



EFFECT OF ADDITION OF BIOACTIVE GLASS TO BULKFILL COMPOSITE ON ITS IMMEDIATE AND DELAYED SHEAR BOND STRENGTH TO DENTIN.

Dental Science

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KEYWORDS

INTRODUCTION:

The increasing demand for aesthetic, tooth-coloured restorations coupled with the patient's concerns with the use of mercury containing restorations, has driven a surge in the resin based composite materials usage.¹ Since their introduction, composite resins have gained popularity as restorative materials, particularly due to their aesthetic properties and minimally invasive preparation for restoration. Due to high demand, there is continuous development in composite technology in the past decades.² Bulk fill composites have been launched to simplify and accelerate the restoration process by allowing thick composite layers up to 4 to 5 mm to be photo-polymerized in one step.³ Another important change in the advancement of composites involved in reinforcing fillers, which has been purposely reduced in size to produce materials that are more easily and effectively polished and demonstrate greater wear resistance. The latter was especially necessary for materials used in posterior teeth, but the former has been important for restorations in all areas of the mouth.⁴ Bioactive glass has revolutionized dentistry with its remarkable properties, offering a promising alternative in various dental applications. Essentially, bioactive glass is a type of glass that can bond with biological tissues through the formation of a hydroxyapatite layer, mimicking the composition of natural bone. The first bioactive glass composition, known as 45S5, was invented by Larry Hench around fifty years ago. The bioactive glass has a 45S5 composition (45 wt%SiO₂, 24.5wt%CaO, 24.5 wt%Na₂O and 6 wt%P₂O₅). 45S5 glass could bond to living bone and stimulate osteogenesis by releasing biologically-active ions. Since then, various bioactive glass compositions have been proposed for innovative biomedical applications, including bone repair, dental defects, soft tissue repair, and drug delivery.⁴ Resin composites modified by admixing various types of bioactive glass into methacrylate resin have demonstrated multiple benefits including hydroxyapatite precipitation, induction of remineralization, sealing of marginal gap, prevention of dental hard tissue demineralization, protection of hybrid layer to achieve both antibacterial and remineralizing effect.⁵ There are no studies reported, where bioactive glass was added to bulkfill composite. Hence in this study, bioactive glass was added to bulkfill composites and its effect on the bond strength to dentin was studied. The null hypothesis tested states that the addition of bioactive glass nanoparticles to bulkfill composite will not affect its immediate and delayed bond strength to dentin.

Materials And Methodology: Forty freshly extracted non carious permanent human molars were collected from the Department of Oral and Maxillofacial Surgery, Krishnadevaraya College of Dental Sciences and Hospital.

List Of Materials And Armamentarium: (Fig 1)

- 1) Nano sized particles of Bioactive glass(99.5%, <100 nm) – nano research lab, Jharkhand, India
- 2) Bulk fill composite-Universal shade(MANI BULK) - Germany
- 3) Etchant - 37%phosphoric acid (Dentsply Sirona) - Germany
- 4) Bonding agent (prime bond NT)- Dentsply Sirona -USA
- 5) Curing light-LED
- 6) Straight hand piece(NSK)

- 7) Diamond disc
- 8) 600 grit Silicon carbide paper
- 9) Applicator tips
- 10) Mylar strips
- 11) Glass plate
- 12) Polystyrene tubes
- 13) Artificial saliva- wet mouth
- 14) Electronic weighing scale
- 15) Teflon coated composite instrument
- 16) Agate spatula
- 17) Universal testing machine
- 18) Scanning electron microscope

Commercial bulk fill composite material was modified with bioactive glass nanoparticles (<100nm) of 20% by weight. The bioactive glass has 45S5 composition (45wt%SiO₂, 24.5wt% CaO, 24.5wt%Na₂O and 6wt%P₂O₅). 2 grams of bulkfill composite was mixed with 0.4 grams of bioactiveglass nanoparticles to obtain modified bulkfill composite containing 20 wt% of BAG. The samples were mixed using a dual asymmetric centrifuge at 3500 rpm for 60s. The modified composite were protected from light until use to prevent premature polymerisation (Fig 2,3).

Exclusion Criteria : Teeth with decay, severe attrition and erosion, Teeth with fractures and cracks , Root canal treated teeth , Teeth with anatomical variation.

