

# *EVALUATION OF AN INDIGENOUS DUAL CURE RESIN CEMENT*

*PROJECT REPORT SUBMITTED BY*

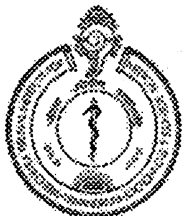
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**CERTIFICATE**

This is to certify that Dr. Pradeep CD, MDS (Prosthodontics) carried out his 1 year postdoctoral project work under my guidance on Evaluation of an indigenously developed dual cure resin cement in Dental Products Laboratory, Biomedical Technology Wing, Sree Chitra Tirunal Institute for Medical Sciences & Technology, Trivandrum during Jan - Dec 2005 and this dissertation is in partial fulfillment of that study

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## LIST OF TABLES

Table I Determination of film thickness

Table II Determination of flexural strength after 24 hours

Table III Determination of flexural strength after 7 days

Table IV Determination of diametral tensile strength of DCRC

Table V Mean shear bond strength with bonding agent

Table VI Mean shear bond strength without bonding

Table VII Statistical analysis of mean bond strength with and without bonding agent using ANOVA

Table VIII Inter comparison of mean bond strength of resin cements without bonding agent-using ANOVA

Table IX Retentive strength of Ni Cr alloy crowns luted with DCRC

Table X Retentive strength of Ni Cr alloy crowns luted with Rely X ARC

Table XI Weight of samples for Water sorption and solubility test

Table XII Water sorption and solubility for DCRC

Table XIII Magnusson and Kligman Scale for Maximization test for delayed hypersensitivity

Table XIV Scores for skin reaction in maximization test for delayed hypersensitivity

Table XV Irritation Score for Intracutaneous intradermal reactivity test

Table XVI Observation for Intracutaneous intradermal reactivity test

## LIST OF FIGURES

Fig I CHITRA DUAL CURE RESIN CEMENT (DCRC)

FIG II FS samples on the jig

FIG III Three point bend test for FS

FIG IV DTS samples and mould

FIG V Test for DTS

FIG VI Brackets attached to tooth surface

FIG VII Shear pull test

FIG VIII Tooth preparation

FIG IX Prepared tooth

FIG X Crown cemented on tooth

FIG XI Test for adhesive strength

FIG XII Dental Thermo cycler

FIG XIII Micro leakage of Test Material

FIG XIV Micro leakage of Control Material

FIG XV Samples for water sorption & solubility

FIG XVI Samples in dessicator

FIG XVII Samples in incubator

FIG XVIII Analytical balance

FIG XIX Sub cutaneous implantation in rats

FIG XX Intra dermal reactivity test in rabbit

FIG XXI Sensitization test in Guinea pigs

FIG XXII Pulp and dentine usage test in pigs

FIG XXIII Graph showing mean shear bond strength

FIG XXIV Graph showing mean adhesive strength

# *INTRODUCTION*

## INTRODUCTION

Resin cements have been used in dentistry for more than three decades<sup>1</sup>. Most of the metal free ceramic restorations are currently being cemented using resin cements. Other applications of resin cements include cementation of orthodontic brackets, resin bonded fixed partial dentures or Maryland bridges, periodontal splints, porcelain veneers, cementation of metallic or non-metallic endodontic posts. Resin bonded fixed partial dentures or Maryland bridges gave superior retention when resin cement was used for luting purpose. Cementation of ceramic veneers, laminates, fixed partial dentures, inlays and onlays have superior retention when cemented with dual cure resin cements.

Resin cements brought forward a new avenue in luting dental restorations. They adhere to tooth structure by presence of a hybrid layer, an intermediate zone obtained by impregnation diffusion and monomer polymerization into etched dentine<sup>2</sup>. Thus the cement promotes bonding to dentine differently when compared with other cements, and has demonstrated superiority in tensile bond strength compared to other luting agents.

Resin cements are basically polymer based cements<sup>3</sup>, the majority of materials in this group being of two types (1) Materials based on methyl methacrylate and (2) Materials based on aromatic dimethacrylates such as BIS-GMA. The properties of acrylic resin cement are comparable to cold curing acrylic resin filling material. They are stronger and less soluble than other type of cements and display low rigidity and viscoelastic property. They have no effective bond to tooth structure in presence of moisture and permits microleakage. The dimethacrylate cements are based on BIS-GMA system. They are combination of an aromatic dimethacrylate with other monomers containing various amounts of filler. They are basically similar to composite restorative materials

The advent of adhesive cements like polycarboxylate and glass ionomer came with the introduction of polyalkanoic acid<sup>4,5</sup>. These cements have chemical adhesion to tooth structure and affinity for metal ions and fluoride release. They are weak in their tensile strength, compressive and

shear strength and are susceptible to failure<sup>6</sup>. This led to the introduction of resin modified glass ionomer cement in 1988 which was a breakthrough in the field of adhesive dentistry. This material resulted in hybridization between glass ionomer and resin system and had many of the advantages of the parent material<sup>7,8</sup>.

## **Classification**

The resin cements are classified according to the type of setting as

**Chemically cured (auto cured):** A two paste system that sets by chemical means. These are used to cement opaque or metal restorations

**Dual cure:** These cements start curing with visible light (440-480 nm) and continue with chemical curing. These are used to cement translucent restorations. Dual cure resin cement achieves only a portion of their polymerization from chemical curing and therefore adequate light exposure is required to achieve complete polymerization.

**Light cured:** These types sets on exposure to light and mainly used to cement thin ceramic veneers.

## **COMPOSITION**

Composition of many of the resin cements is similar to that of resin based composite filling material, but has a low level filler concentration. The tooth surface that comes in contact with resin cement is usually the dentine. So monomers with functional group that are able to induce bonding to dentine are incorporated in it. These include Organophosphates, Hydroxyethyl methacrylate (HEMA) and 4-methacrylethyltrimellitic anhydride (4-META). The two major components required for the preparation of resin cement are resin matrix and silane treated inorganic fillers.

**Resin matrix:** The resin matrix forms a continuous phase in which filler particles are dispersed. Two types of resin matrices are used a) one based on methyl methacrylate and other based on aliphatic or aromatic dimethacrylates such as BIS-GMA, triethyleneglycol dimethacrylate (TEGDMA) and urethane dimethacrylates (UDMA). Presently BIS-GMA is most commonly used as it gives better physical properties, low polymerization shrinkage and therefore reduces marginal leakage which is

highly essential for luting and restorative applications. The disadvantages include its low solubility and high viscosity which makes blending the other components difficult.

**Fillers:** The addition of inorganic filler to resin matrix was introduced in 1950<sup>3</sup>. Fillers reduce the coefficient of thermal expansion and polymerization shrinkage and help to improve the mechanical properties. Resin cements usually contain lower amounts of filler content and are easily flowable. So the effect of fillers upon the above properties will be relatively reduced compared to resin composite. The presence of fillers improves the mechanical properties such as compressive strength, tensile strength, makes cement wear and abrasion resistant and reduces water sorption. In order to achieve translucency similar to tooth, filler particles having refractive index similar to that of resin is used. Commercially used fillers include quartz, silica, and glass. Radiopacity is provided by glass and ceramics containing Barium, Zirconium, or Strontium. The particle size of filler ranges from 100 nanometers to 40 micrometers. The filler used must be resistant to oral fluids, colorless and non-toxic.

### **ACTIVATOR – INITIATOR SYSTEM**

Initiators generate free radical which initiates the additional polymerization reaction. The commonly used free radical initiators are benzoyl peroxide and camphorquinone. Accelerators help to accelerate the formation of free radicals and speed up polymerization. Tertiary amines such as N, N-dimethyl-p-toluidine, dimethylamino phenethyl alcohol etc are used as accelerators.

### **INHIBITORS**

Inhibitor prevents spontaneous polymerization of monomers and also improves shelf life. Commonly used inhibitors include hydroquinone (HQ) and butylated hydroxy toluene (BHT)

### **COUPLING AGENTS**

The organic resin and inorganic filler has to be strongly bonded. Weak bonding results in dislodgement of the filler particles from the resin matrix. Coupling agent provides strong bonding

improves the physical and mechanical properties. 3-trimethoxyvinylpropylsilane is normally used as the coupling agent.

### **DILUENTS**

The high viscosity of BISGMA resin can be reduced by adding other monomers having low viscosities. Dimethacrylate monomers such as TEGDMA, EDMA etc can be used to reduce the viscosity of BISGMA.

### **ADVANTAGES OF RESIN CEMENTS**

Aesthetic, particularly when used under metal free ceramic restorations.

Compared to other luting cement resin cements are virtually insoluble in oral fluids

Prevents micro leakage beneath cemented restorations when used along with dentine bonding agent

Bond strength of resin cement to teeth is superior to any other luting cement used. So can be used to cement inlays onlays 3/4<sup>th</sup> crowns, resin bonded fixed partial denture, ceramic laminates and veneers. Easy to manipulate.

### **DISADVANTAGES**

Resin cements are irritant to pulp. Therefore, pulp protection with calcium hydroxide or glass ionomer is required.

Removal of excess material is difficult

High cost and rarely available

Film thickness changes from brand to brand

Requires a bonding agent

### **APPLICATIONS**

Resin cements are gaining popularity in dental profession. Most of the non-metallic restorations used in present time can be cemented only with resin cement. Resin cements are used in conjunction with enamel and dentine bonding agent, and are capable for micromechanical attachment to both structures through these bonding agents. They can also bond to fitting surface of restorations which

has been microetched. Resin cements can be used for cementation of metallic or nonmetallic endodontic posts.

*EI - Mow Fay et al*<sup>9</sup> in his studies has recommended the use of resin cements along with dentine bonding agent when the length of canal is less than ideal or when the canal is not rounded. When canal is short the extra retention that the bonding produces compensates for reduced length and when the canal is not rounded the resin cement fills the space and eliminates the need for cast post.

*Tama Kanno et al*<sup>10</sup> studied the bond strength of dual-cure resin cement at different regions of root canal dentin using three kinds of curing methods. Thirty-six extracted bovine teeth were used. Each root was sectioned vertically into halves. Their pulpal dentine walls were polished flat and then applied with two dual-cure resin cements (Bistite II, Panavia F), and divided into three curing strategy groups: multi-direction light, one-direction light and no-light. The bonded specimens were sectioned perpendicularly to the long axis of the root into approximately 0.7 mm thick slabs within two-third of the root from the coronal end, and prepared for micro tensile bond strength ( $\mu$ TBS) tests. Knoop hardness of the cements was also measured. Within each curing strategy for both dual-cure resin cements, there were no significant differences between the  $\mu$ TBS values at the coronal third and mid third regions. The effect of curing method on bond strength and KHN was found to be dependent on the material

Ceramic orthodontic brackets can be bonded to teeth surface with resin cement<sup>11</sup>. Bonding of resin to the ceramic orthodontic brackets can be achieved by etching or coating the bracket with organosilane. Plastic brackets can be primed with a solvent containing methyl methacrylate and for metal brackets metallic mesh helps in retention. *Ascension Vicente, et al*<sup>12</sup> in 2003 compared the shear bond strength and the quantity of adhesive remaining on the tooth after the debonding of brackets bonded with two light-cured orthodontic resin adhesive systems (Transbond XT and Light-Bond) and dual-cured resin cement (RelyX, Unicem). Seventy-five premolars were divided into three

groups. In each group, brackets were bonded with one of the adhesives according to the manufacturer's instructions. Shear bond strength was measured using a universal test machine at a crosshead speed of 1mm / min, and adhesive remnant was quantified using image analysis equipment. Results showed that the resin cement produced significantly lower bond strength than the two orthodontic resin adhesive systems. It was also observed that the bond strength produced by Light-Bond was significantly greater than that of Transbond XT. RelyX left significantly less remnant adhesive than Transbond XT and Light-Bond. Between the two orthodontic systems, Light-Bond left significantly less adhesive on the tooth than Transbond XT.

*G J Ward et al* in 1988<sup>13</sup> compared the shear bond strength of nine resins comprising chemically cured light cured and glass ionomer types, 14 days after electrothermal and conventional bonding. The mean shear bond strength ranged from 7.4 MPa to 15.4 MPa. There was no statistically significant difference between electro thermal and conventional bonding method. All the resins produced bond strength adequate for clinical purpose.

*Christina A Mitchell* in 1995<sup>14</sup> compared the maximum load of failure of conventional glass ionomer cement, two resin modified glass ionomer cements, and composite cement used for bracket bonding. Each cement was used to bond the bracket to enamel of human premolars. Tensile shear force was applied and the maximum load for failure was recorded and compared. The results indicated that composite cement was significantly stronger than glass ionomer cement.

