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Research Article

SHEAR BOND STRENGTH OF THREE NATURAL FIBER REINFORCED CONVENTIONAL GLASS IONOMER CEMENTS": AN INVITRO EVALUATION

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A B S T R A C T

Introduction: Glass Ionomer cement has both the properties of fluoride release which is that of silicate cements and adhesion to dental tissues which is a quality of polycarboxylate cement respectively. It is considered as an excellent restorative material based on its bio-compatibility, anti-cariogenicity, chemical bonding and modulus of elasticity. But GIC lacks colour stability and is not as strong as metallic and composite restorations. Evidence shows that GIC is brittle and easily cracks, due to the low flexural strength. Several modified GIC's have been introduced to overcome some of these disadvantages. The most common being resin modified and filler incorporated GIC's. In this study naturally available fillers like silk, flax seed and cellulose are incorporated into conventional GIC. These fibers are biocompatible, economical and with better handling and easy mixing properties. These experimental samples of fiber reinforced GIC's were subjected to shear bond strength and the results compared and concluded.
Methodology: 80 extracted premolar teeth were collected from available sources. After removal of debris, calculus and soft tissue from the tooth surface, the teeth were stored in 10% formalin solution until further use. The teeth were randomly distributed into 20 samples each of 4 groups. Silk particles extracted from Bombyx mori silkworm cocoons were incorporated into conventional type 2 GIC (GC, CORP, JAPAN) at 3% by weight. Cellulose and flax fibers were also collected from sources and incorporated into conventional type 2 GIC at 5% weight and 25% weight respectively. The tooth specimen were then sectioned to expose dentine and polyacrylic acid conditioning was done .Plastic straws of 3mm diameter were cut into mould of 3 mm in height and placed on the conditioned dentine surface. The reinforced GIC materials were manipulated and placed into thede moulds with a condenser. After setting the samples were subjected to shear bond strength using a universal testing machine at a cross head speed of 0.5 mm per minute and values recorded.
Results: Group 4(flax fiber modified GIC) showed the highest shear bond strength (3.4242 Mpa) followed by Group 1(unmodified GIC) with 3.3903 Mpa.Group3 (cellulose modified GIC) showed the least bond strength value (2.2679 Mpa).The intergroup comparison revealed that there was significant difference between the groups ($p<0.05$))
Conclusion: Flax fiber reinforced GIC showed better shear bond strength to dentin than conventional GIC. Further research is needed to advocate the use of natural fibers as fillers in GIC.

INTRODUCTION

The modern era belongs to adhesive dentistry. Following Bowen's resin and Buonocore's acid etching the next leap came with the development of Glass Ionomer cements by Wilson and Kent in the 70's. Glass ionomers used the aluminosilicate powder from silicates and polyacrylic acid liquid of polycarboxylates. Because of the extensive use of this cement as dentin replacement material, it is also referred to as 'Manmade Dentin' or 'Dentin Substitute'. GIC forms strong chemical bonds to the dental hard tissues (dentine and enamel) as well as metal and adhere chemically to prepared tooth structure in moist environments.

However GIC is not a true universal restorative materials due to its many disadvantages. GIC lacks color stability and is not as strong as metallic restorations or composites. Evidence shows that GIC is brittle and easily cracks, due to the low flexural strength and and it lacks wear resistance[1]. Modification, through addition of resins to improve the properties became a major topic of research. Command set or dual cure ionomers were the next big leap. But a true ideal GIC still eluded researches due to mainly its low strength Addition of fillers to improve surface hardness and compressive strength resulted in development of materials like Giomers.

Various modifications of the cement powder and the liquid have been made, such as incorporation of hydroxyapatite, zirconia, zinc, stainless steel, silica, and titanium dioxide, among others. However, such modifications resulted in cost over runs and seemingly offered no improvements with regard to properties[2]. Natural fibers like silk fibers, flax fibers and cellulose fibers, are chosen as fillers in this study as they are

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biocompactible, economical, offer easy handling properties and can be easily mixed with GIC.

Silk fibres from silkworm [Bombyx mori (B. mori)] cocoons are well-known for their magnificent mechanical properties. They are used in textile production and have been successfully used as suture materials for centuries. Silks have been used in the design of biomaterials for medical applications because of their exceptional strength, excellent biocompatibility, and low immunogenicity[3].

