

d) <u>CEMENTATION OF LAMINATES WITH SET PP (FIG. 26)</u>

As the cement is Self-etching, Self- adhesive resin cement, it requires no pre-treatment for the tooth. It is dispensed from the tube and mixed on to the mixing pad for 10 seconds.

It is then applied on to the laminates and then the laminates are placed on the tooth surface. Then it is light cured for 2 seconds and the excess is removed and finally light cured for 20 seconds.

IV. EXPERIMENTAL DESIGN

Total of 48 teeth samples were taken for the study comprising of 24 Anterior teeth (Incisiors and Canines) (FIG. 20) and 24 Posterior teeth (Molar) (FIG. 21)

Total 48 teeth samples were randomly divided into four groups. Each group comprises 6 anterior teeth and 6 posterior teeth.

The four groups are of the following

Group I: LUTED WITH RELY X A1- A2- A3 P1-P2-P3 Group II LUTED WITH VARIOLINK N A4-A5-A6 P4-P5-P6 Group III LUTED WITH CALIBRA A7-A8-A9 P7-P8-P9 Group IV LUTED WITH SET PP A10-A11-A12 P10-P11-P12

V. MEASUREMENT OF SHEAR BOND FAILURE LOADING BY UNIVERSAL TESTING MACHINE (FIG. 27)

After storage in distilled water at 37 degree centigrade for 24 hours, the luted teeth are then thermally cycles around 5000 times between 5 degree and 55 degree centigrade (20 seconds dwelling time)

A Lloyd Universal testing machine (J.J Lloyd instruments Ltd, Warsash, UK) with the Monobevelled chisel placed as close as possible to the junction between the laminate and the tooth was used for the testing (FIG. 28).

A cross head speed of 1.0 mm/min was used and maximum load recorded for each specimen.

The fractured surface of each specimen was examined under optical microscope so that mode of failure could be determined. Failure modes were categorized as

A. Adhesive failure at ceramic- leuting interface

B. Ceramic cohesive failure

C. Cohesive failure of cement

D. Complex A and B

E. Complex B and C

F. Complex A and C

V. STATISTICAL EVALUATION

Statistical analysis of the Maximum load recorded was done with the use of a Software (SPSS Software).

Mean of all the cement group were analyzed using One way ANOVA test with maximum load as the dependent variable and the type of Resin cements as independent variable.

Unpaired T-Test was also done with < 0.05 to indicate significance.

RESULTS

<u>**Table-I**</u> Shows Mean and Standard deviation of Anterior and Posterior teeth sample group for each Cement type.

For Anterior teeth sample **Calibra** shows the highest mean load value of 464.33 and **SeT PP** shows the lowest value of 288.00.

For Posterior teeth sample, **Rely X** shows the highest value of 272.97 while **SeT PP** shows the lowest load value of 154.33.

Among all the cements SeT PP shows the lowest load values.

- <u>Table –II</u> -Shows One Way ANOVA Test for Anterior teeth sample. The table denotes Significance value of 0.295 which shows insignificance of load value among all the cements at 5% of confidence level (p<0.05)
- <u>Table –III</u> -Shows One Way ANOVA Test for Posterior teeth sample. The table denotes Significance value of 0.181 which shows insignificance of load value among all the cements at 5% of confidence level (p<0.05)
- <u>**Table –IV</u>** shows the Mean and Standard deviation of teeth samples with P values.</u>

T- test for Rely X

The T- Test for Rely X shows value of 0.726 which is insignificance of load value among all the anterior and posterior teeth sample at 5% of confidence level (p<0.05).

<u>T- test for Variolink N</u>

T- Test for Variolink N shows value of 0.535 which insignificance of load value among all the anterior and posterior teeth sample at 5% of confidence level (p<0.05).

T- test for Calibra

T- Test for Calibra shows value of 0.001 which **significance** of load value among all the anterior and posterior teeth sample at 5% of confidence level (p<0.05).

T- test for SeT PP

T-_Test for SeT PP shows value of 0.031 which **significance** of load value among all the anterior and posterior teeth sample at 5% of confidence level (p<0.05).

<u>Graph -I</u>

Graph-I shows the Mean values of all Cements against Maximum load in Newtons for Anterior teeth samples.

<u>Graph –II</u>

Graph-II shows the Mean values of all Cements against Maximum load in Newtons for Posterior teeth samples.

<u>Graph-III</u>

Graph-III shows Mean values of Anterior and Posterior teeth samples against Maximum load value for **Rely X** cement. There are no significant differences in Mean values.