*Kitasako et al*<sup>15</sup> compared the tensile and shear bond strengths of one experimental and four commercially available resin cements following the ISO document TR 11405 for bond measurement. Tensile and shear bond tests were performed using bovine enamel and dentin as the tooth substrate with each of the resin cements. Resin composite rods were cemented to the prepared tooth surfaces. The bond strengths were obtained 24 h after cementation, and mode of failure was classified after fracture of the bonds, both visually and by SEM observation. Significant differences

existed between the two bond test methods for all materials with enamel and three of the five cements when bonded to dentin. The shear test results were always the higher of the two test methods. Mode of fracture varied little for the visual classification, but the morphology from SEM observations showed considerable differences.

*Rosen M, Cohen J, Becker PJ et al*<sup>16</sup> evaluated the shear and tensile bond strengths of two resin cements to a high gold alloy with differently prepared surfaces. Eighty buttons were cast from Argident Yellow 1 (gold 83.5 per cent). The surfaces of 40 buttons were sandblasted and the rest were silicoated. Forty cylinders each of F21 and Panavia EX were bonded to the prepared gold alloy surfaces and the specimens stored in distilled water for 24 hours. Half the specimens were then tested to failure using a shear test and the other half using a tensile test. The two cements achieved significantly higher shear and tensile bond strengths to the silicoated metal surfaces. Both cements attained similar shear bond strengths, but F21 achieved significantly stronger tensile bonds than Panavia EX to the differently treated metal surfaces. It is concluded that silicoating of high gold alloys is preferable to sandblasting for resin retention, and that bond strength data from different test methods are not comparable.

*Hassan Z. Movahhed et al*<sup>17</sup> compared the shear bond strengths (SBS) of a light-cured, resin-reinforced glass ionomer and a composite adhesive in combination with a self-etching primer, after different setting times to evaluate when orthodontic wires could be placed. Additionally, the fracture site after debonding was assessed using the Adhesive Remnant Index (ARI). Eighty freshly extracted human premolars were used. Twenty teeth were randomly assigned to each of four groups: (1) brackets bonded with Transbond XT with a Transbond Plus etching primer and debonded within 5 minutes; (2) brackets bonded with Fuji Ortho LC and debonded within 5 minutes; (3) brackets bonded as for group 1 and debonded within 15 minutes; (4) brackets bonded as for group 2 and debonded within 15 minutes. The SBS of each sample was determined with an UTM machine. The

mean SBS were, respectively: (1)  $8.8 \pm 2$  MPa (2)  $6.6 \pm 2.5$  MP; (3)  $11 \pm 1.6$  MP and (4)  $9.6 \pm 1.6$  MP. The findings indicate that brackets bonded with either Transbond XT in combination with Transbond Plus etching primer and Fuji Ortho LC had adequate bond strength at 5 minutes and were even stronger 15 minutes after initial bonding.

*Yoshitaka Kitayama et al*<sup>18</sup> measured the tensile and shear bond strength of resin-reinforced glass ionomer cement (RGIC) to glazed porcelain, to evaluate the durability of RGIC by thermal cycling, and to examine the RGIC remaining on the surface of the porcelain after the bond strength test to evaluate bonding conditions. Three adhesives were used in this study: Concise (CO) as a chemically cured composite resin, Fuji ORTHO (FO) as a chemically cured RGIC, and Fuji ORTHO LC (FOLC) as a light-cured RGIC. Tensile and shear bond strengths were measured 24 hours after bonding orthodontic brackets and also after thermal cycling. Tensile bond strength after 24 hours was  $6.6 \pm 3.2$  MPa in CO,  $7.3 \pm 1.4$  MPa in FO, and  $8.6 \pm 1.9$  MPa in FOLC, and the strength significantly decreased after the thermal cycling test. Shear bond strength after 24 hours was  $32.5 \pm 8.9$  MPa in CO,  $23.3 \pm 6.8$  MPa in FO, and  $24.7 \pm 6.5$  MPa in FOLC, and in contrast to tensile bond strength, no decreases in the strength were detected after the thermal cycling test. CO showed significantly higher shear bond strength than did FO and FOLC. When using the shear bond strength test and CO, destruction of porcelain surfaces frequently occurred after 24 hours and was observed in every specimen after the thermal cycling. RGIC was found to be an advantageous alternative to resin adhesive for bracket bonding to porcelain and to enamel.

*Major PW, Koehler JR, Manning KE*<sup>19</sup> compared the bond strength of three types of adhesion promoters. Ormco Porcelain Primer (OR) (Ormco Corp., Glendora, Calif.), All-Bond2 (AB2) (Bisco Dental Products, Inc., Itasca, Ill.), and Scotchprime Ceramic Primer (SP) (3M, St. Paul, Minn.) with two orthodontic adhesives. Phase II (Reliance, Inc., Itasca, Ill.) and Rely-a-bond (Reliance, Inc., Itasca, Ill.). Eighty ceramometal samples were fabricated and hydrated for 1 week in distilled water before bonding. Next, the samples were etched with 2.5 % HF acid for 90 seconds, rinsed and upper

central incisor metal orthodontic brackets were bonded with the various primers and adhesives. After 24 hours they were sheared off with an MTS machine at a rate of 0.5 mm/min, and the bond strength was measured. The mean shear bond strengths (MPa) with Phase II were as follows; Control 0.44 (sigma = 0.22), AB2 8.40 (sigma = 3.61), OR 13.31 (sigma = 5.79), SP 13.53 (sigma = 3.34). With Rely-a-bond, the shear bond strengths were Control 0.41 (sigma = 0.67), AB2 4.34 (sigma = 1.88), OR 9.73 (sigma = 4.58), and SP 12.40 (sigma = 3.05).

*C.J. Lamour and D.R. Stirups*<sup>20</sup> assessed a resin modified glass ionomer bonding system, Fuji ortho L.C.<sup>TM</sup> using different bonding techniques and compared it with a composite control (Transbond<sup>TM</sup>). Twenty extracted premolar teeth in each group were bonded as follows: (i) Group 1 Control (Transbond); (ii) Group 2 Fuji ortho L.C. without an etch procedure/wet enamel surface; (iii) Group 3 Fuji ortho L.C. without an etch procedure/dry enamel surface; (iv) Group 4 Fuji ortho L.C. using a conventional acid etch technique. Brackets bonded as recommended by the manufacturer (Group 2) have significantly ( $p < 0.001$ ) lower bond strengths compared with the control (Group 1). Bonding with an etch technique (Group 4) will significantly ( $p < 0.001$ ) increase the bond strength compared with the other Fuji groups. All the Fuji groups tended to fail at the enamel/resin interface with lower ARI scores compared with the control. The lower bond strength of Fuji ortho L.C. would limit its use as a routine bonding agent. When bonded with an acid etch technique, the bond strength may be sufficient for low loading situations such as the upper anterior teeth.

*Yi GK, Dunn WJ, Taloumis LJ*<sup>21</sup> compared the shear bond strength of orthodontic brackets bonded to teeth with either an indirect bonding technique and a new adhesive resin or a direct bonding technique and a light-activated adhesive. Fifty-four extracted premolars were mounted in acrylic blocks and randomly divided into 2 groups ( $n = 27$ ). In one group, orthodontic brackets were bonded to premolars with an indirect bonding adhesive system; in the other, brackets were bonded with the direct method. Seventy-two hours later, the brackets were placed in a testing machine and subjected to a shear force with a crosshead speed of 1 mm/minute. The mean shear bond strengths

for the indirect and direct groups were 11.2 and 10.9 MPa, respectively, both exceeding the minimum shear bond strength range of 5.9 to 7.8 MPa often cited in the literature for clinical success. Data were analyzed with Student tests. No significant difference in shear bond strength between the 2 groups was detected ( $P = .76$ ). Resin remnants on orthodontic bracket pads were observed with a dissecting microscope at 30x Magnification and scored with a modified adhesive remnant index. There was no significant difference between groups ( $P > .05$ ). There was also no correlation between shear bond strength and the percentage of adhesive resin remnants left on the orthodontic bracket. Under the conditions of this study, no evidence suggests a difference in shear bond strength of orthodontic brackets bonded to tooth enamel, whether they are bonded with the direct or indirect technique.

*Somsak Sirirungrojying et al*<sup>22</sup> determined the bonding durability when a 'Megabond' self-etching primer is used with 4-META/MMA-TBB resin to bond metal orthodontic brackets to human premolar enamel. Three conditions of enamel were prepared: 'Megabond' self-etching primer without saliva contamination, 'Megabond' self-etching primer with saliva contamination, and repeat 'Megabond' self-etching priming after saliva contamination. Shear bond strengths were measured after immersion in water at 37°C for 24 hours, or after 2000 or 5000 cycles of thermal cycling between 5°C and 55°C. There were no significant differences in shear bond strength among the three groups not only before thermal cycling, but also after thermal cycling. FE-SEM observation revealed the presence of saliva and reduced amount of resin tag formation after saliva contamination. The present study provided the evidence in human teeth that when using 4-META/MMA-TBB resin, 'Megabond' self-etching primer treatment produced tight bonding even when surface was contaminated with saliva.

*Alexander T. H. et al*<sup>23</sup> evaluated the early shear bond strength of enamel-composite-bracket adhesion accomplished without the use of liquid resin. Orthodontic brackets were bonded to the

buccal surfaces of healthy extracted premolars in the test group by Transbond XT ( $n = 8$ ) and Phase II ( $n = 8$ ) composites but not the enclosed liquid resins in these products. Brackets bonded with the same materials ( $n = 8$  for each) along with their corresponding liquid resin served as controls. The specimens were tested for shear bond strength after 24-h storage in water at 37°C. The fractured surfaces were graded with Adhesive Remnant Index (ARI) under a 2x-dissection microscope. Enamel of the randomly selected test and control specimens was dissolved by 20% formic acid. Afterwards, the enamel side of the bonding materials in both groups ( $n = 4$ ) was examined under the scanning electron microscope. ANOVA was used for statistical analyses. Our laboratory data suggest that the enamel adhesion produced by these two commercial materials without the use of liquid resin does not differ significantly in their early in vitro shear bond strength.

Another use of resin cement is in the attachment of resin bonded fixed partial denture where the metal wings are etched to get added retention. El - Mowfay et al<sup>24</sup> in their studies has shown that resin cement used in conjunction with dentine bonding agent can give superior attachment of crowns on teeth with short clinical crown. For teeth with crown preparation having less angle of convergence the attachment of crowns bonded with resin cement was six times higher than the attachment achieved with Zinc phosphate cement. The use of resin cement for luting of ceramic inlays onlays, metal free ceramic crown and three fourth crowns gave superior retention compared to any other luting agent currently available.

In an in-vitro investigation, *Dietschi et al. (1990)*<sup>25</sup> found that ceramic inlays cemented with composite resin were more resistant to fracture than those luted with glass ionomer, although no statistically significant difference was found. *Stenberg & Matsson*<sup>24</sup> reported a failure rate of 23% for 25 Dicor inlays luted with glass-ionomer cement after 2 years. *Van Dijken et al*<sup>26</sup>. (1998) evaluated 118 feldspathic ceramic inlays luted with dual-cured resin composite or glass ionomer cement (GIC)

over a 6-year follow-up. Twelve percent of the resin composite group and 26% of the GIC group were assessed as failures.

*Mitchell et al. (1999)*<sup>27</sup> tested the fracture toughness of six different luting cements (four conventional glass-ionomer cements, one resin-modified glass ionomer and one resin cement), in order to determine their tendency to fail cohesively when loaded. The results indicated that the fracture toughness of the resin-modified glass-ionomer cement was greater than any of the four conventional glass-ionomer cements, and that the fracture toughness of the resin composite cement was significantly greater than any of the other cements tested.

*Yucel yilmaz et al*<sup>28</sup> investigated was to compare the tensile strength, micro leakage, and Scanning Electron Microscope (SEM) evaluations of SSCs cemented using different adhesive cements on primary molars. Sixty-three extracted primary first molars were used. Tooth preparations were done. Crowns were altered and adapted for investigation purpose, and then cemented using glass ionomer cement (Aqua Meron), resin modified cement (RelyX Luting), and resin cement (Panavia F) on the prepared teeth. Samples were divided into two groups of 30 samples each for tensile strength and micro leakage tests. The remaining three samples were used for SEM evaluation. Data were analyzed with one-way ANOVA and Tukey test. The statistical analysis of ANOVA revealed significant differences among the groups for both tensile strength and micro leakage tests ( $p < 0.05$ ). Tukey test showed statistically significant difference between Panavia F and RelyX Luting ( $p < 0.05$ ), but none between the others ( $p > 0.05$ ). This study showed that the higher the retentive force a crown possessed, the lower would be the possibility of micro leakage

*Claus- Peter Ernest et al*<sup>29</sup> studied the retentive strength of cast gold crown using glass ionomer, compomer, and resin cement. Without adhesive system the resin cement F 21 gave the lowest retentive strength and the investigators suggested the use of resin cement along with an adhesive system. *Wu J.C and Wilson*<sup>30</sup> did a review on resin luting cements for full coverage

restorations. They favored the use of adhesive resin cement in restoration of teeth with short tapered clinical crown or other unfavorable geometric configuration. The paper also reviewed the composition, adhesive, physical, biological properties and handling characteristics of Panavia and 4 META containing cements.

