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OPTIMIZING GLASS IONOMER CEMENT FOR PAEDIATRIC DENTISTRY: ENHANCING SHEAR BOND STRENGTH VIA SILK FIBROIN INCORPORATION

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Abstract

Background: The shear bond strength (SBS) is an important parameter to be used to determine the longevity of adhesive restorations. Silk fibroin (SF)-modified glass ionomer cement (GIC) is a promising method in increasing the mechanical characteristics of GIC to be used in paediatric restoration.

Purpose: This study was done to assess the influence of adding SF with 1, 3, and 5 weight percentages on SBS of conventional GIC to dentin.

Methodology: The *in vitro* experiment study was carried out on forty extracted paediatric molars (≤ 16 years). A random selection of specimens ($n=10$) was assigned to each of four groups (Group A, Control conventional GIC; Group B (1% SF-GIC), Group C (3% SF-GIC) and Group D (5% SF-GIC). The GIC powder was thus ball-milled with SF powder. Specimens Preparation: They were prepared by attaching cylindrical cement specimens (4mm diameter) to flat dentin surfaces. A universal testing machine (Shimadzu AG-IS) was used to carry out SBS testing at a crosshead speed of 1 mm/min based on ISO/TS 11405:2015 guidelines.

Results: Group A: 3.30 MPa, Group B: 3.28 MPa, Group C: 4.50 MPa, Group D: 6.30 MPa. One-way ANOVA showed significant differences among groups, which was statistically significant ($p=0.000$). Post-hoc Tukey tests revealed that Groups C and D had a significantly better SBS than the control ($p<0.05$), but Group B did not (p). Conclusion: Addition of silk fibroin at the concentration of 3 per cent and 5 per cent has a significant improvement on the shear bond strength of traditional glass ionomer cement to dentin. This change has the possibility of creating more resistant restorative materials in paediatrics.

Keywords: Biomaterials, Dental adhesion; Glass ionomer cement, Paediatric dentistry, Shear bond strength, Silk fibroin

INTRODUCTION

One of the most important goals in clinical dentistry is the restoration of the lost tooth structure caused by caries, trauma, or other pathologies in an effective way (1). This is especially relevant to the field of paediatric dentistry, in which dental caries is a chronic disease with a high prevalence rate on the global level (2). A perfect paediatric restorative material should be biocompatible, long-lasting, and one that can sustain the special physiological and behavioural needs of young patients. Glass ionomer cements (GICs) are very popular in paediatric dentistry because of their desirable characteristics, which are chemical adhesion on tooth structures, fluoride release, and easy methods of application (3). Nonetheless, they are only applicable in stressful conditions because of intrinsic mechanical limitations, including low fracture toughness and moderate bond strength (4). In order to overcome these restrictions, some changes, such as resin reinforcement, addition of nano-fillers, and introduction of bioactive agents, have been considered (5). One of these promising approaches entails the integration of natural biomaterials e.g. silk fibroin (SF), into the GIC matrix. SF is a fibrous protein that is produced by the cocoons of *Bombyx mori* silkworms, and is known to be exceptionally biocompatible, mechanically strong, and tough (7, 8). High tensile strength, owing to its unique 9-sheet crystalline structure, means that it is an ideal candidate to reinforce dental



materials (9). Early studies indicate that SF may be used to enhance the mechanical characteristics of dental composites, which may increase the lifespan of a restoration (10). The essential factor in clinical success of adhesive restorations is shear bond strength (SBS), which is particularly important in paediatric patients where stresses on restorations can be masticatory and mechanical abuse may occur (11). Better SBS may result in fewer microleakages, less secondary caries, and a greater continuing system of restorations (12).

Since SF has potential and clinical requirements with respect to improved paediatric restorative material, the study attempted to examine the impact of incorporating SF at various levels (1, 3, and 5 percent weight) on the SBS of traditional GIC to dentin. The findings could contribute to the development of advanced, bio-reinforced restorative materials tailored for paediatric dental care.

