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Effect of two Triphala formulations on resin–dentin bond strength and durability

Gopireddy Navya[1], Lavanya Anumula[1], Chinni Suneelkumar[1], Kiranmayi Govula[1], Sannapureddy Swapna[1], Mounika Gaini[1]

Abstract

Objective: This study aimed to evaluate the effect of two 0.2% Triphala (*Phyllanthus emblica: Terminalia chebula: Terminalia bellirica*) formulations: a standard 1:1:1 ratio and a modified 1:2:1 ratio with increased *Terminalia chebula* content, on immediate and six-month-aged microtensile bond strength (μTBS) of a composite resin.

Methods: Twelve extracted caries-free molars were randomly assigned to three groups (n=4), based on the dentin pre-treatment applied after acid-etching: Group 1: control (no pre-treatment), Group 2: 0.2% Triphala 1:1:1, and Group 3: 0.2% Triphala 1:2:1. After applying the bonding agent, the teeth were restored with composite resin. Each tooth was sectioned to obtain multiple resin-dentin beams (1×1 mm), among which 24 were selected per group. In each group, 12 beams were tested after 24 hours, and 12 after 6 months of water storage at 37°C. μTBS was measured using a universal testing machine. Data were analyzed using ANOVA and independent t-tests (α = 0.05).

Results: The control group exhibited a significant reduction in μTBS after 6 months (11.20±4.35 to 6.75±3.48 MPa; P=0.005). The Triphala-treated groups did not show a significant reduction in μTBS over time, with group 2 decreasing from 10.94±4.47 to 9.54±3.50 MPa (P=0.344) and group 3 from 11.06±5.95 to 10.50±8.35 MPa (P=0.866). Intergroup differences in bond strength were not significant at any time point (P>0.05).

Conclusions: Dentin pre-treatment with 0.2% Triphala preserved μTBS over six months, suggesting potential MMP-inhibitory activity. Increasing the proportion of *Terminalia chebula* in the Triphala formulation from 1:1:1 to 1:2:1 did not provide additional benefits concerning bond durability.

Keywords: Composite resins, Dentin bonding, Herbal medicine, Matrix metalloproteinases, Plant extracts, Triphala

Introduction

Composite resin materials have undergone substantial improvements in mechanical properties, wear resistance, and polymerization behaviour, supporting their broad use in contemporary restorative dentistry (1, 2). The long-term clinical success of composite resin restorations depends largely on the stability of the resin–dentin bond, which is formed through micromechanical interlocking within the hybrid layer (3). Despite adequate initial adhesion, this hybrid layer is highly susceptible to hydrolytic breakdown and enzymatic degradation, resulting in progressive loss of bond strength over time (4).

Matrix metalloproteinases (MMPs) are zinc- and calcium-dependent endopeptidases naturally present in dentin. They can be activated by acid etching or bacterial by-products, leading to the degradation of exposed collagen fibrils in the hybrid layer and a subsequent loss of resin-dentin bond durability (5-8). Specifically, MMP-2, MMP-8, and MMP-9 play key roles in this process. Consequently, strategies aimed at inhibiting these endogenous dentin proteases have become an important focus for enhancing bond durability.

Both synthetic and natural MMP inhibitors have been investigated for their effects on the longevity of restorations. Agents such as chlorhexidine, EDTA, and quaternary ammonium methacrylates are effective at inhibiting proteases, but they may cause concerns related to cytotoxicity, chemical reactions with other substances, or limited long-term effectiveness (9). Collagen crosslinkers, such as glutaraldehyde and proanthocyanidins, can also stabilize dentin matrices, although their clinical use is limited by long application times and technique sensitivity (10). These limitations have encouraged interest in naturally derived

[1] Department of Conservative Dentistry and Endodontics, Narayana Dental College and Hospital, Chintareddypalem, Nellore, Andhra Pradesh, India.

*Corresponding Author: Lavanya Anumula
Email: lavanyamds@gmail.com

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biomodifiers with enhanced biological activity and improved biocompatibility (11).

