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Dentistry Section

# Comparative Evaluation of Shear Bond Strength and Fluoride Release of Conventional Glass Ionomer with 1% Ethanolic Extract of Propolis Incorporated Glass Ionomer Cement –Invitro Study

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## ABSTRACT

**Introduction:** Atraumatic restorative treatment is a minimal intervention approach which involves manual removal of caries followed by restoration using adhesive restorative material. Due to incomplete manual caries excavation, there is a high chance of secondary caries under the restoration. Hence, many antibacterial agents have been incorporated in cement to enhance their antibacterial effect. Propolis is one of the natural medicines that has highlighted application in dentistry.

**Aim:** The current study evaluated the shear bond strength and fluoride release of Glass Ionomer Cement (GIC) combined with 1% Ethanolic Extract of Propolis (EEP). The research hypothesis was that the incorporation of 1% EEP in GIC has an effect on shear bond strength and fluoride release.

**Materials and Methods:** A study was conducted among two groups. Group A conventional GIC (control), Group B GIC incorporated with 1% EEP (experimental). Shear bond strength: Thirty samples were prepared. Dentinal surface was restored and bond strength was assessed using a universal testing machine. Fluoride release: Thirty samples were prepared and stored in distilled water at a constant temperature until the time of measurement. The fluoride release was assessed by ion selective electrode after 1st day and 7th day. Data obtained by shear bond strength analysis was subjected to statistical analysis using an unpaired t-test and the data obtained by the fluoride release analysis was subjected to an unpaired t-test and paired t-test.

**Results:** Result showed that there was no statistically significant difference in shear bond strength between the groups (p-value 0.77). A statistically significant difference was noticed in fluoride release among the groups after 1st and 7th day (p-0.001). However, the release was lesser in both the groups after the 1st day.

**Conclusion:** A 1% EEP incorporated GIC enhanced the fluoride release without causing a significant effect on shear bond strength of GIC.

**Keywords:** Ion selective electrode, Mechanical properties, Universal testing machine

## INTRODUCTION

There has been a paradigm shift from the concept of "extension for prevention" to minimal intervention approaches. ART is one such minimal intervention approach with technique using manual instruments for caries excavation and restoring mainly with Glass Ionomer Cement (GIC) [1]. The drawback of the procedure is, some of the residual bacteria do remain under restoration which over time leads to secondary caries and restoration failure [2]. These residual bacteria can be diminished by adding the antibacterial agents to the GIC. Literature studies have proved that the addition of antimicrobial agents to GIC have diminished the counts of residual bacteria under the restoration. Antimicrobials such as metronidazole, ciprofloxacin, cefaclor, triclosan, cetrimide, quaternary ammonium salt have been experimented. These antibiotic modified GIC had a significantly greater antibacterial effect, but gave rise to antibiotic resistance [3]. These modified GIC's failed to maintain the ISO standard for physical and mechanical properties [4]. Hence there is a need of alternative material which doesn't cause changes in physical properties.

In the past few decades, there has been an increased use of natural products for medicinal purposes. These natural products are efficient, less toxic, they serve as an alternative source of medicine [5,6]. One such natural mode of treatment is Apitherapy. Apitherapy is defined as, the art and science of treatment and holistic healing through the honey bee and its products for the benefit of mankind and all the animal kingdom [7].

Propolis is a resin collected by the honey bee known as *Apis mellifera* it is used for protection of the hive against microorganisms. It has been extensively used in medicine for centuries and presents a complex chemical composition. It has antibacterial, antifungal and anti-inflammatory properties. Propolis has been shown to exert antibacterial action against various oral microorganisms [5,6]. Effect of propolis on the physical properties of GIC as a natural antibacterial agent has not been clearly understood. Hence this study aimed to evaluate and compare the effect of bond strength and fluoride release of conventional GIC with EEP incorporated GIC.

## MATERIALS AND METHODS

The present study was carried out at the Bapuji Dental College and Hospital in Department of Pedodontics and Preventive Dentistry between academic year 2011 to 2014. The material used in the study was GC Fuji type IX gold label (GC corporation, Tokyo Japan) GIC. It was selected as its most commonly used posterior restorative material in paediatric patients because of its advantages of high fluoride release, chemical adhesion and good strength. Propolis (Hi Tech natural products INDIA Ltd., New Dehli) was used in the present study. Propolis was subjected to ethanolic extraction and obtained product was incorporated in GIC liquid.

**Shear bond strength:** Fifteen caries free, premolars samples, extracted for orthodontic reasons were cleaned of debris and sectioned mesio-distally using a diamond disc. Hence 30

specimens were obtained which were embedded in self-cure acrylic resin such that occlusal surface is parallel to the resin block [Table/Fig-1]. Later the samples were ground on trimmer to expose a flat dentinal surface, which was followed by manual polishing of the dentinal surface with wet 600 grit silicon carbide paper. The exposed dentin was conditioned with 20% polyacrylic acid for 10 seconds, washed off with water for 10 seconds and blotted dry using blotting paper. These 30 samples were randomly assigned into two groups [8-10].