Cellulose fibers have been studied for several years, with the goal of incorporating them into materials, mainly polymers, as a reinforcing agent. Discontinuous wood pulp fibers (lignocellulosic), were used in this study. These fibers are rigid, non-abrasive, and non-toxic, and they are easily modified by chemical agents[2].

Flax (Linum usitatissimum L.), fibres are strong, nonabrasive, and renewable and offer better handling properties. Genetically modified flax fibres were used as wound dressings to treat infection because of their antibacterial action[4]. Commercially available flax fibers were procured and used as fillers in this study.

This study aimed to incorporate the above mentioned processed natural fibers as filler particles into conventional type II GIC powder by weight percentage. This modified powders were then individually mixed and bonded to tooth samples and subjected to shear bond strength. The values were then tabulated and results drawn.

Aims & Objectives

Aim

To evaluate the shear bond strength of fiber reinforced conventional glass ionomer cement to dentine.

Objectives

To evaluate and compare the shear bond strength of GIC reinforced with three different natural fibers.

METHODOLOGY

Selection of Specimen

80 extracted permanent premolar teeth were collected from available sources .All the teeth were washed using detergents to remove blood and debris. Ultrasonic scaling was done to remove any extrinsic stains and calculus if present. All the extracted teeth were stored in 10 % formalin solution.

Inclusion Criteria

Teeth extracted due to periodontal or orthodontic reasons were included in the study

Exclusion Criteria

Teeth with severe discolouration, cracks, extensive caries, fluorosis and intrinsic stains were excluded from the study

MATERIALS & INSTRUMENTS

- Bombyx Mori Silkworm cocoons (Andraparadesh)
- Natural cellulose fibers (Fiber region, Chennai)
- Natural flax fiber (Fiber region, Chennai)
- Type 2 Glass Ionomer Cement(Fuji universal, GC Corp, Tokyo, Japan) (Fig.3)
- Washing soda
- Self-Cure resin (Fig.4)
- Diamond discs (Fig.3)
- 400-600 grit silicon carbide paper. (Fig.5)
- Cylindrical mould (Fig.3)
- Plastic straw (Fig.5)
- Distilled water
- Universal testing machine(Instron UTM)
- Digital Weighing machine (Fig.13)

Sample preparation for shear bond strength

The samples were divided into 4 groups of: 20 samples each (Fig.1)

Group1: Conventional Type II GIC (+control)
Group2: Silk Fiber reinforced GIC
Group3: Cellulose fiber reinforced GIC
Group4: Flax fiber reinforced GIC

Preparation of Silk GIC

Bombyx mori silk worm cocoons were processed within 48 hrs of purchasing in order to maintain the quality of the silk. (Fig.7) The degumming procedure was carried out by boiling the silk cocoons in hot water along with washing soda. Within few minutes, the fibres started to untangle in the boiling water. (Fig.8) Then the silk fibres were air dried and shredded into finer particles 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 powder was mixed with polyacrylic acid liquid as per manufacturers instructions and used for the study. (Fig.14 and Fig.19)

Preparation of Cellulose GIC

Natural Cellulose fiber made with esters of cellulose obtained from the wood of plants were shredded into finer particles using a blender. The cellulose fiber at 5% mass weight were weighed using a digital weighing scale and was then incorporated into commercially available conventional Type 2 glass ionomer cement. The cellulose fiber reinforced powder was then mixed with cement liquid as per manufacturers instructions and used for the study. (Fig.11 and Fig.18)

Preparation of Flax Fiber GIC

Natural flax fibers were chopped into finer particles using a blender. 25 % mass weight of flax fiber powder was weighed using a digital weighing scale and mixed into commercially available conventional Type 2 glass ionomer powder. This fiber reinforced powder was then mixed with cement liquid as per manufacturers instructions and used for the study. (Fig.12 and Fig.17)

Decoronation of Specimen

The occlusal surface of all the sample teeth 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 the teeth for better handling and testing purposes. (Fig.2)

Material Build-Up Using Template

A 3mm diameter plastic straw was used as a template for build-up of testing material on the decoronated dentin surface of the tooth specimen. The plastic straws were cut into 5mm height templates and were slit vertically to facilitate easy stripping off the straw after the material sets. Dentin conditioning was done using poly acrylic acid initially. Then, fiber reinforced GIC was manipulated according to the manufacturer's instructions and packed or condensed into the plastic template placed on the dentin surface using hand condenser. After the material was set, the straw was stripped off.