Triphala is a traditional polyherbal formulation derived from the dried fruits of *Phyllanthus emblica*, *Terminalia chebula*, and *Terminalia bellirica*. Triphala is typically prepared as a powdered extract or aqueous solution, and is commonly used in herbal medicine for its antioxidant, anti-inflammatory, and antimicrobial properties. This material has recently gained attention for its MMP-inhibitory potential in dental applications (11). Previous studies have demonstrated its capacity to inhibit MMP-9 activity at concentrations of 1500 µg/mL (11). Importantly, *Terminalia chebula* contains high levels of phenolics such as gallic and chebulinic acids, which contribute to strong collagenase and hyaluronidase inhibition (12). This suggests that altering the proportion of *Terminalia chebula* within the formulation may influence its collagen-preserving capability.

Despite evidence supporting Triphala's MMP-inhibitory potential, the available dental literature has almost exclusively evaluated its standard 1:1:1 composition (13). No studies have compared the effects of different Triphala ratios on resin–dentin bond strength or bond durability. Variations in the phytochemical composition (differences in the types and relative concentrations of plant-derived compounds) of Triphala could affect how it interacts with dentin collagen and adhesive systems. These variations could alter collagen crosslinking capacity, MMP activity, and surface chemistry, potentially affecting the overall bond strength and durability (5). T

The present study aimed to compare the effects of two 0.2% Triphala formulations (standard 1:1:1 and modified 1:2:1 ratios) as pre-treatment liners, on immediate and six-month aged µTBS of resin composite to dentin.

Materials and methods

Ethical approval for the use of extracted human teeth was obtained from the institutional ethical committee of Narayana Dental College and Hospital, Nellore, India (IEC/NDCH/2023/May/p-34). All extracted teeth were anonymized and handled according to institutional guidelines for biomedical research involving human tissues.

Tooth selection and storage

Twelve freshly extracted, caries-free human molars were collected. Teeth with cracks, restorations, developmental defects, or previous endodontic treatment were excluded. All teeth were cleaned of soft-tissue debris and stored in 0.1% thymol solution at 4°C for one week, then kept in distilled water until use. Before preparation, the teeth were equilibrated to room temperature.

Specimen Preparation

The occlusal enamel was removed using a diamond bur under continuous water cooling to expose a flat mid-coronal dentin surface. Radiographs were taken to confirm complete enamel removal and uniform dentin exposure. Each surface was standardized using 600-grit silicon carbide paper for 60 seconds to create a uniform smear layer (14).

Preparation of experimental materials

Commercially available powders of *Phyllanthus emblica*, *Terminalia chebula*, and *Terminalia bellirica* were used to prepare two formulations of Triphala:

- 1:1:1 formulation: One gram each of *Phyllanthus emblica*, *Terminalia chebula*, and *Terminalia bellirica* was mixed thoroughly, yielding a total of 3 grams of homogeneous powder.
- 1:2:1 formulation: Two grams of *Terminalia chebula* were mixed with one gram each of *Phyllanthus emblica* and *Terminalia bellirica*, resulting in a 4-gram homogeneous mixture.

Each Triphala formulation was prepared at a 0.2% (w/v) concentration by dissolving 0.2 g of each crude extract in 100 mL of distilled water, with continuous magnetic stirring to ensure uniform dispersion.

The solutions were filtered through muslin cloth to remove coarse particles. Subsequently, the filtrates were heated at 90°C for 3 hours on a mantle plate (a type of lab equipment that provides uniform heating) and allowed to cool to room temperature. The final experimental solutions were labeled as the 1:1:1 and 1:2:1 Triphala formulations.

Concentrations were confirmed gravimetrically, and all preparations were used within 24 hours to ensure chemical stability. The solutions were stored in amber glass bottles at room temperature (~25°C) and protected from direct light until use.

Grouping

Teeth were randomly allocated into three groups based on the dentin pre-treatment liner applied (n=4). Randomization was performed using a computer-generated random number sequence by an operator not involved in specimen preparation or testing. The study groups were as follows:

Group 1: Control (no dentin pre-treatment)
Group 2: 0.2% Triphala in a 1:1:1 ratio
Group 3: 0.2% Triphala in a 1:2:1 ratio

Bonding procedure

All teeth were treated using a conventional etch-and-rinse protocol. A 37% phosphoric acid gel was applied for 15 seconds, rinsed for 15 seconds, and then blot-dried. Subsequently, group 1 received no pre-treatment. In groups 2 and 3, the assigned Triphala solution was applied using a microbrush with gentle agitation for 5 minutes, followed by 10 seconds of air-drying. Afterwards, Tetric N-Bond Universal (Ivoclar Vivadent, Schaan, Liechtenstein) was applied in two coats, each scrubbed for 15 s, followed by gentle air-drying. The bonding agent was light-cured according to the manufacturer's instructions with an LED unit (Coltene Spec 3 LED, Coltene, Korea; light intensity ≥1000 mW/cm²).