Preparation Of Specimens: All forty teeth were prepared by removing 2.0 mm of their occlusal surfaces with the help of single sided diamond disc to obtain flat dentin surface (Fig 4). Uniform smear layer at dentinal surface was obtained by polishing it with 600grit silicon carbide paper (Fig 5). The specimens were washed in distilled water and embedded in acrylic resin till the level of CEJ. These samples were randomly divided into two groups of 20 samples each: Group I - Bulkfill composite and Group II - Bulkfill composite modified with bioactive glass nanoparticles.

The two groups were further divided into two subgroups based on the time interval to check the bond strength. Unmodified bulk fill composites were used as controls. Bulk fill composites modified with bioactive glass nanoparticles were the experimental groups.

Group I: Unmodified Bulk Fill Composite

- IA: Immediate Bond Strength
IB: Delayed Bond Strength

Group II: Bulk fill composite + Bioactive glass nanoparticles II A: Immediate Bond Strength II B: Delayed Bond Strength

Dentinal surface of all specimens were etched with 37% phosphoric acid for 15 sec and rinsed with water for 10 sec. Excess water was gently blot dried with absorbent paper to remove the excess moisture. Then bonding agent was applied and cured. Polystyrene tube which is 5 mm in diameter and 4 mm in height was placed on the flattened occlusal surface (fig 6) and filled with respective composite resin

material as a single increment upto the brim and light cured for 20 seconds. The polystyrene tubes were removed after curing the composite resin.

Test For Shear Bond Strength

These samples were stored in normal-saline at 37°C for 24 hours, thermocycled for 500 cycles for the immediate group. The samples of the delayed group were stored in artificial saliva for 3 months. Shear bond testing was performed using Universal testing machine at a crosshead speed of 0.5 mm/minute(fig 7). The shear bond strength values were calculated as the ratio of fracture load and bonding area, and were expressed in megapascals.

Failure Analysis

After shear bond strength testing, the fractured test specimens were examined under SEM(fig 8) to observe the modes of failure. ANOVA and t-value calculation analysis were applied. Adhesive failures, cohesive failures within dentin, cohesive failures within the composite and the mixed failures were distinguished.

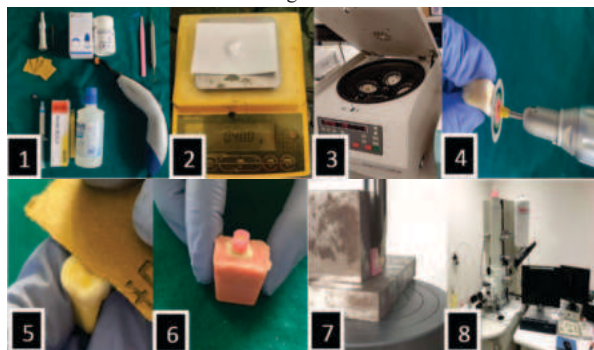


Fig 1: Armamentarium, Fig 2: Weighing scale, Fig 3: Dual asymmetric centrifuge, Fig 4: Creating a flat dentin surface by diamond disc, Fig 5: Polishing the dentin surface, Fig 6: Placing of polyethylene tube of 4mm and composite application, Fig 7: Shear bond strength using universal testing machine, Fig 8: Failure analysis testing using scanning electron microscope.

RESULTS:

The comparison between groups at two-time intervals was performed with a one-way ANOVA test, and there were statistical differences between materials and time intervals.

The mean Shear Bond Strength:

Group IA –Immediate Bond Strength of Unmodified bulk fill composite was 26.77 ± 0.70, Group IB – Unmodified bulk fill composite Delayed Bond Strength was 21.29 ± 0.60

Group IIA – Bulk fill composite modified with bioactive glass Immediate Bond Strength was 30.81 ± 0.80 Group IIB – Bulk fill composite modified with bioactive glass Delayed Bond Strength was 28.96 ± 0.56.

This difference in the mean Shear Bond Strength among four groups were statistically significant at p=0.01.

Table 1: Comparison of Mean Shear Bond Strength(In Mpa) between four groups using One way Anova Test.

Comparison of mean Shear Bond Strength (in MPa) b/w 4 groups using One-way ANOVA Test						
Groups	N	Mean	SD	Min	Max	p-value
Group IA	10	26.77	0.70	25.6	27.9	<0.001*
Group IB	10	21.29	0.60	20.6	22.6	
Group IIA	10	30.81	0.80	29.5	32.0	
Group IIB	10	28.96	0.56	27.9	29.7	

Multiple comparison of mean difference between groups revealed that:

The Group IIA showed significantly highest mean Shear Bond Strength as compared to Group IIB, Group IA & Group IB and the mean differences were statistically significant at p<0.001.

