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# Silk Fibre Reinforced GIC – A Promising Alternative to Conventional GIC

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## Abstract

**Introduction:** Glass Ionomer cements are excellent anti cariogenic restorative materials in the non-stress bearing areas of the teeth. Poor mechanical properties like compressive strength and wear resistance limits its use clinically. In order to overcome these limitations various chemical resin fillers and natural fibres like silk were added to GIC. Natural silk fibres exhibit excellent mechanical properties apart from being highly biocompatible. Hence this in-vitro study was undertaken to analyze the shear bond strength of silk fibre reinforced GIC and compared to conventional GIC.

**Materials and Method:** A total of 60 permanent decoronated premolar teeth were used in this study. The teeth were randomly distributed into 20 samples each of 3 groups. Silk particles extracted from Bombyx mori silkworm cocoons was incorporated into conventional type 2 GIC (GC, CORP, JAPAN) at 3% by weight. Thus, obtained silk reinforced GIC was tested for shear bond strength on dentin surface of the decoronated premolar teeth and the results were compared with shear bond strengths of conventional type 2 GIC (GC CORP, JAPAN) and universal micro-hybrid Composite (CHARISMA, KULZER). The shear bond strength of all the groups was analyzed using a Universal testing machine (ZWICK ROELL Z101, GERMANY) at a cross head speed of 0.5mm per minute and the results were tabulated.

**Results:** Compared to conventional GIC, Silk reinforced GIC showed increased shear bond strength. But, both the GIC materials were inferior to Composite.

**Conclusion:** Within the limitations of this study, it can be concluded that incorporation of silk particles 3% by weight exhibited better shear bond strength compared to conventional GIC. But the values were less comparable to composite. Further studies are required to assess the impact of silk reinforced GICs in clinical situations.

**Keywords:** Bombyx mori silk worms, Silk cocoons, Silk, Glass Ionomer cement, Universal composite, Shear bond.

## Introduction

In the late 1960's the demand for anti-cariogenic restorative materials arose due to the increasing cases of pit and fissure caries on the occlusal surface of the posterior teeth (4,9). This paved the way for Wilson & Kent in 1972 to introduce Glass ionomer cement as a preventive pit & fissure sealant material. The Glass Ionomer cement had both the properties of fluoride release which was of silicate cements and chemical adhesive property which was a quality of poly carboxylate cements respectively. It is considered as an excellent restorative material based on its bio-compatibility, anti- cariogenicity, chemical bonding, modulus of elasticity, etc(4). GIC mimics dentin properties and is often tagged as Man-made dentin.

Glass ionomer cement was primarily intended in class III and class V cavities of anterior teeth due to its better aesthetic appearance than its predecessors. Some of the properties like abrasion resistance and compressive strength make it an unfavorable restorative material especially in stress bearing areas (4). Several types of GICs were formulated for using as a luting agent for crown & bridges and as a restorative material in non-stress bearing areas. Type 2 GICs (conventional restorative GICs) are widely being used as material of choice in carious deciduous teeth due to its active fluoride release (9). Despite some excellent properties, the conventional Glass Ionomer cement failed clinically to withstand mechanical load, making it an unfavorable restorative material in class I and II cavities and stress bearing areas (4,9).

Researchers have been trying to overcome some of these undesirable properties of GIC through addition of various filler particles. (4) Some of these modifications have even been tried as amalgam substitutes and in ART techniques. Recent modifications include the addition of resin which offered command set and increased mechanical properties (2,4).

Materials like Silk fibers, flax seed fibers, silver nano particles, etc. are incorporated into GIC as fillers and are being tested for their mechanical properties (2,4). Natural materials like silks from silkworm and spider has shown extraordinary mechanical properties than many man made synthetic materials (10).

In fact, these natural silk materials are also being used to manufacture bullet proof vests, automobile accessories, etc due to its exceptional mechanical properties. Besides, they also exhibited very interesting biomimetic properties like very weak immune response, low bacterial adherence, etc (3,10). These abilities encouraged many researchers to study their potential for application as implantable materials and cell/tissue scaffolds in more detail (1,8).