METHODOLOGY

STUDY DESIGN AND ETHICAL APPROVAL

This in vitro experimental study was conducted after obtaining ethical approval from the Institutional Review Board of Gandhara University, Peshawar, Pakistan. The study was performed at the Dental Materials Laboratory, Sardar Begum Dental College, Gandhara University, Peshawar. The study methodology is illustrated in Fig. 1.

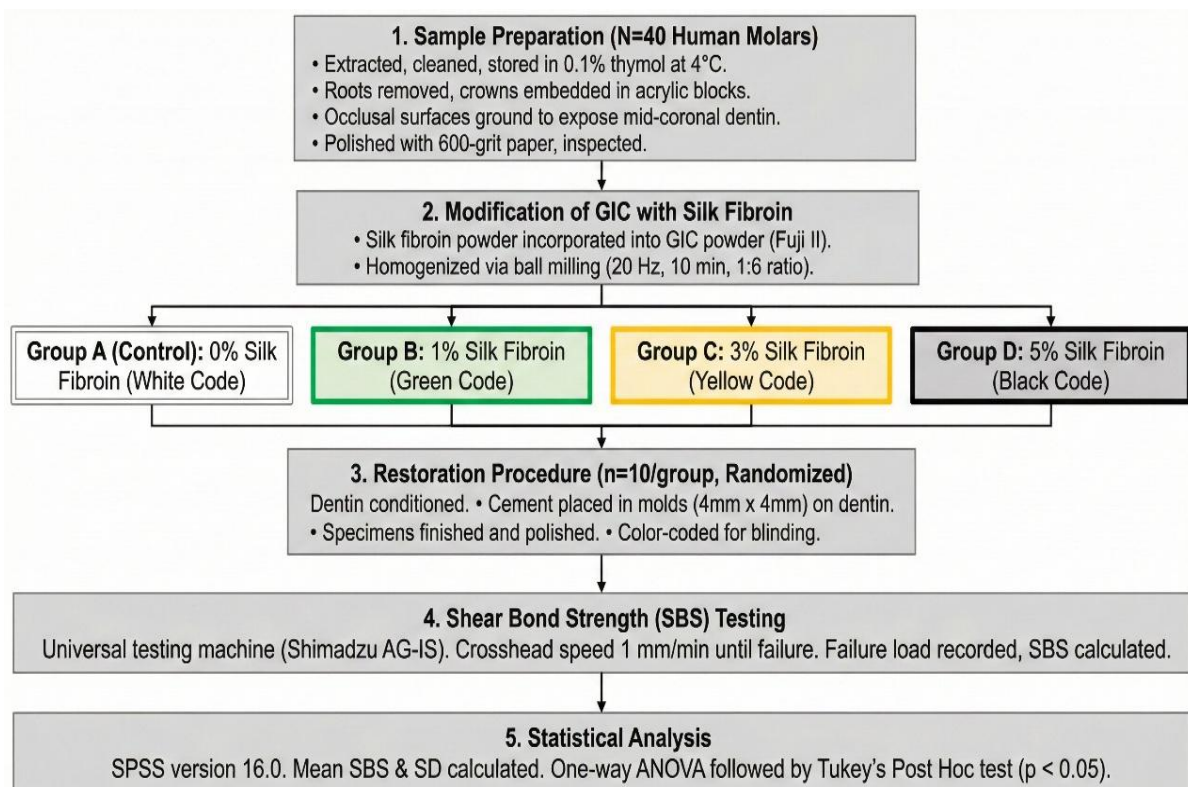


Fig. 1. Flow diagram of study methodology

SAMPLE PREPARATION

Forty non-carious, intact human permanent molars extracted from patients aged ≤ 16 years for orthodontic or periodontal reasons were collected. The teeth were cleaned and stored in 0.1% thymol solution at 4°C until use. The roots were removed, and the crowns were embedded in auto-polymerizing acrylic resin blocks. The occlusal surfaces were ground flat to expose mid-coronal dentin using a slow-speed diamond saw under water cooling. The exposed dentin surfaces were polished with 600-grit silicon carbide paper and examined under a stereomicroscope to ensure absence of enamel or pulp exposure.

