Evaluating the Effect of Different Conditioning Agents on the Shear Bond Strength of Resin‑Modified Glass Ionomers

Abstract

Aim of the Study: This study aims to evaluate the effects three different conditioning agents on the shear bond strength of resin‑modified glass ionomers to human dentin. Materials and Methods: One hundred and twenty recently extracted, caries-free premolars and molars will be cleaned of debris and disinfected in a 0.5% solution of sodium hypochlorite and sterile water for 30 min. The occlusal surface of each tooth will be reduced using conventional model trimmer with water to produce the dentin surface. Then, three different resin-modified glass ionomer cements (GICs) were triturated and mixed according to the manufacturer's instructions, 10 specimens will be made of each group. The excess restorative material will be removed from matrix band dentin interface with a sharp number 25 bard parker blade. Samples were shear tested with Instron universal testing machine with a crosshead speed of 0.5 mm/min. A shearing bar beveled to a 1 mm thick contact surface area will be placed at the junction of dentin and plastic band matrix. The load required for the failure will be recorded in pounds and converted to megapascals. **Results:** Statistical analysis was done with analysis of variance and Tukey's test. Ketac primer as conditioning agent along with Fuji II LC as restorative material had the highest shear bond value whereas intact smear layer which was unmodified dentin had the least value. **Conclusion:** Within the limitations of the present study, it can be concluded that surface conditioning of dentin resulted significantly higher bond strength than unconditioned dentin surfaces. **Clinical Significance:** Resin‑modified glass ionomers have several advantages compared to chemically cured GICs. The advantages include command cure, ease of handling, improved physical properties, and esthetics. Resin-modified glass ionomers have been marketed as direct restorative materials for Class V lesions as well as liners, bases, and luting agents. Several conditioning agents have been evaluated to condition dentin before the application of conventional glass ionomers and resin-modified glass ionomers. These have mainly included polyacrylic acid, citric acid, phosphoric acid, and ethylenediamine tetra-acetic acid. Of late, manufactures have recommended other conditioners to replace polyacrylic acid which includes Ketac primer as one of the conditioning agents.

Keywords: *Glass ionomer conditioning agents, resin‑modified glass ionomer, shear bond strength*

Introduction

Bonded restorations have a substantial importance in modern-day restorative dentistry. Since they are stated to be reliably adhesive to the tooth structure, they considerably reduce the need for removal of tooth structure and also eliminate microleakage thus minimizing discolorations, postoperative sensitivity and the risk of secondary caries formation. Complete bonding of a restorative material to tooth structure is hence of the vital significance which directly stimulates the clinical success.[1]

One of the primary tasks in dentistry has always been to invent an ideal restorative material which has physical properties similar to those of natural tooth structure, adhesion to dentin and enamel, also with resistance to degradation in the oral cavity. In an attempt to reach these characteristics, glass ionomer cement (GIC) was fabricated and presented by Wilson and Kent in 1972.

Its initial formulation underwent several transformations with the intention set to improve handling and physical characteristics. A notable improvement of this class of material occurred almost 15 years ago, with the inception of the resin‑modified GIC (RM‑GIC). This material was refined by the addition of photoactivated methacrylate, and 2‑HEMA or bisphenol‑A‑glycidyl methacrylate, a

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resin to the conventional GIC liquid or powder. The present time, RM-GICs set by two or three setting reactions:

- a. Acid-base reaction, classic reaction of conventional GICs (initiated when the powder and liquid are mixed, occurring without light);
- b. Photo-initiated setting reaction occurs through the methacrylate groups (initiated when exposed to light and occurs only where the light penetrates);
- c. free-radical methacrylate curing that occurs without light.[2]

Resin‑modified glass ionomers have several advantages compared to chemically cured GIC. The merits include command cure, ease of handling, improved strength along with esthetics. Resin-modified glass ionomers are marketed and promoted as direct restorative materials for Class V lesions as well as liners, base, and luting agents.[3]

The tooth surface to which the bond has to occur is most commonly covered with an adherent layer of debris when it is prepared known as smear layer. A smear layer can be because of rotary or hand instruments. When the preparation of a cavity with the bur, the cavity is covered with this layer. The smear layer is believed to be composed of shattered and crushed hydroxyapatite, along with denatured and fragmented collagen. *In vivo* circumstances, a smear layer may also be contaminated by bacteria and saliva. The notable disadvantage of a smear layer covering the bonding surface is its inherently reduced bond to the underlying dentin.[4] Thus, to incapacitate the effect of smear layer and boost bonding, different surface treatment agents have been proposed to remove or modify the smear layer before placing of GIC.^[5]