Table 2: Multiple comparison of mean difference in mean shear bond strength between four groups using Tukey's Post hoc Test.

Multiple comparison of mean diff. in mean Shear Bond Strength b/w 4 groups using Tukey's Post hoc Test					
(I) Groups	(J) Groups	Mean Diff.(I-J)	95% CI for the Diff		p-value
			Lower	Upper	
Group IA	Group IB	5.48	4.67	6.29	<0.001*
	Group IIA	-4.04	-4.85	-3.23	<0.001*
	Group IIB	-2.19	-3.00	-1.38	<0.001*
Group IB	Group IIA	-9.52	-10.33	-8.71	<0.001*
	Group IIB	-7.67	-8.48	-6.86	<0.001*
Group IIA	Group IIB	1.85	1.04	2.66	<0.001*

* - Statistically Significant

This infers that the mean Shear Bond Strength was significantly Highest in Group IIA, followed by Group IIB, Group IA and least in Group IB.

The Failure modes in all the four groups were predominantly Mixed type:

60% in group I, 70%each in group II and 80% in group III. This was then followed with Adhesive failure with 40% in group I, 20% each in group III & IV & 10% in group II. The Cohesive failure was least found in all 4 groups, which varied between 0 to 20%. However, there was no significant difference in the failure modes between 4 groups.

Table 3: Comparison of failure modes between different groups using Chi Square Test.

Comparison of Failure modes between different groups using Chi Square Test									
Failure Mode	IA		IB		IIA		IIB		p-value
	n	%	n	%	n	%	n	%	
Adhesive	4	40.0%	1	10.0%	2	20.0%	2	20.0%	0.42
Mixed	6	60.0%	7	70.0%	8	80.0%	7	70.0%	
Cohesive	0	0.0%	2	20.0%	0	0.0%	1	10.0%	

DISCUSSION :

Composite restorative materials represent one of the many successes of modern biomaterials research, since they replace biological tissue in both appearance and function.6 Demands on these restorations with regard to mechanical properties, placement, and need for in situ curing leave significant room for advancements.7 Clinical evaluations and laboratory based studies focused on composite durability and also continue to highlight the need for new materials with improved properties.8,9 Bulk fill composite resins were designed to facilitate restorative procedure, since they enable dentists to use thicker layers of composite filling materials, in increments of 4–5 mm. Various studies have concluded that modification of light cured composite resin with certain amounts of nanoparticles (3% and 7% of ZrO2 and 3% of TiO2 and SiO2) can be beneficial in improving flexural strength and wear resistance while hardness of composite resin was increased with all nanoparticles additions. Nanoparticles of bioactive glasses appear to be promising due to their capability to simultaneously release remineralizing ions (Ca2+, PO43-, F-), neutralize acids, and precipitate calcium phosphates on their surface.10

Mechanical properties such as compressive strength, flexural strength and microhardness and good bond strength are important for the load bearing applications and longevity of composite resin. Incorporation of bioactive glass nanoparticles into the composite resin to achieve antibacterial and remineralizing functions should not sacrifice the mechanical properties of the composite resin. Hence in this study the effect of addition of bioactive glass nanoparticles to bulkfill composite on immediate and delayed bond strength to dentin was evaluated. In a study done by Han X et al. experimental composite resins containing 0 to 23 wt % BAG particles have shown to fulfil the requirement of ISO 4049 for a minimum flexural strength of 80 mpa10. Also bioactive glass fillers have been reported to have a direct inhibitory effect on the polymerization of Bis-GMA resin systems because of surface oxides 34 but data showed that the DC for the prepared resin system was not compromised by the addition of BAG fillers up to 23 wt %. Thus in the present study 20 wt% bioactive glass nanoparticles (<100 nm) was added to bulk fill composite material.

In the present study mid coronal sections of the teeth were selected as the bond strength is known to be dependent on the dentin depth, as it is affected by the diameter of dentinal tubules and dentin water content as well.1

Adhesion between materials can be checked by different tests such as macro-tensile bond strength (TBS), micro shear bond strength (μ SBS), micro-tensile bond strength test (μ TBS), mold-enclosed shear bond strength (ME-SBS/EM-SBS), or lever-induced mold enclosed shear bond strength (LIME-SBS) tests.¹² The shear bond strength method has been used in the literature for evaluating adhesive strength at the interface among dental restorative materials, adhesive, and the substrate.¹³ Because its application is relatively simple and reproducible compared to other methods in which the steps to align the specimen inside the machine are more difficult without affecting the stress distribution,¹⁴ and are easy to perform, requiring minimal equipment and specimen preparation, and provide an overview of the adhesion strength; that presently, they are still extensively used for adhesion evaluation of dental materials. They can simulate realistic loading conditions, provide uniform stress distribution, and are sensitive to adhesive characteristics. They can also yield valuable information for assessing the long-term performance of dental restorations.¹¹ This in vitro study evaluated the immediate and delayed bond strength of bulk fill composites modified with bioactive glass at 24 h and 3 months. The shear bond strength results showed statistically significant differences among all the groups. Therefore, the first null hypothesis was rejected.