Finally, a 4-mm composite resin block (IPS Empress Direct, Ivoclar Vivadent) was built incrementally in 1-mm layers, each light-cured for 20 seconds.

Specimen sectioning and aging

After 24 hours, specimens were sectioned perpendicular to the bonded interface using a slow-speed diamond saw to obtain beams of approximately 1 × 1 mm. Each tooth yielded 12–15 beams. From these, 24 beams with uniform dimensions were selected per group (n=24 beams per group).

The specimens in each group were then divided into two subgroups (n=12 beams per subgroup). One subgroup was tested after 24 hours (immediate subgroup), and the other subgroup was tested after storage in distilled water at 37°C for six months (aged subgroup). The water was renewed weekly in the aged subgroup.

Microtensile bond strength testing

Beams were attached to a Geraldeli jig (a device specifically designed to align microtensile specimens and ensure uniform tensile loading) using cyanoacrylate adhesive. The specimens were loaded in tension using a universal testing machine (ElectroPlus E3000, Instron Industrial Products, Grove City, PA, USA) at 0.5 mm/min until failure. µTBS was calculated in MPa by dividing the maximum load by the cross-sectional area of the bonded surface, which was measured using a digital caliper.

Statistical analysis

A priori power analysis was performed using the G*Power 3.1 software (Heinrich Heine University, Düsseldorf, Germany) based on a one-way analysis of variance with three groups, an alpha level of 0.05, a statistical power of 80%, and an effect size of 0.55. This analysis indicated that a total of 36 specimens, or 12 specimens per group, would be required. Considering that each group included two subgroups, the sample size was adjusted to 24 specimens per group, resulting in a total of 72 specimens for evaluating immediate and aged bond strength.

The normality of the data was confirmed using the Shapiro-Wilk test (P>0.05). A two-way analysis of variance (ANOVA) was performed to assess the effects of group and aging condition on the bond strength of composite resin to dentin. The statistical analysis was conducted using IBM SPSS Statistics for Windows, Version 25.0 (IBM Corp., Armonk, NY, USA) at a significance level of $P < 0.05$.

Results

Table 1 presents the mean and standard deviation (SD) of bond strength values (MPa) in the study groups under immediate and aged conditions. A two-way ANOVA revealed a significant interaction between groups and time ($P < 0.05$). Therefore, intergroup comparisons at each time point were conducted using one-way ANOVA, while independent t-tests were used to compare bond strength between immediate and aged conditions within each group.

The mean µTBS dropped from 11.20 ± 4.35 MPa at 24 hours to 6.75 ± 3.48 MPa after six months in the control

Table1. Mean ± standard deviation (SD) of microtensile bond strength (µTBS, MPa) for the study groups under immediate (at 24 hours) and aged (after 6 months) conditions

Groups	At 24 hours	After 6 months	P-value
Group 1 (Control)	11.20 ± 4.35	6.75 ± 3.48	0.005*
Group 2 (Triphala 1:1:1 ratio)	10.94 ± 4.47	9.54 ± 3.50	0.344
Group 3 (Triphala 1:2:1 ratio)	11.06 ± 5.95	10.50 ± 8.35	0.866
P-value	0.992	0.275	