Previously used agents-citric, polyacrylic, tannic, and ethylenediaminetetraacetic acid (EDTA). Polyacrylic acid has been the mainstay for conditioning dentin earlier to the application of CGIs and RMGICs. Recently, other recommendations as conditioners to replace polyacrylic acid have been proposed; they include Ketac Primer (3M ESPE, St Paul MN, USA) and Self Conditioner (GC, Tokyo, Japan).[3]

The liquid of glass ionomer is the most commonly used conditioner for conventional GICs because the polyacrylic acid content is capable of cleansing the dentin surface without completely unplugging the dentinal tubules. The increase in bonding efficiency results from a cleansing effect which removes loose cutting debris following cavity preparation, partial demineralization effect which increases the surface area and creates micro porosities, chemical interaction of the polyalkenoic acid with residual hydroxyapatite.^[6]

EDTA does not alter the fibrillar structure of collagen, allowing the mineral content of collagen to bond with the ionic component of RMGIC's. EDTA did not aggressively decalcify the dentin surface or widely open the dentinal tubules, ensuing in the formation of long and thin resin tags .[7]

Resin-modified GICs are incorporated with polymerizable monomer and a crosslinking agent, resulting in longer working time and less sensitivity to water contamination than conventional GIC. These are also called hybrid ionomer cement. It has advantages of both GIC and also better fracture toughness than conventional GIC.^[8]

Natural and normal tooth structure transfers external biting loads through enamel into dentin as compression forces that are spread over a large internal volume and thus local stresses are lower whereas a tooth which is filled with a restorative material reacts to stress much differently than a natural intact tooth. Any force on the restoration produces tension, compression, or shear stress along the tooth/ restoration interface, leading to complex stress distributions; a mixture of compressive, tensile, and shear stresses. Since the process of mastication is one of indentation, basically related to shearing or cutting away phenomenon, the true nature of adhesive strength of the materials at the interface is portrayed by the shear bond strength.^[9]

Materials and Methods

One twenty freshly extracted, intact, noncarious, and unrestored human premolars and molars were collected from the Department of Oral and Maxillofacial Surgery, A. J. Institute of Dental Sciences, Mangalore, and stored under 10% neutral buffered formalin solution at room temperature. After cleaning the teeth, of all blood, calculus and surface deposits, the teeth were examined by transillumination to rule out any cracks or defects in them. The selected specimens were used within a month of extraction and storage.

Tooth selection and preparation

In this study, 120 caries free, nonrestored recently extracted human premolars and molars were collected. The teeth were thoroughly cleaned for debris and disinfected in a 0.5% solution of sodium hypochlorite and sterile water for 30 min. The teeth were mounted in cold cure acrylic resin prepared with brass jigs with 5 cm \times 2 cm dimensions and the occlusal surfaces were ground flat using a conventional model trimmer with water to expose the dentin.

The specimens are then randomly divided into 12 groups of 10 specimens each. Each group then received a different surface treatment as follows. The experimental group being four and a control group with intact smear layer.

Each of the three experimental groups was treated with conditioning agents whereas the control group was not treated with any conditioning agent. First group with liquid of glass ionomer, second with Ketac primer, and the third with EDTA 17% liquid [Table 1].

In Group I, the dentin surface was conditioned using liquid of glass ionomer for 30 s, rinsed with a copious air/water spray for 10 s and blot dried using sterile cotton, leaving the dentin surface visibly moist (wet bonding). Sub Group I: tooth was conditioned with liquid of glass ionomer and restored with Fuji II LC. In the second subgroup the teeth were conditioned with liquid of glass ionomer and restored with Ketac Nano followed by a group which was restored with vitremer whereas, in GROUP II, Ketac Primer liquid was used as a conditioning agent. KetacN100 primer was applied with a disposable micro brush to the conditioned dentin surfaces for a period of 30 s and light cured. 10 specimens each were conditioned with ketac primer and restored with the three different RMGICS. In GROUP III, EDTA 17% liquid was used as the conditioning agent. The dentin surface was conditioned with EDTA solution for 30 s, rinsed for 20 s and blot dried, followed by restoration with resin-modified GICs. Group four with intact smear layer without conditioning served as the negative control. Plastic band matrices 5 mm in diameter and 2 mm in height were held on the dentin surface by grasping with cotton forceps to hold the band steady. Then resin‑modified GIC was triturated and mixed according to the manufacturer's instructions using finger pressure to compress the restorative materials against dentin [Table 2]. Then, the specimens were light cured for 20 s with an intensity of 5^2 , 10 specimens were made of each group. The excess restorative material was removed from matrix band dentin interface with a sharp No. 25 bard parker blade. Samples were stored for 48 h prior testing, initially for 24 h in 100% humidity at 37°C and then placed in 37°C deionized water for 24 h. The testing assemblies were then mounted in a test jig for the determination of shear bond strength evaluation using Instron Universal Testing Machine. A shearing load is to be applied to the specimens in universal testing machine at a cross-head speed of 0.5 mm/min. A shearing bar beveled to a 1 mm thick contact surface area was placed at the junction of dentin and plastic cylinder interface.