A total of 80 samples were prepared and divided into 20 in each of four groups: (Fig.20 and Fig.23)

Group1: Conventional GIC (+control).
Group 2:Silk Fiber reinforced GIC
Group3:Cellulose fiber reinforced GIC
Group4:Flax fiber reinforced GIC

Shear Bond Strength Test

The samples were loaded individually on Instron universal testing machine. A wedge shaped tip was placed as close as possible at the junction between the dentin surface and the testing material. A crosshead speed of 0.5 mm/min was applied until the testing material completely dislodged 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 four groups were recorded respectively. (Fig.26 and Fig.27)

RESULTS

Statistical Analysis

The statistical software IBM SPSS statistics 20.0(IBM corporation, Armonk, NY, USA) was used for the analyses of the data. Descriptive and inferential statistical analyses were carried out in the present study. Results on continuous measurements were presented as Mean (SD) and Standard deviation (SD). Oneway ANNOVA was used to find the significance of study parameters between the groups and posthoc comparison was done using Bonferroni posthoc test. Level of significance was fixed at p<0.05 and any value less than or equal to 0.05 was considered to be statistically significant.

Table 1 Descriptives

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minim um	Max imum
					Lower Bound	Upper Bound		
Group 1(GIC)	20	3.3903	1.35583	.30317	2.8757	4.1448	1.61	6.45
Group 2 (SILK GIC)	20	2.9108	1.14358	.25571	2.3456	3.4160	1.61	5.38
Group 3 (CELLU LLOSE GIC)	20	2.2679	0.72114	.16125	1.8804	2.5554	1.43	3.76
Group 4 (FLAX GIC)	20	3.4242	1.24070	.27743	2.8435	4.0049	2.15	5.92
Total	80	2.9983	1.23361	.13792	2.7338	3.2828	1.43	6.45

Table 1 shows the descriptive shear bond strength values between the groups in descending order namely , Group 4(flax GIC) > Group 1(GIC) >Group 2(silk GIC) > Group 3 (cellulose GIC)

Table 2 Comparison between Groups

ANOVA	Sum of Squares	df	Mean Square	F	P value
Between Groups	21.318	3	7.106	5.461	0.002*
Within Groups	98.903	76	1.301		
Total	120.221	79			

Table 2 shows comparison between the groups with F VALUE (5.461) and P VALUE (0.002).

Table 3 Multiple Comparisons

Dependent Variable:						
(I) VAR0000 1	(J) VAR00001	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	Upper Bound
Group 1 (GIC)	Group 2	.62945	.36074	.085	-.0890	1.3479
	Group 3	1.29235*	.36074	.001	.5739	2.0108
	Group 4	.08605	.36074	.812	-.6324	.8045
Group 2 (SILK GIC)	Group 1	-.62945	.36074	.085	-1.3479	.0890
	Group 3	.66290	.36074	.070	-.0556	1.3814
	Group 4	-.54340	.36074	.136	-1.2619	.1751
Group 3 CELLULOSE GIC)	Group 1	-1.29235*	.36074	.001	-2.0108	-.5739
	Group 2	-.66290	.36074	.070	-1.3814	.0556
	Group 4	-1.20630*	.36074	.001	-1.9248	-.4878
Group 4 (FLAX GIC)	Group 1	-.08605	.36074	.812	-.8045	.6324
	Group 2	.54340	.36074	.136	-.1751	1.2619
	Group 3	1.20630*	.36074	.001	.4878	1.9248

Multiple comparison using Bonferroni post hoc test showed that there was a statistically significant difference (p< 0.05)in mean shear bond value of cellulose GIC with both TYPE II GIC and flax GIC group.

DISCUSSION

Fom the time dentistry evolved as a medical science, materials employed in the human body, especially those in the oral cavity, have been expected to be passive, stable materials that have no interactions with the environment. These qualities are typically found in cements, amalgams, and composite resins. Sadly, none of these materials yet meets all the criteria for the perfect restorative material. GIC was one of the revolutionary material introduced by Wilson and kent in the 70's.These cements combine the properties of silicates and polycarboxylate cements to offer considerable advantages over traditional restorative material.

Glass ionomer cements have special qualities that make them effective as luting and restorative materials. They release fluoride, adhere to slightly moist enamel and dentin without the aid of an adhesive system, and possess longlasting anti-cariogenic properties.