*Christophe Azevedo, et al*<sup>31</sup> compared the effects of time on the flexural strength of a resin-reinforced glass ionomer and a composite adhesive system, specifically at three time frames corresponding to the three stages of polymerization of Fuji Ortho LC. Ten rectangular specimens of each material were prepared in a metal mold (25 × 2 × 2 mm) and then stored at 37°C and 100% humidity in an incubator. Six test groups were created, in which each specimen was fractured using a 3-point-bending test at a crosshead speed of 0.5 mm/min. The test results indicated that there were significant differences among the groups ( $P = .0001$ ). The flexural strengths were significantly higher in the two groups (III and VI) that were fractured after seven days. This was true for both the Fuji Ortho LC ( $\bar{x} = 77 \pm 6.1$  MPa) and the Concise ( $\bar{x} = 103.9 \pm 4.2$  MPa). The flexural strength of the resin-modified glass ionomer adhesive was significantly lower than that for the composite whatever the time of fracture, 10 minutes, one hour, or seven days. The analysis of the strength-deformation curve of the group of Fuji Ortho LC, which was fractured within 10 minutes after setting (group I), showed viscoelastic behavior, whereas that of all the others groups showed elastic behavior. According to this study, clinicians must consider the mechanics of Fuji Ortho LC setting and, when this material is used, at least one hour waiting is required to ligate initial or repaired arch wires.

*Miyazaki M et al*<sup>32</sup> investigated the curing characteristics of light-cured composites and their related mechanical properties. Single-edge notch specimens [25 mm x 2.5 mm x 5 mm with a 5 mm notch ( $a/W = 0.5$ )] were prepared for fracture toughness measurements. For flexural strength testing, a stainless steel mold (25 mm x 2 mm x 2 mm) was used. Light-cured composites were condensed into the mold, and the middle third of the specimen was first activated for 30 s with 400 mW/cm<sup>2</sup>, for

60 s with 200 mW/cm<sup>2</sup>, or for 120 s with 100 mW/cm<sup>2</sup>. Then the remaining thirds were activated at the same intensity and curing time as the middle third. After 24 h storage in 37 degrees C water, three-point bending tests were performed with a span length of 20 mm at a crosshead speed of 0.5 mm/min. A one-way ANOVA, followed by a Newman-Keuls test ( $p < 0.05$ ), were used to compare the data obtained from each group to test the effect of the curing conditions. Fracture toughness, flexural strength, and flexural modulus varied with resin composites. Among the three curing conditions for each material, there were no significant differences in fracture toughness, flexural strength, or flexural modulus. The fracture toughness and the flexural strength were the same when irradiations with the same amount of energy (light intensity multiplied by curing time) were used. It was found that, at lower light intensity, longer curing was required to provide comparable mechanical properties. Accumulated irradiation energy obtained through a product of the light intensity and curing time may serve as a guideline to produce samples exhibiting equivalent fracture toughness as well as flexural strengths.

*Walker MP*<sup>33</sup> determined changes in flexural properties of resin cement under cyclic loading and aqueous aging: flexural modulus and strength of Panavia F were measured by static loading to failure after 48-h, 31-d, and 60-d aqueous aging at 37 degrees C with and without cyclic loading at clinical stress levels. Six specimens were used for each experimental condition. Scanning electron microscopy (SEM) was also used to characterize the morphology of the fractured surfaces to potentially identify resin cement components particularly susceptible to degradation under simulated clinical function. A two-factor ANOVA ( $p \leq 0.05$ ) and least significant difference post hoc test indicated that cyclic loading produced a significant increase in the flexural modulus with no significant effect on the flexural strength.

A *Piwowarczyk, et al*<sup>34</sup> evaluated the compressive and flexural strengths, surface hardness and film thickness of different cement systems in their recommended modes of curing. RelyX Unicem cement achieved high strength values. Results show the strength values comparing resin

and non-resin cement systems. A *Piwowarczyk*<sup>34</sup> determined the amount of water that will be absorbed into cured cement samples after one month. There is a direct correlation to the amount of water the cement absorbs to the amount of expansion or swelling of the cement. RelyX Unicem cement shows very low levels of water uptake after being stored in water for one month. The uptake is considerably less than resin-modified glass ionomer and compomer cement systems and is comparable to other resin cement systems.

*Yap A, Lee CM*<sup>35</sup> compared the water sorption and solubility of several resin-modified polyalkenoate cements. The materials evaluated included Variglass used as a restoration, base on liner; Fuji II LC, Fuji Liner; Vitrebond; Vitremer and Photac-Bond. Z100, a composite resin, was used as control. All specimens were manipulated according to the manufacturers' instructions and then subjected to water sorption and solubility tests based on the ISO 4049 requirements. Data were subjected to one-way ANOVA and Duncan's test at a 0.05 significance level. Results show that the composite resin control had significantly less water sorption than any of the resin-modified polyalkenoate cements evaluated. The degree of water sorption was product dependent and appeared to be influenced by the resin (HEMA) content. There was no apparent correlation between water sorption and solubility. Some of the resin-modified polyalkenoate cements retained water in their set structure and hence solubility could not be assessed.

*Ortengren U*<sup>36</sup>, Assessed water sorption and solubility of six proprietary composite resin materials and monomers eluted from the organic matrix during water storage identified. Water sorption and solubility tests were carried out with the following storage times: 4 h, 24 h and 7, 60 and 180 days. After storage, water sorption and solubility were determined. Eluted monomers were analyzed by high performance liquid chromatography (HPLC). Correlation between the retention time of the registered peak and the reference peak was observed, and UV-spectra confirmed the identity. The results showed an increase in water sorption until equilibrium for all materials with one exception. The solubility behavior of the composite resin materials tested revealed variations, with

both mass decrease and increase. The resin composition influences the water sorption and solubility behavior of composite resin materials. The HPLC analysis of eluted components revealed that triethyleneglycol dimethacrylate (TEGDMA) was the main monomer released. Maximal monomer concentration in the eluate was observed after 7 days. During the test period, quantifiable quantities of urethanedimethacrylate (UEDMA) monomer were observed, whereas 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy)-phenyl]propane (Bis-GMA) was only found in detectable quantities. No detectable quantities of bisphenol-A were observed during the test period.

*Keyf F, Yalcin F*<sup>37</sup> investigated weight changes of seven different light-cured composite restorative materials, one polyacid glass ionomer compomer, and one light-cured glass-ionomer cement following short-term and long-term storage in water. Two pack able composites, three universal (hybrid) composites, one micro glass composite, one polyacid glass ionomer resin composite (compomer), one micro hybrid low-viscosity (flow able) composite, and one light cured glass ionomer composite cement were evaluated in this study. The weight changes of these specimens were measured daily (short-term storage), and they were measured after six weeks (long-term storage) using an electronic analytical balance. A significant difference was found in Ionoliner, Dyract AP, Opticor flow, Charisma, and Solitare 2, but no significant difference was found in the others (Filtek Z 250, Filtek P60, TPH Spectrum, and Valux Plus). Weight change showed a tendency to increase with the time of water storage. The greatest weight change occurred in light-cured glass ionomer composite cement (Ionoliner), which is followed in order by the weight changes in Dyract AP, Opticor Flow, Charisma, Solitare 2, Filtek Z250, Filtek P60, TPH Spectrum; Valux Plus had the least amount of change.

*Jacques LB, Ferrari M, Cardoso PE*<sup>38</sup>. Evaluated the sealing ability of single-unit all-ceramic and gold-electroformed crowns luted with resin cement under laboratory conditions. Thirty extracted maxillary premolars were selected. Standardized abutments were prepared for full-crown restorations with cervical margins located 1 mm below the cemento-enamel junction. Samples were

divided into two groups (n = 15) based on type of restorative system: Group 1: a heat-pressed all-ceramic system (IPS Empress 2, Ivoclar-Vivadent); Group 2: a gold-electroformed porcelain-fused-to-metal system (Gramm Technik). Crowns were luted with Excite DSC bonding system (Ivoclar-Vivadent) and Variolink II resin cement (Ivoclar-Vivadent) following manufacturer's instructions. After luting procedures and 700 thermal cycles (5 degrees C and 55 degrees C, 1 min dwell time), 20 specimens (n = 10 per group) were randomly selected and processed for micro leakage evaluation at cervical sites. The remaining specimens (n = 5 per group) were prepared for SEM investigation on film thickness. Micro leakage and film thickness data were statistically evaluated using the Mann-Whitney U-test ( $p < 0.05$ ). Fifty percent of group 1 and 60% of group 2 samples showed leakage. For group 1, dye penetration was mostly observed between metal and resin material, while group 2 specimens showed leakage mainly between ceramic margins and resin cement. Samples from group 1 showed a cement thickness of 82 micron, while 71 micron was found for group 2. Statistically significant differences were not found between samples from the two groups. Regarding micro leakage and film thickness, both restorative systems evaluated in this study performed similarly.

*Levine WA*<sup>39</sup> Acid-etched cast metal resin-bonded retainers require resin luting agents with film thicknesses similar to dental cements to allow complete seating of frameworks without the need for post-bonding adjustments. Conventional cements, restorative resins, and resin cements were compared in terms of film thickness by using the American Dental Association Specification No. 8 for zinc phosphate cement. Most of the resin luting agents had film thicknesses of 20 to 40 microns. Working time was seen to affect film thickness. The particle size of one resin was also examined under scanning electron micrograph.

*Daniela Francisca Gigo et al*<sup>40</sup> analyzed the diametral tensile strengths of three resin-modified restorative glass ionomer cements - Vitremer, Fuji II LC and Photac Fil and one compomer - Dyract. They were tested at 1 hour, 1 day and 1 week. Kratos testing machine was used to load the specimens at a cross-head speed of 0.5 mm/min. The data were analyzed by two-way ANOVA and

Tukey's test that showed statistically significant differences among the materials. The tested materials presented an increase in strength from 1 hour to 1 week and were as follows for each material respectively: Vitremer (19.22-27.29), Fuji II LC (23.91-28.67), Photac Fil (19.35-22.86), Dyract (28.83-46.95). Dyract presented the highest strengths.

*Piemjai M, et al*<sup>41</sup> evaluated micro leakage of direct composite inlays cemented with acid-base cements and methyl methacrylate resin cement. Sixty Class V box preparations (3 mm x 3 mm x 1.5 mm) were precisely prepared in previously frozen bovine teeth with one margin in enamel and another margin in dentin. Direct composite inlays (EPIC-TMPT) for each preparation were divided into 4 groups of 15 specimens each and cemented with 3 acid-base cements (control group): Elite, Ketac-Cem, Hy-Bond Carbo-Cem, and 1 adhesive resin cement: C&B Metabond. All specimens were stored in distilled water for 24 hours at 37 degrees C before immersion in 0.5% basic fuchsin for 24 hours. The dye penetration was measured on the sectioned specimens at the tooth-cement interface of enamel and cementum margins and recorded with graded criteria under light microscopy (Olympus Vanox-T) at original magnification x 50, 100, and 200. A Kruskal-Wallis and the Mann-Whitney test at  $P < .05$  were used to analyze leakage score. All cementum margins of the 3 acid-base cements tested demonstrated significantly higher leakage scores than cementum margins for inlays cemented with the resin cement tested ( $P < .01$ ). No leakage along the tooth-cement interface was found for inlays retained with the adhesive resin cement. Within the limitations of this study, the 3 acid-base cements tested exhibited greater micro leakage at the cementum margins than did the adhesive resin cement that was tested.

*Erdilek N et al*<sup>42</sup> performed an in vitro dye leakage study to compare the sealing ability of glass ionomer, composite resin and glass ionomer/resin cement when used as restorative materials for G.V. Black class V cavities. In this research, standard Class V cavities were prepared in sound premolar teeth extracted for orthodontic reasons. The cavities were randomly divided into 3 groups. After filling the cavities with glass ionomer cement, composite resin and light curing glass

ionomer/resin cement, the specimens were immersed in silver nitrate solution. Marginal micro leakage at the interface between the cavity wall and restoration was evaluated. The results were analyzed by using Kruskal-Wallis and Mann Whitney U tests. Results of this in vitro study indicate that composite resin and glass ionomer/resin cement provide a better seal than glass ionomer cement.