With an understanding that Mulberry silk had been a boon in bio-medical applications, this study aimsto evaluate the shear bond strength of silk-reinforced GIC in extracted teeth and compare the results with conventional type 2 GIC and universal composite respectively.

**Aim**

To evaluate and compare the shear bond strength of silk fibre reinforced glass Ionomer cements with type 2 Glass Ionomer cement and composite.

**Objectives**

To evaluate the bond strength of silk fibre reinforced glass Ionomer cement on permanent teeth under shear forces.

To measure and compare the shear bond strength of silk reinforced glass ionomer cement, type 2 glass ionomer cement and composite.

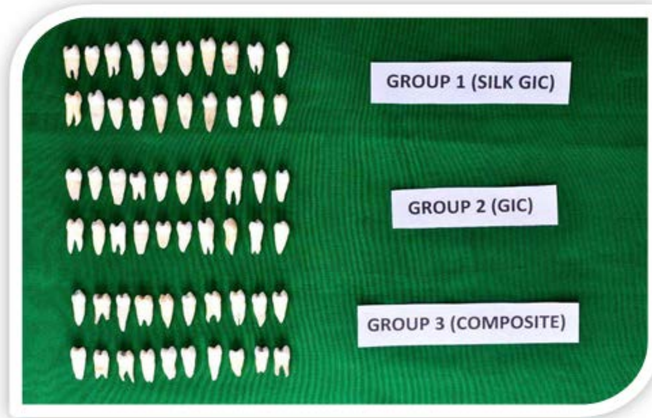
**Methodology**

The materials and methods used for this study are described below:

- Selection of specimen
- Materials & Instrumentation
- Preparation of Silk GIC
- De-coronation of specimen
- Material build up using template
- Shear bond strength test

**Selection of Specimen**

60 freshly extracted permanent premolar teeth were collected from available sources including the Department of Oral & Maxillofacial Surgery, Mahe Institute of Dental Sciences, Mahe (U.T of Puducherry). All the teeth were washed using detergents to remove blood and debris. Ultrasonic scalingwas done to remove any extrinsic stains and calculus. All the samples were disinfected with 10 % formalin solution for 2 weeks.




**Inclusion Criteria**

Teeth extracted due to periodontal, orthodontic reasons and extracted impacted teeth.

**Exclusion Criteria**

Severely discolored teeth, Teeth with cracks, extensive caries, huge restorations, teeth with fluorosis and Root canal treated teeth**.** The specimens were randomly divided into 3 groups (n = 20 for each group).

Group 1 (SILK GIC) : 20 SAMPLES (S1- S20)

Group 2 (TYPE 2 GIC): 20 SAMPLES (G1 – G20)

Group 3 (COMPOSITE): 20 SAMPLES (C1- C20)

**Materials & Instruments**

- Bombyx Mori Silkworm cocoons
- Type 2 Glass Ionomer Cement
- Universal composite
- Washing soda
- Composite Instruments
- Self-Cure resin
- Diamond discs
- 400 – 600 grit silicon carbide paper.
- Cylindrical mould
- Plastic straw
- Distilled water
- Universal testing machine
- Weighing machine

**Preparation of Silk GIC**

Best quality Bombyx mori silk- worm cocoons were purchased from cottage industries of Madanapalle silk town (Andhra Pradesh) for preparation of Silk GIC. The cocoons were processed within 48 hrs of purchasing in order to maintain the quality of the silk. The degumming procedure was carried out by boiling the silk cocoons in hot water along with washing soda. Within few minutes, the fibre starts to untangle in the boiling water. Then, a metal fork was used to unroll the silk fibre from the cocoons.

Thus, obtained silk fibre is rinsed multiple times in running tap water. The silk fibre is dried under the shade and it was then shredded into finer particles using a blender. The silk fibre particles were weighed using a digital weighing scale and was then incorporated 3% by weight into commercially available conventional Type 2 Glass ionomer Cement. Then, the Silk GIC was transferred into the same GIC container and labelled accordingly.