<u>Graph-IV</u>

Graph-IV shows Mean values of Anterior and Posterior teeth samples against Maximum load value for **Variolink N** cement. There are no significant differences in Mean values.

Graph-V

Graph-V shows Mean values of Anterior and Posterior teeth samples against Maximum load value for **Calibra** cement. There are is significant differences in Mean values.

<u>Graph-VI</u>

Graph-VI shows Mean values of Anterior and Posterior teeth samples against Maximum load value for **SeT PP** cement. There are is significant differences in Mean values.

Table-I

Maximum Load in Newtons (N)

| | | Tooth | | | | | | |
|---------|-------------|--------|--------|-----------|--------|--|--|--|
| | | Ante | erior | Posterior | | | | |
| | | Mean | SD | Mean | SD | | | |
| Cements | Rely X | 318.00 | 181.25 | 272.97 | 101.38 | | | |
| | Variolink N | 322.67 | 132.19 | 267.97 | 45.36 | | | |
| | Calibra | 464.33 | 18.15 | 191.00 | 51.07 | | | |
| | Set PP | 288.00 | 17.09 | 154.33 | 69.01 | | | |

Table-II

ANOVA

| Maximum Load in Newtons (N) | | | | | | | |
|-----------------------------|-------------------|----|-------------|-------|------|--|--|
| | Sum of Squares | df | Mean Square | F | Sig. | | |
| Between Groups | 56024.917 | 3 | 18674.972 | 1.466 | .295 | | |
| Within Groups | 101893.33 3 | 8 | 12736.667 | | | | |
| Total | 157918.25 0 | 11 | | | | | |

Table-III

ANOVA

Maximum Load in Newtons (N)

| | Sum of Squares | df | Mean Square | F | Sig. |
|----------------|-------------------|----|-------------|-------|------|
| Between Groups | 30748.687 | 3 | 10249.562 | 2.081 | .181 |
| Within Groups | 39410.320 | 8 | 4926.290 | | |
| Total | 70159.007 | 11 | | | |

Table-IV

Maximum Load in Newtons (N)

| Cements | | | | | |
|-------------|--------|--------|--------|--------|---------|
| | Ante | erior | Post | erior | P Value |
| | Mean | SD | Mean | SD | |
| Rely X | 318.00 | 181.25 | 272.97 | 101.38 | 0.726 |
| Variolink N | 322.67 | 132.19 | 267.97 | 45.36 | 0.535 |
| Calibra | 464.33 | 18.15 | 191.00 | 51.07 | 0.001** |
| Set PP | 288.00 | 17.09 | 154.33 | 69.01 | 0.031* |

Note: ** Denotes significance at 1% level * Denotes significance at 5% level

<u>T-Test- Rely X</u>

Group Statistics

| | Tooth | Ν | Mean | Std. Deviation | Std. Error Mean |
|------------------------|-----------|---|----------|----------------|--------------------|
| Maximum | Anterior | 3 | 318.0000 | 181.24845 | 104.64384 |
| Load in Newtons (N) | Posterior | 3 | 272.9667 | 101.37556 | 58.52920 |

Independent Samples Test

| | t-test for Equality of Means | | | | | | |
|--------------------------------|------------------------------|-------|-----------------|--------------------|--------------------------|--------------------------|---------------------------|
| | t | df | Sig. (2-tailed) | Mean Difference | Std. Error Difference | 95% Confide of the Di | ence Interval fference |
| | | | | | | Lower | Upper |
| Maximum Load in Newtons (N) | .376 | 4 | .726 | 45.0333 | 119.89996 | - 287.86233 | 377.92900 |
| | .376 | 3.140 | .731 | 45.0333 | 119.89996 | - 327.10827 | 417.17494 |

T-Test- Variolink N

Group Statistics

| | Tooth | N | Mean | Std. Deviation | Std. Error Mean |
|------------------------|-----------|---|----------|----------------|--------------------|
| Maximum | Anterior | 3 | 322.6667 | 132.19052 | 76.32023 |
| Load in Newtons (N) | Posterior | 3 | 267.9667 | 45.36324 | 26.19048 |

Independent Samples Test

| | t-test for Equality of Means | | | | | | |
|--------------------------------|------------------------------|-------|-----------------|--------------------|--------------------------|-------------------------|----------------------------|
| | t | df | Sig. (2-tailed) | Mean Difference | Std. Error Difference | 95% Confide of the D | ence Interval ifference |
| | | | | | | Lower | Upper |
| Maximum Load in Newtons (N) | .678 | 4 | .535 | 54.7000 | 80.68903 | - 169.32865 | 278.72865 |
| | .678 | 2.465 | .556 | 54.7000 | 80.68903 | - 236.72059 | 346.12059 |