MODIFICATION OF GIC WITH SILK FIBROIN

Silk fibroin powder was prepared and incorporated into conventional GIC powder (Fuji II, GC Corp) at concentrations of 1%, 3%, and 5% by weight (Table I). The powders were homogenized using a ball milling machine at a vibration frequency of 20 Hz for 10 minutes, maintaining a ball-to-powder weight ratio of 1:6.

Table I. Composition of experimental groups

Groups	Composition (GIC: Silk fibroin)	% SF by weight
A (Control)	10.0 g: 0.0 g	0%
B	9.9 g: 0.1 g	1%
C	9.7 g: 0.3 g	3%
D	9.5 g: 0.5 g	5%

RESTORATION PROCEDURE

Specimens were randomly assigned to four groups (n=10). The dentin surfaces were conditioned according to the GIC manufacturer's instructions. The cement was mixed and placed into a cylindrical plastic mold (4mm internal diameter, 4mm height) positioned on the prepared dentin surface. After setting, the mold was removed, and the specimens were finished and polished. Groups were color-coded to blind the operator performing the shear bond strength (SBS) test (Table II).

Table II. Group identification and coding

Groups	Color code	% Silk fibroin
A	White	0%
B	Green	1%
C	Yellow	3%
D	Black	5%

SHEAR BOND STRENGTH TESTING

SBS testing was performed using a universal testing machine (Shimadzu AG-IS) following ISO/TS 11405:2015 guidelines. A chisel-shaped shearing blade was applied at the cement-dentin interface at a crosshead speed of 1 mm/min until failure. The failure load (N) was recorded, and SBS (MPa) was calculated using the formula: $SBS = \text{Load (N)} / \text{Bonding Area (mm}^2\text{)}$.

STATISTICAL ANALYSIS

The analysis of data was done via SPSS version 16.0. Each group was provided with mean SBS and standard deviation. The one way ANOVA was used to compare the mean values between the groups and Tukey post Hoc test was used to compare a pair of means. A p-value < 0.05 was considered statistically significant.

RESULTS

The mean SBS values and standard deviations for all groups are presented in Table III and Fig. 2. Group D (5% SF-GIC) demonstrated the highest mean SBS (6.30 ± 0.15 MPa), followed by Group C (3% SF-GIC) at 4.50 ± 0.12 MPa. The control Group A had a mean SBS of 3.30 ± 0.10 MPa, while Group B (1% SF-GIC) showed the lowest value (3.28 ± 0.11 MPa).

Table III. Mean Shear Bond Strength (MPa) of experimental groups

Group	% Silk fibroin	Mean SBS (MPa) \pm SD
A (Control)	0%	3.30 ± 0.10
B	1%	3.28 ± 0.11

Group	% Silk fibroin	Mean SBS (MPa) \pm SD
C	3%	4.50 \pm 0.12
D	5%	6.30 \pm 0.15

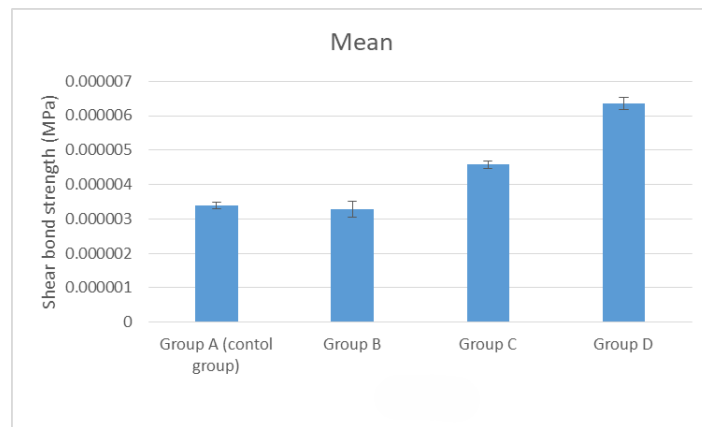


Fig. 2. Mean and standard deviation of shear bond strength value

One-way ANOVA revealed a statistically significant difference in SBS among the groups ($F = 774.977$, $p = 0.000$) (Table IV). Post-hoc analysis confirmed that Groups C and D had significantly higher SBS than Groups A and B ($p < 0.05$). No significant difference was found between Group A and Group B ($p > 0.05$).