Bombyx mori silk worm cocoons

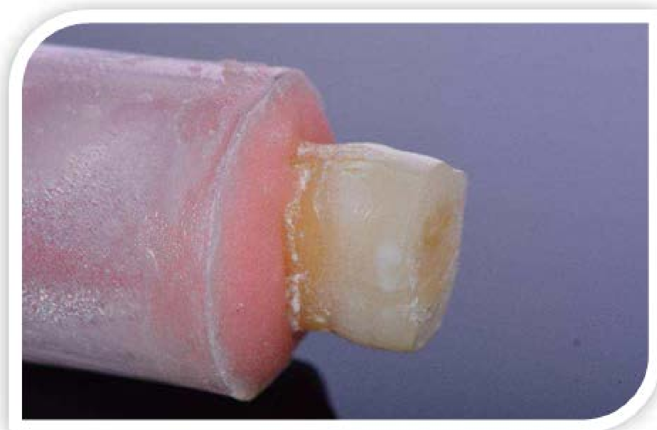
Degumming process with boiling water and washing soda

Shredded Silk Particles

**De-coronation of Specimen**

The occlusal surface of all the specimens were horizontally sectioned using diamond discs to expose fresh and even dentin surface at a depth of around 1 - 1.5 mm from cusp tip. The samples were subsequently polished using 400 – 600 grit silicon carbide paper for 30 seconds. The decoronated specimen teeth were embedded inside a cylindrical mould along with self-cure resin to stabilize theteeth for better handling and testing purposes.

Decoronated Specimen



Specimen Closer View

Material Build-Up Using Template

A plastic straw was used as a template for build-up of testing material on the decoronated dentin surface of the tooth specimen. The straw was 3mm in diameter and 5mm in length and was slit vertically to facilitate easy stripping off the straw after the material sets.

For Group 1 (SILK GIC) & Group 2 (TYPE 2 GIC), Dentin conditioning was done using poly acrylic acid initially. Then, the materials were manipulated respectively according to the manufacturer's instructions and loaded into the plastic straw. The loaded straw was condensed along with the testing material on the decoronated dentin surface. After the material was set, the straw was stripped off. Similarly, all the specimen were made ready.

For Group 3 (Composite), the etching was done with 37% orthophosphoric acid. Bonding agent was applied and light cured. The composite material was loaded into the straw and was then condensed on the dentin surface. It was then light- cured and the straw was stripped off.

All the specimen was stored in distilled water till shear bond strength testing.

