

**COMPARATIVE EVALUATION OF TENSILE BOND
STRENGTH OF ZIRCONIA AND RESIN CEMENT
USING TWO DIFFERENT BONDING AGENTS
- AN *IN VITRO* STUDY**

Dissertation submitted to

**MAHARASHTRA UNIVERSITY OF HEALTH SCIENCES,
NASHIK**

In the partial fulfilment of regulations

for the award of the degree of

MDS

IN

**PROSTHODONTICS INCLUDING REMOVABLE, FIXED,
MAXILLOFACIAL AND IMPLANTOLOGY**

BRANCH –I

2019

CONTENTS

SR NO.	TITLE	PAGE NO.
1.	INTRODUCTION	1 - 5
2.	AIMS AND OBJECTIVES	6 - 7
3.	REVIEW OF LITERATURE	8 - 27
4.	MATERIALS AND METHOD	28 - 38
5.	RESULT	39 - 46
6.	DISCUSSION	47 - 56
7.	SUMMARY	57 - 59
8.	CONCLUSION	60 - 61
9.	BIBLIOGRAPHY	62 - 68
10.	TABLES AND GRAPHS	i - iv
11.	ANNEXURE	v

LIST OF TABLES

TABLE NO.	TITLES
1.	Mean tensile bond strength in three study groups along with different measures of variation
2.	Estimated Mean Tensile bond strength in three study groups along with 95% Confidence Interval (CI)
3.	Comparison of Mean Tensile bond strength across three study groups by one-way ANOVA (Analysis of Variance)
4.	Pair-wise comparison of Mean Tensile bond strength of three study groups by Bonferroni Multiple Comparison test

LIST OF GRAPHS

GRAPH NO.	TITLES
1.	Mean and standard deviation for tensile bond strength in three groups
2.	Comparison of variation in tensile bond strength across three groups
3.	Mean difference in three pairs of groups

LIST OF COLOR PLATES

PLATE NO.	FIG NO.	TITLES
I	1.	Aluminium channel
	2.	Cold-cure polymethyl methacrylate monomer & polymer
	3.	Petrolleum jelly
	4.	3% Hydrogen peroxide
	5.	Formalin solution
	6.	Nickel chromium pellets
	7.	Inlay wax
	8.	10-methacryloyloxy-decyl dihydrogenphosphate (MDP)(Z prime Plus)
	9.	10-methacryloyloxy-decyl dihydrogenphosphate (MDP) and 3-methacryloxy propyltrimethoxy silane (MPS) (Monobond Plus)
	10.	Zirconia blank (Cercon)
	11.	Etchant (microgel sf)
	12.	Resin cement (Variolink N Dual cure)
	13.	Aluminium oxide partcles
II	14.	Air rotor
	15.	Surveyor
	16.	Digital scanner
	17.	CADCAM Milling unit
	18.	Sandblaster
	19.	Universal testing machine
	20.	Miscellaneous armamentarium
	21.	Tooth preparation burs
	22.	Light curing unit
	23.	Test-tube holder
	24.	Milling unit
	25.	Digital weighing machine

PLATE NO.	FIG NO.	TITLES
III	26.	30 Mandibular first molars
	27.	Tooth with retention groove and wire
	28.	Tooth mounted in acrylic
	29.	Mounted assembly on surveyor for tooth preparation
	30.	Axial preparation with round tapered diamond bur
	31.	Occlusal preparation with wheel diamond bur
IV	32.	Prepared tooth
	33.	Wax pattern of bucco-occluso-lingual templete
	34.	Wax pattern of mesio-occluso-distal templete
	35.	Casted bucco-occluso-lingual metal templete
	36.	Casted mesio-occluso-distal metal templete
	37.	30 prepared mandbular first molars
V	38.	Scanning of prepared tooth
	39.	Scanned image
	40.	Digital image of designed coping
	41.	Milling of copings
	42.	30 milled copings
	43.	Sand blasting
VI	44.	Z prime plus bonding agent application
	45.	Monobond plus bonding agent application
	46.	Application of resin cement
	47.	1 kg weight
	48.	Zirconia coping reinforced with acrylic
	49.	30 samples with acrylic reinforcement
	50.	Retention testing in utm
	51.	Debonded 30 samples

LIST OF ABBREVIATIONS USED

SR NO.	ABBREVIATIONS	FULL FORM
1.	ZrO ₂	Zirconium Dioxide
2.	°C	Degrees Celsius
3.	Al ₂ O ₃	Aluminium Oxide
4.	CAD-CAM	Computer-Aided Design And Computer-Aided Manufacturing
5.	%	Percentage
6.	H ₃ PO ₄	Phosphoric Acid
7.	HF	Hydrofluoric Acid
8.	Er:YAG	Erbium-Doped Yttrium Aluminium Garnet Laser
9.	MDP	10-Methacryloyloxydecyl Dihydrogen Phosphate
10.	MPS	3-Methacryloxy Propyltrimethoxy Silane
11.	META	Methacryloyloxyethyl Trimellitate Anhydride
12.	MPTS	Methacryloyloxy Propyltrimethoxy Silane
13.	cm	Centimetre
14.	µm	Micrometre
15.	Kg	Kilogram
16.	hrs	Hours
17.	/min	Per Minute
18.	ANOVA	Analysis Of Variance
19.	SD	Standard Deviation
20.	vs	Versus
21.	P value	Probability of happening of an event
22.	i.e	That is
23.	N	Newton
24.	CI	Confidence Interval
25.	Y-TZP	Yttria Stabilized Zirconia Polycrystals
26.	MPa	Mega Pascal
27.	DPI	Dental Products of India
28.	Inc.	Incorporated
29.	Ltd.	Limited

INTRODUCTION

The true function of philosophy is to educate us in the principles of reasoning and not to put an end to further reasoning by the introduction of fixed conclusions.

- George Henry Lewes

Introduction:-

Advancements in the course of the last 10– 15 years in ceramic materials science for dental applications have prompted a class of high strength materials (i.e. zirconia-based ceramics) which conceivably give better fracture resistance and long term viability when contrasted with porcelain and other inorganic, non-metallic alternatives¹.

Recently, Zirconia (ZrO_2) has been introduced in dentistry because of its superior esthetics potential compared to metal-ceramic prosthesis. Zirconia has been

explored as a dental material for a range of clinical applications: Orthodontic brackets, dental posts/dowels and abutments, single crowns and fixed partial dentures. There's a wealth of data within the scientific literature concerning the employment of zirconia (ZrO_2) in dental applications. Zirconia (ZrO_2), or zirconium oxide, could be a metal chemical compound that was known as a reaction product of heating the gem, Zircon, by the German chemist Martin Heinrich Klaproth in 1789. Zirconia is polymorphic in nature, which means that it displays totally different equilibrium (stable) crystal structure at different temperatures with no modification in chemistry. It exists in three crystalline forms:

1. Monoclinic at low temperatures
2. Tetragonal above 1170°C
3. Cubic above 2370°C

In recent years, ZrO_2 has gained attention as a biomaterial because of superior mechanical properties, compared to Aluminium oxide (Al_2O_3), chemical and biological inertness that creates it very biocompatible. Exploration into ZrO_2 as a biomaterial began within the 1960s, with most of the work over the years targeted on the utilization of ZrO_2 in medical science, specifically within the area of limb, heads for total hip replacements¹. Zirconia frameworks are often fabricated according to two completely different CAD/CAM techniques.

In soft machining technique, CAD/CAM systems form pre-sintered blocks that involves machining enlarged frameworks during a so-called green state. The enlarged pre-sintered zirconium dioxide frameworks are then sintered during a sintering furnace to their full strength which is accompanied by shrinkage of

the milled framework by 25% to the required dimensions. In hard machining technique, fully sintered blocks are shaped².

Although superior in terms of mechanical performance (strength, toughness, fatigue resistance), bonding to ZrO₂ has become a subject of interest in recent years³, which is the inherent issue. It has been reportable that full-coverage zirconium dioxide ceramic restoration and FPDs may not need adhesive cementation which they can be cemented with conventional cements, including zinc phosphate or glass ionomer cement. However, the benefits of resin luting agents, e.g. marginal seal, good retention, and improvement of fracture resistance have created them progressively frequently used, even for high-strength ceramics. Moreover, an adequate resin bond has the aforesaid advantages and may become necessary in some clinical situations, like for compromised retention and short abutment teeth⁴.

Bonding of ZrO₂ to tooth structure or different substrates needs a strong organic compound bond. The success of organic compound bonding depends on mechanical bonding through micromechanical interlocking from surface roughening, and if possible, chemical bonding between ceramic and cement. Phosphoric acid (H₃PO₄) or hydrofluoric acid (HF) etching is normally recommended ways used to surface roughen silica-based ceramics. This creates a rough, clean surface that improves wettability and will increase surface area offered for mechanical interlocking. Unfortunately, H₃PO₄ and HF cannot be used effectively on non-silica-based ceramics, like ZrO₂, creating it difficult to roughen the surface for mechanical retention. The lack of silicon oxide additionally removes the chemical bonding between silica-silane necessary for silanization¹.

In search of solutions, completely different procedures to improve the bond of the resin cement to the inner surface of zirconium dioxide are tested, like surface preparation with erbium-doped and yttrium-aluminium-garnet laser (Er: YAG), grinding with diamond rotary instruments, selective infiltration etching, surface roughening by aluminium oxide blasting of different particle sizes before or when sintering, surface roughening by alumina-silica particles before silanization, and application of a liner. All these strategies obtain to enhance the mechanical and micromechanical interlocking through the increase in the roughness of the surface. However, some of these treatments have tried to be ineffective, and in many cases, they'll cause surface damage⁵.

A different approach to improve the bond strength of resin cement to zirconia is to develop a chemical interaction between the surface and the applied resin cement. For this task, research has focused on the use of primers that contain phosphate monomers that have an affinity for metal oxides with 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) being one of the most used monomers⁴. The monomer 10-methacryloyloxydecyl dihydrogen phosphate (MDP) was originally designed to bond to metal oxides and its use has been extended to oxide ceramics. MDP-containing resin cements appear to be the foremost acceptable due to the chemical interaction between the hydroxyl groups of the passive zirconium dioxide surface and also the phosphate ester group of the MDP³.

The current silane coupling agents (one-liquid or two-liquid type primers) contain some acidic monomers, together with 4-methacryloyloxyethyl trimellitate anhydride (4-META) or MDP activated γ -MPTS. Therefore, acidic monomers have

improved the wettability of the ceramic surface and have additionally increased bond strength. However, there are frequent discussions regarding how silane coupling agents will have an effect on the resin bond to conventional silica based dental ceramics, leading to failing resin bonds to oxide-based dental ceramics. Similarly, the silanization of zirconia will improve the wetting ability of the surface and thereby lead to increased bond strength values⁴.

Tensile stresses are the longitudinal stress that leads to dislodgment forces on the prosthesis. In many clinical situation, where the prepared tooth structure is comparatively less or tapered, compromises the retention of the prosthesis. It is also well established that the stresses in cement, which develop during mastication, are exceedingly complex and generally greater forces are required to dislodge prosthesis cemented with luting agents that have high tensile strength⁶.

Because of less known information regarding the bonding mechanism and effect of silane and phosphate ester monomer on zirconia, more studies needs to be done to evaluate the bond strength of zirconia and resin cement. Therefore this study was planned to determine the tensile bond strength of zirconia and resin cement using two different bonding agents.

AIMS AND OBJECTIVES

This study was aimed to evaluate and compare the tensile bond strength of zirconia and resin cement using two different bonding agents.

The bonding agents which were used were: 1) Z prime plus 2) Monobond plus

The objectives were determined to achieve this aim. Those were

1. To evaluate the tensile bond strength of zirconia and resin cement without using bonding agent.
2. To evaluate the tensile bond strength of zirconia and resin cement using bonding agent containing 10-methacryloyloxy-decyl dihydrogenphosphate (MDP).

3. To evaluate the tensile bond strength of zirconia and resin cement using bonding agent containing combination of 10-methacryloyloxy-decyl dihydrogenphosphate (MDP) and 3-methacryloxy propyltrimethoxy silane (MPS).

4. To compare the tensile bond strength of zirconia and resin cement without using bonding agent and with using bonding agent containing 10-methacryloyloxy-decyl dihydrogenphosphate (MDP) and bonding agent containing combination of 10-methacryloyloxy-decyl dihydrogenphosphate (MDP) and 3-methacryloxy propyltrimethoxy silane (MPS) respectively.

REVIEW OF LITERATURE

“Literature is the garden of Wisdom”

— *James Ellis*

Hiroki Ohno, Yoshima Araki, Masahiro Sagara (1986)⁷, evaluated the adhesion mechanism of dental adhesion resin to alloy. A dental adhesive resin containing 4-META was bonded to three different surface states, an as-polished surface and two oxidized surfaces, of a 70 mass % Co-Cr alloy. The adhesion was examined by a tensile test with and without a thermal cycle using liquid nitrogen. The relationship between the bonding strength and surface structures was discussed on the basis of the adhesion mechanism models. They concluded that, the resin bond to the as-polished surface is stronger than that to the two oxidized surfaces. The thermal cycle showed clear differences in the surface states affecting the adhesion. There are no differences in adhesion for the two oxidized surfaces. The presence of chemical

bonds including a several-molecule thick layer of adsorbed water between the oxidized surface and the 4-META side-chain was considered to be the reason why adhesion to the oxidized surface was inferior to that to the as-polished surface.