<u>T-Test- Calibra</u>

Group Statistics

| | Tooth | N | Mean | Std. Deviation | Std. Error Mean |
|------------------------|-----------|---|----------|----------------|--------------------|
| Maximum | Anterior | 3 | 464.3333 | 18.14754 | 10.47749 |
| Load in Newtons (N) | Posterior | 3 | 191.0000 | 51.06858 | 29.48446 |

Independent Samples Test

| | | t-test for Equality of Means | | | | | | | |
|-----------------|-------|------------------------------|-----------------|--------------------|--------------------------|-------------------------|----------------------------|--|--|
| | | | | | | 95% Confide of the D | ence Interval ifference | | |
| | t | df | Sig. (2-tailed) | Mean Difference | Std. Error Difference | Lower | Upper | | |
| Maximum Load in | 8.735 | 4 | .001 | 273.3333 | 31.29075 | 186.45628 | 360.21039 | | |
| Newtons (N) | 8.735 | 2.497 | .006 | 273.3333 | 31.29075 | 161.38941 | 385.27726 | | |

T-Test- SeT PP

Group Statistics

| | Tooth | N | Mean | Std. Deviation | Std. Error Mean |
|------------------------|-----------|---|----------|----------------|--------------------|
| Maximum | Anterior | 3 | 288.0000 | 17.08801 | 9.86577 |
| Load in Newtons (N) | Posterior | 3 | 154.3333 | 69.00966 | 39.84275 |

Independent Samples Test

| | t-test for Equality of Means | | | | | | |
|-----------------|------------------------------|---|------|-------------------------|----------------------------|-----------|-----------|
| | t | t df Sig. (2-tailed) Mean Std. Error 95% Con Difference Difference of th | | 95% Confide of the D | ence Interval ifference | | |
| | | | | | | Lower | Upper |
| Maximum Load in | 3.257 | 4 | .031 | 133.6667 | 41.04604 | 19.70458 | 247.62876 |
| Newtons (N) | 3.257 | 2.244 | .071 | 133.6667 | 41.04604 | -25.73629 | 293.06962 |

DISCUSSION

Dental ceramics are appreciated as highly esthetic restorative materials with optimal esthetic properties that better simulate the appearance of natural dentition^{1,24}. Other desirable characteristics include translucence, fluorescence, chemical stability, biocompatibility, high compressive strength, and the coefficient of thermal expansion similar to that of tooth structure².

The ceramo-metal restoration, which combines the strength of metal with the esthetics of ceramic, improved the success of dental ceramics^{41,42}. The ceramo-metal restorations enjoy wide clinical use; however, the metal core can reduce the translucency of the restoration.

With development of high alumina and zirconia which can be used as core material, metal-free all-ceramic restorations has got popularity as there will not be compromise with translucency like ceramo-metal restorations⁴³.

The cementation process is vital for the clinical success of all-ceramic restorations²⁴. The restoration may be cemented with zinc phosphate, glass ionomer, or composite resin cements. When zinc phosphate or glass ionomer cements are used, mechanical retention is necessary. Such water based cements work mainly by frictional force. On the other hand, when

mechanical retention is compromised, adhesive luting systems are recommended.

Resin luting agents are required for luting of ceramic crowns to assure high bond strengths⁴⁴, and their translucency and minimal film thickness provide optimum esthetics and enhance the clinical survival rate of allceramic restorations⁴⁵.

Gorodowsky et al (1992)⁴⁶ and **Tjan et al (1992)**⁴⁷ reported reduced microleakage with resin cement as compared to conventional cements like zinc phosphates or glass ionomer cements.

In search of high-strength ceramic materials 'new' production methods are used: as casting, pressing, and milling of ceramic caps²⁶. Automatic production methods may exclude the variance in strength related to manual manipulation of ceramic materials. CAD/CAM technology is an example of a method for making dental ceramic restorations without manual interference.

Even with the application of automated ways of production, the cement can be the 'Achilles heel' of an all-ceramic restoration⁴⁸. For brittle materials, as ceramics are, the integrity and longevity of the tooth–cement–ceramic interface is the main importance for the risk of fracture of the restoration.