Slit Plastic Straw And It's Dimensions

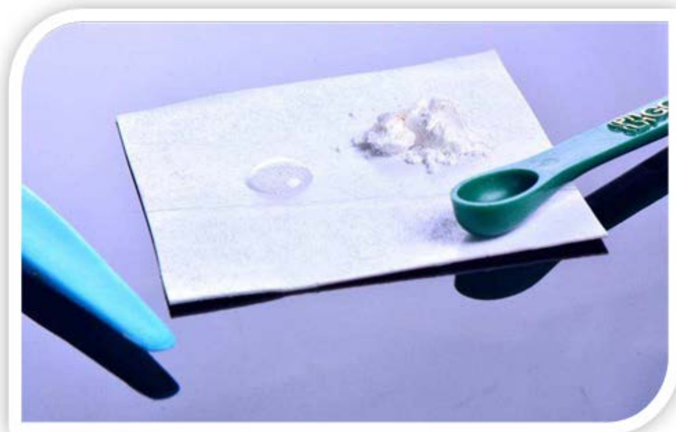

Silk Incorporated Glass Ionomer Cement


Etchant & Bonding Agents


Etching with 37% ortho phosphoric acid

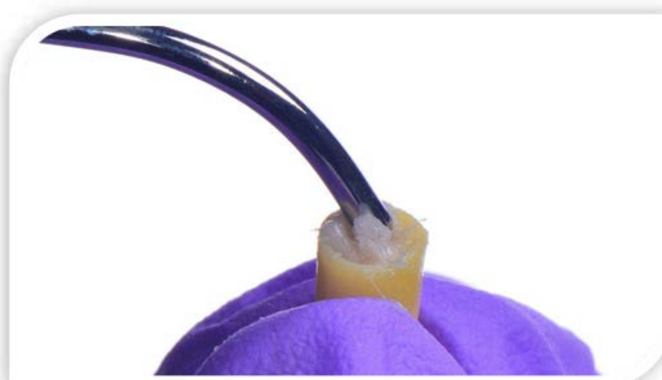

Bonding Agent Application

Dentin conditioning with polyacrylic acid



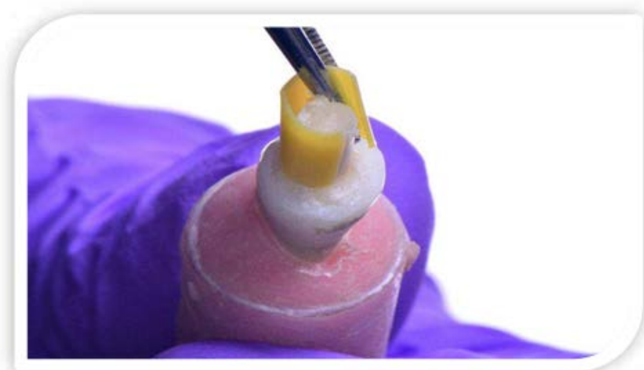
GIC Manipulation as per Manufacturer's Instructions

Loading into the template

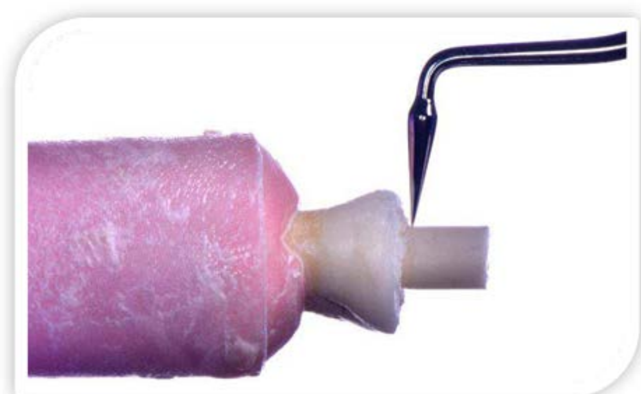


Material Condensed

Condensed on dentin surface



Template Stripped Off



Shear load application site

**Shear Bond Strength Test**

The teeth samples were subjected to shear bond strength using the Universal test machine (ZWICK ROELL UNIVERSAL TESTING MACHINE – Z010). A wedge-shaped tip is placed as close as possible at the junction between the dentin surface and the testing material at a crosshead speed of 0.5 mm/min until the testing material completely disengages from the dentin surface. The shear force at which the material disengages was recorded automatically by the computerized sensors in MPa. Similarly, the shear bond strength of all the specimens of all the 3 Groups was recorded respectively.

Zwick roell universal testing machine

Automated Jig Load Application

**Results**

The data were analyzed using one way ANOVA test and inter comparison analysis was doneusing Bonferroni test.

The mean Shear bond strength values obtained for experimental groups 1, 2 & 3 are given inTable 1.

Descriptives

MPa

| | | | | | 95% Confidence Interval for Mean | | | |
|---|---|---|---|---|---|---|---|---|
| | N | Mean | Std. Deviation | Std. Error | Lower Bound | Upper Bound | Minimum | Maximum |
| Composite | 20 | 58.62807560340 | 13.829832489356 | 3.092444556364 | 52.15551476001 | 65.10063644679 | 28.842088700 | 77.972221370 |
| Silk GIC | 20 | 48.13264466300 | 10.921968647679 | 2.442226434433 | 43.02100598942 | 53.24428333658 | 30.090589520 | 65.546357280 |
| GIC | 20 | 42.06119669900 | 11.185387106488 | 2.501128592476 | 36.82627439178 | 47.29611900622 | 25.249362950 | 61.635242800 |
| Total | 60 | 49.60730565513 | 13.708538708588 | 1.769764737307 | 46.06601459548 | 53.14859671479 | 25.249362950 | 77.972221370 |