William D. Gates et al (1993)⁸, studied the tensile bond strengths of two base metal alloys and two noble metal alloys, tin-plated and non-tin-plated, with an adhesive resinous cement. Two tin platers were compared for their effectiveness in enhancing the composite resin-to-metal bond. Cylinders of the alloys were bonded end to end with the adhesive cement, thermocycled for 24 hours, stored in distilled water for 27 days, and tested for tensile bond strength.

The following conclusions were drawn:

1. The mean tensile bond strength of non-tin-plated noble and high noble alloys was significantly lower than both the tin-plated noble and high noble alloys and the non-tin-plated base metal alloys.
2. The mean tensile bond strength of the tin-plated alloys was not significantly different from the tensile bond strength of the base metal alloys.
3. The mean tensile bond strengths of the samples tin plated with the different tin platers were not significantly different.

Johnny P. Salonga et al (1994)⁹, evaluated the influence of chromium content on bond strength and durability between nickel-chromium alloys and an adhesive resin that contained 4-methacryloxyethyl trimellitate anhydride. Three nickel-chromium alloys with different chromium content, as well as pure chromium and pure nickel metals, were bonded and tested for shear strength. After repeated thermocycling,

shear bond strength decrease was lower in alloys containing high chromium content. Pure chromium metal demonstrated a 15.2% decrease, whereas pure nickel metal demonstrated the greatest (53.7%) decrease. The results suggest that nickel-chromium alloys with higher chromium content are desirable for 4-methacryloxyethyl trimellitate anhydride resin-bonded restorations.

Matthias Kern, Van P. Thompson (1995)¹⁰, evaluated the durability of alternative methods of adhesive bonding to In-Ceram ceramic. The tensile bond strength of six bonding systems to In-Ceram ceramic was tested after up to 150 days of storage in isotonic artificial saliva solution and thermal cycling. They concluded that, a long-term durable bond to In-Ceram ceramic was achieved with either the combination of tribochemical silica coating and conventional BIS-GMA composite resin or with the combination of sandblasting and composite resin modified with a phosphate monomer. These bonding methods appeared clinically suitable for bonding In-Ceram ceramic restorations. 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 to evaluate the durability of chemical bonds.

Matthias Kern, Stefan M. Wegner (1998)¹¹, evaluated the tensile bond strength and durability of yttrium-oxide–partially-stabilized zirconia ceramic (YPSZ) by different bonding methods. Plexiglass tubes filled with resin composite were bonded to YPSZ discs following various adhesion protocols. Groups of 16 samples were bonded using seven different bonding methods. Subgroups of eight bonded samples were tested for

tensile strength following storage in distilled water (37⁰C) for either 3 or 150 days. In addition, the 150 day samples were thermal cycled 37,500 times as a method to stress the bond interface. They concluded that, sandblasting alone, the additional use of a silane or acrylizing resulted in an initial bond of a conventional BisGMA resin composite to YPSZ which failed spontaneously over storage time. The use of the BisGMA resin composite after tribochemical silica coating of YPSZ and the use of a polyacid-modified resin composite after sandblasting of YPSZ resulted in an initial bond which decreased significantly over storage time ($p = 0.05$). A durable resin bond to YPSZ was achieved only after sandblasting the ceramic and using one of two resin composites containing a special phosphate monomer.

K. Yoshida, K. Kamada, M. Atsuta (1999)¹², evaluated the effect of five adhesive primers on the shear bond strength of a self-curing resin to cobalt–chromium (Co-Cr) alloy. The adhesive primers Acryl Bond® (AB, Shofu), Cesead Opaque Primer® (COP, Kuraray), Metacolor Opaque Bonding Liner® (MOBL, Sun-Medical), Metal PrimerII® (MPII, GC) and MR. Bond® (MRB, Tokuyama) were used. A brass ring which was placed over the casting alloy disk surface non -primed or primed with each primer was filled with the self-curing MMA-PMMA resin. The specimens were stored in water at 37 °C for 24 h and then immersed alternately in water baths at 4 °C and 60 °C for 1 min each for up to 50000 thermal cycles before shear mode testing at a crosshead speed of 0.5 mm/min. All of the primers examined, except MOBL, improved the shear bond strength between the resin and Co-Cr alloy compared with non-primed specimens prior to thermal cycling. After 50000 thermal cycles, the bond strengths of resin to Co-Cr alloy primed with COP or MPII were significantly greater than those of specimens primed with AB, MOBL or MRB and non-primed controls.

S. Nakamura et al (2004)¹³, evaluated the shear bond strengths of three dual-cured resin luting cements (Linkmax HV, Panavia Fluoro Cement, and RelyX ARC) to glass-infiltrated alumina-reinforced ceramic material and the effect of four silane coupling agents (Clearfil Porcelain Bond, GC Ceramic Primer, Porcelain LinerM, and Tokuso Ceramic Primer) on the bond strength. The two type-shaped of In-Ceram alumina ceramic glass-infiltrated specimens were untreated or treated with one of the four ceramic primers and then cemented together with one of the three dual cured resin luting cements. Half of the specimens were stored in water at 37⁰ C for 24 h and the other half thermocycled 20,000 times before shear bond strength testing. Surface treatment by all silane coupling agents improved the shear bond strength compared with non-treatment. When the alumina-reinforced ceramic material was treated with any silane coupling agent except GC Ceramic Primer and cemented with Linkmax HV, no significant differences in bond strength were noted between after water storage and after 20 000 thermocycles.

Markus B. Blatz et al (2004)¹⁴, evaluated and compared the bond strengths of different bonding/silane coupling agents and resin luting agents to zirconia ceramic before and after artificial aging. Composite cylinders (2.9 mm33.0 mm) were bonded to airborne-particle-abraded intaglio surfaces of Procera All Zirkon specimens (n=80) with either Panavia F (PAN) or Rely X ARC (REL) resin luting agents after pretreatment with Clearfil SE Bond/ Porcelain Bond Activator (Group SE). In another group, Rely X ARC was used with its bonding/silane coupling agent (Single Bond/Ceramic Primer, Group SB). PAN without any bonding/silane agent (Group NO) was the control. Subgroups of 10 specimens were stored in distilled water for either 3 or 180 days before shear bond strength was tested. One hundred eighty-day-

old specimens were repeatedly thermal cycled for 12,000 cycles between 5 and 60⁰C with a 15-second dwell time. They stated that, artificial aging significantly reduced bond strength. A bonding/silane coupling agent containing an adhesive phosphate monomer can achieve superior long-term shear bond strength to airborne-particle-abraded Procera All Zirkon restorations with either one of the 2 resin luting agents tested.

Luiz Felipe Valandro et al (2005)¹⁵, evaluated the effect of silica coating on a densely sintered alumina ceramic relative to its bond strength to composite, using a resin luting agent. Blocks of ceramic and composite were made. The ceramic (Procera AllCeram) surfaces were polished, and the blocks were divided into 3 groups (n = 5): SB, airborne-particle abrasion with 110- μ m Al₂O₃; RS, silica coating using Rocatec System; and CS, silica coating using CoJet System. The treated ceramic blocks were luted to the composite (W3D Master) blocks using a resin luting agent (Panavia F). Specimens were stored in distilled water at 37⁰C for 7 days. The specimens were loaded to failure in tension in a universal testing machine. They concluded that tribochemical silica coating systems increased the tensile bond strength values between Panavia F and Procera AllCeram ceramic.

Keiichi Yoshida, Yukiko Tsuo, Mitsuru Atsuta (2006)¹⁶, evaluated the shear bond strength between dual-cured resin luting cement and pure zirconium (99.9%) and industrially manufactured yttrium-oxide-partially stabilized zirconia ceramic, and the effect of MDP (10-methacryloyloxydecyl dihydrogen phosphate) primer (MP) and zirconate coupler (ZC) on bond strength. Two different-shaped pure zirconium and zirconia ceramic specimens were untreated or treated with various primers, including

different concentrations of MP containing phosphoric acid ester monomer (MDP) in ethanol, ZC containing a zirconate coupling agent in ethanol, or a mixture of MP and ZC. The specimens were then cemented together with dual-cured resin luting cement (Claparl DC). The bond strengths of resin luting cement to both the zirconium and zirconia ceramic were enhanced by the application of most MPs, ZCs, and the mixtures of MP and ZC. The application of the mixture of MP and ZC was effective for bonding between zirconia ceramic and dual-cured resin luting cement.

Saadet Saglam Atsu et al (2006)¹⁷, evaluated the effects of airborne-particle abrasion, silanization, tribochemical silica coating, and a combination of bonding/silane coupling agent surface treatment methods on the bond strength of zirconium-oxide ceramic to a resin luting agent. The ceramic surfaces were airborne-particle abraded with 125 μ aluminium-oxide (Al_2O_3) particles and then divided into 6 groups (n=10) that were subsequently treated as follows: Group C, no treatment (control); Group SIL, silanized with a silane coupling agent (Clearfil Porcelain Bond Activator); Group BSIL, application of the adhesive 10-methacryloyloxydecyl dihydrogen phosphate monomer (MDP)-containing bonding/silane coupling agent mixture (Clearfil Liner Bond 2V/ Porcelain Bond Activator); Group SC, silica coating using 30-mm Al_2O_3 particles modified by silica (CoJet System); Group SCSIL, silica coating and silanization (CoJet System); and Group SCBSIL, silica coating and application of an MDP-containing bonding/silane coupling agent mixture (Clearfil Liner Bond 2V/Porcelain Bond Activator). The composite resin cylinders were bonded to the treated ceramic surfaces using an adhesive phosphate monomer-containing resin luting agent (Panavia F). After the specimens were stored in distilled water at 37 °C for 24 hours, their shear bonding strength was tested using a universal

testing machine at a crosshead speed of 0.5 mm/min. They concluded that tribochemical silica coating (CoJet System) and the application of an MDP-containing bonding/ silane coupling agent mixture increased the shear bond strength between zirconium-oxide ceramic and resin luting agent.

Jukka P. Matinlinna et al (2006)¹⁸, evaluated and compared the effect of three trialkoxysilane coupling agents on the bond strength of a Bis-GMA-based unfilled resin and a dimethacrylate-based resin composite luting cement to a zirconia ceramics. The specimens in each group were all assigned to air-borne alumina particle abrasion followed by tribochemical silica-coating and silanization with 1 vol% solutions of 3-methacryloyloxypropyltrimethoxysilane, 3-acryloyloxypropyltrimethoxysilane, or 3-isocyanatopropyltriethoxysilane in an ethanol–water mixture. The sample stubs were made of a Bis-GMA/MMA/DMAEMA resin or a commercial resin composite luting cement (RelyXTM ARC, 3M ESPE, Seefeld, Germany). They were bonded to the conditioned and silanized silicacoated zirconia specimens using polyethylene molds. All specimens were tested at dry and thermo-cycled (6000, 5–55°C, 30 s) conditions. The shear bond strength of resin stubs to zirconia was measured in a universal testing machine (cross-head speed 1 mm/min). They stated that, in dry conditions, the highest shear bond strength was 9.7MPa (S.D. 3.3MPa), and for thermo-cycled samples 7.4MPa (S.D. 2.4MPa) was obtained with RelyXTM ARC cement with 3-methacryloyloxypropyltrimethoxysilane. In general, thermo-cycling decreased the bond strengths significantly for the Bis-GMA resin. All samples silanized with 3-isocyanatopropyltriethoxysilane de-bonded during thermo-cycling. De-bonding was dominantly due to adhesive failure.

Mona Wolfart et al (2007)¹⁹, evaluated of the bond strength and its durability of two composite resins to zirconia ceramic after using different surface conditioning methods. Plexiglas tubes filled with composite resin were bonded to zirconia ceramic discs (Cercon) which were either in their original state as supplied by the manufacturer only cleaned in isopropanol or were cleaned with an air–powder–water spray with sodium hydrocarbonate solution or were air abraded. Groups of 20 specimens each were bonded either with a conventional composite resin (Variolink II) or with a phosphate monomer (MDP)-containing resin (Panavia F) to the ceramic discs. Subgroups of 10 bonded specimens were stored in distilled water (37⁰C) for either 3 days or for 150 days. Additionally, the 150 days specimens were thermal cycled 37,500 times. Air abrasion resulted in significantly higher TBS ($p \leq 0.01$) than the two other surface conditioning methods. After 150 days storage, only the air abraded specimens bonded with Panavia F showed high bond strengths of 39.2mpa, whereas most other specimens debonded spontaneously or showed very low bond strengths.

DP Senyilmaz et al (2007)²⁰, evaluated the bond strength of modern “self-adhesive” resin cements to a zirconium- based dental ceramic following different surface preparations and storage conditions. The surface of zirconium-based ceramic discs were either left untreated, prepared using alumina grit-blasting or tribochemical treatment. Resin composite cylinders were bonded to ceramic specimens using Panavia-F, RelyX Unicem or Maxcem resin cements. The shear bond strength of specimens was tested “dry,” following 24-hour water immersion or a thermocycling regime. They concluded that the pre-treatment of a zirconium based ceramic surface with grit-blasting and tribochemical treatment improves the bond strength of resin

cements. Following “wet” storage conditions, Panavia-F and Unicem demonstrated superior bond strength compared with Maxcem.