All ceramic restoration are broadly of two types namely Silica-based and Non-Silica based viz Zircornia/Alumina ceramics etc. Both types require different type of surface treatment. For Silica-based Porcelain Hydrofluoric acid etching is recommended whereas the Non-Silica based Porcelain like Zircornia/Alumina requires sandblasting as Acid etchants used for silica-based dental ceramics do not sufficiently roughen the surface of aluminum-oxide ceramics⁴⁹. Airborne particle abrasion with Al2O3 is effective and practical for creating an activated and roughened surface on aluminum-oxide ceramics⁵⁰.

Simonsen RJ et al (1983)⁵¹ and Chen et al (1998)⁵² reported that acid etching of ceramics using Hydrofluoric acid increases the surface area and enhance the potential for micromechanical retention of resin cement.

Yen TW et al $(1993)^{53}$ reported that etching with acid tends to remove surface cracks and round off the bottoms of cracks, thereby reducing stress concentration and increasing the overall strength.

Suliman et al (1993)⁵⁴ found that surface roughness has no significant effect on bond strength.

Ozden et al (1994)⁵⁵ reported that silane application on mechanically roughened ceramic surface to the most effective.

Roulet et al (1995)⁵⁶ reported that acid-etching with 10% hydrofluoric acid gel and 10% Ammonium biflouride was much more rougher than air-abrasion/grinding.

Chen et al (1998)⁵² also reported that if glass surface is over etched, the shear bond strength can be adversely affected.

Kamada et al (1998)⁷ reported that silane coupling agent with or without phosphoric acid –etching improves shear bond between ceramic and four luting agents.

Madani et al $(2000)^{57}$ reported that an increased concentration of hydrofluoric acid form 5% to 9.5% results in decreased shear bond strength values.

In present study, Four commercially available dual cure cements was selected namely **Rely X U100, Calibra, Variolink N** and **SeT PP.** Rely X U100 is self adhesive resin cement whereas SeT PP is self etching, self adhesive resin cement. Variolink N and Calibra have separate steps for etching, silanization and adhesive application.

The purpose of selecting the four cements, of which two cements with multistep applications and two with single step application is to evaluate and compare the significant differences in the shear bond strength. As the multistep application via etch and rinse as compared to self-etch approach is time consuming and when used clinically there is increased chance of saliva or moisture contamination.

The four luting cements tested in this study were dual cure cements; all have been used extensively to bond ceramic restorations to enamel and dentin surfaces. Dual cure cement is a two-paste system that provides both light-curing and chemical-curing capabilities. The main advantage of this type of cement is the control of working and setting times.

Cementing a veneer facing can be time-consuming because the shade needs to be matched, and excessive cement is easier to remove before setting¹⁷. The dual-cure cement gives plenty of working time because it should not set before being light cured, which can be controlled by the operator. Even though the facing is cemented permanently, it can still be removed if necessary, because the cement will not reach maximum bonding strength until 24 hours later.

During fabrication of the veneer facings, sometimes opaque is added to mask the heavy stain that comes from tetracycline or fluorosis, or sometimes the thickness is increased to improve contouring of the teeth. In this situation, light cure is less effective, and the chemical-cure component is more important to ensure that the cement is completely cured. This is another advantage of using dual-cure cement with veneer facings because it has both components and it does not rely on light cure only¹⁷.

A previous studies^{3,4,6,9,38} on the shear bond comparison was done on posterior teeth only because they provided necessary bonding area. In this study, an attempt was made to study the shear bond strength on anterior teeth (incisor and canine tooth) and as well as posterior teeth (molar tooth). The ideology of including is that in clinical scenario, laminates are most frequently indicated for anterior teeth.

The occlusal forces applied to a restoration are complex and made up of a combination of forces such as shear, tension, compression, and flexure^{6,7,21}. Accordingly, it follows that no single test can satisfactorily predict the intraoral behavior of an adhesive system⁸. The tests most widely used to examine the bond strength of composite resin to dentin are shear and tensile tests^{8,17,21}. Both can be measured by Universal testing machine.

In this study, A Lloyd Universal testing machine (J.J Lloyd instruments Ltd, Warsash, UK) with the Monobevelled chisel placed as close as possible to the junction between the laminate and the tooth was used for the testing at the cross head speed of 1mm/min.

In this study, Shear test was used for the following few reasons. First, shear strength values are higher than those obtained by the tensile test and hence easy to record. Second, shear stress is considered to be more representative of the clinical situation⁸.

In contrast **Sano et al. (1994)**⁵⁸ reported that tensile testing is very critical. If not carefully conducted, the specimen undergoes torque stress, which reduces the bond strength value.

The ideology of using distilled water instead of artificial saliva for storage of prepared tooth is to simulate the effect of moisture on the resin cements; not the effect of other ions present in artificial saliva.