In the present study highest shear bond strength was seen in Group IIA (immediate bond strength of modified bulkfill composite) followed by group IIB, IA and IB respectively. The shear bond strength of the modified bulkfill composites are higher than the unmodified bulkfill composites irrespective of the time of testing. This could be due to the increased overall filler content which has reduced the polymerization shrinkage. Bioactive glass nanoparticles also promotes hydroxyapatite formation, biocompatible in nature and its reduces microleakage which creates favourable bond characteristics.¹⁵ Therefore addition of bioactive glass nanoparticles gave good shear bond strength results in group IIB as well.

The results of the current study showed statistically significant differences in the shear bond strength values of composite modified with bioactive glass when compared with unmodified composite. In a study done by Laura Martínez-Sabio et al.¹⁵ there were no statistically significant differences in the shear bond strength values of composite, Bulk-fill, and ACTIVA™ bioactive-Restorative™ groups. However, ACTIVATM (bioactive restorative) has given increased bond strength in the study which is in accordance with the results of our study.

In the present study, immediate bond strength (IA, IIA) have given better results than delayed bond strength (IB, IIB) and significant differences were observed in shear bond strength values irrespective of the group/material. It could be due to the plasticization of composite due to the uptake of water which reduced its brittleness, thus making the adhesive/ composite interface more prone to fracture. The higher shear bond strength values in immediate groups may be attributed due to the presence of oxygen inhibition layer formed after the material has been light cured. The degradation of this layer overtime after light curing of the material may be the reason for the lower shear bond strength values in delayed groups.¹⁶

Bulkfill composite used in the present study contains UDMA which has a distinct capability to polymerize through chain transfer reactions unlike Bis-EMA and BisGMA. By this reaction mechanism, the growth centre at the end of a polymeric chain can be transferred to -NH- groups of the UDMA molecule, rendering that molecule a new initiation site. In that way, chain growth sites can migrate onto new monomer molecules instead of being immobilized on long and crosslinked polymeric chains.¹⁷ In the present study SBS values were within the range of 20 to 30 mpa. Pashley et al.¹⁸ suggested maintaining long-term values above 20 mpa is desirable for long term survival of restoration.

In the present study the failure modes in all four groups were predominantly mixed type followed by adhesive failures and cohesive failures respectively and there was no significant difference in the failure modes among the four groups. However, we found the mixed fracture to be the most frequent with bulkfill composites modified with bioactive glass nanoparticles in both immediate and delayed shear bond strength groups followed by cohesive fracture with the modified bulk-fill composites.

results for achieving both antibacterial and remineralizing activity with low cytotoxicity and good bond strength providing insights into better design and intelligent fabrication of bioactive composite resins. From a clinical perspective, the researchers' primary goal is to enable the use of direct resin composite resins (DRCs) for both anterior and posterior restorations by enhancing the mechanical properties of the resins, such as hardness, fracture resistance, and abrasion resistance, by adding small amounts of inorganic filler particles.¹⁹

In this study single type of direct resin composite was used, simulation of clinical and oral condition was not done which are the limitations. Additional research utilizing various composite materials is advised in addition to clinical studies in various oral conditions to corroborate the in-vitro results. Further studies should be done using various concentrations, type and silanization of BAG nanoparticles in order to balance the mechanical and optical qualities of the dental composite.¹⁹

CONCLUSION:

Based On The Results The Following Conclusions Can Be Drawn :

- 1) The bulkfill composite's SBS to dentin was improved by the addition of BAG.
- 2) Modified bulkfill composite containing 20 wt% BAG nanoparticles have higher SBS to dentin than unmodified bulkfill composite.
- 3) Irrespective of the addition of bioactive glass nanoparticles, immediate SBS of the bulkfill composite was better than delayed SBS.