Table 1: Mean Shear Bond Values Between Group 1, Group 2 and Group 3

**ANOVA**

MPa

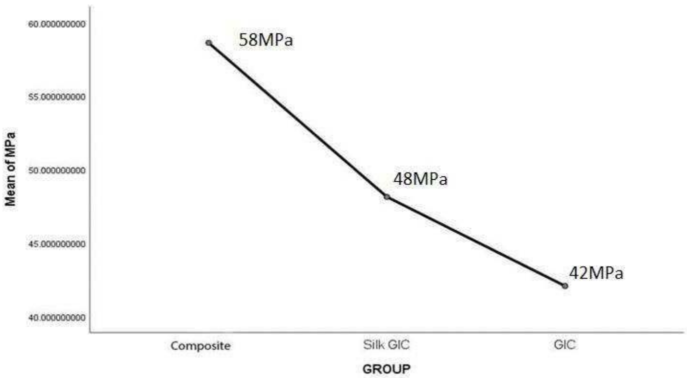
| | Sum of Squares | df | Mean Square | F | Sig. |
|---|---|---|---|---|---|
| Between Groups | 2809.854 | 2 | 1404.927 | 9.674 | .000 |
| Within Groups | 8277.664 | 57 | 145.222 | | |
| Total | 11087.518 | 59 | | | |

**There is a significant difference in the mean shear bond value between the three groups.**

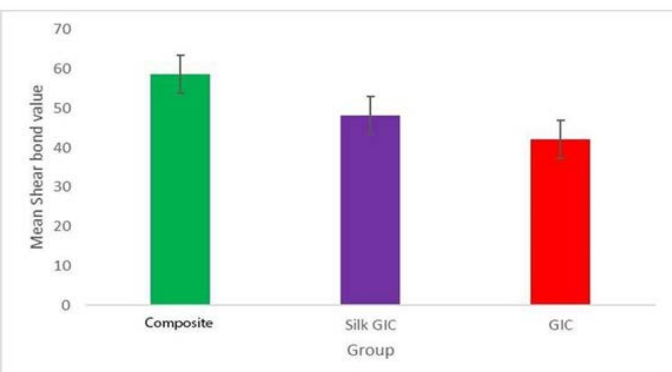
Table 2: Comparision between Groups

According to ANOVA test, it is observed that there is a significant difference in mean shear bond strength between the 3 groups ($p < 0.05$ ).

Composite is having the highest shear bond strength followed by Silk GIC and Conventional type 2 GIC respectively.




Graph 1: Mean shear bond strength graph (MPa)




Graph 2: Mean shear bond strength bar diagram (MPa)

**Multiple Comparisons**

Dependent Variable: MPa

Bonferroni

| (I) GROUP | (J) GROUP | Mean Difference (I-J) | Std. Error | Sig. | 95% Confidence Interval Lower Bound | 95% Confidence Interval Upper Bound |
|---|---|---|---|---|---|---|
| Composite | Silk GIC | 10.495430940400* | 3.810802848686 | .024 | 1.09537310587 | 19.89548877493 |
| | GIC | 16.566878904400* | 3.810802848686 | .000 | 7.16682106987 | 25.96693673893 |
| Silk GIC | Composite | -10.495430940400* | 3.810802848686 | .024 | -19.89548877493 | -1.09537310587 |
| | GIC | 6.071447964000 | 3.810802848686 | .350 | -3.32860987053 | 15.47150579853 |
| GIC | Composite | -16.566878904400* | 3.810802848686 | .000 | -25.96693673893 | -7.16682106987 |
| | Silk GIC | -6.071447964000 | 3.810802848686 | .350 | -15.47150579853 | 3.32860987053 |

*. The mean difference is significant at the 0.05 level.