A. *Thaveeratana,etal*<sup>43</sup> Evaluated the micro leakage and Nanoleakage at the interface of dentinoenamel junction (DEJ), enamel and dentin bonded with either Superbond C&B (S), Panavia F (P) or Variolink II (V) resin cements. Methods: Extracted human molars were cross-sectioned at the middle third of coronal part to prepare 2x4x1 mm specimens with DEJ aligning in the middle, 1 mm of enamel and dentin on each side. Ninety-six specimens were divided into 6 subgroups of 8 specimens for each control and sample groups. Samples were cemented to composite veneers: Groups 1, 4; 2, 5; 3, 6 using S; P; V respectively. Groups 1, 2, 3 were measured the penetration distance of 0.5% basic fuchsin using stereoscopy and digital micrometer. Groups 4, 5, 6 were measured silver penetration using stereoscopy, scanning electron microscopy (backscatter and EDX modes). Results: Control group, dentin had significantly higher dye penetration distance than DEJ and enamel ( $P<0.01$ ). No dye penetration in enamel was found. The distance of silver penetration in DEJ was not significant difference compare with in dentin but significantly higher than in enamel. No leakage was found at enamel-resin interface in all groups. No leakage at the tooth-cement interface was found in groups 1S, 2P and 4S. Groups 3V and 6V had highest leakage at resin interface of dentin and DEJ. Conclusion: The components and structures of tooth influence its permeability. Hybridized enamel with resin could resist natural nano-permeability of enamel. Complete hybridization via dry bonding using Superbond C&B cement could prevent leakage at resin interface of enamel, DEJ and dentin. Highest leakage suggested that wet bonding using Variolink II with total phosphoric etching could not provide reliable bonding to DEJ and dentin. Self etching and priming

using Panavia F could resist micro leakage but not nanoleakage at the DEJ- and dentin-resin interfaces

*Maximiliano Sérgio et al*<sup>44</sup>. Studied the ability of different adhesive materials to prevent micro leakage in bonded amalgam restorations. Standard cavities were prepared in both buccal and lingual surfaces of 35 bovine incisors. The gingival wall was located in cementum/dentin and the occlusal wall in enamel. Teeth (n=35) were divided into 5 groups, according to material employed (one glass-ionomer cement, two resin cements, one adhesive system, and Copalex varnish as a control). Following restoration, the teeth were submitted to thermal cycling. The teeth were subsequently immersed in methylene blue dye and sectioned to allow assessment of micro leakage. Non-parametric statistical analysis indicated that all materials demonstrated less leakage than the control group ( $p < 0.01$ ). No leakage was found using the resin-modified glass-ionomer cement, which was significantly different from the other adhesive materials ( $p < 0.05$ ). Leakage in enamel was lower than in cementum/dentin margins. It was concluded that bonded amalgam was an effective technique, since all materials prevented micro leakage in enamel and cementum/dentin, when compared to the control group, except Panavia in cementum/dentin margins

*Prabhakar A et al*<sup>45</sup> carried out a study to compare the marginal micro leakage of some newer materials viz. a flowable composite, an injectable resin modified glass-ionomer and a compomer in Class 1 cavities of 30 non carious primary molars. After 0.5% basic fuchsin dye penetration and sectioning, the teeth were studied under Stereomicroscope. The results obtained revealed that flowable composite showed significantly lower micro leakage ( $p < 0.05$ ) as compared to injectable resin-modified glass ionomer and compomer. However, no significant difference was observed when injectable resinmodified glass-ionomer cement was compared to compomer. This concludes that flowable composite materials adhere better to the primary teeth than resin modified glass ionomer and compomer.

Jung WY et al <sup>46</sup> compared the micro leakage of the IPS Empress ceramic crown according to 1) margin types such as rounded shoulder and bevel, 2) margin locations such as enamel and dentine, 3) resin cement types such as Variolink and Bistite. For this study, 80 extracted premolar teeth were used. After 10 experimental dies were prepared for each group, the IPS Empress ceramic crowns were fabricated on the dies and cemented with one of two types of resin cement. After 1,000 cycles of thermal changes were given with the thermo cycling unit, each specimen was sectioned with a low speed diamond saw in both the buccolingual and the mesiodistal direction. The micro leakage was then measured with a stereo microscope. The obtained results were as follows: Every experimental group showed micro leakage. The micro leakage of the crowns which had a rounded shoulder on enamel cemented with Variolink was the lowest of all. The micro leakage of rounded shoulder finish line was lower than that of bevel finish line. The statistically significant differences were found with respect to the type of finish lines. The micro leakage of the finish line on enamel was lower than that of finish line on dentine. The statistically significant differences were found with respect to the location of the finish lines. The micro leakage of the Variolink cement was lower than that of the Bistite cement. The statistically significant differences were found with respect to the resin cements.

## **PROPERTIES OF DUAL CURE RESIN CEMENT**

Generally International specification does not classify polymer based luting material into types. Instead, the dental polymer based filling, restorative and luting materials are placed under following classes

Class 1 : Materials whose setting is effected by mixing an initiator and activator ("self curing" materials )

Class 2: Materials whose setting is affected by application of energy from an external source, such as blue light or heat ("external energy activated" materials) they are subdivided as follows :

Group I: Materials whose use requires the energy to be applied intra orally.

Group II: Materials whose use requires the energy to be applied extra orally. When fabricated, these materials will be luted in place

Certain materials may be claimed by the manufacturer to be both Group I & Group II in such cases the material should full fill the requirements of both group.

Class 3: Materials that are cured by application of external energy and also have a self curing mechanism present ("dual cure" materials)

The following are some of the important properties stipulated by international standards organization

### ***BIOCOMPATIBILITY***

It is the ability of a material to elicit an appropriate biological response in a given application in a body.

***WORKING TIME:*** For Class I and Class II luting material the material when tested shall be capable of forming a thin layer; during its formation there shall be no detectable change in homogeneity.

***SETTING TIME:*** This is the time required for a material to harden into a solid starting from commencement of mixing .Generally for class 3 material the setting should not be more than 10 minutes.

***FLEXURAL STRENGTH:*** It is the force per unit area at the point of fracture of a test specimen subjected to flexural loading. Generally the flexural strength shall be greater than or equal to 50 MPa

### ***WATER SORPTION AND SOLUBILITY***

As per ISO 4049 the water sorption of all materials when determined shall be less than or equal to  $40\mu\text{g}/\text{mm}^3$ . The solubility of all materials shall be less than or equal to  $7.5\mu\text{g}/\text{mm}^3$

### ***FILM THICKNESS:***

Height of space between two surfaces that are separated by cement denotes film thickness. A property of luting agent, this dimension is measured after pressure is applied between two flat surfaces that are separated by the cement layer. The film thickness of luting cement when

determined shall be no more than 10µm above any value claimed by the manufacturer and in any event shall be no greater than 50 µm

**MICRO LEAKAGE:** Flow of oral fluids and bacteria into the microscopic gap between a prepared tooth surface and the restorative material.

### **Current status in India**

Developments of chemical and light cure dental composites have been reported earlier by Dental Products Group of Sree Chitra Tirunal Institute for Medical Sciences and Technology. Subsequently, developments of radiopaque material have also been reported by the same group. Studies on development of unique bonding agent system suitable for resin composites have also reported. In this work, a newly developed dual cure resin cement based on BISGMA has been evaluated for its biological, physical and mechanical properties. This material is recommended for luting of metal free ceramic restorations, metal full crowns, 3/4<sup>th</sup> crowns, endodontic posts and orthodontic brackets. The imported material in market is extremely costly and rarely available. Development of this material gives a better choice in quality and price.

### **AIM AND SCOPE OF THE WORK:**

The aim of the study was to evaluate the physical, mechanical and biological properties of indigenously developed dual cure resin cement, developed at Dental Products Laboratory, SCTIMST, Trivandrum. The following properties were evaluated as per

- a) ISO 4049:2000(E), Polymer based filling restorative and luting materials
- b) ISO 7405 Pre clinical evaluation of biocompatibility of medical devices used in dentistry- test methods for dental materials read with
- c) ISO 10993 - 1, Biological evaluation of medical devices – Part 1: Guidance on selection of test.
  1. Biocompatibility
  2. Film Thickness
  3. Flexural Strength

#### 4. Water Sorption and solubility

In addition to this the other properties evaluated includes

#### 5. Shear Strength

#### 6. Adhesion to tooth surface

#### 7. Diametral Tensile Strength

#### 8. Micro-leakage

*MATERIALS AND METHODS*

## MATERIALS AND METHODS

The newly developed Chitra dual cure resin cement (DCRC, FIG I) is a low viscosity two paste system. The paste A contains the initiator benzoyl peroxide and the paste B contains the inhibitor BHT. Equal amounts of paste A and Paste B is mixed in a clean mixing pad using a plastic spatula, the mixed cement is exposed to visible light for 40 seconds, for light curing and the chemical reaction starts along with the light exposure and complete curing takes place at around 3minutes from the start of mixing.

The following studies were carried out using DCRC as test material and Rely X ARC as control

### DETERMINATION OF FILM THICKNESS OF DCRC

**AIM:** To measure the film thickness of indigenously made dual cure resin cement.

#### **Materials:**

Paste A & Paste B of DCRC

Two glass plates 2cmx1cmx0.5cm

Universal testing machine

Cellophane tape

Light cure unit

Micrometer

#### **Methods**

Two glass plates of above mentioned size was cleaned and was kept above one another. Its height was measured at five different points and its mean was calculated. The glass plates were then separated and kept aside. Equal amount of paste A & paste B of DCRC was dispensed into a mixing pad and was mixed for 10 seconds using a clean plastic spatula. One drop of the mixed cement was placed onto one of the cleaned glass plate the other glass plate was placed over it and the whole assembly was stabilized using a cellophane tape. One minute after mixing the whole assembly was placed on the platform of a universal testing machine INSTRON 1011, and a weight of 150 Newtons

was applied for three minutes and then the whole unit was removed and was light cured for 120 seconds. After complete curing height was again measured at five different points and the average was noted .The procedure was repeated in five sets of glass plates and the mean difference between the initial and final height gave the film thickness of the dual cure resin cement.

## **DETERMINATION OF FLEXURAL STRENGTH OF DCRC**

### ***AIM***

To determine the flexural strength of an indigenously developed DCRC.

### ***Materials and methods***

Stainless Steel Mold –  $(25 \pm 2)$  mm ×  $(2 \pm 0.1)$  mm ×  $(2 \pm 0.1)$  mm

Cellophane sheet

DCRC paste

C – Clamp

Incubator

Metal sheet

### ***Procedure***

The mould was placed on a cellophane sheet on a metal plate. Equal amounts of Paste A and paste B of DCRC was mixed and the mould cavity was filled with it. Another cellophane sheet was placed on it, over which a metal sheet was placed. The entire assembly was clamped using a C clamp and was kept aside for 3 minutes. After three minutes the clamp was removed and the samples were light cured for 40 seconds each at three points along its length from both the sides .The samples were again clamped and kept in an incubator at 37°C for 15 minutes after 15 minutes the samples were removed from the mould .Ten such samples were made and five of the samples were stored in distilled water in an incubator at 37°C for 23 hours, one hour before testing the samples were brought to testing room temperature of 22°C. The next five samples were stored in distilled water in an incubator at 37°C for 7 days, in an incubator.

Width and depth of the samples were measured. It was then placed horizontally on two metal rods fixed 20mm apart on an aluminum plate (FIG II) and another aluminum plate was kept on it. Load was applied at the center of the sample using universal testing machine with a crosshead speed of 1mm per minute. (FIG III) Maximum load at which the break occurred was recorded and flexural strength was calculated using the formula  $FS = 3FL / 2BD$  where F is Load at break (newtons), L is Span length, B is Width and D is Thickness (all in mm)

## **DETERMINATION OF DIAMETRAL TENSILE STRENGTH OF DCRC**

### ***AIM***

To determine the diametral tensile strength of an indigenously developed DCRC

### ***Materials and methods***

Mould of 6 mm diameter and 3 mm height

Cellophane sheet

DCRC paste

C – Clamp

Incubator

Metal sheet

### ***Procedure***

The mould was placed on a cellophane sheet on a metal plate. Equal amounts of Paste A and Paste B of DCRC was mixed and the mould cavity was filled with it. Another cellophane sheet was placed over it over which a metal sheet was placed. The entire assembly was clamped using a C Clamp and was kept aside for 3 minutes. After three minutes the clamp was removed and the samples were light cured for 40 seconds from both the sides. The samples were again clamped and kept in an incubator at 37°C for 15 minutes after 15 minutes the samples were removed from the mould. Ten such samples were (FIG IV) made and all the samples were stored in distilled water in

an incubator at 37°C for 23 hours, one hour before testing the samples were brought to testing room temperature of 22°C

Testing was carried out in a universal testing machine. (FIG V) The diameter and heights of the samples were measured and were placed between the plates of the UTM. Compressive load was applied with a cross head speed of 10 mm per minute until the samples broke at the center. The maximum load (P) required for the break was recorded and the diametral tensile strength was calculated using the formula  $2P/\pi DL$ . Where P is the maximum load, D is the diameter of the sample and L is the height

All the samples were tested and six best values were recorded to get the mean value.