Figure 1 Total Samples -Eighty Teeth

Figure 2 Decoronation of Tooth (Horizontal View]

Figure 3 Plastic Mould and Diamond Dics

Figure 4 Self Cure Resin

Figure 5 Plastic Straw And Silicon Carbide Paper

Figure 6 Conventional Type 2 Gic (Gc Corp,Japan)

Figure 7 Bombyx Mori Silk Worm Coccoons

Figure 8 Degumming Process With Washing Soda

Figure 9 Silk Fiber Air Dried

Figure 10 Silk Fiber Closer View

Figure 11 Natural Cellulose Fibers

Figure 12 Natural Flax Fibers

Figure 13 WeighingScale

Figure 14 3% Silk Fiber Measured Using Weighing Scale

Figure 15 25% Flax Fiber Measured Using Weighing Scale

Figure 16 Blended Flax Fiber And Cellulose Fiber

Figure 17 Flax Fiber Reinforced Gic Powder

Figure 18 Cellulose Fiber Reinforced Gic Powder

Figure 19 Silk Fiber Reinforced Gic Powder

Figure 20 Group 1 DecoronatedSamples

Figure 21 Group 2 Decoronated Samples

Figure 22 Group 3 Decoronated Samples

Figure 23 Group 4 Decoronated Samples

Figure 24 GIC Manipulation

Figure 25 Dentin Conditioning

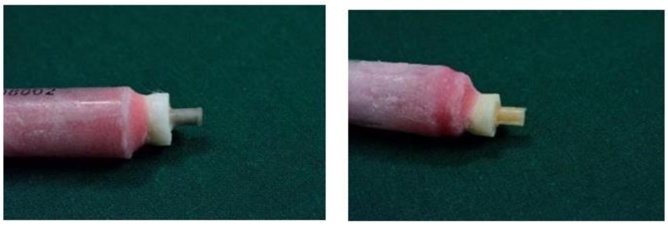

Figure 26 Specimen Ready For Shear Bond Test

Figure 27 Universal Testing Machine

Additionally, they are biocompatible, less sensitive to dentin moisture, and have thermal expansion that is comparable to enamel. It is frequently used as a lining and base, for fissure sealants, and as an atraumatic restorative treatment (ART) material in paediatric dentistry[5].

Since GIC's elastic modulus is comparable to that of dentin, it is frequently referred to as "artificial dentin." Additionally, it has lower thermal conductivity and shrinkage. As a result, it can be used in deep cavities to thicken the remaining dentin without causing pulp damage.

Despite these advantages, the existing conventional formulation's weak mechanical strengths restrict their use in high-stress applications, such as class I and II restoration. GIC, however, has a limited use as a restorative material of choice in stress-bearing areas because of its poor mechanical characteristics, including compressive strength and surface hardness[1].

The primary drawback of GIC materials is their sensitivity to water contamination during the initial phase of the setting reaction, which results in a porous, brittle, and soft cement surface that is prone to cracking[1]. This causes the restoration's surface hardness to decrease There have been numerous attempts to enhance the mechanical properties of glass ionomer cements, including the use of metal powders for reinforcement, resin for modification, SiC whiskers or short fibres for incorporation, Hydroxyapatite and fluoroapatite nanobioceramics, forsterite nanoparticles, and various filler particles such as silver alloy, zirconia, wollastonite, natural fibres, synthetic fibres, etc[2].

Many natural fibres, including silk, cellulose microfibres, jute have recently been used as filler particles in GICs to enhance their mechanical properties. Various attempts have been made to incorporate fibres as agents into the composition of these materials to reinforce their physical structure in order to obtain better mechanical strength and an increase in elasticity modulus.

Silk fibres from Bombyx mori (B. mori) cocoons are well known for their use in textile dentistry due to its magnificient mechanical properties. Nearly 30% of the weight of a silk cocoon is made up of sericin, which holds the fibres together. They have been widely used in wound healing, drug delivery, and other biomedical applications,. Silks are used in the design of biomaterials for medical applications because of their remarkable strength, excellent biocompatibility, and low immunogenicity. Superior strength has been demonstrated in numerous studies involving the addition of silk fibres to composites.

According to S. Alizadeh *et al*. [2016] using 1, 3 and 5 wt.% of silk fibre to GIC respectively, produced the highest compressive strength, flexural strength, and diametral tensile strength. However, all three strength measures demonstrated a significant improvement over commercial GIC at 3 weight percent silk fiber. Studies on the biomedical uses of silk fibre came to the conclusion that silk proteins are excellent candidates for scaffolds in tissue regeneration and cell therapy[3].