R. Tanaka et al (2008)²¹, evaluated the efficiency of silica-coating by tribochemical modification of Yttrium-oxide-partially stabilized zirconia (YPSZ) ceramics. They concluded that the silica-coating of YPSZ ceramics by tribochemical modification was not efficient, given the higher mechanical toughness of the densely sintered ceramics. Stable shear bond strength was achieved on silica-coated YPSZ ceramics with the cooperative interaction of phosphate monomer and silane coupling.

Mutlu Özcan, Henk Nijhuis, Luiz Felipe Valandro (2008)²², evaluated the effect of chairside and laboratory types of surface conditioning methods on the adhesion of dual cure resin cement with MDP functional monomer to zirconia ceramic after thermocycling. Disk-shaped Y-TZP ceramics were used and finished with wet 1200-grit silicon carbide abrasive paper. Specimens were randomly divided into four experimental groups according to the following surface conditioning methods: Group 1—Chairside airborne particle abrasion with 50- μm Al_2O_3 + Alloy Primer; Group 2—Airborne particle abrasion with 50- μm Al_2O_3 + Cesead II Opaque Primer; Group 3—Airborne particle abrasion with 50- μm Al_2O_3 + Silano-Pen + silane coupling agent; Group 4—Laboratory tribochemical silica coating (110- μm Al_2O_3 + 110- μm SiOx) (Rocatec) + silane coupling agent. Adhesive cement, Panavia F 2.0, was bonded incrementally to the ceramic surfaces using polyethylene molds. All specimens were thermocycled (5 and 55°C, 6,000 cycles) and subjected to shear bond strength test (1 mm/min). They concluded that after 6,000 cycles of thermal cycling, no significant

differences in the adhesion of resin cement to zirconia were found upon comparing the chairside methods against the laboratory method.

AN Cavalcanti et al (2009)²³, evaluated the influence of surface treatments and metal primers on the bond strength of resin cements to a yttrium-stabilized tetragonal zirconia (Y-TZP) ceramic. The combination of surface treatment (none, air abrasion with Al₂O₃ particles, Er:YAG laser irradiation), metal primer (none, Alloy Primer, Metal Primer II or Metaltite) and resin cement (Calibra [Bis-GMA-based] or Panavia F2.0 [MDP-based]) was done on yttrium-stabilized tetragonal zirconia (Y-TZP) ceramic. The micro-shear bond test was carried out at a 1mm/minute speed until failure. They concluded that, air abrasion with Al₂O₃ particles and the application of metal primers increased bond strength to Y-TZP surfaces for both resin cements.

Raquel Castillo de Oyagüe et al (2009)²⁴, evaluated the effect of surface conditioning on the microtensile bond strength of zirconium-oxide ceramic to dual-cured resin cements. Eighteen cylinder-shaped zirconium-oxide ceramic blocks (Cercon® Zirconia, Dentsply) were treated as follows: (1) Sandblasting with 125µm aluminium-oxide (Al₂O₃) particles; (2) tribochemical silica coating using 50µm Al₂O₃ particles modified by silica; (3) no treatment. Each ceramic cylinder was duplicated in composite resin (Tetric Evo Ceram, Ivoclar-Vivadent) using a silicon mold. Composite cylinders were bonded to conditioned ceramics using: (1) Calibra (Dentsply Caulk); (2) Clearfil Esthetic Cement (Kuraray); (3) Rely×Unicem (3M ESPE). After 24h bonded specimens were cut into microtensile sticks that were loaded in tension until failure. They stated that, the phosphate monomer-containing

luting system (Clearfil Esthetic Cement) is recommended to bond zirconia ceramics and surface treatments are not necessary.

Hesam Mirmohammadi et al (2010)²⁵, evaluated the bond strength values and the ranking order of three phosphate monomer containing resin cements using microtensile (TBS) and microshear (SBS) bond strength tests. Zirconia discs (Procera Zirconia) were bonded to resin composite discs (Filtek Z250) using three different cements (Panavia F 2.0, RelyX UniCem, and Multilink). Two bond strength tests were used to determine zirconia resin bond strength; microtensile bond strength test (TBS) and microshear bond strength test (SBS). Based on the findings of this study, the data obtained using either TBS or SBS could not be directly compared. TBS was more sensitive to material differences compared to SBS which failed to detect such differences.

Shuzo Kitayama et al (2010)²⁶, evaluated and compared 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 and failure modes were examined. They

stated that, 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. On the other hand, the use of primers containing a phosphonic acid monomer or a phosphate ester monomer improved resin bonding to zirconia ceramic.

Pascal Magnea, Maria P.G. Paranhos, Luiz H. Burnett Jr (2010)²⁷, evaluated the effect of a new experimental primer, a mixture of organophosphate and carboxylic acid monomers, on the zirconia-to-resin shear bond strength (SBS). Forty Y-TZP blocks (15×4×2mm) were embedded in an acrylic resin base, polished, Al₂O₃-sandblasted and randomly divided into eight groups. Three different resin-based luting agents (BisCem, Duo-Link, Panavia F) were used to build 2.4 mm-diameter cylinders ($n = 15$) onto the zirconia surface with and without the new experimental zirconia primer. The new primer was also tested with Z100 restorative composite resin cylinders. In addition, Panavia was used with its own primer (Clearfil Ceramic Primer). SBS testing was carried out after 24h of storage in water. He stated that, the use of the new zirconia experimental primer based on organophosphate/ carboxylic acid monomers increased the bond strength of different resin-based luting agents including Z100 restorative material.

Jeong-yeon Yun et al (2010)²⁸, evaluated the effect of sandblasting and metal primers on the shear bond strength of three commercial resin cements to Yttria-Tetragonal Zirconia Polycrystal (Y-TZP) ceramics. One hundred and twenty Y-TZP ceramic cylinders (Ø7mm×12mm) were embedded in polytetrafluoroethylene (PTFE) molds using PMMA. The specimens were divided randomly into 12 groups ($n = 10$),

according to the surface treatments (control; sandblastonly; metal primer-only; sandblast + metal primer) and metal primer-resin cements (Alloy primer – Panavia F 2.0, V-primer – Superbond C&B, Metaltite – M bond) rendered. The mixed resin cements were placed onto the treated zirconia surfaces in cylindrical shape ($\text{Ø}3\text{mm}\times 3\text{mm}$) using PTFE molds. All specimens were thermocycled (5 and 55°C , 5000 cycles) and subjected to shear bond strength test by a universal testing machine with a crosshead speed of 0.5 mm/min. They stated that, metal primers are not always effective for bonding between Y-TZP ceramics and resin cements. Even though a metal primer is not enough to be used alone, combined application with sandblasting seems to be an appropriate pretreatment for improving the bond strength of resin cement to Y-TZP ceramics, especially in Panavia F 2.0.

Michael Behr et al (2011)²⁹, investigated the shear bond strength (SBS) and the tensile bond strength (TBS) of the zirconia-to-resin interface using different cement bonding concepts. Coplanar zirconia specimens were bonded to CoCr-cylinders. All bonding areas were first sandblasted with $110\ \mu\text{m}\ \text{Al}_2\text{O}_3$. SBS and TBS were determined after 24 h and 90 d of water storage as well as after 12,000 thermal cycles. The bonding concepts consisted of the application of a silane coupling agent, tribological silica coating (Rocatec system), cements or primers containing phosphone, mono-phosphate, or di-phosphate, and a combination of silica coating and primer. They concluded that, the application of silane coupling agent alone showed very low values in the TBS test. The SBS values were high and stable even after 90 d water storage. However, in the TBS test, MDP-containing systems did not surpass a mean value of 10 MPa.

Chenfeng Chen, Cornelis J. Kleverlaana, Albert J. Feilzera (2012)³⁰, evaluated the adhesive properties of a MDP-containing resin cement to a colored zirconia ceramic, using an experimental zirconia–silica coating technique with different priming conditions. 18 zirconia ceramic discs were divided into two groups: the control group and the experimental zirconia–silica coating group. Specimens in each group were further divided into 3 subgroups (n = 3) according to the priming conditions: no primer, a MDP-containing primer (ED Primer II) or a silane coupling primer (RelyXTM Ceramic Primer). Then resin-composite discs (FiltekTM Z250) were bonded to the treated surface using a MDP containing resin cement (Panavia F 2.0). The bi-layered specimens were cut into microbars and 20 microbars were randomly selected from each specimen, half of which were stored in 37⁰C water bath for 24 h, and the other half were stored for 30 days. After water storage, the samples were exposed to a micro tensile bond strength test (MTBS). They concluded that, the combination of zirconia–silica coating with silane coupling can improve the bonding of resin cement to this colored zirconia.

Liang Chen, Hong Shen, Byoung In Suh (2013)³¹, investigated the effect of incorporating BisGMA resin on the bonding properties of silane and zirconia primers. Silica-base lithium disilicate was etched and treated with BisGMA-incorporated Porcelain Primer, unmodified Porcelain Primer, or resin-containing Kerr Silane. Zirconia ceramic was airborne-particle abraded and treated with BisGMA-incorporated Monobond Plus, unmodified Monobond Plus, or BisGMA-containing Z Prime Plus. Shear bond strength tests was also performed to measure the adhesion strength between resin cements and ceramic surfaces. They concluded that, the

addition of BisGMA resin significantly inhibited the efficacy of silane-containing porcelain primers but did not affect that of phosphate-containing zirconia primers.

Yusuke OBA et al (2014)⁴, evaluated the effects of primers on the bond strength and durability of an acrylic resin luting agent bonded to zirconia. Disk specimens were fabricated from zirconia partially stabilized with yttrium oxide. The disks were primed with one of the following materials: Alloy Primer (AP), Ceramic Primer (CP), Liquid A of the Porcelain Liner M (PLM-A), Liquid B of Porcelain Liner M (PLM-B), Porcelain Liner M (PLM-A+PLM-B), Monobond Plus (MP), and mixture of AP and PLM-B. The specimens were bonded with a tri-n-butylborane (TBB)-initiated luting agent. The shear bond strengths were determined both before and after thermocycling. The highest post-thermocycling bond strength was generated from the groups primed with MP, CP, and AP. They concluded that the application of three phosphate primers is recommended for bonding the zirconia with the TBB-initiated luting agent.

Bruno Seabra, Sofia Arantes-Oliveira, Jaime Portugal (2014)³², evaluated the influence of 2 new multimode MDP-containing adhesives and several application protocols of a zirconia primer on the shear bond strength (SBS) of composite resin to zirconia. Sixty zirconia (3Y-TZP) blocks were abraded (50 μm Al_2O_3) and divided into 6 experimental groups : one Z-Prime Plus coat without light polymerization; one Z-Prime Plus light-polymerized coat; two Z-Prime Plus coats without light polymerization; two Z-Prime Plus light-polymerized coats; All-Bond Universal; and ScotchBond Universal Adhesive. They concluded that, the new multimode adhesives tested were effective in promoting adhesion between composite resin and zirconia. Z-

Prime Plus should be applied in 2 light-polymerized coats to promote SBS values similar to those of the new multimode adhesives.

Haifeng Xie et al (2016)³³, evaluated the bonding of resin-cement to yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) via silica coating followed by silanization, and three one-bottle universal adhesives, with or without prior conditioning using a zirconia primer. Y-TZP specimens (n = 160) were conditioned by tribochemical silica coating and silanization (CS), or alumina sandblasting with one of the following MDP containing adhesives or primers: Z-Prime Plus TM(zirconia primer, ZP), Single Bond Universal TM(SU), Clearfil Universal Bond TM(CU) or All-Bond Universal TM(AU). Additionally, some specimens (ZPSU,ZPCU and ZPAU) received Z-Prime Plus TM followed by one of the three adhesives. After 24 h water storage and “aging” (20,000 thermocycles plus additional 40-day water storage), shear bond strength (SBS) was measured. They concluded that the CS and ZPCU groups showed higher SBS than the other six groups. Aging led to significantly decreased SBS for all groups except CS and ZPCU.

Raphael Pilo et al (2016)³⁴, evaluated the changes in surface chemistry of Y-TZP frameworks induced by zirconia primer treatments. Polished Y-TZP discs (Lava, 3M ESPE), ultrasonicated for 10 min in ethanol, water-rinsed and air-dried were treated as follows: A: Reference (no treatment), B: Treatment with Z Prime Plus (Bisco), and C: Treatment with Z-Bond (Danville Materials). The primer films formed on Y-TZP surfaces were air-dried, left intact for 5 days (dark storage, 37⁰C, 40% RH),rinsed with 10 ml acetone to remove the loosely bound fractions, air-dried and studied by: (a)reflection optical microscopy, (b) reflection Fourier transform infrared microscopy

(RFTIRM) and (c) scanning electron microscopy/energy dispersive X-ray microanalysis (SEM/EDX). They concluded that, an amorphous thick film was observed on primed and acetone rinsed Y-TZP surfaces after B treatment, whereas C treatment formed a thinner film with phase-separated aggregates. The RFTIRM study showed that both primers induced carboxylate salt formation on Y-TZP. Phosphate groups in a dissociative form have been identified on Y-TZP, as well, indicating formation of phosphate salts. EDX analysis showed increased C, O and P content in the films, which masked the substrate contributions.