## **DETERMINATION OF SHEAR BOND STRENGTH OF DCRC**

**Aim:** Is to evaluate and compare the shear bond strength of orthodontic bracket bonded using three different resin cements namely Rely X ARC from 3M, Vario link II light cure, and DCRC.

Also to compare the shear bond strength of orthodontic bracket bonded using three different resin cements with and with out application of bonding agent.

## **MATERIALS & EQUIPMENTS**

Beggs Bracket Orthotec

37% Phosphoric Acid

Single Bond 3M Bonding Agent

Mounted extracted teeth

Rely X Arc 3M

Vario Link Light cure Resin Cement – Vivadent

DCRC

Self Cure Acrylic Resin

Universal Testing Machine

## **PROCEDURE**

Bracket bonding was done on extracted incisors, the teeth was cleaned with 3% hydrogen peroxide and was stored in distilled water. Root surface of teeth was grooved with diamond wheel for added retention in acrylic. The tooth was then mounted in self cure acrylic resin till the cemento-enamel junction such that the crown portion remained out of acrylic and the labial surface remained parallel to the vertical plane. These were then divided into two groups Group I and Group II. In Group I bracket bonding was done after acid etching and application of two consecutive layers of single bond bonding agent 3M and in Group II bracket bonding was done after acid etching and without application of bonding agent. Each group namely Group I and Group II was further sub divided into Group IA (GRIA) Group IB (GRIB) Group IC (GRIC) similarly Group II was again sub divided into Group IIA (GRIIA), Group II B (GRIIB), Group IIC (GRIIC). Bracket bonding was done as follows

In Group IA (GRIA) the labial surface of teeth was etched with 37% phosphoric acid for one minute cleaned in running water for ten seconds and was dried with blotting paper. Two consecutive layers of single bond bonding agent was applied, after each application the surface was dried by gentle blow of air from an air syringe. It was then cured for ten seconds with a light source from an LED. Beggs bracket Orthotec with mesh at the bonding surface was used; bonding agent was applied on the mesh and was cured for ten seconds. Equal amount of paste A and paste B of Rely X ARC was mixed on a mixing pad and applied to the bonding surface of the bracket. The brackets were then gently pressed to the tooth surface excess cement was removed and light curing was done for forty seconds using an LED

In Group IB (GRIB) Bracket bonding was done as same as in Group IA (GRIA) except for the use of Vario link II light cure resin cement .curing was done initially for ten seconds removal of flash followed by curing for forty seconds .

In Group IC (GRIC) Bracket bonding was done as same as in Group IA (GRIA) except for the use of DCRC

In Group II bracket bonding was done after acid etching and without application of bonding agent all other procedures remained same as in Group I. In Group IIA (GRIIA) RelyXARC dual cure resin cement was used.

Group IIB (GRIIB) Vario link II light cure resin cement was used.

In Group IIC (GRIIC) Bracket bonding was done as same as in Group IIA (GRIA) except for the use of DCRC (FIG IV)

All the specimens were then stored in room temperature (22° c) for 15 minutes. It was then stored in distilled water at 37° in an incubator for 23 hours one hour before testing the samples were kept at testing room temperature for one hour. Testing was carried out in a universal testing machine INSTRON 1011. The machine worked with a cross head speed of 1mm per minute. The samples were clamped at the lower grip and a stainless steel wire loop passing through the bracket slot was held by upper grip. (FIG VII) The upper grip was free to move in vertical plane applying the shear force to simulate oral condition. The machine stops as soon as the break occurs. In order to obtain the peak stress, the peak load was divided by area of bracket base. Following de bonding, the fracture sites were visually inspected with a magnifying glass to determine the location of bond failure.

## **DETERMINATION OF RETENTIVE STRENGTH OF Ni Cr ALLOY CROWNS**

### ***Materials and Method***

Freshly extracted human premolars

Auto polymerizing resin

Hydrogen peroxide

Distilled water

Resin cements

### ***Sample preparation***

Freshly extracted human pre molars were used for the study. The extracted tooth was cleaned with 3% hydrogen peroxide to remove the debris and was stored in distilled water. Grooves were made in the radicular portion of the teeth with diamond bur for added retention of teeth in acrylic resin. Self cure acrylic resin was mixed and poured into a 25mm diameter & 25mm height polyethylene cup, at about 5 minutes from the start of mixing the stored tooth was mounted on to the acrylic resin in such a way that the root portion remained within the acrylic, the crown projected out of acrylic & the long axis of the teeth remained parallel to the vertical plane. In order to prepare the teeth according to the specification centering of the teeth in the acrylic resin base was required. This was achieved by Gedee Weiler India conventional lathe model LZ300G. The tooth crown was gripped in the collet of the lathe and the long axis of the teeth being considered as the long axis of the specimen all excess acrylic was removed in concentric fashion till the diameter reduced to 20 mm. This reduction facilitates the attachment of the specimen in the collet of HMT model GTC-28 universal tool and cutter grinder machine.

### ***Tooth preparation***

Tooth preparation was done in a HMT model GTC-28 universal tool and cutter grinder machine. (FIG VIII) This has a work head collets which can hold the acrylic base of the specimen and a 180 mm diameter and 13 mm thick diamond wheel rotating at a speed of 2800 RPM. The machine has a tilting work head which can move around 360° in both vertical and horizontal plane. Two degree taper was set in horizontal plane to achieve an overall taper of 4° to the preparation. The embedded specimens were held in the collets such that the long axis of the teeth was in horizontal plane. Depending on the size of the individual teeth reduction was carried out on all aspects namely the buccal, lingual, mesial & distal till a definite margin was achieved at the cervical area. The occlusal aspect of the tooth was made flat for study purpose. Each specimen had the crown prepared such that the preparation

resembled an inverted cone with greater diameter at the cervical area and lesser at the occlusal aspect. (FIG IX) The specimens were numbered properly and were then stored in distilled water.

### ***Preparation of Ni Cr alloy crowns***

Ni Cr alloy crowns were fabricated in a dental laboratory. Wax patterns were made conventionally with flat occlusal aspect with a loop which facilitate testing the numbers of the corresponding specimens were marked superficially on the wax pattern the patterns were invested and cast using Ni Cr alloy .The crowns thus obtained was sand blasted on the inner surface with aluminum oxide 110µm and finished . Fit of the crown was checked on the corresponding teeth, the crowns were not less than 1mm thickness all around.

### ***Trimming of the acrylic base***

In order to facilitate the attachment of the specimen on to the holding device of the universal testing machine the acrylic base of the specimen was trimmed flat to a thickness of less than 6mm. Trimming was done using stone disc in a conventional dental lathe. All the specimens were measured for average height, greater and lesser diameter and total surface area was calculated using the formula  $A = \{\pi S (R+r)\} + \pi r^2$

### ***Conditioning of tooth surface for luting of crowns***

Luting of the crown to the prepared tooth surface was done With Relay X ARC 3M and DCRC. Acid etching was carried out on prepared tooth surface with 37% phosphoric acid for 15 seconds, Washed with distilled water, excess water was removed with tissue paper. First thin coat of single bond bonding agent was applied kept aside for 30 sec excess bonding agent was removed with gentle blow of air and was subjected to light curing for 10 sec. The second coating of single bond bonding agent was applied as mentioned above and cured for 10 seconds.

### ***Luting with Rely X ARC***

Equal amount of paste A and paste B of Rely X ARC was dispensed into a clean mixing pad and was mixed with a clean plastic spatula for 60 seconds the inside surface of the crown was

coated with the cement using a plastic carrying instrument . The crown was then seated on to the tooth surface with gentle pressure till it was completely seated .light curing was carried out at the margins for 10 seconds. Excess cement was removed from the margins with a sharp instrument curing was done for another 30 seconds. Separate mix of cement was used for cementing each crown.

### ***Luting with DCRC***

Equal amounts of paste A and paste B of DCRC was dispensed into a clean mixing pad and was mixed with a clean plastic spatula for 60 seconds. Cementation was carried out as mentioned above.

All the cemented specimens were kept at room temperature for 15 minutes. It was then stored in distilled water at 37°C for 23 hours one hour before testing the specimens were brought to room temperature of 22°C. Testing was carried out in a universal testing machine Instron 1011 series with a cross head speed of 1mmper minute. (FIG X)

The UTM has a movable upper head and a stationary lower head. The specimens were clamped to the lower head at the acrylic base. A stainless steel wire was passed through the loop in the crown and tensile load was applied at a cross head speed of 1mm/minute till the break occurred (FIG XI) .A minimum of ten specimens were tested for each cement and the and standard deviations were calculated.

### **MICRO LEAKAGE**

The study was done on extracted human premolars ten maxillary premolars were selected for the study. Class V cavities were made on the buccal aspect using an airtor hand piece and diamond burs. The axial wall of the cavity was placed in dentine. The cavities were washed with distilled water, dried with a gentle blow of air. Etching was carried out with 37% phosphoric acid for 15 seconds. It was then washed and dried with distilled water. Excess water was removed from the surface. The first layer of bonding agent was applied, gently blowed with air to remove the excess,

and was cured with light for 10 seconds. Second layer of bonding agent was applied and cured as mentioned above. Equal amount of base and catalyst paste of DCRC was mixed and the cavities were restored using the cement and one minute after mixing the restorations were subjected to light curing for 40 seconds. Five cavities were restored with DCRC. The same protocol was followed for RelyX ARC resin cement. The entire restored tooth was stored independently.

### ***Thermo cycling of the samples***

All the restored samples were subjected to thermo cycling 500 cycles in Haake W 15 at 5, 22, & 55 c° with dwell time of 20 seconds each (FIG XII).

**Staining :** After thermo cycling the root apex was sealed using green stick impression compound, clear nail polish was applied all over the tooth surface except 1mm around the restoration and the coronal portion with the restoration was dipped in 50% silver nitrate solution in a dark container and was stored in a dark space for two hours. After two hours the samples were washed thoroughly with running water and then with distilled water. It was then developed in a developing solution and was exposed to light for 10 hours.

### ***Embedding and sectioning***

The stained samples were then embedded in Epoxy resin. After 24 hours the samples were sectioned buccolingually through the middle of the restorations. Sectioning was done in a slow speed diamond cutter. It was then polished in an ecomet polisher using 10 µm alumina and was viewed under a stereo microscope and was graded as follows.

Score 0 = No leakage

Score 1 = Dye penetrated to one half of external wall.

Score 2 = Dye penetrated to the entire external wall but not up to axial wall.

Score 3 = Dye penetrated up to axial wall.

## **WATER SORPTION AND SOLUBILITY TEST FOR DCRC**

**AIM** To determine the water sorption and solubility of dual cure resin cement.

### **Materials and methods**

Teflon mould 15mm diameter and 1mm height

Desiccators

Silica gel

DCRC paste

Light cure unit

Cellophane sheet

C Clamp

### **Procedure**

The Teflon ring was placed over a cellophane sheet supported by a metal plate. Equal amount of paste A & paste B of DCRC was mixed on a mixing pad, the mould was filled with it. Another cellophane sheet was placed over it and was covered with a metal sheet. The entire assembly was then clamped using a c-clamp and was kept aside for three minutes. After three minutes the clamp was removed and light curing with LED was done in an overlapping fashion around the circumference on both sides. Each exposure was made for 40 seconds on each sides. Five such specimens were made. It was removed from the mould. Fins were removed by abrading with 1000 grit emery paper (FIG XV). All the specimens were cleaned in soap solution washed with distilled water and was ultrasonically cleaned for 10 minutes. The specimens were moped with tissue paper to remove excess water, and was stored in a desiccators containing dried silica gel at 37c° for 23 hours and then stored in a desiccators at 22 c° for one hour (FIG XVI). The weight was then measured to an accuracy of 0.1mg. The cycle was repeated until a constant mass was obtained and was recorded as M1. The diameter of the specimen is measured by taking two measurements at

right angles. The mean is taken. The thickness at the center and four points at periphery were taken and the mean calculated. The mean diameter and thickness is calculated and the volume V of the specimen in cubic millimeter is determined. The specimen were immersed in distilled water such that they are kept vertical. It was stored for 7 days at 37°C in an incubator (FIG XVII). After seven days the specimen were taken out and the visible water was blotted away. The samples were waved in air for 15 seconds and the weight was record at as M2. After weighing recondition the specimen as mentioned before till a constant mass is obtained (FIG XVIII). Weigh this and record it as M3. Calculate the water sorption and solubility using the following formula.

$$\text{Water sorption } W_{sp} = \frac{M2 - M1}{V}$$

$$\text{Water solubility } W_{sl} = \frac{M1 - M3}{V}$$

### **BIOCOMPATIBILITY:**

It is the ability of a material to elicit an appropriate biological response in a given application in a body. The following tests are carried out to check the biocompatibility of the material as per ISO 7405 read with ISO 10993. (1)Pulp and dentine test on pig (2)Subcutaneous implantation on rats (3)Skin irritation test on guinea pigs (4) Sensitization and delayed hyper sensitivity on rabbits.