According to Kobayashi *et al*. [2000] adding fibers to GIC depend on length and concentration of the fibres which could affect the GIC's strength. The larger the aspect ratio, the better the reinforcement. and crack arresting[6].

Cellulose fibers has unique thermomechanical properties and is biocompatible, it has been used in various biomedical applications. Extremely crystalline cellulose-derived structures called cellulose nanocrystals (CNCs) exhibit exceptional mechanical strength. Any natural source of cellulose is acid hydrolyzed to create CNCs, which range in size from 5 to 15 nm in diameter and 100 to 250 nm in length.

Traditional restorative glass-ionomer cement had its bond strength to dentin strengthened by the addition of cellulose fibres to its formula. Cellulose fibres when added to glass ionomer cement, the material's structure was altered, allowing it to be used in restorations of posterior teeth, particularly those requiring the ART technique.

According to Silva *et al*. SILVA *et al* that addition of cellulose fibers (average length 1 mm) resulted in improved mechanical properties for conventional GICs. Cellulosic fiber-modified GIC demonstrated syneresis and imbibition properties, as well as water solubility and disintegration and diametral tensile strength comparable to conventional cement. However, the cellulose fibres increased the GIC compressive strength and abrasion resistance, as well as its bonding strength to dental structures. [7-10].

R.M. Silva *et al*. (2016) came to the conclusion that the addition of a small amount of cellulose nanocrystals (0.4 wt%) resulted in a significant improvement of all of the evaluated mechanical properties, including: compressive strength increased up to 110%, elastic modulus increased by 161%, and diametral tensile strength increased by 53% compared with the control group, and wear resistance decreased from 10.95 to 3.87%[8].

Flax (Linum usitatissimum L.), which is a member of the Linaceae family, which includes thirteen families and 300 species. Cellulose (70–82%), hemicellulose (15–18%), pectin (2–5%), lignin (3– 5%), waxy substances (2–3%), and dust (1%) make up the majority of the flax fibers. Because of its fibres and oil, flax is a significant product that has been grown for many years. Flax fibres have been regarded as multipurpose fibres and are an example of biologic material (also known as cellulosic material). They are frequently used for non-textile purposes, primarily in composites, as well as industrial uses in sectors like the automotive and construction industries. When broken, flax fibres are easy to extend and have a high tensile strength.

The presence of flexible flax fibers, could absorb some of the stresses or energy required for cracks propagation redistribute these stresses or energy to the nearby GIC matrix, thus suppressing the strain localization. Incorporation of flax fibres was associated with a significant increase in surface roughness and stiffness.

According to Abou Neel EA *et al*. [2017][4] that adding flax fibres significantly increased compressive and yield strengths only at high weight percentages (25 FFRGIC). Low fibre content significantly enhanced the compressive strength produced by the silane treatment of flax fibres[4]. However, the behaviour of the material changed from brittle to ductile when flax fibres were added, increasing it by 1 weight percent. The set materials underwent no morphological changes as a result of the fibre addition.

While parameters such as compressive strength, diameteral tensile strength and flexural strength of fiber reinforced GIC's were studied extensively, very little data was available on the shear bond strength of fiber reinforced GIC's .Hence this study was undertaken to shed new light on how addition of natural fibers can affect the shear bond strength of GIC to dentine.

When compared to group 1, group 2 (silk GIC) had a mean shear bond strength of 2.91Mpa, with no significant mean difference. Mechanical studies from S. Alizadeh *et al* (2016) revealed that increasing the proportion of silk fibre up to 3 wt.% increased compressive strength by 44%. The silk fibre GIC sample had a smoother surface and fewer cavities than the control sample.

Group 4 (flax fibre GIC) has highest shear bond strength when compared with all groups. According to Abou Neel EA *et al*. [2017][4] concluded that flax fibres at high weight percentages (25FFRGIC) showed a significant increase in compressive and yield strength. When flax fibres are introduced in high concentration (25 weight%) an totally coated with the GIC matrix and fill voids and prevent crack propagation.

The results from this study showed that there is a slight improvement in the shear bond strength values of the experiment at GIC when compared to conventional GIC. Among the experimental groups, Flax reinforced GIC shows superior bond strength.

CONCLUSION

Within the limitation of this study it can be concluded that adding natural fibers, doesn't significantly inmprove the shear bond strength Of GIC. Among these flax fiber reinforced GIC's showed better shear bond strength to dentin than conventional GIC. Further research is needed to advocate the use of natural fibers as fillers in GIC.

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