Group I conventional GIC (control group).

Group II GIC incorporated with 1% EEP (experimental group).

**Prepration of 1% Ethanolic Extract of Propolis Incorporated GIC:** A 20% propolis in 60% ethanol was used in the present study. Filtration was carried and obtained product was dried which was sticky in consistency. Dilutions of propolis were prepared to get a final concentration of 1% EEP [11]. To achieve a final concentration of 1% EEP, 0.08ml of EEP was added to 7.92 ml of glass ionomer liquid [Table/Fig-2].

Material for control and experimental group was manipulated according to manufacturer's directions and placed on the prepared dentin surface with the help of a teflon mould of standard dimension (3mm x 4mm). The specimens were stored in distilled water at 37°C for 24 hours. Shear bond strength was assessed using a knife-edge blade in a universal testing machine (Instron) at a crosshead speed of 1 mm/minute. The shear bond strength values were calculated as the ratio of fracture load and bonding area. The results obtained in kilograms were converted into mega pascals (MPa) [Table/Fig-3] [8-10].

**Fluoride release:** Fifteen disc shaped cement samples for each group (group 1 conventional GIC and group 2 GIC incorporated with 1% GIC) were prepared, using a standardized brass mould of inner diameter 10 mm and height 2 mm. Mylar strip was secured on a glass plate to form the base of the mould. The material was manipulated according to manufacturer's instructions and condensed into the mould. The mould was then covered with a second mylar strip, on which another glass plate was placed to apply pressure and remove excess material [Table/Fig-4].

The samples were carefully removed from the brass moulds and placed individually in tightly closed plastic containers containing 5ml double de-ionized water and maintained at a constant temperature of 37±0.5°C until the time of measurement. De-ionized water was renewed after 24 hours for seven days.

**[Table/Fig-1]:** GIC Liquid incorporated with 1% EEP.
**[Table/Fig-2]:** Preparation of shear bond strength specimen.
**[Table/Fig-3]:** Measurement of shear bond strength by universal testing machine.


**[Table/Fig-4]:** Cement specimen preparation for fluoride estimation.
**[Table/Fig-5]:** Specific ion electrode for fluoride release estimation.

| Groups | Sample | Shear bond strength | | | Mean | p-value |
|---|---|---|---|---|---|---|
| | | Mean ±SD | Minimum | Maximum | | |
| Control | 15 | 6.73±1.23 | 5.38 | 10.08 | 0.21 | p=0.77 |
| Experimental | 15 | 6.94±2.50 | 3.38 | 11.42 | | |

**[Table/Fig-6]:** Descriptive statistics of shear bond strength values between control and experimental group.

| Groups | N | 1st day | 7th day | Difference (1st to 7th day) | t | p-value |
|---|---|---|---|---|---|---|
| | | Mean ±SD | Mean ± SD | Mean ± SD | | |
| Control | 15 | 5.42 ± 0.46 | 0.55 ± 0.04 | 4.87 ± 0.47 | 40.00 | <0.001 |
| Experimental | 15 | 10.96 ± 2.01 | 5.24 ±1.03 | 5.71 ± 1.72 | 12.88 | <0.001 |
| Experimental v/s control | t | 10.38 | 17.58 | 1.82 | | |
| | p | <0.001,HS | <0.001,HS | <0.09, NS | | |

**[Table/Fig-7]:** Descriptive statistics showing comparison of fluoride release at 1st and 7th day.

Fluoride release was evaluated by taking 1 ml of double de-ionized water from each container after 1:1 dilution with TISAB (Total Ionic Strength Adjustment Buffer). Ion selective electrode was used to check the release on the 1st and 7th day [12,13] [Table/Fig-5].

## RESULTS

The obtained data for shear bond strength was collected, tabulated accordingly and was subjected to statistical analysis. Mean and standard deviations were calculated for each group and analysed using t-test. [Table/Fig-6] shows Mean±S.D shear bond strength of control and experimental groups. The mean difference of control and experimental groups was 0.21 which is not statistically significant (p-value 0.77). Thus, on comparison from [Table/Fig-6], it can be concluded that addition of 1%EEP had no effect on shear bond strength of conventional GIC.

The obtained data for fluoride release was collected, tabulated accordingly and was subjected to statistical analysis. Mean and standard deviations were calculated for each group and analysed using Paired Sample t-test and unpaired sample t-test [Table/Fig-7]. The experimental group showed higher fluoride release on 1st day and the 7th day ($p < 0.001$) which was statistically significant.