### **TEST FOR LOCAL EFFECTS AFTER IMPLANTATION**

#### ***Sub cutaneous implantation in rats***

**AIM** To access the biological response of subcutaneous tissue on implanted material

#### ***Materials***

Chitra dual cure resin cement: Test material : Disc of 1cm diameter & 1mm thickness

Light cure composite: Control material: Disc of 1cm diameter & 1mm thickness

#### **PROCEDURE**

##### ***Preparation of samples***

The Teflon ring was placed over a cellophane sheet supported by a metal plate. Equal amount of paste A & paste B of DCRC was mixed on a mixing pad, the mould was filled with it. Another

cellophane sheet was placed over it and was covered with a metal sheet. The entire assembly was then clamped using a c-clamp and was kept aside for three minutes. After three minutes the clamp was removed and light curing with LED was done in an overlapping fashion around the circumference on both sides each exposure was made for 40 seconds on each side. Specimens were removed from the mould and flash was removed by abrading with 1000 grit emery paper. 80 such samples were made with DCRC which was used as a test material, and another 80 samples were made with chitra light cure dental composite was used as control. All the specimens were cleaned in 5% soap solution washed with distilled water and was ultrasonically cleaned for 10 minutes. The samples were dried in an oven at 37°C for 24 hours and were packed in an air tight container.

### ***Subcutaneous implantation***

Adult rats of 200 – 300 gm of both sex were used for implantation. Total of 18 animals were used for the study and was divided into three groups of six each. Subcutaneous implantation was done on the dorsal aspect incision was made on either side of mid line such that the base of the subcutaneous pocket were more than 1mm from the line of incision (FIGIX). The samples were implanted in such a way that they do not contact each other. Test material was implanted on the left side and control was implanted on right side one sample was placed on each pocket. Two interrupted sutures were placed to close the incision. The animals were housed separately. After a period of Seven days the first groups of animals were sacrificed and the implanted material along with surrounding skin and subcutaneous tissue was removed and stored in formalin for histopathological evaluation. The other two groups were sacrificed after a period of 28 and 84 days. In all the three groups, the implanted site did not show any visible signs of inflammation or hemorrhage.

The implanted material both control and test was excised along with the surrounding skin and subcutaneous tissue and was stored in 10% formalin for histopathological evaluation.

## MAXIMIZATION TEST FOR DELAYED HYPERSENSITIVITY

**AIM** To determine the potential for a substance or material under test to produce skin sensitization in guinea pigs.

### **PROCEDURE**

#### ***Preparation of samples***

Equal amount of Paste A and Paste B of DCRC was mixed in a clean mixing pad using a clean spatula the mix was then filled into a circular mould which was kept on a cellophane sheet and metal plate, the mould was then covered with a cellophane sheet and metal plate. It was then clamped with a C- clamp and was kept aside for 3 min, it was then subjected to light curing for 40 seconds. The disc was then removed from the mould. 40 such disc was made it was then washed in 5% soap solution ultrasonically cleaned in distilled water for 10 minutes and was dried in an incubator at 37 c° for 24 hours. The dried samples were then powdered in a ball mill for 3hours around 40 gm of powder was obtained. Extract of the test material was made and the solutions for intradermal injections were as follows:

- (1) 50:50 (v/v) mixture of Freund's complete adjuvant mixed with physiological saline.
- (2) The test material extract /control alone.
- (3) The test material extract /control emulsified in 50:50 (v/v) mixture of Freund's complete adjuvant mixture.

***Intra dermal induction phase:*** Pair of 0.1ml intra dermal injections of each of the above solutions was made at three respective sites on either side of clipped intracarpular region of guinea pigs.

***Topical induction phase:*** Seven days after intra dermal injections, the test and control extracts (in physiological saline) were topically applied to intracarpular region of each guinea pig. 48 hours later the dressings and patches were removed. (24 hours prior to topical application, the intra dermal sites were pre treated with 10% sodium lauryl sulfate.) (FIGXXI).

**Challenge phase:** Fourteen days after topical application, the test and control animals were challenged with test material extract. The patches and dressings were removed after 24 hours.

The appearance of challenge skin sites of test and control animals were observed after 24, 48, & 72h after removal of dressings and patches. The skin reactions for erythema and oedema were scored and recorded the numerical grading as per ISO 10993-10: 2002.

## **INTRACUTANEOUS (INTRADERMAL) REACTIVITY TEST**

**AIM:** To assess the potential of the material under test to produce irritation following intradermal injection of the material extract in rabbits.

Physiological saline (NS) and cotton seed oil (CSO) extract of the powdered material was used as the test material and physiological saline and cotton seed oil was used as control. Healthy active adult albino rabbits of 2 kg not used for any other purpose were used for the study. Physiological saline (NS) and cotton seed oil (CSO) test extract was aseptically injected into 5 sites (0.2ml/site) each on upper left hand side (NS) and upper right hand side (CSO) of two rabbits. The (NS) and (CSO) control extract was injected into five sites each on lower left hand side (NS) and lower right hand side (CSO) of the same rabbit. The grading of erythema and oedema of test and control sites of all animals at 24, 48 and 78 h were recorded (FIGXX).

## **PULP AND DENTINE TEST IN PIGS**

The pulp and dentine test is part of the preclinical mandatory requirements prescribed for evaluation of biocompatibility of medical devices used in dentistry as per ISO7405 read with ISO 10993. The objective is to assess the response of the dental pulp and dentine to clinical procedures involved with dual cure flowable resin cement.

### **METHODS**

The pulp and dentine test is part of the preclinical mandatory requirements prescribed for evaluation of biocompatibility of medical devices used in dentistry as per ISO 7405 read with ISO 10993. The objective is to assess the response of dental pulp and dentine to clinical procedures

involved with dual cure flowable resin cement. Animals stipulated for this mandatory test as per ISO 7405 constitute Monkeys, Dogs or miniature pigs in which all permanent teeth other than third molars has been erupted. Three adult pigs one for each time period nearly 40 -60 Kg weight was selected for the study. The time period for each animal is  $7 \pm 2$  days,  $28 \pm 3$  days, and  $70 \pm 5$  days. Each animal should have at least seven teeth containing test material, and four teeth containing imported control material.

The animal was anesthetized and following procedures are carried out. The cheliotomy was performed in order to gain access to the posterior teeth. All calculus debris from teeth surface is removed mechanically. The surfaces of the teeth is then scrubbed using 3% hydrogen peroxide and disinfected with povidone iodine, Class V cavities were made on the buccal aspect of molars and premolars using straight fissure diamond bur in a micro motor contra angle hand piece with adequate cooling with saline externally, care was taken to place the pulpal floor in dentine. Seven cavities were made for the test material and four for the control material. The cavities were then washed with distilled water excess water was removed using a dry gauze piece. After proper isolation the cavities were then dried using a gentle blow of air from a chip blower.

Etching of the walls of the cavities were carried out using 37% phosphoric acid for 15 seconds, washed with distilled water to remove the acid layer, excess water was removed using gauze. First coat of Single bond bonding agent was applied and excess was removed with a gentle blow of air and curing was done with a light source from an LED for 10 seconds. The second coat was applied and cured as mentioned above. Equal amount of base paste and catalyst paste of DCRC test material was mixed in a clean paper pad using a clean plastic spatula. The cavities were restored using this and was cured using a light from an LED for 40 seconds. Total of seven cavities were restored using test material, and two cavities were restored at a time. The same procedure was carried out for the RelyX ARC test material and total of four cavities were restored using control.

The same procedures were carried out in all the animals. The animal was caged separately and was kept under observation. Autopsy of each animal to retrieve the restored teeth was carried out after a period of  $7 \pm 2$  days,  $28 \pm 3$  days, and  $70 \pm 5$  days. (FIGXXII).

## *RESULTS AND DISCUSSION*

# RESULTS AND DISCUSSION

## DETERMINATION OF FILM THICKNESS OF DUAL CURE RESIN CEMENT

From the study done it was found that the film thickness of DCRC was found to be 40µm. This fulfills the specification as per ISO 4049:2000(E) for polymer based filling restorative and luting materials , which states that the film thickness of luting materials when determined shall be no more than 10 µm above any value claimed by the manufacturer and in any event shall be no greater than 50 µm.(Table I)

## DETERMINATION OF FLEXURAL STRENGTH OF DCRC

Mean flexural strength of DCRC after 24 hrs is  $50.18 \pm 7.17$  MPa. (Table II) And the mean flexural strength of DCRC after 7days is  $52.79 \pm 5.43$  MPa.(Table III) Both these values are found to fulfill the requirements specified by ISO 4049:2000(E) for polymer based filling restorative and luting materials , which states that the flexural strength of luting materials Type 2 Class3 should be a minimum of 50 MPa.

## DETERMINATION OF DIAMETRAL TENSILE STRENGTH OF DCRC

The diametral tensile strength of DCRC was found to be  $29.96 \pm 2.1$  MPa. (Table IV)

## COMPARISON OF SHEAR BOND STRENGTH

The mean shear bond strength of DCRC when used with bonding agent was  $4.322 \pm 0.589$  MPa. The mean shear bond strength of Rely X ARC was  $5.393 \pm 0.749$  MPa and The mean shear bond strength of Vario link light cure was  $5.254 \pm 1.568$  MPa. (Table V) With out the use of bonding agent DCRC gave the mean value of  $6.616 \pm 1.629$  The mean shear bond strength without bonding agent for Rely X ARC was  $5.565 \pm 0.965$  MPa. And the mean shear bond strength of Vario link light cure was  $5.597 \pm 0.968$  MPa. (Table VI)

## **COMPARISON OF RETENTIVE STRENGTH Ni Cr ALLOY CROWNS LUTED WITH DCRC & Rely X ARC**

The mean retentive strength of Ni Cr alloy crowns luted with DCRC is  $4.1175 \pm 0.618$  MPa (Table IX) The mean retentive strength of Ni Cr alloy crowns luted with RelyXARC is  $4.109 \pm 0.769$  MPa (Table X) Statistical analysis of the above mentioned result did not show any significant difference in mean shear bond strength.

### **MICRO LEAKAGE**

On examination under stereo microscope all the samples in test as well as control group showed dye penetration up to the axial wall. This gave a score of 3 for all the samples. Thus both the test as well as the control material showed equal amount of micro leakage when used for restoration. (FIG XIII, XIV)

### **WATER SORPTION AND SOLUBILITY TEST FOR DCRC**

Water sorption of DCRC  $38.5 \pm 2.11 \mu\text{g} / \text{mm}^3$  (Table XI, XII)

Water solubility of DCRC  $2.58 \pm 0.589 \mu\text{g} / \text{mm}^3$

As per ISO 4049 the water sorption of all materials shall be less than or equal to  $40 \mu\text{g} / \text{mm}^3$  and the water solubility should be less than or equal to  $7.5 \mu\text{g} / \text{mm}^3$  As per the results obtained DCRC fulfills the requirements as mentioned in ISO 4049

### **SUB CUTANEOUS IMPLANTATION IN RATS**

The general physical condition of all experimental animals was normal. The increase in body weight and feed intake were normal and none of the animals showed any abnormality or behavioral changes during the experimental period. Histopathological report says that there was no encapsulation, infection or necrosis around the test and control implanted materials at one, four, and twelve weeks of observation period. The biological response around the test implant site was comparable with the biological response around the control implant sites at one, four, and twelve

weeks of post implantation period. There is evidence of repair with formation of collagenous capsule at 12 weeks post implantation in both test and control group.

### **MAXIMIZATION TEST FOR DELAYED HYPERSENSITIVITY IN GUINEA PIGS**

*Evaluation criteria:* Grades of 1 or greater in the test group generally indicate sensitization, provided grades of less than 1 are seen on control animals. If grades 1 or greater are noted on control animals, then the reactions of test animals that exceed the most severe control reaction are presumed to be due to sensitization. If the response is equivocal, a re-challenge is recommended to confirm the results from the first challenge. (Table XIII)

The results indicate that none of the animals in the test or control groups showed any adverse skin reaction during the induction or challenge period. (Table XIV) The extract of test material induced a numerical grading of '0' for erythema and oedema. Hence the physiological saline extract of the material meets the requirements of the test as per ISO 10993-10:2002(E) Clause 7.4. Maximization test for delayed hypersensitivity.

### **INTRACUTANEOUS INTRADERMAL REACTIVITY TEST (RABBITS)**

The results indicate that the physiological saline extract of test material i.e. DCRC did not produce any irritation following intradermal injection. Whereas the cotton seed oil extract of the material induced an average irritation score of 0.017 which is under acceptable level. (Table XV, XVI) Hence the test material DCRC meets the requirements of test as per ISO 10993-10: 2002(E) – Biological evaluation of medical device: Part 10: Tests for irritation and delayed hypersensitivity: Annex B Additional irritation test: B.2: Intra cutaneous reactivity test.