MLL Alves et al (2016)³⁵, evaluated the effects of different adhesive strategies on the adhesion of zirconia to dentin using conventional and self-adhesive cements and their corresponding adhesive resins. The teeth and zirconia cylinders were randomly divided into eight groups according to the factors “surface conditioning” and “cement type”. One conventional (CC: RelyX ARC, 3M ESPE) and one self-adhesive (SA: RelyX U200, 3M ESPE) and their corresponding adhesive resin (for CC, Adper Single Bond Plus; for SA, Scotchbond Universal Adhesive-SU) were applied on dentin. Zirconia specimens were conditioned either using chairside (CJ: CoJet, 30 μ m, 2.5 bar, four seconds), laboratory silica coating (RC: Rocatec, 110 μ m, 2.5 bar, four seconds), or universal primer (Single Bond Universal-UP).

Specimens were stored in water (37⁰C, 30 days) and subjected to the shear bond strength (SBS) test (1 mm/min). They concluded that,

1. Both chairside and laboratory air-abrasion protocols and the use of universal primer without air-abrasion improved the bond strength of zirconia to dentin

with conventional and self-adhesive cements compared to non-conditioned control groups.

2. Conventional resin and self-adhesive cements showed similar mean bond strength of zirconia to dentin after 30 days of water storage.
3. While conventional resin cement presented more frequent failures between cement and zirconia, self-adhesive cement showed mainly adhesive failures between cement and dentin.

AE Llerena-Icochea et al (2017)⁵, evaluated the influence of adhesives with different 10-MDP concentrations on the shear bond strength of a resin cement to zirconia. Six experimental adhesives were prepared with the following composition: camphorquinone, 1,2-diaminobenzene, butylhydroxytoluene, diphenyliodonium hexafluorophosphate, 2-hydroxyethyl methacrylate triethylene glycol dimethacrylate, ethoxylated bisphenol A glycol dimethacrylate, urethane dimethacrylate, bisphenol A diglycidyl methacrylate, and ethanol. The 10 - methacryloyloxydecyl dihydrogen phosphate (10-MDP) monomer was added at 0wt%, 3wt%, 6wt%, 9wt%, 12wt%, or 15wt%. Three commercially available adhesives were evaluated: Single Bond Universal, Single Bond 2, and Signum Zirconia Bond. Resin cement cylinders made with RelyX Ultimate were bonded to yttria stabilized tetragonal zirconia polycrystal with one of the evaluated adhesives and were subjected to the shear bond strength evaluation. They concluded that, the highest shear bond strength values were obtained with the Signum Zirconia Bond and Single Bond Universal. Single Bond 2 showed the lowest values.

Zafar A et al (2017)³⁶, described the review article on Perfectly Bonded Zirconia: Clarifying Confusion under following headings: Historical perspective, Concept behind zirconia ceramics, Importance of zirconia bonding, Surface abrasion or roughening, Chloro-silane treatment, Plasma oxyfluoride / gas – phase fluorination process, Application of a tribochemical silica coating, Silicocoating, Pyrosilpen technology, Selective infiltration etching, Coating of low melting temperature porcelain micro pearls, Nanostructured alumina coating, Zirconia particle deposition, Molecular vapour deposition tool (mvd), D-zirconia and metal primers, Laser application, Application of phosphate primer.

And summarized that, the incorporation of zirconia in clinical dentistry offers a new alternative to metal-free esthetic dentistry. Zirconia has superior esthetic but demands good adhesion. Recreating the DEJ is a function of addressing the needs of the individual substrates involved (enamel, dentin, and indirect materials such as zirconia). Silica coating with aluminum oxide particles is the surface treatment of choice for improving the bonding to Y-TZP ceramics with resin cements. The use of special functional monomers can chemically bond to zirconium dioxide which further improves the quality of the bond between the ceramic and resin cement.

MATERIAL METHOD

Art and science have their meeting point in method.

- *Edward G. Bulwer-Lytton*

This study was carried out to evaluate and compare tensile bond strength of zirconia and resin cement using two different bonding agents.

The description of this was divided into following headings:

1. Materials
2. Equipment
3. Armamentarium
4. Method

1. MATERIALS

Materials used were as follows:-

Material used for mold preparation for mounting tooth:-

Color Plate 1 :- Fig. 1,2,3

SR NO.	TYPE OF MATERIAL	MANUFACTURER	BATCH NUMBER
1.	Aluminium channel	--	--
2.	Cold-cure polymethyl methacrylate monomer	DPI RR Dental products of India Ltd	41815
3.	Cold-cure polymethyl methacrylate polymer	DPI RR Dental products of India Ltd	41814
3.	Petroleum jelly	--	--

Solutions used:-

Color Plate 1 :- Fig. 4,5

SR NO.	TYPE OF SOLUTION	MANUFACTURER	BATCH NUMBER
1.	3% hydrogen peroxide	HYPO SEPT	HY019
2.	Formalin Solution	IOBA cherrie	LB241809

Samples:-

Color Plate 3 :- Fig. 26

SR NO.	SAMPLES
1.	30 extracted mandibular first molar teeth

Metal template fabrication:-**Color Plate 1 :- Fig. 6,7**

SR NO.	MATERIAL	MANUFACTURER	BATCH NUMBER
1.	Nickel chromium pellets	Ruby dental product Inc.	290290
2.	Inlay wax	Surana industries	492098

Bonding agents and copings:-**Color Plate 1 :- Fig. 8,9,10,11,12,13**

SR NO.	TYPE OF MATERIAL	MANUFACTURER	BATCH NUMBER
1.	10-methacryloyloxy-decyl dihydrogenphosphate (MDP)	Z prime Plus	1700007565
2.	10-methacryloyloxy-decyl dihydrogenphosphate (MDP) and 3-methacryloxy propyltrimethoxy silane (MPS).	Monobond Plus	W97518
3.	Zirconia blanks	Cercon	103774
4.	Etchant	Microgel SF	3181502
5.	Resin cement	Variolink N Dual cure	642979AN
6.	Aluminium oxide particles	KOREX 110	460575

1. EQUIPMENTS

Color Plate 2 :- Fig. 14,15,16,17,18,19

SR NO.	MATERIAL	MANUFACTURER
1.	Air rotor	Kavo Kerr, India
2.	Surveyor	MARATHON-103, Saeyang company, Korea
3.	Digital scanner	Identica hybrid, Medit co, Korea
4.	CAD-CAM milling unit	Arum 3D Dental solutions
5.	Sand blaster	Tissi Dental
6.	Universal testing machine	INSTRON

2. ARMAMENTARIUM

Color Plate 2 :- Fig. 20,21,22,23,24,25

Miscellaneous armamentariums used were:

- Silicon mixing jar
- Scaler
- Acrylic trimming bur
- Wax knife
- Lacron's carver
- Tooth preparation burs
- Light curing unit
- Test-tube holder
- Milling Unit

- Digital weighing machine

The study was carried out in the following order.

- I. Sample collection.
- II. Mounting of tooth.
- III. Tooth preparation.
- IV. Metal Template fabrication.
- V. Copings fabrication and cementation
- VI. Tensile bond testing of the samples

1. METHOD

i) SAMPLE COLLECTION:- Color Plate 3 – Fig. 26

30 sound, freshly extracted mandibular first molar teeth were collected from the Department of Oral and Maxillofacial Surgery of the institute during the period from 01/02/2018 to 30/04/2018. The teeth with caries, restorations and hypo plastic defects were discarded. Any calculus deposits and soft tissues were removed with hard scaler. They were cleaned of debris by placing them in 3% hydrogen peroxide solution and stored in formalin solution.

ii) MOUNTING OF TOOTH:- Color Plate 3 – Fig. 27,28,29

Commercially available Aluminium channels were cut into desired dimension of (1×1) inch. These mold were used to prepare acrylic block in which the tooth was mounted. The mold was opened on both the ends. To prevent dislodgment of tooth from acrylic, a hole was drilled in the root and a orthodontic wire was looped from the hole. In this way, tooth position was secured in the acrylic mold. Petroleum jelly was applied on all the surfaces of the rectangular mold. Autopolymerising monomer was

dispensed in to the mixing jar, autopolymerising polymer was sprinkled into the monomer until saturation. The mix was stirred thoroughly and poured in the mold which was kept on a platform on which petroleum jelly was applied for separation. An extracted molar was embedded in the autopolymerising resin after initial curing in such a way that the tooth should be 1mm above from the CEJ. The long axis of the tooth was kept perpendicular to the horizontal plane. The excess was trimmed with the lecron's carver. After the complete polymerisation, the tooth along with the acrylic block was separated from the mold. The excess was trimmed with acrylic trimmer. The samples were kept in water. To standardize the tooth preparation, a surveyor was used. Milling unit was attached to the vertical arm of the surveyor which is spring loaded and used to attach the surveying tool. The hole in the milling unit used to hold the air rotor, a test-tube holder was inserted vertically into it. And it was tightened with the tightening screw to avoid movement during use. An air rotor handpiece was inserted horizontally in such a way that the bur was kept parallel to the long axis of the tooth, in to the holding part of the test-tube holder and it was secured using of autopolymerising acrylic. The air rotor handpiece was attached to the compressor for the use.

iii) TOOTH PREPARATION :-

Color Plate 3 – Fig. 30,31 Color Plate 4 – Fig. 32

An acrylic mount (5×5×1) cm was prepared to hold the acrylic block on the surveying table. In the centre portion of the block a space was kept with same dimensions of acrylic block on which the tooth was mounted. The tooth mounted block was snugly fitted into the acrylic block which was used to fix the tooth on the

surveying table. The assembly of acrylic block and the mounted tooth was kept on the surveying table and it was secured with tightening screw. Tapered round end diamond bur was attached to the air rotor handpiece for the tooth preparation. The bur was kept parallel to the long axis of the tooth and was secured in the proper position with the adjusted assembly. Deep chamfer margin was prepared as needed for zirconia restorations. Round end tapered diamond bur was inserted half the depth for axial tooth preparation to get deep chamfers margin and angle of convergence of 6 degrees, as recommended for ideal tooth preparation. The occlusal preparation was kept flat at the height of 5 mm using wheel diamond bur.

The surveying arm was moved during the tooth preparation and the mounted tooth was kept stable.

II) METAL TEMPLETE FABRICATION:-

Color Plate 4 :- Fig. 33,34,35,36,37

To standardize the tooth preparation, bucco-occluso-lingual and mesio-occluso-distal metal templates were fabricated. Wax pattern of the template was made on the molar tooth which was prepared first, using the inlay wax. The pattern of template was such that for bucco-occluso-lingual, it covered the whole surface of buccal side in continuation with occlusal and lingual. Similarly, for mesio-occluso-distal, it covered the whole surface of mesial side in continuation with occlusal and distal. On both the templates, a provision of handle was kept on occlusal surface to hold and check during the preparation. The pattern was invested and casted in usual manner, using nickle-chromium alloy. These metal templates were used as guide during tooth preparation. The remaining teeth were also prepared and everytime the

two metal templates were used to check the tooth preparation. The prepared teeth were stored in water.

III) COPING FABRICATION AND CEMENTATION:-

Color Plate 5 – Fig. 38,39,40,41,42,43

After completion tooth preparation was completed, 30 samples were randomly divided into 3 groups such as,

Group I = 10 samples

Group II = 10 samples

Group III = 10 samples

All the samples were sent to the laboratory for the fabrication of the zirconia copings.

Each prepared tooth was scanned with CAD-CAM machine optical scanner from all the surfaces to get the appropriate digital image of the tooth, which was used to design the full veneer crown. The coping was designed on the image of the prepared tooth over which a loop was also designed with the internal diameter of 3mm. After designing, a programme was set for milling the copings. The milling machine started the process of carving the copings from the zirconia blanks as per the scanned image.

Pre sintered enlarged framework was created. These were then sintered in sintering furnace to get their full strength that was accompanied by shrinkage of the milled framework by 25% to the desired dimensions, as per machine's manual/programme.

The zirconia copings were sandblasted with 110µm aluminium oxide particles in the sandblaster.

These copings were randomly divided as follows:-

Sr. No.	Groups	Description	Samples
1.	Group I (Control)	Zirconia copings luted with resin cement to tooth without using bonding agent.	10
2.	Group II (MDP)	Zirconia copings luted with resin cement to tooth using bonding agent containing MDP i.e, Z Prime Plus	10
3.	Group III (MDP & MPS)	Zirconia copings luted with resin cement to tooth using bonding agent containing combination of MDP and MPS i.e, Monobond Plus	10
Total Samples			30

1. Treatment to the Zirconia coping:- Color Plate 6 – Fig. 44,45

- For Group I samples, no bonding agent was applied.
- For Group II samples, using the applicator tip the bonding agent containing MDP (Z Prime Plus) was applied to the intaglio surface of zirconia as per manufacturer’s instructions.
- For Group III samples, using the applicator tip the bonding agent containing combination of MDP and MPS (Monobond Plus) was

applied to the intaglio surface of zirconia as per manufacturer's instructions.

Excess solvent from bonding agent was removed with gentle, oil free air for 5 seconds.