## DISCUSSION

Glass ionomer cement (GIC) has been widely used in restorative dentistry since its invention. It provides a significant anticariogenic property, through fluoride release, but the reduction in the bacterial counts obtained by placing the conventional Glass ionomer cements is not reliable for ART restorations. Therefore, antibacterial modified GIC would provides an alternative approach to overcome this problem [14].

Propolis is a natural raw material with complex structure. It has the potential of use in the treatment of bacterial disease. It is effective against dental caries as it causes inhibition of glucosyltransferase activity in cariogenic bacteria [15]. Hence, it can be a preferred alternative to overcome the drawback of the previous materials.

Propolis has a complex structure, hence cannot be used directly. Solvents used for extraction of propolis are water, methanol, ethanol, chloroform, dichloromethane, ether and acetone. These solvents help in the removal of inert material while preserving the desired compounds [16]. There are different forms of propolis such as ethanolic or lyophilized [3]. Troca et al., in a study mentioned that the incorporation of lyophilized propolis leads to difficulty in the manipulation of GIC. EEP is the most often used material for experiment, hence it was used in the present study. Authors have also stated that incorporation of 2% of EEP increases the water sorption of the material which affects the physical property of the

cement [3]. Hence, 1% was used in the present study. A study done by Steinberg Kaine and Gedalia concluded that 0.4% is the lowest concentration of propolis to have desirable antibacterial activity, hence 1% EEP which has acceptable antibacterial effect was preferred [11].

Adhesion of restorative materials to enamel is the most important aspect of restorative dentistry. Dentinal adhesion is proved to be much difficult due to the complex histological structure and variable composition of the dentine [17]. Ideal restoration should thus be strong to counteract the forces of mastication acting on the tooth and restoration. Various mechanical tests have been proposed to assess the bonding performance of restorative materials. Measurement of shear bond strength is a relatively simple, reproducible and widely accepted test [18].

In the present study, control group samples showed shear bond strength values ranging from 5.38 Mpa to 10.08 Mpa with a mean of 6.73±1.23Mpa. In the experimental group samples, shear bond strength ranged from 3.88Mpa to 11.42Mpa with a mean of 6.94±2.50Mpa. The reason may be because of increased ionic exchange and interaction in the experimental group within first 24 hours leading to increase in bond strength.

Fluoride is efficient in inhibiting the demineralization process and promotes the remineralization process [19,20]. Ion selective electrode is an accurate method to estimate the amount of fluoride release, gives a direct estimate of the free fluoride ions present. In the present study, for fluoride release estimation distilled water was used as a medium as suggested by various investigators. Since, there are no existing ions in the medium it gives an exact estimate of the fluoride ions released [21]. In the present study, both groups showed initially a high fluoride release, that declined sharply and gradually diminished to a nearly constant level.

In the present study the control group showed a mean fluoride release of 5.42±0.46 ppm after 1$^{st}$ day that decreased to 0.55 ± 0.04 ppm on the 7$^{th}$ day. The experimental group showed a mean fluoride release of 10.96±2.01 ppm after 24 hours, which decreased to 5.24±1.03 ppm at seven days.

The pattern of fluoride release in this study was in similar to the results of another study by Tay and Braden [22]. They explained the mechanism of fluoride reason and the reason for decline of release over time. In the first process, there is short term burst of fluoride release from the surface after which the elution was markedly reduced. Later it is accompanied by second process where there is bulk diffusion process in which small amounts of fluoride ions continue to be released into the surrounding medium for periods of 2 years to 5 years [22].

A positive finding in the study was the increase in fluoride release of propolis containing glass ionomers when compared to conventional glass ionomer alone. The reason could be attributed to the presence of propolis in the matrix of glass ionomer system which may have created a pathway for the release of fluoride ions. It could also be because as propolis, is obtained from plants and some of these plants do contain fluorides. When this is mixed with GIC the fluoride from GIC and Propolis could have released into the surrounding area, hence there was an increase in fluoride release when compared to the conventional GIC.

In the present study only 1% of EEP was incorporated into GIC. Effect of different concentration of EEP on physical properties of the cement can be considered. Other mechanical properties of EEP incorporated to GIC can be studied. The study calculated fluoride release for 24 hours and seven days. Long time fluoride release should be studied. The main reason for failure of ART is secondary caries. Incorporation of EEP into GIC has improved the antibacterial effects with additional benefits of increased fluoride release without effecting shear bond strength.

## LIMITATION

The present study considered effect of 1% ethanolic extract of propolis on physical properties, different concentration of propolis on GIC can be considered. Flouride release and shear bond strength were only measured. The influence of propolis on other mechanical property have to be studied. Only the etanolic extract of propolis was used in the study other forms of propolis should be incorporated to GIC and studied.

## CONCLUSION

Incorporation of 1% EEP to conventional GIC increased the fluoride release and did not affect the shear bond strength of the cement. Propolis incorporated GIC may be seen as an alternative restorative material for use in atraumatic restorative procedures.

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