Statistical analysis

The results are statistically analyzed using one‑way analysis of variance (ANOVA) and Tukeys tests (α = 0.05). Results were regarded as statistically significant if *P* < 0.05 [Tables 3 and 4].

Discussion

Teeth need restorative interruption for different reasons. Primary is the need to mend a tooth after destruction from caries^[10] whereas traditionally, more extensive restorations on teeth were executed using nonadhesive techniques. The materials of choice were gold, porcelain, and metallic ceramics. These were placed either intra‑ or extra‑coronally and depend on the cavity preparation having near-parallel walls, with the help of a luting cement to fill the marginal gap and aid with the retention process.[11] With the growth of new materials and facilities for adherence to the tooth, there has been a confusion of the various methods being pragmatic; often restorations rely on a collection of factors

for retention which incorporates both mechanical and adhesive principles.[12]

"Extension for prevention" has been an integral part of dentistry for over 100 years. Since this notion advocated the elimination of sound tooth structure, it was not in total agreement at the turn of the century. The arrival of the gold casting catapulted extension for prevention into general acceptance. In 1883, Webb presented a concept of "prevention of extension of decay." The extension of the margins, along with proper contact and contours, was thought to elevate natural and biological cleansing of the embrasures with fluids in the diet and saliva. GV Black's 1891 idea of "extension for prevention" was to provide extension of the preparation to the facial and lingual line angles with the intention to bring about "self-cleansing" margins through food excursion.^[13]

Although tooth preparations for operative procedures formerly stuck to the concept of "extension for prevention," increased knowledge of prevention methods, improved clinical techniques and advanced restorative materials now have provided more conservative approach. Now, no longer primal "extension for prevention" is practiced but has changed to "constriction with conviction."

Bonded restorations have the highest importance in this day and age of restorative dentistry. Since they are claimed to be adhesive to the tooth structure, they considerably reduce the need for removal of tooth structure and also eliminate microleakage, thus minimizing the discolorations, postoperative sensitivity, and risk of secondary caries formation. Total bonding of a restorative material to tooth structure is hence most vital which directly influences the clinical success.[14]

The smear layer is described as a layer of debris which is created by cutting a tooth.[15]

HEMA: Hydroxyethyl methacrylate; EDTA: Ethylenediaminetetraacetic acid

During treatment of a root canal, either by rotary or hand instruments, such smeared contaminants reduce the surface energy and therefore decrease the reactivity of the substrate surface.^[15] The awareness of the structural qualities of cut surfaces of teeth is a key to formulating adhesive restorative systems.[16]

Scanning electron microscopy reveals the smear layer as a 1–2 µm layer of debris with a granular substructure that completely covers the dentin. The orifices of the dentin tubules are blocked by debris tags, called smear plugs, which could well extend into the tubule to a depth of $1-10 \mu m$.^[16]

Smear layer varies in thickness, roughness, density, and degree of attachment to the underlying tooth structure according to the surface preparation.^[17] In restorative procedures, the smear layer must be removed, modified, or enclosed by the resin to allow for better bonding between the tooth and the restorative material.

The smear layer has the potential to create a negative effect on dentin bonding.[18] The smear layer adheres poorly to dentin, and its removal by an acid demineralizing agent before the usage of a bonding system has been reported to have stronger bonds.

Clinically, after carious dentin has been eliminated or any other kind of dentin instrumentation leads to smear layer. The characteristics of the smear layer depend on the type of bur used. Besides, a different pressure applied and the speeds of the bur may influence the kind of smear layer.^[19]

Coarse and superfine diamond burs each create a different form of smear layer, which can interfere with the bonding of the adhesive because it's not easy for some adhesive monomers to penetrate dentin smears and impregnate the underlying dentin. Differences in the smear layer created by burs and abrasive papers have been reported to affect the bond strengths of resins to dentin.