REFERENCES:

1. Chesterman J, Jowett A, Gallacher A, Nixon PJ. Bulk-fill resin-based composite restorative materials: a review. *British dental journal*. 2017 Mar 10;222(5):337-44.
2. Demarco FF, Collares K, Coelho-de-Souza FH, Correa MB, Cenci MS, Moraes RR, Opdam NJ. Anterior composite restorations: A systematic review on long-term survival and reasons for failure. *Dental materials*. 2015 Oct 1;31(10):1214-24.
3. Dieckmann P, Mohn D, Zehnder M, Attin T, Tauböck TT. Light transmittance and polymerization of bulk-fill composite materials doped with bioactive micro-fillers. *Materials*. 2019 Dec 7;12(24):4087.
4. Bellantone M, Coleman NJ, Hench LL. Bacteriostatic action of a novel four component bioactive glass. *J Biomed Mater Res*. 2000 Sep 5;51(3):484-90.
5. Par M, Spanovic N, Tauböck TT, Attin T, Tarle Z. Degree of conversion of experimental resin composites containing bioactive glass 45S5: The effect of post-cure heating. *Scientific Reports*. 2019 Nov 21;9(1):17245.
6. Sadowsky SJ. An overview of treatment considerations for esthetic restorations: a review of the literature. *The Journal of prosthetic dentistry*. 2006 Dec 1;96(6):433-42.
7. Ferracane JL. Developing a more complete understanding of stresses produced in dental composites during polymerization. *Dental Materials*. 2005 Jan 1;21(1):36-42.
8. Bernardo M, Luis H, Martin MD, Leroux BG, Rue T, Leitão J, DeRouen TA. Survival and reasons for failure of amalgam versus composite posterior restorations placed in a randomized clinical trial. *The Journal of the American Dental Association*. 2007 Jun 1;138(6):775-83.
9. Drummond JL. Degradation, fatigue, and failure of resin dental composite materials. *Journal of dental research*. 2008 Aug;87(8):710-9.
10. Han X, Chen Y, Jiang Q, Liu X, Chen Y. Novel bioactive glass-modified hybrid composite resin: mechanical properties, biocompatibility, and antibacterial and remineralizing activity. *Frontiers in Bioengineering and Biotechnology*. 2021 Jun 1;9:661734.
11. Tarle Z, Hickel R, Ilie N. Dentin Bond Strength of Experimental Composites Containing Bioactive Glass: Changes During Aging for up to 1 Year. *Journal of adhesive dentistry*. 2018 Jul 1;20(4).
12. Jin XZ, Homaei E, Matinlinna JP, Tsoi JK. A new concept and finite-element study on dental bond strength tests. *dental materials*. 2016 Oct 1;32(10):e238-50.
13. Van Meerbeek B, Yoshihara K, Van Landuyt K, Yoshida Y, Peumans M. From Buonocore's pioneering acid-etch technique to self-adhering restoratives. A status perspective of rapidly advancing dental adhesive technology. *Journal of Adhesive Dentistry*. 2020 Jan 1;22(1):7-34.
14. Placido E, Meira JB, Lima RG, Muench A, de Souza RM, Ballester RY. Shear versus micro-shear bond strength test: a finite element stress analysis. *Dental Materials*. 2007 Sep 1;23(9):1086-92.
15. Martínez-Sabio L, Peñate L, Arregui M, Veloso Duran A, Blanco JR, Guinot F. Comparison of shear bond strength and microleakage between activa™ bioactive restorative™ and bulk-fill composites—an In vitro study. *Polymers*. 2023 Jun 27;15(13):2840.
16. Candan M, Altınay Karaca FK, Öznurhan F. Evaluation of the Shear bond strength of immediate and delayed restorations of various calcium silicatebased materials with fiber-reinforced composite resin materials. *Polymers*. 2023 Oct 2;15(19):3971.
17. Pfeifer CS, Wilson ND, Shelton ZR, Stansbury JW. Delayed gelation through chain-transfer reactions: mechanism for stress reduction in methacrylate networks. *Polymer*. 2011 Jul 7;52(15):3295-303.
18. Pashley DH, Sano H, Ciucchi B, Yoshiyama M, Carvalho RM. Adhesion testing of dentin bonding agents: a review. *Dental Materials*. 1995 Mar 1;11(2):117-25.
19. Azmy E, Al-Kholly MR, Fattouh M, Kenawy LM, Helal MA. Impact of nanoparticles additions on the strength of dental composite resin. *International Journal of Biomaterials*. 2022;2022(1):1165431.

The BAG-modified composite resins obtained highly promising