2. Treatment to the tooth:-

Each prepared tooth was etched, with 37% phosphoric acid which is etchant as well as dentin conditioning agent for 15 seconds, thoroughly rinsed and dried as per manufacturer's instructions. After that each zirconia coping was luted to tooth with the resin cement (Variolink N Dual cure).

FOR CEMENTATION:- Color Plate 6 – Fig. 46,47,48,49

Equal quantity of 2mm base and catalyst paste of resin cement was dispensed on the mixing pad. It was mixed thoroughly for 10 seconds with a plastic spatula in circular motion to form a uniform mixture. Using the cement carrier, uniform paste was carried and applied evenly to the intaglio surface of the zirconia coping. The zirconia coping loaded with cement was luted over the prepared tooth.

This sample was kept on digital weighing machine and TARE button was pressed for zeroing of the weight. Then finger pressure of 1 kg was applied to the coping for 5mins and the weight was seen on the digital screen to monitor exact weight. It was first light cured for 5 seconds and excess was removed from all sides with a clean instrument. Light curing was completed for 40 seconds each on all surfaces.

As zirconia is brittle material, the loop which was made over the coping may break during tensile load. To overcome this problem, the zirconia coping was reinforced with acrylic sparing the margins. The loop of the coping was encapsulated into the acrylic keeping 3mm of internal hole of the loop. All the cemented samples were stored in water till the test was performed.

IV) TENSILE BOND TESTING PROCEDURE:-

Color Plate 8 – Fig. 50,51

After luting procedure, samples were stored in distilled water at room temperature for 24 hrs. All the samples were carried to PRAJ Metallurgy laboratory for the tensile bond testing procedure. The tensile bond test was carried out with the Universal testing machine (Instron U.S.A). The specimens were loaded axially in the universal testing machine. The hook was engaged in the loop of the coping to apply force opposite to the path of insertion. The samples were subjected to tensile stress and removed along the path of insertion using universal testing machine at a cross head speed of 0.5 mm / min and separation force was recorded. The maximum load at the debonding was measured, which was determined on the computer. The tensile bond strength was calculated in Newton.

PLATE I



Fig. 1 Aluminium channel



Fig. 2 Cold-cure polymethyl methacrylate monomer & polymer



Fig.3 Petroleum jelly

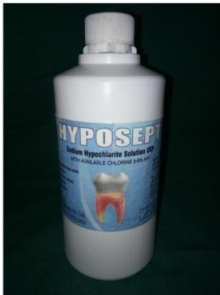


Fig. 4 3% Hydrogen peroxide



Fig 5. Formalin Solution



Fig. 6 Nickel chromium pellets

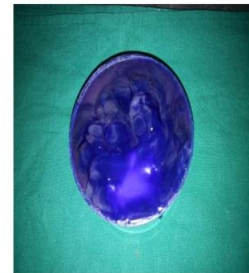


Fig. 7 Inlay wax



Fig. 8 10-methacryloyloxy-decyl dihydrogenphosphate (MDP)(Z prime Plus)



Fig. 9 10-methacryloyloxy-decyl dihydrogenphosphate (MDP) and 3-methacryloxy propyltrimethoxy silane (MPS) (Monobond Plus)



Fig. 10 Zirconia blank (Cercon)



Fig. 11 Etchant (Microgel SF)



Fig. 12 Resin cement (Variolink N Dual cure)



Fig. 13 Aluminium oxide particles

PLATE II



Fig. 14 Air rotor



Fig. 15 Surveyor

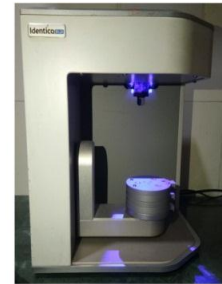


Fig. 16 Digital scanner



Fig. 17 CAD-CAM Milling unit



Fig. 18 Sandblaster



Fig. 19 Universal testing machine



Fig. 20 Miscellaneous armamentarium



Fig. 21 Tooth preparation burs



Fig. 22. Light curing unit



Fig. 23 Test-tube holder



Fig. 24 Milling Unit



Fig. 25 Digital weighing machine

PLATE III



Fig. 26. 30 Mandibular first molars



Fig. 27 Tooth with retention groove and wire



Fig. 28 Tooth mounted in acrylic



Fig. 29 Mounted assembly on surveyor for tooth preparation



Fig.30 Axial preparation with round tapered diamond bur



Fig. 31 Occlusal preparation with wheel diamond bur

PLATE IV



Fig. 32. Prepared tooth

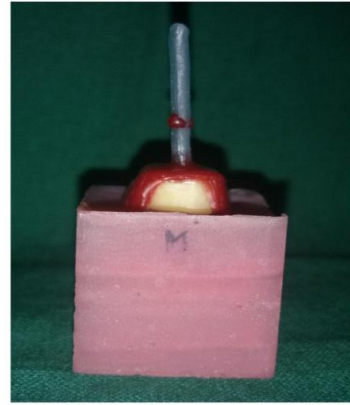


Fig. 33 Wax pattern of bucco-occluso-lingual templete



Fig. 34 Wax pattern of mesio-occluso-distal templete



Fig. 35 Casted bucco-occluso-lingual metal templete



Fig. 37. 30 Prepared mandibular first molars

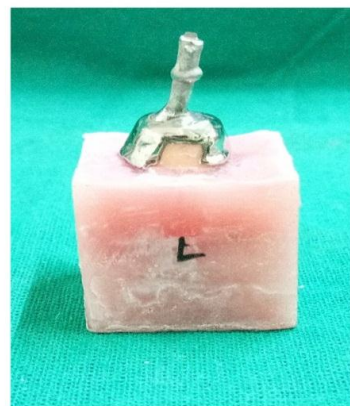


Fig. 36 Casted mesio-occluso-distal metal templete

PLATE V



Fig. 36 Casted mesio-occluso-distal metal template

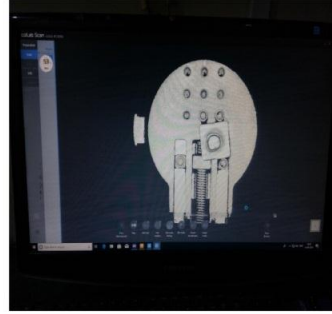


Fig. 39 Scanned image

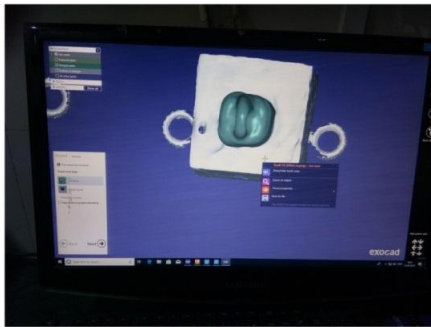


Fig. 40 Digital image of designed coping



Fig. 41 Milling of copings



Fig. 42. 30 Milled copings



Fig. 43 Sand blasting

PLATE VI



Fig. 44 Z Prime Plus Bonding agent application

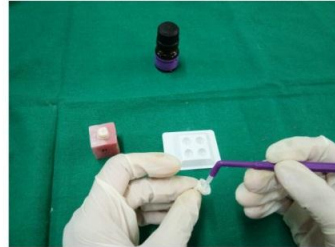


Fig. 45 Monobond Plus bonding agent application



Fig. 46 Application of resin cement

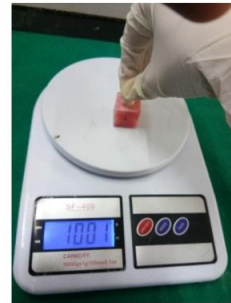


Fig. 47 1 Kg weight



Fig. 48 Zirconia coping reinforced with acrylic



Fig. 49. 30 Samples with acrylic reinforcement



Fig. 50 Retention testing in UTM



Fig. 51 Debonded 30 samples

RESULTS

The result you achieve will be in direct proportion to the effort you apply.

- Denis Waitley.

In this study, tensile bond strength of zirconia and resin cement was evaluated using two different bonding agents.

Standardize tooth preparation was done on 30 mandibular first molars. Zirconia copings were fabricated using CAD-CAM. All the 30 samples were subjected to retention testing on Universal Testing Machine at 0.5 mm/min cross-head speed, a vertical tensile force was applied on the crowns consistently. Retentive force values at which the cemented crowns were dislodged from the prepared teeth were calculated in Newton, on Universal Testing Machine attached with computerized software (Master Chart).

These copings were randomly divided as follows:-

Sr. No.	Groups	Description	Samples
1.	Group I (Control)	Zirconia copings luted with resin cement to tooth without using bonding agent.	10
2.	Group II (MDP)	Zirconia copings luted with resin cement to tooth using bonding agent containing MDP i.e, Z Prime Plus	10
3.	Group III (MDP & MPS)	Zirconia copings luted with resin cement to tooth using bonding agent containing combination of MDP and MPS i.e, Monobond Plus	10
Total Samples			30

STATISTICAL ANALYSIS

Statistical analysis was done with Statistical Package for the Social Sciences (SPSS version 20, IBM, USA). Data comparison was done by applying specific statistical tests to find out the statistical significance of the results. Since the data was of continuous type, parametric tests were used for analysis. Mean and Standard Deviation (SD) were calculated. Statistical tests employed for the obtained data in this study were:

One way Analysis Of Variance (ANOVA) test followed by Pair-wise comparison of three study groups by Bonferroni Multiple Comparison test.

The hypothesis of no difference (Null Hypothesis) was:

H₀: There is statistically no significant difference in retention of zirconia copings luted with resin cement using bonding agent containing 10-methacryloyloxy-decyl dihydrogen-phosphate (MDP) i. e. Z prime plus and zirconia copings and resin cement bonding agent containing combination of 10-methacryloyloxy-decyl dihydrogenphosphate (MDP) and 3-methacryloxy propyltrimethoxy silane (MPS) i.e. Monobond plus.

Alternate hypothesis was:

H₁: There is statistically significant difference in retention of zirconia copings luted with resin cement using bonding agent containing 10-methacryloyloxy-decyl dihydrogen phosphate (MDP) i.e. Z prime plus and zirconia copings and resin cement bonding agent containing combination of 10-methacryloyloxy-decyl di-hydrogen phosphate (MDP) and 3-methacryloxy propyltrimethoxy silane (MPS) i.e Monobond plus.

The formulations and method used in this study are briefly described below:

Mean and Standard deviation

These statistical measures of central tendency and dispersion were obtained for the continuous variables included in the study showed in the Graph. The expressions for the two are given as below:

a) Sample mean for the set of observations was given by

$$\bar{x} = \frac{1}{n} \sum_{i=1}^n x_i$$

Where x_i = observation on each object, n = number of objects

b) Sample standard deviation was given by

$$s = \sqrt{\frac{1}{(n-1)} \sum_{i=1}^n (x_i - \bar{x})^2}$$

ONE WAY ANALYSIS OF VARIANCE: (one way ANOVA)

Analysis of Variance (ANOVA) is a statistical method used to test differences between two or more means. It may seem odd that the technique is called “Analysis of Variance” rather than “Analysis of Means” because inferences about means are made by analyzing variance. ANOVA is used to test general rather than specific differences among means.

$$\text{Variance Ratio (F)} = \frac{\text{Mean square between samples}}{\text{Mean square within samples}}$$

Mean square between samples = sum of squares for variance between the Samples / (k-1)

$$\text{Sum of squares for variance between the samples} = \sum n_i(X_i - \bar{X})^2$$

k- 1 represents degree of freedom

Mean square within samples = sum of squares for variance within the Samples / (n - k)

Sum of squares for variance within the samples

$$= \sum (x_{1i} - \bar{x}_1)^2 + \sum (x_{2i} - \bar{x}_2)^2 + \dots + \sum (x_{ki} - \bar{x}_k)^2$$

n - k = degree of freedom

n = number of items in all samples.

$\bar{x}_1, \bar{x}_2, \dots, \bar{x}_k$ = mean of each sample, when there are k samples.

$\bar{\bar{x}}$ = mean of the sample means.

$i = 1, 2, 3, 4, \dots$

ANOVA is performed as there were more than 2 comparison groups in the study. ANOVA results indicated that overall there is a significant between-the-group difference in mean tensile bond strength across the three comparison groups.

Which one of the mean difference out of three-pair-wise comparisons (**Group I vs II, or I vs III or II vs III**) contributed to the above significant result was established by Bonferroni Multiple Comparison test.

Bonferroni Multiple Comparison test:

A Bonferroni test is a type of multiple comparison test used in statistical analysis. When an experimenter performs enough hypothesis tests, he or she will eventually end up with a result that shows statistical significance of the dependent variable, even if there is none. If a particular test yields correct results 99% of the time, running 100 tests could lead to a false result somewhere in the mix. The Bonferroni test attempts to prevent data from incorrectly appearing to be statistically significant by lowering the alpha value.

The Bonferroni test, also known as the "Bonferroni correction" or "Bonferroni adjustment" suggests that the "p" value for each test must be equal to alpha divided by the number of tests.

To perform a **Bonferroni** correction, divide the critical P value (α) by the number of comparisons being made.