According to Arcoria CJ et al(1990)⁵⁹ and Ferrari M, et al(2002)⁶⁰, thermocycling is the only in vitro test for simulating thermal stress in teeth. So in this study the luted teeth are then thermally cycles around 5000 times between 5 degree and 55 degree centigrade (20 seconds dwelling time) so as to simulate intra oral condition in laboratory.

The Statistical data was analyzed by using statistical software SPEE 15.0 for the four resin cements. Mean and Standard deviation of Anterior and Posterior teeth sample group for each Cement type was done as shown in table I.

From the Statistical data, the results shows that bond strength of resin based self-adhesive cement namely SeT PP was weaker than conventional resin cements Calibra and Variolink N. **One Way ANOVA Test** for **Anterior** teeth sample denotes Significance value of 0.295 which shows insignificance of load value among all the cements at 5% of confidence level (p<0.05) **[Table II].**

One Way ANOVA Test for Posterior teeth sample denotes Significance value of 0.181 which shows insignificance of load value among all the cements at 5% of confidence level (p<0.05) [Table III].

Due to high standard deviation for Rely X and Variolink N, the result shows **no significant differences** among the all cements for maximum load failure.

Unpaired T- Test was done for intra-group comparison for Anterior and Posterior teeth samples. The test shows insignificance level for Rely X and Variolink N at p-value of 0.726 and of 0.535 at 5% of confidence level (p<0.05).

The test shows **Significance** level for Calibra and SeT PP at p-value of 0.001 and of 0.031 which shows significance of load value among all the anterior and posterior teeth sample at 5% of confidence level (p<0.05).

Because of greater standard deviation for Variolink, there is insignificant difference between Variolink and Calibra cements so our **first null hypothesis** Differences exist in long-term durability to human dentin between the cementing agents with their respective bonding system get rejected.

Due to limited penetration of self etch, self adhesive resin cement namely SeT PP shows lower load values for anterior and posterior teeth samples as compared to other cements; this makes **second null hypothesis accepted.**

The efficacy of this study needs to be done in larger scale to enhance the results for application in Clinical condition.

SUMMARY AND CONCLUSION

This study was done to Compare and Evaluate the Shear bond strength of four commercially available resins cements namely Rely X, Variolink N, Calibra and SeT PP to human dental hard tissue and ceramic.

The number of teeth sample for each group was six in number (three anterior and three posterior teeth). Ceramic Laminates was fabricated and luted to the respective human teeth samples according to manufactures instructions.

The samples were stored for 24hrs in distilled water followed by thermo cycling. The sample was tested for maximum load failure using **Universal Testing Machine**. The data obtained was analyzed statistically by **One Way ANOVA** and **Unpaired T-test**.

For Anterior teeth sample Calibra shows the highest mean load value of 464.33 and SeT PP shows the lowest value of 288.00. For Posterior teeth sample, Rely X shows the highest value of 272.97 while SeT PP shows the lowest load value of 154.33. Among all the cements SeT PP shows the lowest load values.

One Way ANOVA Test for teeth sample shows insignificance of load value among all the cements at 5% of confidence level (p<0.05).

Within the limitation of this study following conclusions was made:

(1) There is **no significance differences** exist in long-term durability to human dentin between the cementing agents with their respective bonding system.

(2) Simplifying the application procedures of the corresponding adhesives following three step total-etch, two step total-etch, one-step selfetch, or no use of adhesives, affect the effectiveness of the bond to human dentin.

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Graph -I Anterior Teeth Sample



Maximum Load in Newtons (N)

Graph -II Posterior Teeth Sample



Maximum Load in Newtons (N)



<u>Graph III – Rely X</u>





<u>Graph IV -Variolink N</u>



Graph V -Calibra



<u>\Graph VI- SeT PP</u>





SET PP









FIG. 5 TEETH SAMPLES EMBEDED IN ACRYLIC BLOCKS



























FIG. 19 RING IMMEDIATELY AFTER PRESSING



FIG. 20 LAMINATES FOR ANTERIOR TEETH SAMPLES



FIG. 21 LAMINATES FOR POSTERIOR TEETH SAMPLES







FIG. 24 GROUP-II LUTED WITH VARIOLINK N CEMENT



FIG. 25 GROUP-III LUTED WITH CALIBRA CEMENT



FIG. 26 GROUP-I LUTED WITH SET PP CEMENT





FIG. 28 SHEAR LOADING OF TEETH SAMPLES USING UNIBEVEL CHISEL IN UNIVERSAL TESTING MACHINE