Table IV. One-way ANOVA results for shear bond strength

Source of variation	Sum of squares	df	Mean square	F	<i>p-value</i>
Between Groups	45.420	3	15.140	774.977	0.000
Within Groups	0.704	36	0.0196		
Total	46.124	39			

DISCUSSION

This study investigated the reinforcement of conventional GIC with silk fibroin, a natural biomaterial, for potential paediatric dental applications. The results demonstrate a dose-dependent improvement in SBS, with significant enhancements observed at 3% and 5% SF incorporation. The insignificant difference between 0.01 and 0.01 SF (Group B) indicates that a certain concentration is needed to establish an effective reinforcing network in the GIC matrix. The high rise in SBS of Groups C and D could be attributed to the inherent mechanical properties of SF. This high tensile strength and the ability to form a large number of intermolecular hydrogen bonds and β -sheet structures probably form a supporting fibrous network that enhances the cohesion and adhesion of the cement to the dentin substrate (15). These results are consistent with other studies on biopolymer-modified GICs. As an example, the improvement of the mechanical properties of GIC upon the addition of chitosan was also reported (16). Najeeb *et al.*, 2025 (17) and Jowkar and Alizadeh Oskoe, 2019 (18) also showed that the mechanical and adhesive properties of GIC were enhanced with the addition of nano-fillers, such as cellulose nanocrystals and bioactive glass. The biocompatibility and biodegradability of SF, as put forward by Altman *et al.*, 2003 (19), further justify its use as paediatric restorative materials where safety is the utmost consideration. Biodegradability is especially important when it comes to the paediatric population, where the stability of the materials used should be balanced against the natural process of primary tooth exfoliation and the growing child's dynamic physiological environment. Clinically, the enhanced SBS of SF-modified GIC may be translated to longer-

lasting restorations in the primary and young permanent teeth, which have been shown to have fewer failures because of debonding or fracture under masticatory forces (20). This is especially when it comes to the field of paediatric dentistry where the survival of a restoration is difficult over the long term. Moreover, by potentially reducing restoration failure and subsequent caries, such durable materials could lessen the need for antibiotic prescriptions linked to dental infections, aligning with crucial antimicrobial stewardship efforts in paediatric healthcare (21-24). Study Limitations although the preliminary research mentioned in this paper offers important insights, the in vitro nature of the study has a number of limitations. The complex intraoral conditions, such as saliva, biofilm, and variable pH are not possible to be recreated in the controlled laboratory setting. The sample size, which was sufficient in preliminary comparison, is a hindrance to statistical strength, and they need to be justified using bigger samples. Thermocycling and cyclic loading to simulate both thermal and masticatory stresses were not done, which influenced assessment of long-term durability. Critical factors of clinical translation, such as fluoride released, handling characteristics (reported to be difficult with 3 and 5 per cent mixes), and overall mechanical profiles (compressive, flexural, fracture toughness), are not characterized. Also, failure mode analysis was not done. Thus, the clinical effectiveness, biocompatibility and long-term success of SF-modified GIC in children are still unfamiliar and will have to be assessed with the help of properly planned clinical trials.

CONCLUSION

In the constraints of the in vitro research, the addition of silk fibroin at 3 per cent and 5 per cent by weight enhances the shear bond strength of traditional glass ionomer cement to dentin considerably. This development is an interesting prospect of creating stronger and, maybe, longer-lasting restorative materials to be used in paediatric dentistry.

Conflict of interest:

Authors declared no conflict of interest.

Authors' contribution:

HI and TAK Conceptualized the study & conducted the literature review. HH and KR Contributed to data analysis & interpretation; ME Assisted in drafting the manuscript; AK Supervised the study, provided critical revisions & finalized the manuscript.

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