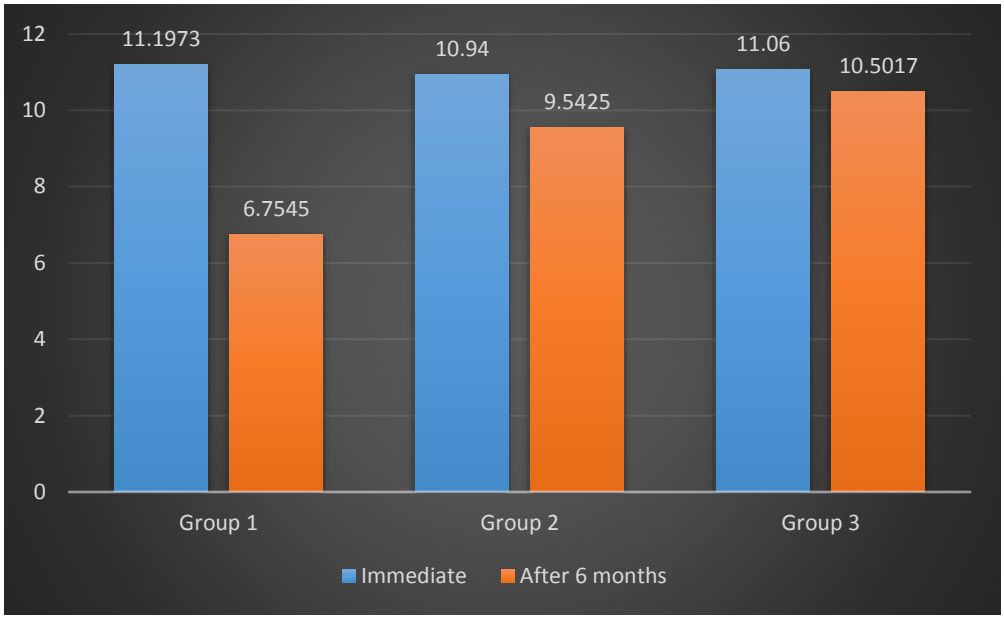

Figure 1. Comparison of mean microtensile bond strength values (MPa) in the study groups under immediate (at 24 hours) and aged (after 6 months) conditions

group. The independent t-test revealed that this reduction in bond strength was statistically significant (P = 0.005; Table 1). Group 2 (0.2% Triphala, 1:1:1 ratio) experienced a non-significant decrease in μTBS from 10.94 ± 4.47 MPa at 24 hours to 9.54 ± 3.50 MPa after six months (P = 0.344). Similarly, group 3 (0.2% Triphala, 1:2:1) maintained comparable values over time, with 11.06 ± 5.95 MPa at 24 hours and 10.50 ± 8.35 MPa after six months (P = 0.866).

At 24 hours, there were no statistically significant differences in microtensile bond strength among the three groups (P = 0.992). After six months, group 1 showed a lower μTBS value compared to groups 2 and 3; however, the difference between groups was not statistically significant (P = 0.275; Table 1). Figure 1 illustrates the bond strength values of the study groups under immediate and aged conditions.

Discussion

This study evaluated the effects of two 0.2% Triphala formulations (1:1:1 and 1:2:1 ratios) used as dentin pre-treatment liners on the immediate and aged bond strength of resin composite to dentin. The results demonstrated that both Triphala formulations maintained resin–dentin microtensile bond strength over six months, whereas the control group experienced a significant decline in bond strength. However, no significant difference in bond strength was observed between the groups, either immediately or after 6 months of storage.

Water storage at 37°C was selected as the aging protocol in this study, as it simulates intraoral hydration and activates endogenous MMPs (15). Although thermocycling and enzymatic challenges may better replicate clinical conditions, long-term water storage remains a validated and reproducible method for assessing adhesive degradation (16). The microtensile bond strength (μTBS) used in this study remains the most sensitive and widely accepted method to evaluate immediate and aged bond performance, allowing reliable assessment of adhesive interface degradation (17).

In the present study, the control group exhibited a significant loss of bond strength, decreasing from 11.20 ± 4.35 MPa at 24 hours to 6.75 ± 3.48 MPa after aging, corresponding to an approximate 40% reduction in μTBS. This decline is consistent with the findings of Hashimoto et al. (4), who demonstrated that water storage activates endogenous proteases and accelerates collagen degradation and hydrolysis within the hybrid layer, thus leading to bond deterioration (4).

The significant reduction in bond strength observed in the control group can be attributed to the degradation of the hybrid layer over time. Although contemporary adhesive systems provide satisfactory immediate bonding, the resin–dentin interface remains susceptible to hydrolytic breakdown and enzymatic degradation during aging. Water storage activates endogenous dentinal proteases, particularly matrix MMPs, which break down exposed and poorly infiltrated collagen fibrils within the hybrid layer (18). Weakening of the

collagen structure reduces support for the adhesive resin, leading to a decline in resin–dentin bond strength.