There are principally two options to overcome lesser bond strengths due to smear layer, i.e., elimination of smear layer before bonding, or the usage of bonding agents that can penetrate beyond the smear layer while incorporating it. For the abolition of this elusive smear layer until now, many acids or/and calcium chelators have been tried and tested. Some of them being nitric acid (2.5%), citric acid (10%) , maleic acid (10%) , pyruvic acid (10%) , polyacrylic acid (20%), ferric chloride, aluminum chloride, and oxalic acid $(1.5\% - 3.5\%)$.^[1]

To find a restorative material with similar characteristics of the natural tooth, adhesion to enamel, dentin along with strength and resistance to degradation has always been the major challenge in dentistry. In a push to reach these goals, GIC was developed and first presented by Wilson and Kent in 1972. GIC has been popularly used as restorative materials for most of the conservative procedures along with restoration of cervical lesions.^[20]

The application of surface altering solutions to dentin before bonding with glass ionomers has a long history, and it remains a topic of research as now resin containing glass ionomer products have been introduced into the market.

A remarkable betterment of this class of material occurred, with the pioneer of the RMGIC. The addition of a small amount of resin, such as 2‑HEMA or bisphenol a‑glycidyl methacrylate and photoactivated methacrylate, to the conventional GIC liquid or powder, RMGIC when compared with CGIs have several advantages which include increased working time, decreased setting time, ease of handling, and improved physical properties and esthetics. The actual bonding mechanisms of RMGIC to the tooth tissue have been determined to be two fold by micromechanical interlocking and by chemical interaction. The effective contact between the restorative material and the dental tissue is restrained by the smear layer, impairing satisfactory adhesion. The surface intermediate layer includes not only smear layer (remnants or intact) but also demineralized collagen fibrils, depending on the aggressiveness of the conditioning protocol. Different conditioners have been investigated to improve the clinical performance of RMGIC.

SD: Standard deviation; GIC: Glass ionomer cement; EDTA: Ethylenediaminetetraacetic acid

Table 4: Represents comparative evaluation between each of the 12

Contd...

After Tukey analysis Ketac Primer with Fuji II LC (7.78 MPa) shear bond strength values were significantly better than all the other groups expect for EDTA with Fuji II LC (7.002 MPa). EDTA with Fuji II LC (7.002 MPa) was significantly better than liquid of glass ionomer with Ketac Nano (3.24 MPa) and Vitremer (3.858 MPa), Ketac primer with Ketac Nano (4.302 MPa) and Vitremer (4.14 MPa), EDTA with Vitremer (3.952 MPa) and all the three control groups control Fuji II LC (3.333 MPa) control Ketac Nano (1.466 MPa) and control Vitremer (2.718 MPa). Liquid of glass ionomer with Fuji II LC (5.127 MPa) was significantly better than control group of Ketac Nano (1.466 MPa). Control group of Ketac Nano (1.466 MPa) was having significantly lower shear bond strength values than all of the groups expect of liquid of glass ionomer with Vitremer (3.858 MPa), and Ketac Nano (3.24 MPa), control groups of Vitremer (2.718 MPa) and Fuji II LC (3.333 MPa). GIC: Glass ionomer cement; EDTA: Ethylene diaminetetraacetic acid; CI: Confidence interval, ?: *P*<0.05 level of significance. q is range distribution

This study design had 12 groups, the liquid of glass ionomer was one of the three conditioning agents used were the presence of polyacrylic acid in major concentration is known to have a significant effect on bonding by affecting the surface roughness and partial removal of smear layer without totally unplugging the tubules.^[7]

The second conditioning agent used in this study was Ketac primer with Ph. 3, which may be helpful in removing smear layer in a partial way is also known to improve the wettability of dentin increasing monomer penetration into hydrophilic dentin substrate.[3]

Calcium chelators are used to remove/modify the smear layer without demineralizing the surface dentin layer. Most commonly used chelator is EDTA.[21]

EDTA 17% the third agent used. EDTA decalcifies the underlying dentin, improving the diffusing ability of RMGIC through the decalcified dentin surface. EDTA is known not to aggressively decalcify the dentin surface or widely open the dentinal tubules, resulting in the development of long and thin resin tags, thinner hybrid layer, and much fewer filler distributions. It is an agent which in an aqueous form chelates divalent cations such as $Ca++$, $Mg++$, $Fe++$, and $Pb++$ at neutral pH. Negligible and nonuniform effect on enamel whereas on dentinal surfaces, EDTA caused widening of dentinal tubule orifices and the demineralization extended into the depth of tubules.^[22] It has been used to dissolve the mineral phase of dentin without altering dentin proteins.[23]

After surface treatments with three conditioning agents, the specimens were tested for shear bond strength. A significant interaction between the RMGICS and the conditioning agents was hence indicated by a one‑way ANOVA and Tukey's test which followed. All the fractured specimens exhibited predominant adhesive failures which suggest that the bonding configuration of the materials surpassed the inherent strength of RMGI and dentin.