DISCRIPTIVE ANALYSIS:-

Table 1 & 2 gives the descriptive statistics of the mean retention value with standard deviation. The results of the study showed that the mean retention value for **Group I (Control)** was **219.15 N**, for **Group II (MDP)** was **449.13 N**, and for **Group III (MDP+MPS)** was **535.38 N**. Amongst these three groups, the mean retention value of zircona copings luted with resin cement with bonding agent containing (MDP + MPS) i.e Monobond plus (**Group III**) was highest.

Table 3 provides the description of statistical assessment of mean retention values using ANOVA test. Here **Group I (Control)** showed significantly less mean retentive value than **Group II** and **Group III** respectively. Whereas there was no statistically significant difference ($p > 0.05$) between the mean retentive values of **Group II** and **Group III**. Since p-value for the ANOVA was found out to be less than 0.05 ($p < 0.05$); indicates significance of difference between the means of three groups. Hence, the Null Hypothesis H_0 is rejected and the alternate hypothesis H_1 is accepted.

The results also stated that using bonding agent for zirconia coping luted with resin cement significantly increased the retention of zirconia copings. The mean retention value for **Group III (MDP + MPS)** was found out to be higher than **Group II (MDP)**. However the increase in the mean retention value was not statistically significant. So it has been proved that use of bonding agent for zircona coping luted with resin cement on the internal surface significantly increases the retention.

Table 2 gives description of estimated mean tensile bond strength in three study groups along with 95% Confidence Interval (CI). Both the summary measures viz. Mean and standard deviation values of tensile bond strength were showing increasing trend from **Group I** to **Group III** (**Graph 1**). Though the standard deviation (which is a measure of absolute variation) was the lowest for **Group I**, but coefficient of variation (which is a measure of relative variation) was found the lowest for **Group II**, thereby indicating that **Group II** has the least variability among all the three comparison groups. Standard error of the mean was found the lowest in **Group III** indicating sampling error was the least and results are more precise in this group as compared to other two groups (**Graph 2**). Observed (or estimated) mean values obtained from three groups are expressed along with respective 95% CIs. The CIs represent the probability (i.e. 95%) with which true parameter values (i.e. Population mean) would fall in the specified range. Width of the CI depends on the standard error and sample size. Narrower CIs observed here represent good precision of the sample estimates. Three 95% CIs are found non-overlapping with each other thereby indicating that the estimates are significantly different across the three comparison groups (Table 3).

Table 4 shows descriptive analysis of Pair-wise comparison of Mean Tensile bond strength of three study groups by Bonferroni Multiple Comparison test. Results from Bonferroni multiple comparison test suggested the significance of all the three possible pair-wise differences. However, the mean difference in mean tensile bond strength for **Group I versus II** was observed to be **229.98** and **p value of 0.001** which suggest highly significant, the lowest in third pair i.e. **Group II versus III** with the mean difference of **86.25** and **p value of 0.030** which suggest significant. Mean

difference was found the highest in the pair of **Groups I versus III** i.e. **316.22** and **p value of 0.001** highly significant (**Graph 3**). Therefore, mean differences for all the three pairs were found significant.

DISCUSSION

If there are differences of views or divergence of ideas, they can be resolved through discussion and dialogue.

- Azim Premji

The increasing demand for esthetic and durable metal free restorations led to the development of core-strengthening ceramics that are used as frameworks for crowns and fixed dental prostheses³⁷. The yttria-stabilized polycrystalline tetragonal zirconium dioxide (Y-TZP) ceramic is employed wide within the clinic for esthetic restorations, due to its excellent esthetic performance and biocompatibility, additionally as its high strength and fracture toughness, all of that demonstrate its superiority to other all-ceramic materials. Zirconium dioxide is additionally used for partial fixed dental prostheses (PFDP), monolithic crowns within the anterior and posterior regions, and all-ceramic

crowns³⁸. Additionally, the combination of the good mechanical properties of zirconium dioxide ceramics, adhesive cementation techniques, and therefore the introduction of computer-aided design/computer-aided producing (CAD-CAM) technology has paved the means for the fabrication of latest non-invasive treatments for tooth replacement with resin bonded ceramic restorations³⁷.

For the long-term success of esthetic restorations using zirconium dioxide, it's essential to reinforce retention and resistance of the restoration through strong bonding with the tooth structure. The strategies usually used to achieve this outcome include the following: (i) increasing the area of the ceramic to reinforce the bond strength with the tooth structure; (ii) acid etching to form a stronger micro-mechanical interlock; and (iii) the utilization of silane primer to attain chemical bond strength³⁸.

Because of some considerations for adhesive bonding of zirconium oxide and other high strength ceramics to resin cements, conventional cementation strategies using zinc phosphate or polycarboxylate cements are suggested by the manufacturers³⁹. However, adhesive resin cements give durable bonding to zirconium oxide ceramics, increasing retention, fracture strength, and marginal seal of the definitive restoration. Many surface treatments are accustomed improve resin bonding to zirconium oxide ceramic, like airborne-particle abrasion and tribochemical silica coating. However, acid etching can't be performed on zirconium oxide because it doesn't contain glass, and silane primer cannot give sufficient bond strength by itself as no reaction occurs because of the lack of silica in zirconium oxide³⁸.

There are adhesive monomers and metal primers available which will improve chemical adhesion between resin cements and base or noble metals. The adhesive metal primers have an affinity for metallic oxides and bond powerfully to pure metals and alloys. It's additionally possible to use metal primers to increase the bond strength between a metallic oxide, zirconia, and resin cement. **Blatz MB et al (2004)**, **Yang Bet al (2010)**, **Dias de Souza GM et al (2011)**, **Kitayamaa S et al (2010)**, have reported the effectiveness of using metal primers for improvement of the bond durability to zirconia⁴⁰. However, alternative investigators rumoured that none of the bonding concepts they used provided adequate bond strength values, though contradictory results are obtained. Also, the bond durability between resin cements and zirconium dioxide retainers from one aspect and completely different core materials from the opposite aspect is unclear. Therefore, it is necessary to evaluate and compare the bond performance of various resin cement/primer systems to zirconia⁴⁰. In this study, tensile bond strength was compared between zirconia and resin cement using two different bonding agents.

Attia and kern (2011)³⁷ investigated the tensile bond strength of adhesive luting cement to zirconia ceramic after the application of a new universal primer and stated that silica coating or air-borne particle abrasion and primer containing combination of 10-methacryloyloxy-decyl dihydrogenphosphate (MDP) and 3-methacryloxy propyltrimethoxy silane (MPS) improved long term resin bonding to zirconia ceramics.

Similarly, **Baltz MB et al (2004)**¹⁴, stated that bonding/silane coupling agent containing an adhesive phosphate monomer can achieve superior long-term shear

bond strength to airborne-particle-abraded Procera AllZirkon restorations with resin luting agents.

Many factors that influence the retention of full veneer crowns with varied magnitude are very difficult to control and standardize in an in-vitro study. However, all possible efforts were made to standardize the samples used in the study.

Here 30 freshly extracted, sound, non-carious & non restored human mandibular first molar teeth were used. Sample standardization was done by ensuring relatively same dimensions tooth of anatomic crown length 7.5 ± 1 mm, bucco-lingual dimension of 11.0 ± 1 mm & mesio-distal diameter of 11.5 ± 1 mm. Aluminium blocks of 1x1 inch were used for mounting of molars using auto polymerizing acrylic resin. The teeth were mounted in centre and cement-enamel junction was maintained 1 mm above the resin surface. On the root portions of molars, a hole was made and orthodontics wire was looped to the hole to achieve mechanical retention. This was done to facilitate attachment of the specimen to lower member of Universal Testing Machine.

Tooth preparation was done using Air-rotor handpiece mounted on a Dental surveyor. This was performed so that diamond points are held parallel to long axis of the tooth. Occlusal reduction was done using round wheel diamond bur to make the occlusal surface flat. Axial reduction was performed by tapered round ended diamond point so as to have constant taper of 6° and deep chamfer finish line was established. Two metal templates were made, one bucco-occluso-lingual and other mesio-occluso-distal to check each tooth preparation for all samples, to get uniform surface area, retention & resistance form⁴¹.

The prepared teeth were scanned at the lab using a model scanner and copings were designed using the CAD software. The occlusal portion of the copings was designed in such a way, that a vertical extension of 6 mm. with a hole in the centre was made, for engaging the attachment of Universal Testing Machine. The resultant copings were milled in a milling machine using a zirconia disc. The zirconia used was yttria-stabilized. After milling, the copings were subjected to a standard sintering cycle of 7½ h as per manufacturer's instructions. After sintering, the copings were checked for the accuracy of fit.

Sand blasting was done with the 110µ aluminium oxide particles for 21 seconds at 0.2 MPa. Sandblasting increases the surface area for bonding and decreases the surface tension, which allows a better wetting by the luting agent⁴². 30 zirconia copings were randomly divided into 3 groups, 10 samples in each group.

Each prepared tooth was etched, with 37% phosphoric acid which is etchant as well as dentin conditioning agent for 15 seconds, thoroughly rinsed and dried as per manufacturer's instructions. Resin cements mainly adhere to the tooth structure through micromechanical retention. To achieve this micromechanical retention, the usual adhesive steps of etching, priming, and bonding should be performed on the enamel and dentin to form a stable hybrid layer⁴³.

Group I was the control group where the samples were luted with resin cement without any bonding agent. Group II & III were the experimental groups where the samples were luted with resin cement with 2 different bonding agents. The bonding agent which was used for group II was 10-methacryloyloxy-decyl dihydrogenphosphate (MDP) i.e. Z Prime Plus and for group III was combination of

10-methacryloyloxy-decyl dihydrogenphosphate (MDP) and 3-methacryloxy propyltrimethoxy silane (MPS) i.e. Monobond Plus.

Dual cure resin cement was used as it cures by both light-cure as well as self-cure. The initial set is usually achieved with light curing to quickly seal the gingival margins. The self-curing component ensures that the cement will cure underneath restorations that are too thick or too opaque to allow transmission of light through it⁴³.

Luting agent was mixed according to manufacturer's instructions and a constant seating Force of 1 kg was maintained during cementation to standardize the pressure for cementing all copings. Generally, removing of cemented crowns by pulling them along the long axis of preparation with an increasing statically loaded model is the measure of the retentive strength. On Universal Testing Machine, the loop of zirconia copings was engaged to upper jaw and the lower jaw grasped the acrylic block having cemented specimen. The maximum load required to dislodge the crown was recorded in Newton on Universal Testing Machine, attached with computerized software.

After statistical analysis, the results of the present study showed that the mean retention value for **Group I (Control)** was found **219.15 N**, for **Group II (MDP)** it was found **449.13 N**, and for **Group III (MDP+MPS)** it was **535.38 N**. The results stated that using bonding agent for zirconia coping luted with resin cement significantly increased the retention of zirconia copings. Hence, the Null Hypothesis was rejected for **Group I-II** and **Group I-III** but it was accepted for **Group II - III**.

The mean difference in mean tensile bond strength for **Group I versus II** was observed **229.98** and **p value of 0.001** which suggest highly significant, the lowest in third pair i.e. **Group II versus III** with the mean difference of **86.25** and **p value of 0.030** suggesting significant values. Whereas mean difference was found the highest in the pair of **Groups I versus III** i.e. **316.22** and **p value of 0.001** highly significant. Therefore, the mean retention value for Monobond Plus bonding agent (**Group III**) was found out higher than that of Z Prime Plus bonding agent (**Group II**). Thus, reinforced concept of using bonding agent for zirconia coping, luted with resin cement on the internal surface of full veneer crowns showed significantly increased retention.

These results were similar to the study of **Tamis M et al (2015)** where they concluded that when an MDP-containing silane primer is applied after sandblasting to increase the surface roughness; this is because the primer binds directly to hydroxyl groups on the zirconia surface and enhances the stability against hydrolysis, which in turn improves bond strength with an adhesive⁴⁴.

The results were also in accordance with **Al-Harbi et al (2015)** who in their study concluded that the tensile bond strength of Y-TZP ceramics to the core material was more with primer containing combination of silane and phosphate monomer⁴⁰.

In **(2016) Byeon S** studied the effects of 3 mol % yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP) ceramic surface treatments on the tensile bond strength and surface characteristics of enamel. He concluded that surface treatment with 110 µm alumina sandblasting and MDP-containing silane primer is suitable for clinical

applications, as it considerably improves the bond strength between 3Y-TZP and enamel³⁸.

The cement is a bridge between the tooth and the restoration. Whereas tooth bonding procedures make sure that the cement adheres well to the tooth, pre-treatment of the internal surface of the restoration ensures that the cement can adhere to the restoration as well. A good adhesion to the interior surface of the restoration needs (1) roughening of the interior surface of the restoration to extend the extent for bonding and (2) increasing the wettability of the cement to the restoration and forming chemical bonds between the restoration and therefore the cement⁴³.

Non-silica-based ceramics like alumina and zirconia have crystalline phase and will not be etched as they're extremely resistant to chemical attack from HF. The popular pre-treatments for alumina or aluminium oxide ceramics embrace (1) airborne abrasion with 50–110µm aluminium oxide particles at 2.5 bars, (2) use of an MDP-containing resin cement (3) silica coating through tribochemical surface followed by application of a conventional bis-GMA resin cement. Resin cements and primers containing the acidic monomer 10-MDP are the suggested cements for zirconia ceramics as MDP will chemically bond with zirconia⁴³. Without using the bonding agent between the restoration and resin cement, there will not be any chemical bond between the two. Lack of such chemical bond could be the reason resulting in less retentive force for de-bonding of the restoration as seen in **Group I (Control)**.