In the present study, both Triphala formulations demonstrated stable μTBS values after six months, with no statistically significant reduction compared with their immediate measurements. The absence of bond deterioration in the Triphala groups suggests a protective effect on the collagen matrix during aging. These findings align with Abraham et al. (11), who demonstrated significant MMP-9 inhibition by Triphala at a concentration of 1500 μg/mL. In the present study, the Triphala concentration was slightly increased to 0.2%, or approximately 2000 μg/mL. This slight increase aimed to enhance the availability of active phenolic compounds without compromising adhesive performance.

The comparable performance of the two Triphala formulations suggests that the collagen-preserving effect is primarily related to the overall concentration of bioactive polyphenols rather than to the relative predominance of a single component. Both formulations were applied at the same total concentration of 0.2% (approximately 2000 μg/mL), which may have provided sufficient levels of active compounds such as gallic acid, ellagic acid, and chebulinic acid to achieve effective collagen stabilization and MMP inhibition. Although the 1:2:1 formulation contained a higher proportion of *Terminalia chebula*, increasing its relative content did not result in additional improvement in bond durability. This finding suggests that the standard 1:1:1 formulation may already reach a saturation threshold beyond which further enrichment with *Terminalia chebula* does not enhance enzymatic inhibition or collagen crosslinking (12). Therefore, changing the formulation ratio alone, without increasing the total concentration or adjusting application conditions, is unlikely to enhance bond preservation.

In the current study, no significant differences in microtensile bond strength were observed among the study groups under immediate conditions, indicating that dentin pre-treatment with Triphala did not adversely affect initial adhesive performance. Although the Triphala-treated groups exhibited higher bond strength values than the control group after six months of aging, these differences did not reach statistical significance. This lack of significance may be due to the small sample size and the inherent variability of dentin substrates, which can lead to a wide range of μTBS values. Consequently, although a protective trend was observed for both Triphala formulations, the variability of the data may have masked statistically significant differences.

The findings of this study align with previous research showing the efficacy of various plant-derived biomodifiers in maintaining bond durability. Polyphenol-rich natural extracts such as green tea catechins, proanthocyanidins, Moringa, and *Centella asiatica* have all demonstrated collagen crosslinking, MMP inhibition, or hybrid layer stabilization in previous studies (19, 20). A recent study by Cho et al. (21) found that pretreatment with the flavonoid kaempferol significantly enhanced resin–dentin bond stability after thermocycling by inhibiting matrix metalloproteinase activity and promoting collagen crosslinking at the bonded interface.

Despite these promising findings, several limitations must be acknowledged. The study did not include a positive control, such as chlorhexidine or EDTA, which are well-established MMP inhibitors and could provide a benchmark for evaluating Triphala's efficacy. Additionally, failure mode analysis and microstructural assessment were not performed, limiting the understanding of how Triphala affects hybrid layer morphology or failure patterns. Furthermore, the beams subjected to tensile testing were not statistically independent, as multiple beams were derived from each tooth. Future investigations should focus on characterizing Triphala's active components, evaluating shorter and clinically acceptable application times, and exploring a broader range of concentrations. It is also suggested that future research consider tooth-level statistical analysis, advanced aging protocols, and microstructural evaluation to better assess the bonding interface. Ultimately, well-designed clinical studies will be required to determine whether Triphala can provide durable benefits in vivo.

Conclusions

Within the limitations of this in vitro study:

1- Dentin pre-treatment with 0.2% Triphala did not adversely affect immediate resin–dentin bond strength, indicating compatibility with the adhesive procedure.

2- Both the standard 1:1:1 and the modified 1:2:1 Triphala formulations preserved microtensile bond strength after six months of water storage, whereas the control group showed a significant reduction in bond strength. These findings suggest that Triphala may function as a natural dentin biomodifier capable of mitigating collagen degradation.

3- Increasing the proportion of *Terminalia chebula* in the Triphala formulation from 1:1:1 to 1:2:1 did not provide additional benefits concerning bond durability.

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None.

Conflicts of interest

The authors declare no conflict of interest.

Ethical considerations

Ethical approval for the use of extracted human teeth was obtained from the institutional ethical committee of Narayana Dental College and Hospital, Nellore, India (IEC/NDCH/2023/May/p-34). All extracted teeth were anonymized and handled according to institutional guidelines for biomedical research involving human tissues.

Author contributions

G.N. contributed to the study concepts, study design, and manuscript review; L.A. contributed to data analysis and manuscript editing and served as the guarantor; C.S., K.G., S.S., and M.G. contributed to data collection and manuscript preparation. All authors read and approved the final manuscript.

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