## **PULP AND DENTINE TEST IN PIGS**

Autopsy was performed for all the three animals at respective observation period of 7, 28, & 70days. All the restorations were found to be intact in all animals. The animal was euthanized and the individual teeth were removed by open method using a chisel and mallet. Care was taken not to damage the radicular portion or the pulp. The extracted tooth was then stored in individual bottles containing 10% formalin and was transferred to the histopathology lab. Results waited.

**Table I Determination of film thickness**

SLNO	Thickness of glass plates With out cement mm	Thickness of glass plates With cement mm	Film thickness of the cement DCRC
1	9.56	9.61	.04mm = 40µm
2	9.56	9.6	
3	9.57	9.62	
4	9.74	9.77	
5	9.71	9.74	
Mean	9.628	9.668	

**Table II Determination of flexural strength after 24 hours**

SI NO	Width	Depth	Flexural strength	Mean FS
1	1.98	2.11	57	50.18±7.17MPa
2	2.22	2.41	54.09	
3	2.08	2.2	45.44	
4	2.12	2.36	53.6	
5	2.24	2.36	39.98	

**Table III Determination of flexural strength after 7 days**

SL NO	Width	Depth	Flexural strength	Mean FS
1	2.22	2.42	53.07	52.79±5.43MPa
2	2.23	2.47	44.93	
3	2.42	2.56	58.4	
4	2.26	2.17	50.41	
5	2.75	2.30	57.13	

**Table IV Determination of diametral tensile strength of Chitra dual cure resin cement**

SL NO	Diameter	Thickness	Area	Load N	DTS MPa	Mean MPa
1	5.92	2.86	26.58	790.5	29.74	29.96±2.1
2	5.82	2.85	26.04	727.5	27.94	
3	5.84	2.89	26.49	793.5	29.95	
4	5.82	2.90	26.4	862.5	32.67	
5	5.84	2.94	26.95	740	27.46	
6	5.82	2.93	26.77	858	32.05	

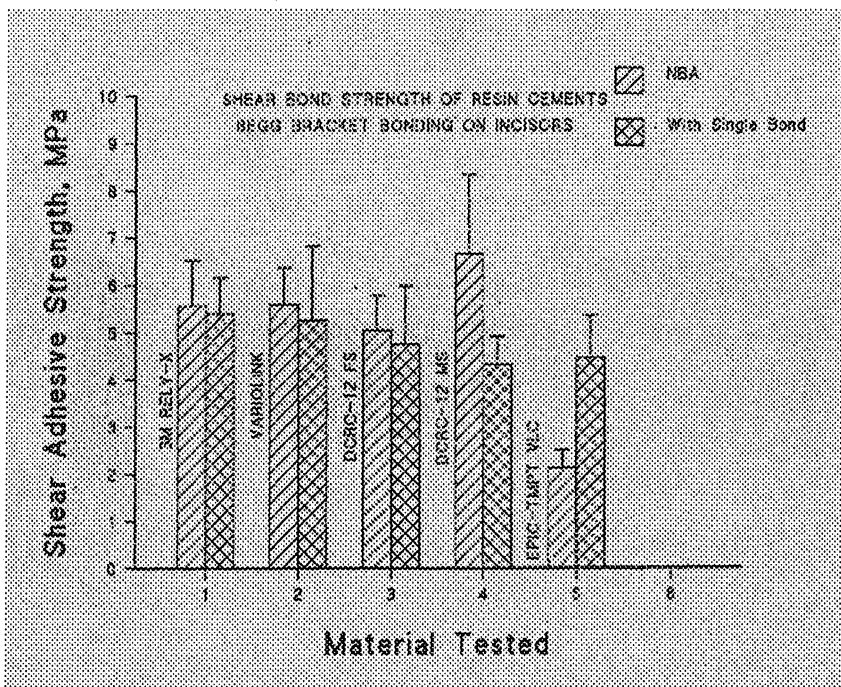
**Table V Mean shear bond strength with bonding agent**

Groups	Resin Cement	Bonding Agent	Mean	Std Dev
GRIA	Rely X 3M	Single bond	5.393	0.749
GRIB	Vario link light cure	Single bond	5.254	1.568
GRIC	Chitra dual cure resin cement	Single bond	4.322	0.589

**Table VI Mean shear bond strength without bonding agent**

Groups	Resin Cement	Bonding Agent	Mean	Std Dev
GRIIA	Rely X 3M	No bonding agent	5.565	0.965
GRIIB	Vario link light cure	No bonding agent	5.597	0.768
GRIIC	Chitra dual cure resin cement	No bonding agent	6.616	1.629

**FIG (XXIII) Graph showing the mean shear bond strength**



**Table VII Statistical analysis of mean bond strength with and without bonding agent using ANOVA**

Resin Cements	Mean Without bonding Agent MPa	Mean With Bonding Agent MPa	P Value
RelyX 3M	5.565 ±0.965	5.400 ± 0.749	0.750
Vario Link LC	5.597± 0.768	5.254 ± 1.568	0.641
Chitra dual cure resin cement	6.616 ± 1.69	4.322 ± 0.589	0.0093

**Table VIII Inter comparison of mean bond strength of resin cements without bonding agent using ANOVA**

Group	Mean	Comparison Between Groups	P Value	Significance
GRIIA	5.565	GRIIA - GRIIB	0.948	No Significance
GRIIB	5.597	GRIIA - GRIIC	0.197	No Significance
GRIIC	6.616	GRIIC - GRIIB	0.190	No Significance

**Table IX Retentive strength of Ni Cr alloy crowns luted with Chitra dual cure resin cement**

Sample no	Load at break	Surface area	Adhesive strength	Mean
1	3.115	81	3.845	4.11±0.61MPa
2	497	127.65	3.89	
3	374	98.73	3.78	
4	229.5	54.88	4.07	
5	272	72.19	3.76	
6	322.8	60.17	5.36	

**Table X Retentive strength of Ni Cr alloy crowns luted with Rely X ARC**

Sample no	Load at break	Surface area	Adhesive strength	Mean
1	311.5	81	3.845	4.10± 0.769MPa
2	497	127.65	3.89	
3	374	98.73	3.78	
4	229.5	54.88	4.07	
5	272	72.19	3.76	
6	322.8	60.17	5.36	

**Table XI Weight of samples for Water sorption and solubility test**

Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
0.35152	0.3338	0.3647	0.3530	0.3736
0.3504	0.3326	0.3635	0.3519	0.3725
0.3500	0.3317	0.3622	0.3515	0.3720
0.3499	0.3320	0.3628	0.3511	0.3716
0.3493	0.3315	0.3624	0.3508	0.3713
0.3492	0.3315	0.3622	0.3507	0.3711
0.3489	0.3313	0.3620	0.3505	0.3709
<b>M1<sub>1</sub></b>	<b>M1<sub>2</sub></b>	<b>M1<sub>3</sub></b>	<b>M1<sub>4</sub></b>	<b>M1<sub>5</sub></b>
0.3491	0.3313	0.3620	0.3505	0.3710

**M2 of five specimens**

<b>M2<sub>1</sub></b>	<b>M2<sub>2</sub></b>	<b>M2<sub>3</sub></b>	<b>M2<sub>4</sub></b>	<b>M2<sub>5</sub></b>
0.3572	0.3394	0.3703	0.3588	0.3793

**Table XI Weight of samples for Water sorption and solubility test**

Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
0.35152	0.3338	0.3647	0.3530	0.3736
0.3504	0.3326	0.3635	0.3519	0.3725
0.3500	0.3317	0.3622	0.3515	0.3720
0.3499	0.3320	0.3628	0.3511	0.3716
0.3493	0.3315	0.3624	0.3508	0.3713
0.3492	0.3315	0.3622	0.3507	0.3711
0.3489	0.3313	0.3620	0.3505	0.3709
<b>M1<sub>1</sub></b>	<b>M1<sub>2</sub></b>	<b>M1<sub>3</sub></b>	<b>M1<sub>4</sub></b>	<b>M1<sub>5</sub></b>
0.3491	0.3313	0.3620	0.3505	0.3710

**M2 of five specimens**

<b>M2<sub>1</sub></b>	<b>M2<sub>2</sub></b>	<b>M2<sub>3</sub></b>	<b>M2<sub>4</sub></b>	<b>M2<sub>5</sub></b>
0.3572	0.3394	0.3703	0.3588	0.3793

0.3525	0.3345	0.3656	0.3540	0.3747
0.3512	0.3333	0.3644	0.3528	0.3733
0.3500	0.3321	0.3632	0.3517	0.3718
0.3496	0.3318	0.3630	0.3512	0.3717
0.3494	0.3317	0.3625	0.3508	0.3714
0.3494	0.3315	0.3620	0.3508	0.3712
0.3491	0.3313	0.3621	0.3506	0.3709
0.3491	0.3313	0.3621	0.3506	0.3711
0.3490	0.3312	0.3620	0.3504	0.3709
0.3488	0.3311	0.3617	0.3502	0.3707
0.3486	0.3310	0.3616	0.3500	0.3705
0.3484	0.3309	0.3616	0.3500	0.3704
<b>M3<sub>1</sub></b>	<b>M3<sub>2</sub></b>	<b>M3<sub>3</sub></b>	<b>M3<sub>4</sub></b>	<b>M3<sub>5</sub></b>
0.3484	0.3308	0.3616	0.3500	0.3703

**Table XII Water sorption and solubility for Chitra dual cure resin cement**

NO of samples	Volume of samples	Water sorption	Water solubility
1	231.21mm <sup>3</sup>	35 μ g/mm <sup>3</sup>	3 μ g/mm <sup>3</sup>
2	199.2 mm <sup>3</sup>	40.6 μ g/mm <sup>3</sup>	2.5 μ g/mm <sup>3</sup>
3	215.51mm <sup>3</sup>	38.5μ g/mm <sup>3</sup>	1.8 μ g/mm <sup>3</sup>
4	213.73 mm <sup>3</sup>	38.8μ g/mm <sup>3</sup>	2.3 μg /mm <sup>3</sup>
5	209.32 mm <sup>3</sup>	39.6μg /mm <sup>3</sup>	3.3μg /mm <sup>3</sup>

**Table XIII Magnusson and Kligman Scale for Maximization test for delayed hypersensitivity**

Patch test reaction	Grading Scale
No visible change	0
Discrete or patchy erythema	1
Moderate or confluent erythema	2
Intense erythema and swelling	3

**Table XIV Scores for skin reaction in maximization test for delayed hypersensitivity**

Animal No	Group	Skin Reaction					
		Erythema			Oedema		
		24h	48h	72h	24h	48h	72h
1	Test	0	0	0	0	0	0
2	Test	0	0	0	0	0	0
3	Test	0	0	0	0	0	0
4	Test	0	0	0	0	0	0
5	Test	0	0	0	0	0	0
6	Test	0	0	0	0	0	0
7	Test	0	0	0	0	0	0
8	Test	0	0	0	0	0	0
9	Test	0	0	0	0	0	0
10	Test	0	0	0	0	0	0
11	Control	0	0	0	0	0	0
12	Control	0	0	0	0	0	0
13	Control	0	0	0	0	0	0
14	Control	0	0	0	0	0	0
15	Control	0	0	0	0	0	0