Group II (MDP), chemically, this monomer bonds to oxide metals and tooth substrates. It's an amphiphilic structure with the vinyl group because the

hydrophobic half and therefore the phosphate group because the hydrophilic half. The 10-MDP monomer is an efficient bonding agent between the organic compound cement, zirconia, and alternative metal oxide materials. The interaction of hydroxyl groups of the phosphate half with the hydroxyl groups on the zirconia surface through van der Waals forces or hydrogen bonds could be reason for acceptable retentive bond between the zirconia copings and resin cement⁵.

Group III (MDP+MPS), the addition of a MDP-containing bonding/silane coupling agent to enhance bonding of MDP resin cements has produced positive results. It was shown that particle air-abrasion or tribochemical coating, followed by the application of MDP-containing bonding/silane coupling agent, resulted in increased retentive bond strength compared to MDP-containing cements only. It is known that acidic monomers rapidly hydrolyze silane coupling agents, producing the siloxane bonds necessary for chemical bonding. It is thought that the acidic nature of MDP enhances the polysiloxane bonding produced by silane coupling agents and results in improved retention of resin cements to ZrO_2 ¹.

Clinical Implications:-

The use of bonding agent is suggested between resin cement and zirconia for chemical bond between them. Without any chemical bond the restoration will fail. The present study was performed on the molar teeth with standardized crown height, suggests the use of both the bonding agents i.e. MDP and MDP+MPS as there is no statistically significant difference between the results of the two. So, in normal clinical situations, where the full veneer zirconia crowns are suggested, either of the two bonding agents can be used for their cementation.

Retention of full veneer crown is mainly dependent on sufficient prepared tooth surface. Clinical situations, where the crown height is less especially in molars, could not provide sufficient retention form to the restorations, after cementation. Therefore use of bonding agents containing silane coupling agent and an adhesive phosphate monomer to enhance resin bond to sandblasted surface of zirconia is recommended.

Limitations of the study:-

There were some limitations in this study, being an in-vitro study it could not replicate all the conditions present in the oral environment. The dislodging forces to which a fixed crown is subjected in the oral cavity are multidirectional may it be vertical, lateral or oblique, whereas the dislodging force exerted by the Universal Testing Machine is unidirectional. So, a direct comparison between dislodging forces encountered in the oral cavity and those exerted by the universal testing machine is ambiguous.

Scope for further studies:-

1. Other parameters in tensile testing like the thermal cycling or aging can be considered to mimic the oral conditions.
2. Bonding of zirconia laminate luted with resin cement using different bonding agents.
3. SEM analysis can also be done using similar parameters.

SUMMARY

Perhaps the best test of a man's intelligence is his capacity of making the summary.

- Lytton Starychey.

The present investigation was done with an aim to evaluate and compare the tensile bond strength of zirconia and resin cement using two different bonding agents.

Bonding of zirconia to tooth had been studied in the literature but still is controversial. Because zirconia does not react to acid etching, and lack of silica particles in zirconia prevents the use of silane.

Different bonding agents – Z Prime Plus and Monobond Plus were chosen. For the purpose of the investigation, 30 freshly extracted, sound, non-carious & non-restored human mandibular first molar teeth were mounted on auto polymerizing acrylic resin block. Standardized tooth preparation for full veneer crown was

accomplished using Airotor handpiece mounted on a Dental Surveyor. Each prepared tooth was scanned with CAD/CAM machine optical scanner. 30 zirconia copings were milled.

These copings were randomly divided into 3 Groups of 10 samples in each group. Zirconia copings luted with resin cement to tooth without using bonding agent (Group I- Control). Zirconia copings luted with resin cement using bonding agent containing 10-methacryloyloxy-decyl di-hydrogenphosphate (Group II- MDP) i.e. Z prime plus and Zirconia copings and resin cement bonding agent containing combination of 10-methacryloyloxy-decyl dihydrogenphosphate (MDP) and 3-methacryloxy propyltrimethoxy silane (MPS) Group III (MDP & MPS) i.e. Monobond plus. Retention testing was done on universal testing machine at cross head speed of 0.5 mm/min. and the maximal tensile force used to separate the crown was recorded in Newton.

Statistical analysis was done to calculate Mean and Standard Deviation (SD). One way analysis of variance (ANOVA) was performed to compare mean values between three groups. The results of the study showed that the mean retention value for **Group I (Control)** was **219.15 N**, for **Group II 449.13 N**, and for **Group III 535.38 N**.

The result showed that the use of bonding agent significantly increased the retention of zirconia copings. Further, pair wise comparison between different groups was done using Bonferroni Multiple Comparison test. The mean difference in mean tensile bond strength for **Group I versus II** was observed to be **229.98** and **p value of 0.001** which suggest highly significant, the lowest in third pair i.e. **Group II versus**

III with the mean difference of **86.25** and **p value of 0.030** which suggest significant. Mean difference was found the highest in the pair of **Groups I versus III** i.e. **316.22** and **p value of 0.001** highly significant.

The result also revealed that the use of bonding agent significantly improves the tensile bond strength between zirconia and resin cement. The use of bonding agent to lute the zirconia crown to tooth with resin cement was found satisfactory method to improve retention.

CONCLUSION

A conclusion is simply the place where you got tired of thinking.

— Dan Chaon

The aim of the study was to evaluate and compare the tensile bond strength of zirconia and resin cement using two different bonding agents.

Within the limitation of the study, following conclusions can be drawn:

1. Bonding agent is essential for retention of zirconia copings to the natural tooth.
2. The retention of zirconia coping increases when bonding agent containing 10-methacryloyloxy-decyl dihydrogenphosphate (MDP) i.e. Z prime plus is used.

3. The retention of zirconia coping significantly increases when bonding agent containing combination of 10-methacryloyloxy-decyl dihydrogenphosphate (MDP) and 3-methacryloxy propyltrimethoxy silane (MPS) i.e. Monobond plus is used.

4. Between the two bonding agents used in the study, namely: 10-methacryloyloxy-decyl dihydrogenphosphate (MDP) i.e. Z prime plus and bonding agent containing combination of 10-methacryloyloxy-decyl dihydrogenphosphate (MDP) and 3-methacryloxy propyltrimethoxy silane (MPS) i.e. Monobond plus, the tensile bond strength of zirconia and resin cement was highest with Monobond plus.

BIBLIOGRAPHY

1. Thompson JY, Stoner BR, Piascik JR, Smith R. Adhesion/ cementation to zirconia and other non-silicate ceramics: where are we now? *Dent Mater.* 2011 Jan;27(1):71-82.
2. Özkurt-Kayahan Z. Monolithic zirconia: A review of the literature. *Biomedical Research* 2016; 27 (4): 1427-1436.
3. Gowida MA, and Aboushelib MN. Bonding to Zirconia (A Systematic Review). *J Dental Sci* 2016, 1(1): 000102.
4. Oba Y, Koizumi H, Nakayama D, Ishii T, Akazawa N, Matsumura H. Effect of silane and phosphate primers on the adhesive performance of a tri-n-butylborane initiated luting agent bonded to zirconia. *Dent Mater J.* 2014 ;33(2):226-32.

5. Llerena-Icochea AE, Costa RM, Borges A, Bombonatti J, Furuse AY. Bonding Polycrystalline Zirconia With 10-MDP-containing Adhesives. *Oper Dent*. 2017 May/Jun;42(3):335-341.
6. Anusavice K, Phillips R, Shen C, Rawls H. Phillips' science of dental materials. 11th ed. Elsevier; 456-458.
7. Ohno H, Araki Y, Sagara M. The adhesion mechanism of dental adhesive resin to the alloy-relationship between Co-Cr alloy surface structure analyzed by ESCA and bonding strength of adhesive resin. *Dent Mater J*. 1986 Jun;5(1):46-65.
8. Gates WD, Diaz-Arnold AM, Aquilino SA, Ryther JS. Comparison of the adhesive strength of a BIS-GMA cement to tin-plated and non-tin-plated alloys. *J Prosthet Dent*. 1993 Jan;69(1):12-6.
9. Salonga JP, Matsumura H, Yasuda K, Yamabe Y. Bond strength of adhesive resin to three nickelchromium alloys with varying chromimcontent. *J Prosthet Dent*. 1994 Dec;72(6):582-4.
10. Kern M, Thompson VP. Bonding to glass infiltrated alumina ceramic: adhesive methods and their durability. *J Prosthet Dent*. 1995 Mar;73(3):240-9.
11. Kern M, Wegner SM. Bonding to zirconia ceramic: adhesion methods and their durability. *Dent Mater*. 1998 Jan;14(1):64-71.

12. Yoshida K, Kamada K, Atsuta M. Adhesive primers for bonding cobalt-chromium alloy to resin. *J Oral Rehabil.* 1999 Jun;26(6):475-8.
13. Nakamura S, Yoshida K, Kamada K, Atsuta M. Bonding between resin luting cement and glass infiltrated alumina-reinforced ceramics with silane coupling agent. *J Oral Rehabil.* 2004 Aug;31(8):785-9.
14. Blatz MB, Sadan A, Martin J, Lang B. In vitro evaluation of shear bond strengths of resin to densely-sintered high purity zirconium oxide ceramic after long-term storage and thermal cycling. *J Prosthet Dent.* 2004 Apr;91(4):356-62.
15. Valandro LF, Della Bona A, Antonio Bottino M, Neisser MP. The effect of ceramic surface treatment on bonding to densely sintered alumina ceramic. *J Prosthet Dent.* 2005 Mar;93(3):253-9.
16. Yoshida K, Tsuo Y, Atsuta M. Bonding of dual-cured resin cement to zirconia ceramic using phosphate acid ester monomer and zirconate coupler. *J Biomed Mater Res B Appl Biomater.* 2006 Apr;77(1):28-33.
17. Atsu SS, Kilicarslan MA, Kucukesmen HC, Aka PS. Effect of zirconium-oxide ceramic surface treatments on the bond strength to adhesive resin. *J Prosthet Dent.* 2006 Jun;95(6):430-6.
18. Matinlinna JP, Heikkinen T, Ozcan M, Lassila LV, Vallittu PK. Evaluation of resin adhesion to zirconia ceramic using some organosilanes. *Dent Mater.* 2006 Sep;22(9):824-31.

19. Wolfart M, Lehmann F, Wolfart S, Kern M. Durability of the resin bond strength to zirconia ceramic after using different surface conditioning methods. *Dent Mater.* 2007 Jan;23(1):45-50.
20. Senyilmaz DP, Palin WM, Shortall AC, Burke FJ. The effect of surface preparation and luting agent on bond strength to a zirconium-based ceramic. *Oper Dent.* 2007 Nov-Dec;32(6):623-30.
21. Tanaka R, Fujishima A, Shibata Y, Manabe A, Miyazaki T. Cooperation of phosphate monomer and silica modification on zirconia. *J Dent Res.* 2008 Jul;87(7):666-70.
22. Ozcan M, Nijhuis H, Valandro LF. Effect of various surface conditioning methods on the adhesion of dualcure resin cement with MDP functional monomer to zirconia after thermal aging. *Dent Mater J.* 2008 Jan;27(1):99-104.
23. Cavalcanti AN, Foxton RM, Watson TF, Oliveira MT, Giannini M, Marchi GM. Bond strength of resin cements to a zirconia ceramic with different surface treatments. *Oper Dent.* 2009 May-Jun;34(3):280-7.
24. De Oyagüe RC, Monticelli F, Toledano M, Osorio E, Ferrari M, Osorio R. Influence of surface treatments and resin cement selection on bonding to dense ly-sintered zirconium-oxide ceramic. *Dent Mater.* 2009 Feb;25(2):172-9.

25. Mirmohammadi H, Aboushelib MN, Salameh Z, Feilzer AJ, Kleverlaan CJ. Innovations in bonding to zirconia based ceramics: Part III. Phosphate monomer resin cements. *Dent Mater.* 2010 Aug;26(8):786-92.
26. Kitayama S, Nikaido T, Takahashi R, Zhu L, Ikeda M, Foxton RM, Sadr A, Tagami J. Effect of primer treatment on bonding of resin cements to zirconia ceramic. *Dent Mater.* 2010 May;26(5):426-32.
27. Magne P, Paranhos MP, Burnett LH Jr. New zirconia primer improves bond strength of resin-based cements. *Dent Mater.* 2010 Apr;26(4):345-52.
28. Yun JY, Ha SR, Lee JB, Kim SH. Effect of sandblasting and various metal primers on the shear bond strength of resin cement to Y-TZP ceramic. *Dent Mater.* 2010 Jul;26(7):650-8.
29. Behr M, Proff P, Kolbeck C, Langrieger S, Kunze J, Handel G, Rosentritt M. The bond strength of the resin-to-zirconia interface using different bonding concepts. *J Mech Behav Biomed Mater.* 2011 Jan;4(1):2-8.
30. Chen C, Kleverlaan CJ, Feilzer AJ. Effect of an experimental zirconia-silica coating technique on micro tensile bond strength of zirconia in different priming conditions. *Dent Mater.* 2012 Aug;28(8):e127-34.
31. Chen L, Shen H, Suh BI. Effect of incorporating BisGMA resin on the bonding properties of silane and zirconia primers. *J Prosthet Dent.* 2013 Nov;110(5):402-7.