Table 3: Multiple Comparision

From multiple comparisons using Bonferroni test, it was observed that

There was a significant difference in the mean shear bond value between Composite and Silk GIC

There was a significant difference in the mean shear bond value between Composite and GIC

There was no significant difference in the mean shear bond value between GIC and Silk GIC

**Discussion**

From time immemorial, mankind has suffered from dental caries. The earliest evidences of restorations date back to over 2500 years. Carious teeth were filled with ground mastic, alum and honey over 1000 years ago according to Rhazes (11). Since then, many other materials had been tried as restoratives like bee wax, molten metals, etc. Since the 19th century, the modern restorative materials were developed like direct filling gold, silver amalgam, restorative cements, porcelain and dental composites (11). Unfortunately, none of these restorative materials till date fulfill all the requisites of an ideal restorative material. Glass Ionomer cement was introduced by Wilson & Kent in 1972 which is a combination of silicates and polycarboxylate cements. The Glass Ionomer cement exhibits excellent properties like chemical adhesion to tooth structure. It is a tooth-colored material and was being used as material of choice in class 3 and class 5 cavities in anterior teeth. It has an active fluoride release mechanism which protects the teeth from dental caries. Therefore, it was indicated to restore teeth with high caries index. GIC has high elastic modulus similar to that of dentin and is often called as man-made dentin. Besides, it also has lesser shrinkage and lesser thermal conductivity (9). Hence, it can be used in deep cavities to increase the remaining dentin thickness without damaging the pulp. Considering all these advantages, the Glass Ionomer cement should have been an ideal or near novel restorative material. But, GIC has limited use as a restorative material of choice in stress bearing areas due to its poor mechanical properties including compressive strength and surface hardness. To overcome these disadvantages, researchers have been trying to reinforce the GIC by the addition of different types of filler particles like silver alloy, zirconia, wollastonite, natural fibres, synthetic fibres, etc(4). RMGIC is one of the modified forms of GIC with improved mechanical properties. But it cannot be used in deep cavities as it might induce pulpal damage. China had dominated the Silk Industry for many years and was only available for the privileged classes of the community. Silk was even used a currency and the cost was measured in lengths of silk fibre. Later, the silk was traded to different countries through “The Silk Route” which connected the east to the western countries. Today, India is the second largest producer of silk in the world. India is known as the only country to produce five different types of Silk namely, Mulberry, Eri, Muga, Tropical Tasar & Temperate Tasar. Mulberry silk almost sums upto 79% of the country’s silk production (6,8). In India Sericulture is mostly a cottage industry-based establishment in many parts of Karnataka, Andhra Pradesh, Tamilnadu, etc. The Indian government has been promoting innovations in the field of sericulture for its applications in textile industry, biomedical and pharmaceutical industries through various statutory bodies. One such famous statutory body is “The Central Silk Board” with its headquarters at Bengaluru under the administrative control of the Ministry of Textiles, Govt. of India. In India, this statutory body has been undertaking, assisting and encouraging scientific and technological research of Silk applications with special emphasis on the biomedical applications of these natural fibres. Recently, many natural fibres like silks, cellulose microfibres, jute, etc are being used as filler particles in GICs to improve the mechanical properties (4). Out of so many natural silk fibres, silk worm cocoon silk is one of the most easily available due to its higher production. Bombyx mori silk worms produce best quality silk fibre in India. The silk has a protein called fibroin and sericin (silk fibroin of 75%–83%, sericin of 17%– 25%, waxes of about 1.5%, and others of about 1.0% by weight) (7). According to Abadi et al, adding silk fibres (3% by weight) into the powder part of Glass Ionomer cement, has improved the compressive strength up to around 44%, Flexural Strength up to 157%, and Diametrical tensile strength up to 61% as compared to the commercial product (2). Even though, the mechanical properties of the silk reinforced GIC has improved, there is no evidence that the newer material ‘s bonding to the tooth has improved or not. Silk has many favorable properties, such as biocompatibility, adaptable mechanical properties and stability under a wide range of conditions of humidity and temperature (7,8). They also stated that Silk materials can be tailored to various forms of architecture and morphology, depending on the application, suggesting their potential for dental applications, such as in dentin and periodontal tissue engineering (8). The literature reviewed suggests that silk has wide scope in dental applications including periodontal tissue regeneration, engineering scaffolds for alveolar bone, synthetic bone grafts with bioactive glass, hydrogel delivery of drugs and macromolecule signals to the dental pulp (6,8). Studies on biomedical applications of Silk fibre concluded that silk proteins are promising candidates which can be used for scaffolds in tissue regeneration and cell therapy. In vitro and in vivo experiments have shown that silk materials exhibit a wide spectrum of advantages such as low/absence of toxicity, low degradation, wide pore-size distribution and elastic properties, low/absence of immunoreactivity and the possibility to functionalize silk proteins with cell-adhesion domains (3,6). Despite the advantages of the silk reinforced GIC, the material could become clinically insignificant, if its bond to the tooth structure is compromised. Hence, this study was undertaken to evaluate the shear bond strength of silk modified GIC and compare it with the shear bond strengths of conventional type 2 GIC and universal composite. The results of this study revealed that composite withstood highest amount of shear force to debond from the dentin surface, followed by silk reinforced GiC and conventional type 2 GIC. The shear bond strength of conventional type 2 GIC has improved after reinforcing with silk 3% by weight. We are postulating that a biological bond is responsible for the improved bonding of Silk GIC to the dentin surface. During the testing phase using the universal testing machine, all the restorative specimens of conventional GIC (G1- G20) and Composite (C1- C20) showed adhesive failure at the interface of dentin and the restorative material. Surprisingly, all the specimens of silk GIC (S1- S20) showed combination of adhesive and cohesive failure at the dentin and restoration interface. Our assumption that the silk GIC possesses a biological bond with the dentin surface seems much possible as many researches had concluded that Silk can be successfully used as an implantable material in cell-to-cell adhesion domains. It was also suggested that Silk can be used as a scaffold in periodontal regenerative procedures, alveolar bone, etc. However, much more research is needed to confirm our assumption. Statistical analysis of this study reveals that there was significant difference among the shear bond strengths of Silk GIC and conventional GIC ($p < 0.05$) when compared to composite. Whereas the intergroup comparison reveals that there is no significant difference between the mean shear bond strengths of Silk GIC (48 MPa) and conventional type 2 GIC ( 42 MPa). This might be due to non- homogenous particle size of the silk particles used in our study. Silk was shredded into finer particles randomly in our study. If the silk particles can be processed to much finer and homogenous size, we can expect much more promising results from Silk reinforced GIC. Studies are being undertaken to produce silk nano particles and to isolate the silk proteins to incorporate into biomedical applications to make skin grafts for burn injuries (5). Even though, Silk has been proven to be a promising material in biomedical applications, extensive and long-term research is needed to use silk reinforced GIC as a better restorative material commercially than conventional type 2 GIC. More research on Silk reinforced GIC should be undertaken to evaluate the clinical parameters like long term structural integrity of silk particles, effect of humidity and thermocycling, polishability of the surface, etc, when compared with other filler reinforced GICs.

**Summary**

Glass Ionomer cement is an excellent restorative material except for its poor mechanical properties which makes it an undesirable material of choice in class II cavities and stress bearing areas. Various researches are ongoing to improve its undesirable mechanical properties. One such modification was to incorporate silk particles as fillers to reinforce the conventional type 2 Glass Ionomer cement.

Natural materials like silks from silkworm and spider had shown extraordinary mechanical properties than many man made synthetic materials. This study was undertaken to evaluate the shear bond strength of silk reinforced GIC to dentin substrate. The findings of this study seem to suggest that silk modified GICs have improved bond strength when compared to conventional GIC. Further trials have to be undertaken to evaluate various other clinical properties of silk GIC before it's acceptance as clinically viable material.

**Conclusion**

Within the limitations of this study, it can be concluded that Silk reinforced GIC (3% silk by weight) had exhibited superior shear bond strength to dentin compared to type 2 GIC. But composite resin remains the material of choices as far as posterior restorations are concerned. Silk GIC had shown affinity towards better bonding to the dentin surface compared to the conventional GIC. Further studies are needed to prove the clinical acceptability of silk modified GICs.

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