**Table XV Irritation Score Intracutaneous intradermal reactivity test**

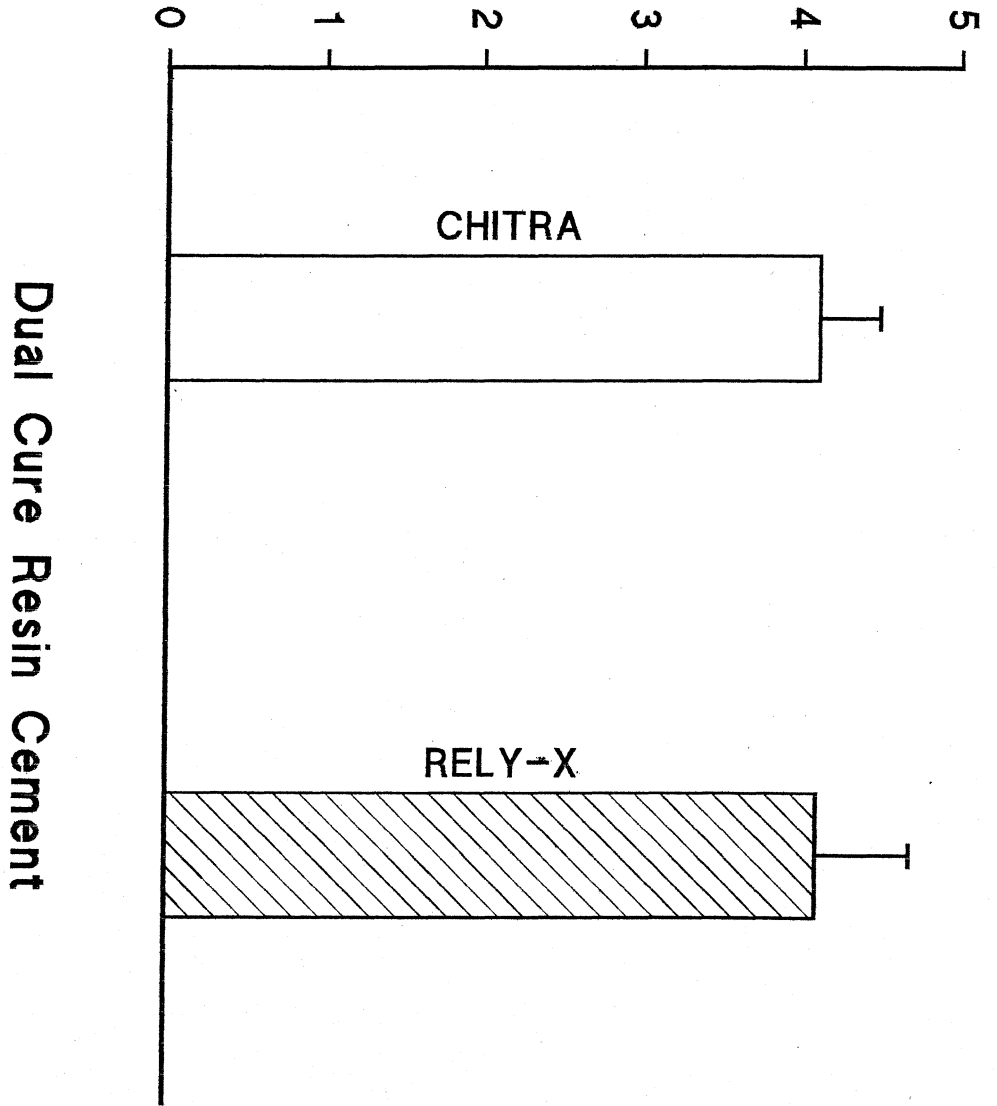
Particulars	Erythema		Oedema	
	NS	CSO	NS	CSO
Animal 1	0	0.2	0	0
Animal 2	0	0	0	0
Average irritation score	0	0.017	0	0

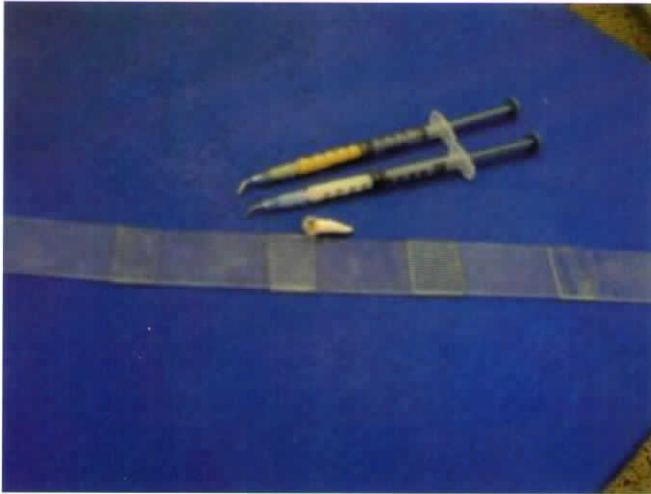
**Table XVI Observation for Intracutaneous intradermal reactivity test**

SL NO	Animal id No		Observation at		
			24 h	48 h	72 h
1	445 ♀	Erythema	<u>Test site</u> NS = 0,0,0,0 CSO = 0,1,0,0	NS = 0,0,0,0 CSO = 0,0,0,0	NS = 0,0,0,0 CSO = 0,0,0,0
			<u>Control site</u> NS = 0,0,0,0 CSO = 0,0,0,0	NS = 0,0,0,0 CSO = 0,0,0,0	NS = 0,0,0,0 CSO = 0,0,0,0
		Oedema	<u>Test site</u> NS = 0,0,0,0 CSO = 0,0,0,0	NS = 0,0,0,0 CSO = 0,0,0,0	NS = 0,0,0,0 CSO = 0,0,0,0
			<u>Control site</u> NS = 0,0,0,0 CSO = 0,0,0,0	NS = 0,0,0,0 CSO = 0,0,0,0	NS = 0,0,0,0 CSO = 0,0,0,0
2	457 ♀	Erythema	<u>Test site</u> NS = 0,0,0,0 CSO = 0,0,0,0	NS = 0,0,0,0 CSO = 0,0,0,0	NS = 0,0,0,0 CSO = 0,0,0,0
			<u>Control site</u> NS = 0,0,0,0 CSO = 0,0,0,0	NS = 0,0,0,0 CSO = 0,0,0,0	NS = 0,0,0,0 CSO = 0,0,0,0
		Oedema	<u>Test site</u> NS = 0,0,0,0 CSO = 0,0,0,0	NS = 0,0,0,0 CSO = 0,0,0,0	
			<u>Control site</u> NS = 0,0,0,0 CSO = 0,0,0,0	NS = 0,0,0,0 CSO = 0,0,0,0	NS = 0,0,0,0 CSO = 0,0,0,0

FIG XXIV

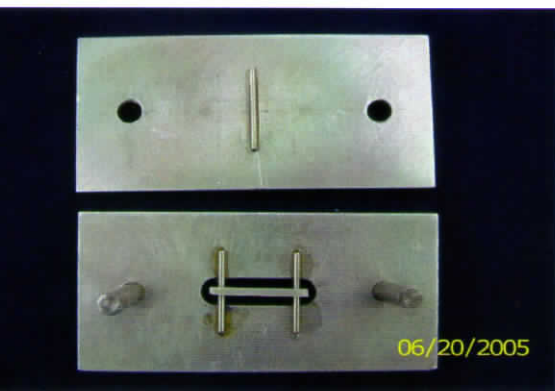
Adhesive Strength, MPa



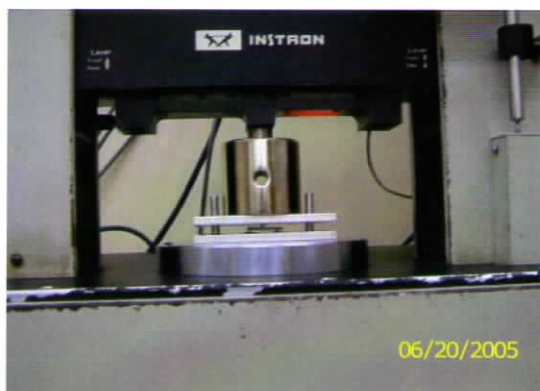


**FigI CHITRA DUAL CURE RESIN CEMENT (DCRC)**

**Determination of Flexural strength of DCRC**



**FIG II FS samples on the jig**



**FIG III 3-Point bend test for FS**

**Determination of diametral tensile strength of DCRC**



**FIG IV DTS samples and mould**



**FIG V Test for DTS**

**Determination of shear bond strength of DCRC**

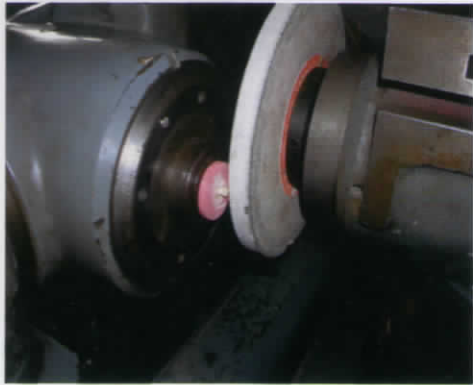


**FIG VI brackets attached to tooth surface**



**FIG VII Shear pull test**

**Determination of adhesive strength of DCRC**



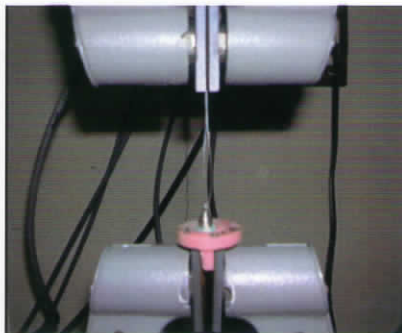
**FIG VIII Tooth preparation**



**FIG IX Prepared tooth**



**FIG X Crown cemented on tooth**

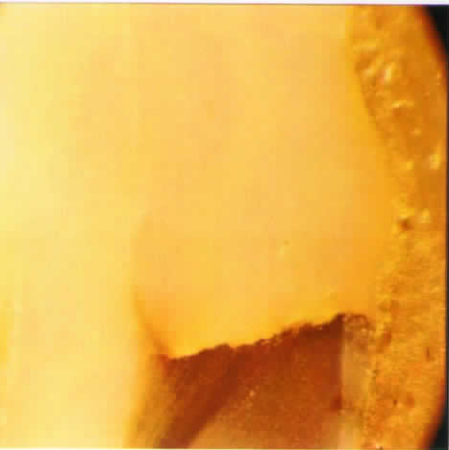


**FIG XI Test for adhesive strength**

## MICRO LEAKAGE STUDY



**FIG XII Dental Thermocycler**

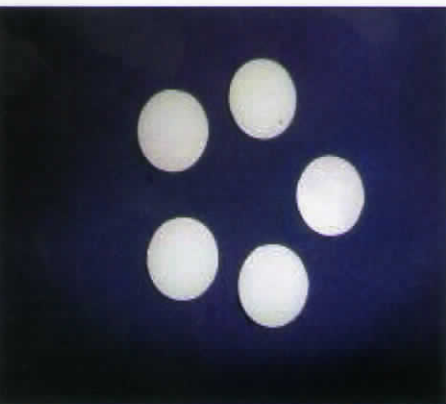


**FIG XIII Microleakage of test material**



**FIG XIV Microleakage of Control Material**

## WATER SORPTION AND SOLUBILITY TEST



**FIG XV Samples for water sorption & solubility**



**FIG XVI Samples in desiccator**



**FIG XVII Samples in incubator**



**FIG XVII Analytical Balance**

**TEST FOR BIOCOMPATIBILITY**



**FIG XIX Subcutaneous implantation in rats**



**FIG XX Intradermal reactivity test in rabbits**



**FIGXXI Sensitization test in Guinea pigs**



**FIG XXII Pulp and dentine usage test in pigs**

## *CONCLUSION*

## CONCLUSIONS

Physico-chemical evaluation of dual cure resin cement (DCRC) developed indigenously was carried out initially. The bond strength of beggs brackets bonded using DCRC (6.61 MPa ) was found to be statistically comparable to that of Rely-X ARC ( 5.57 MPa) and Variolink II (5.60MPa) which were imported controls. Use of bonding agent did not show any appreciable difference in bond strength in case of control materials though they made a significant difference in case of DCRC. It was found that that there is a significant reduction in bond strength ( 4.32 MPa) when Single Bond adhesive was used along with DCRC. So it is advisable to use DCRC without bonding agent for orthodontic bracket applications.

Crown adhesion studies revealed comparable adhesive values for DCRC (4.11 MPa) compared to imported control Rely-X ARC (4.10 MPa). Other properties studied such as flexural strength, diametral tensile strength, film thickness, water sorption and solubility for DCRC gave values which were above the minimum specified values as per international specification ISO 4049. Microleakage studies after thermocycling for 500 cycles at 5, 22 and 55°C using both DCRC and Rely-X ARC on human premolar class V cavities revealed same degree of microleakage in both cases. The results shows that the values obtained for the DCRC are comparable to the values of imported controls and also meets the requirements as per ISO4049.

The toxicological studies of DCRC was carried out as per ISO 7405 & 10993. Tests for local effects after implantation in subcutaneous tissue of albino rats revealed no encapsulation, infection or necrosis around the test material even after 1, 4 and 12 weeks of observation period. The biological response around test implant sites was comparable with the biological response around the control implant sites at 1, 4 and 12 weeks post implantation. Maximization test for delayed hypersensitivity in guinea pigs revealed that the physiological saline extract of DCRC powder meets the requirements of test as per ISO 10993-10. Intracutaneous (intradermal) reactivity test on albino rabbits revealed that the physiological saline extract and cotton seed oil extract of DCRC powder

meets the requirements of the test as per ISO 10993-10. Pulp and dentine evaluation on swine model for 7, 28 and 70 days were carried out and initial observations after autopsy revealed no substantial difference in inflammatory response between DCRC and Rely X-ARC control samples. Detailed histopathological evaluation is in progress.

These observations indicate that the dual cure resin cement developed is meeting all international requirements for further clinical evaluation and commercialization. All properties studied are in compliance with the required standard values. The novel dual cure resin cement can be recommended for crown adhesion, bracket bonding and luting applications in dentistry.

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