32. Seabra B, Arantes-Oliveira S, Portugal J. Influence of multimode universal adhesives and zirconia primer application techniques on zirconia repair. *J Prosthet Dent*. 2014 Aug;112(2):182-7.
33. Xie H, Li Q, Zhang F, Lu Y, Tay FR, Qian M, Chen C. Comparison of resin bonding improvements to zirconia between one-bottle universal adhesives and tribochemical silica coating, which is better? *Dent Mater*. 2016 Mar;32(3):403-11.
34. Pilo R, Kaitsas V, Zinelis S, Eliades G. Interaction of zirconia primers with yttria-stabilized zirconia surfaces. *Dent Mater*. 2016 Mar;32(3):353-62.
35. Alves M, Campos F, Bergoli CD, Bottino MA, Özcan M, Souza R. Effect of Adhesive Cementation Strategies on the Bonding of Y-TZP to Human Dentin. *Oper Dent*. 2016 May-Jun;41(3):276-83.
36. Zafar A, Bangar B, Kamble S, Agroya P. Perfectly bonded zirconia : clarifying confusion. *University J Dent Scie* 2017;3(2):4-8.
37. Attia A, Kern M. Long term resin bonding to zirconia ceramic with a new universal primer. *J Prosthet Dent*. 2011 Nov;106(5):319-27
38. Byeon SM, Jang YS, Lee MH, Bae TS Improvement in the Tensile Bond Strength between 3YTZP Ceramic and Enamel by Surface Treatments. *Materials (Basel)*. 2016 Aug 18;9(8).
39. Mahmoodi N, Hooshmand T, Heidari S, Khoshro K. Effect of sandblasting, silica coating, and laser treatment on the

- microtensile bond strength of a dental zirconia ceramic to resin cements. *Lasers Med Sci.* 2016 Feb;31(2):205-11.
40. Al-Harbi FA, Ayad NM, Khan ZA, Mahrous AA, Morgano SM. In vitro shear bond strength of Y-TZP ceramic to different core materials with the use of three primer/resin cement systems. *J Prosthet Dent.* 2016 Jan; 115(1):84-9.
41. A. Taneja, M. Subash, M. Dayalan, K.R. Nagaraj. Comparative evaluation of tensile bond strength of surface treated zirconia copings luted with a resin cement - An In vitro study. *Research Journal of Pharmaceutical, Biological and Chemical Sciences.* 2014 jan;5(3):1028-1044
42. Su N, Yue L, Liao Y, Liu W, Zhang H, Li X, Wang H, Shen J. The effect of various sandblasting conditions on surface changes of dental zirconia and shear bond strength between zirconia core and indirect composite resin. *J Adv Prosthodont.* 2015 Dec;7(6):506.
43. Sunico-Segarra M, Segarra A. *A Practical Clinical Guide to Resin Cements.* springer; 9 – 22
44. Tanış MÇ, Akay C, Karakış D. Resin cementation of zirconia ceramics with different bonding agents. *Biotechnol Biotechnol Equip.* 2015 Mar 4;29(2):363-367.

Tables and Graphs:-

Table 1: Mean tensile bond strength in three study groups along with different measures of variation

Groups	n	Mean	Standard Deviation	Standard error of Mean	Coefficient of Variation (%)
I	10	219.15	52.00	16.44	23.73
II	10	449.13	63.52	20.09	14.14
III	10	535.38	88.08	27.85	16.45

Table 2: Estimated Mean Tensile bond strength in three study groups along with 95% Confidence Interval (CI)

Groups	n	Mean	95% Confidence Interval	
I	10	219.15	183.46	254.84
II	10	449.13	405.54	492.72
III	10	535.38	474.93	595.82

Table 3: Comparison of Mean Tensile bond strength across three study groups by one-way ANOVA (Analysis of Variance)

Source	Sum of Squares	Degrees of Freedom	Mean Sum of Squares	F-value	P-value
Between groups	534420.5	2.00	267210.25	55.30	0.001**
Within groups	130467.3	27.00	4832.12		
Total	664887.8	29.00	22927.16		

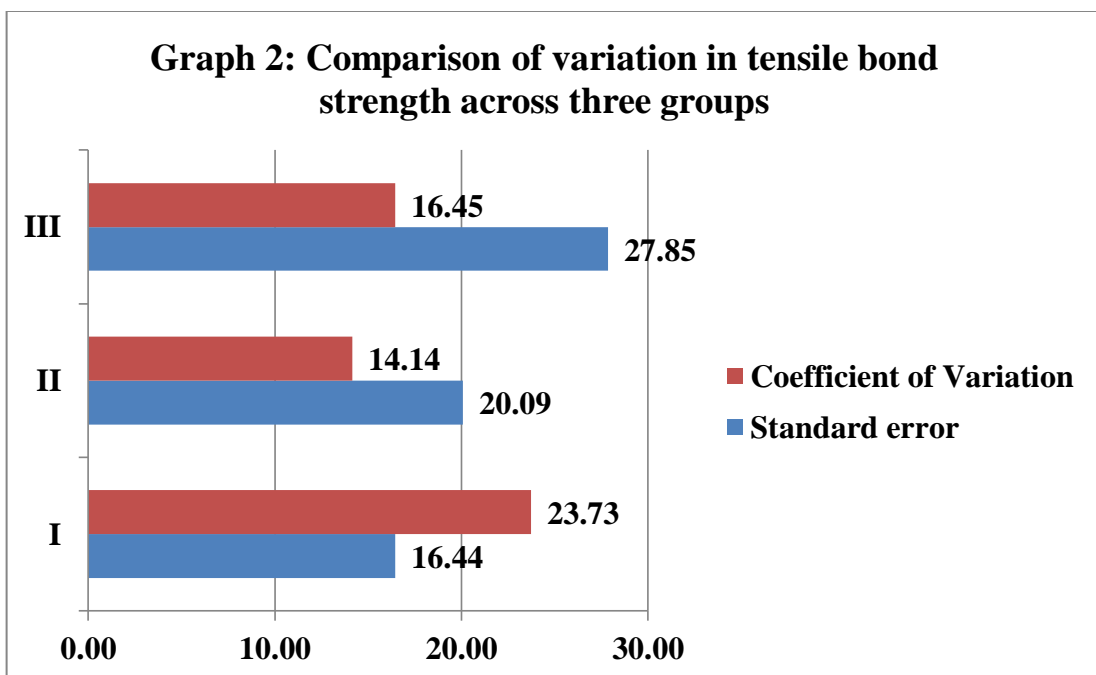
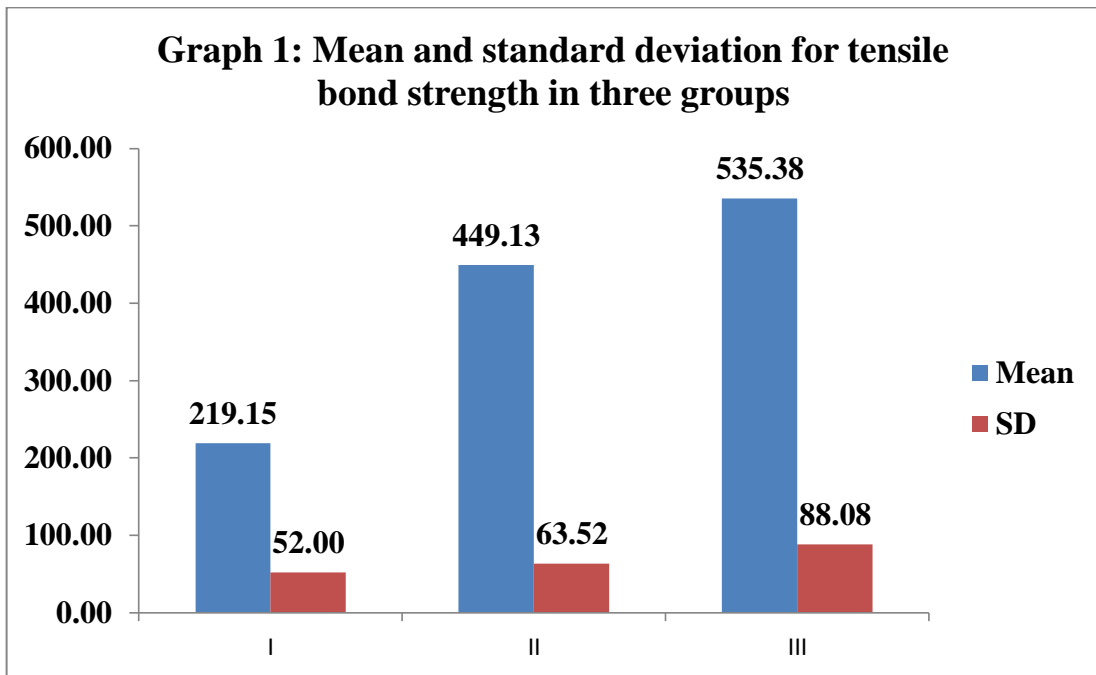
** Highly Significant

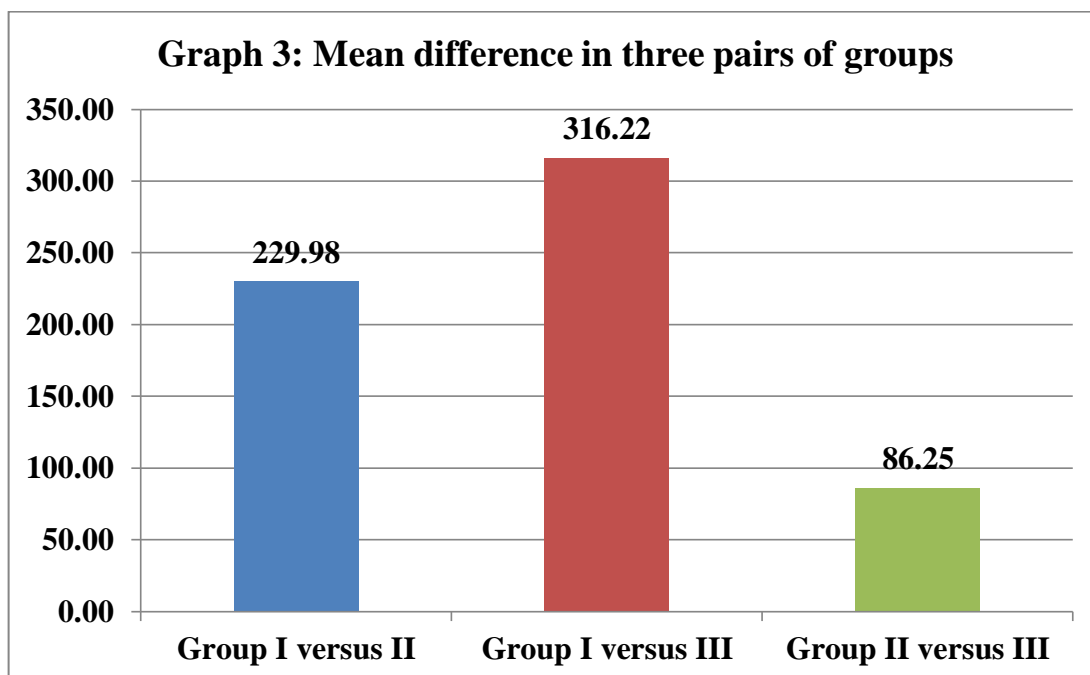
Table 4: Pair-wise comparison of Mean Tensile bond strength of three study groups by Bonferroni Multiple Comparison test

Group-wise Pairs	Means of two comparison groups	Mean difference (95% CI)	P-value
Group I versus II	219.15 versus 449.13	229.98 (175.44 – 284.52)	0.001**
Group I versus III	219.15 versus 535.38	316.22 (248.28 – 384.18)	0.001**
Group II versus III	449.13 versus 535.38	86.25 (14.11 – 158.39)	0.030*

** Highly Significant

*Significant





ANNEXURE
MASTER CHART

Tensile bond strength of 10 samples included in three study groups

GROUP : I			GROUP : II			GROUP : III		
Sr. No.	Sample No.	Tensile Bond Strength (N)	Sr. No.	Sample No.	Tensile Bond Strength (N)	Sr. No.	Sample No.	Tensile Bond Strength (N)
1	No.1	211.9	1	No.1	323.45	1	No.1	619.5
2	No.2	164.2	2	No.2	499.95	2	No.2	438.65
3	No.3	134.1	3	No.3	425.2	3	No.3	537.05
4	No.4	245.25	4	No.4	517.05	4	No.4	566.55
5	No.5	216.3	5	No.5	480.4	5	No.5	539.75
6	No.6	281.55	6	No.6	457.55	6	No.6	458.8
7	No.7	160.25	7	No.7	416.35	7	No.7	582.6
8	No.8	281.75	8	No.8	526.1	8	No.8	410.75
9	No.9	261.98	9	No.9	382.05	9	No.9	499.05
10	No.10	234.23	10	No.10	463.2	10	No.10	701.05
Minimum		134.10			323.45			410.75
Maximum		281.75			526.10			701.05
Range		147.65			202.65			290.30