Of The three conditioning agents used for Fuji II LC, Ketac primer with Fuji II LC (7.78MPa) showed the highest shear bond strength followed by EDTA (7.00MPa) and liquid of glass ionomer (5.127MPa) [Tables 5 and 6]. All the three conditioning agents used with Fuji II LC showed higher values than the negative control (3.33MPa) similar to studies by Hajizadeh *et al*. [6]

The next material in the study Ketac Nano N100 had the better result with EDTA (4.79MPa) as its conditioning agent followed by Ketac primer (4.30MPa) and liquid of glass ionomer (3.24MPa) [Graph 1]. All the three conditioning agents used with Ketac Nano N100 showed higher values than the negative control (1.46), similar to results obtained by Imbery *et al*. [3]

The third material used in this study, Vitremer the conditioning agent Ketac primer (4.14MPa) showed a similar value along with EDTA (3.95MPa) and liquid of glass ionomer (3.85MPa), as in the previous groups conditioning agent proved to be effective compared to the unconditioned dentin which was the negative control (2.71MPa)

EDTA: Ethylenediaminetetra‑acetic acid

[Graph 2]. In comparison with the materials, Fuji II LC had consistently more mean values followed by Ketac and Vitremer with the lowest among the three materials. Comparing the three resin‑modified glass ionomers in the negative control group Fuji II LC was significantly stronger to VITREMER and Ketac Nano, similar to results obtained by Fagundes *et al*. [24] Ketac Nano being the lowest in shear bond value among all the groups. Among the conditioning agents, Ketac primer with Fuji II LC had the highest mean value of all. EDTA is very effective for all the three materials, liquid of glass ionmer also improved the strength of the cements. All the three conditioning agents had a significant effect on the strength of the materials as compared to intact smear layer. This result in suggestive of effective modification removal of the smear layer, exposure of collagen network and opening of dentinal layer, exposure of collagen network and opening of dentinal tubule which promotes a better resin monomer penetration within the underlying dentin. This increased surface energy would contribute to providing a better moisture of dentin surfaces, thus creating an interdiffusion zone between the cement and the dentin matrix contributing to micromechanical retention, in addition to the RMGIC'S chemical adhesion to the dentin. The lowest shear bond strength means were

found in the control group of Ketac Nano and vitremer which did not use any conditioning agents. The use of both mild and aggressive conditions in this study was aimed to evaluate whether the preconditioning step can improve the bond strength. The null hypothesis was rejected based on our results because the preconditioning of dentin was found to improve the bond strength of RMGIC significantly. The difference in methodology and technique can affect the results. Initially, prefabricated copper rings were planned to be used to build the resin‑modified glass ionomer 5 mm \times 2 mm, but then it was decided to use plastic rings of the same dimensions instead because they allowed better flow of the viscous RMGIC. Studies have also shown that micro tensile and micro‑shear bond strength studies have shown much better values than what we have received here, 5 mm wide restorations account for a large size leading to larger flaws and voids with higher stress concentration leading to lower values. The inherent weakness of an *in vitro* study is that the results cannot be extrapolated to what the expected bond strengths will be *in vivo*. And however, though there is no clear correlation between the materials *in vitro* and *in vivo*. However, it can be assumed that if a restorative material exhibits lower bond strength under ideal laboratory test conditions. It is very likely that

Graph 1: (a and b) Represent the mean and standard deviation. Conditioning agent ethylene diamine tetra-acetic acid with Fuji II LC and agent Ketac Primer with Fuji II LC with better shear bond strength values

it may not be retained successfully in the oral environment, and thus, the additional need for retention should be thought of when applying clinically.

Conclusion

Within the limitations of the present study, it can be concluded that:

- Surface conditioning of dentin resulted significantly higher bond strength than unconditioned dentin surfaces
- Surface conditioning of dentin with Ketac primer followed by restoration with Fuji II LC resulted in significantly higher bond strength than other groups
- • Surface conditioning of dentin using 17% EDTA showed good shear bond strength with all the three materials used. Initial conditioning with 17% EDTA followed by usage of primer for a particular material as manufacturers instruction might possibly yield even better bond strength
- • Fuji II LC was concluded as the material with better shear bond strength followed by Ketac Nano and vitremer.

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Conflicts of interest

There are no conflicts of interest.

Graph 2: Represents mean in an ascending order (lowest to highest value)

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