ORIGINAL ARTICLE

Effect of *Averrhoa bilimbi L.* extract gel as a natural enamel etching agent on the tensile bond strength of composite resin: an in vitro study

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ABSTRACT

Introduction: Composite resin is a restorative material that bonds to tooth structure through micromechanical interlocking. Etching removes the smear layer and creates microporosities, enhancing mechanical retention. Although 37% phosphoric acid is the gold standard, it may cause inflammatory cell infiltration. *Averrhoa bilimbi L*. contains citric, oxalic, and lactic acids, which may serve as a natural alternative etching agent for enamel. This study aimed to analyze the effect of *Averrhoa bilimbi L.* extract gel as a natural enamel etching agent on the tensile bond strength of composite resin restorations. **Methods:** This was an in vitro laboratory true experimental study that employed a post-test-only control group design. Twenty-eight extracted human premolars were divided into four groups (n = 7): three experimental groups etched with 2.5%, 5%, and 7.5% *Averrhoa bilimbi L.* extract gel, and one control group etched with 37% phosphoric acid. After etching, bonding agent and microhybrid composite resin were applied. Tensile bond strength was tested using a Universal Testing Machine, and data were analyzed using the Shapiro–Wilk, Levene's, and Kruskal–Wallis tests. **Results:** The highest mean tensile bond strength was observed in the 37% phosphoric acid group (7.56 ± 3.87 MPa), followed by 7.5% (6.71 ± 1.71 MPa), 5% (6.17 ± 1.39 MPa), and 2.5% (6.04 ± 3.01 MPa) Averrhoa bilimbi L. extract groups. The Kruskal-Wallis test showed p=0.874, indicating no statistically significant difference between the groups. **Conclusion:** *Averrhoa bilimbi L.* extract gel demonstrated a tensile bond strength comparable to that of 37% phosphoric acid. The extract gel demonstrated promising potential as a natural etching agent; however, further laboratory and clinical studies are required before it can be recommended for clinical application.

KEYWORDS
Organic acid, *Averrhoa bilimbi L.*, etching, tensile bond strength, composite resin

INTRODUCTION

The growing demand for restorative materials that are strong, aesthetic, and biocompatible has driven continuous innovation in dental materials, particularly composite resin. The success of composite restorations depends largely on the quality of adhesion between the material and the tooth surface.[1-3] Tensile bond strength is one of the key indicators used to evaluate the success of composite resin restorations, as it measures the ability of the adhesive material to maintain the bond when subjected to tensile forces, thereby making it essential for ensuring the long-term durability of the restoration.[4,5]

Etching procedures create microporosities in the enamel to enhance resin penetration and micromechanical retention.[6] While 37% phosphoric acid is effective in creating micromechanical retention, it may increase dentin permeability, irritate soft tissues, and cause pulp inflammation.[6-9] This may result in weaker adhesion and an increased risk of premature restoration failure.[8] Therefore, biocompatible alternatives are needed to minimize postoperative sensitivity.[9]

Natural materials have been widely studied for their potential to modify or replace chemically based dental materials.[10] Various natural organic acids have also been investigated for their potential use as alternative etching agents.[11] Studies have shown that weak acids such as citric, lactic, and oxalic acids can selectively demineralize enamel with minimal adverse effects.[12,13]

Averrhoa bilimbi L. (bilimbi) is rich in these organic acids and functions as a mild chelating agent that selectively cleans the smear layer.[14] It has a milder pH of approximately 2–3 compared with phosphoric acid (pH approximately 1), offering a safer biological approach. It is abundant in tropical regions and traditionally used in herbal medicine, making it an economical and readily available natural resource.[15]

While many natural etchants such as phytic acid and grape seed extract have been studied, the use of Averrhoa bilimbi L. has primarily been investigated for its ability to remove the smear layer in root canal treatments.[16] However, its specific application as an enamel etchant for composite restorations remains unexplored. The novelty of this research lies in evaluating whether the natural acidic constituents of *Averrhoa bilimbi L.* are sufficient to produce adhesive retention comparable to that achieved with conventional phosphoric acid. This study aims to analyze the effect of *Averrhoa bilimbi L.* (bilimbi) extract gel as an etching agent on the tensile bond strength of composite resin restorations.

METHODS

This in vitro true experimental study employed a post-test-only control group design. Based on the Federer formula for sample size calculation [(t-1) (n-1) ≥ 15], where t= 4 treatments, a minimum sample size of six samples per group was required. To account for potential dropouts, the sample size was increased by 10% using the formula N=n/(1-f), resulting in seven samples per group. Twenty-eight extracted human premolars, meeting the inclusion criteria (intact crowns, absence of caries, no previous restorations, and no evidence of abrasion or attrition), were randomly divided into four groups (n=7) using simple random sampling.

The experimental groups were etched with 2.5%, 5%, and 7.5% Averrhoa bilimbi L. extract gel, and the control group was etched with 37% phosphoric acid (Any-Etch, Korea). The concentrations were selected based on a previous study demonstrating that 5% natural tamarind extract gel, which shares a similar organic acid profile, produced comparable tensile bond strength to 37% phosphoric acid; therefore, 2.5% and 7.5% concentrations were included to evaluate dose-dependent efficacy.[17]

The raw material used was fresh fruit of Averrhoa bilimbi L. obtained from Padang, Indonesia (identified by the Herbarium of Universitas Andalas, No. 280/K-ID/ANDA/V/2025). The green and yellowish-green fruits were washed, sliced thinly, and dried in a food dehydrator at 40-50 °C for 5-6 hours. The dried fruits were then blended into a powder. The plant extract was prepared using the maceration method, with the powder soaked in 96% ethanol at a 1:7.5 ratio for 3x24 hours at room temperature to effectively isolate the active organic acids.

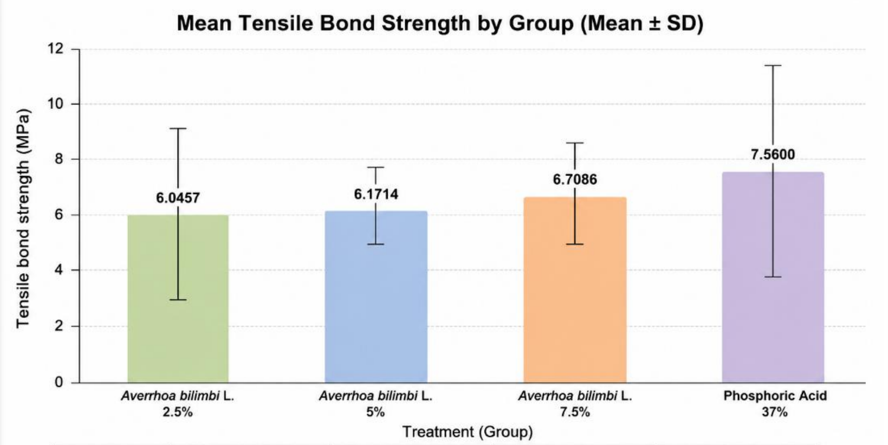
The filtrate was concentrated using a rotary evaporator at 40-50 °C. Finally, it was formulated into a gel using 5% Carboxymethylcellulose Sodium (CMC-Na) and distilled water.[18] The gel had a pH between 2 and 3 as measured using litmus paper.

The extracted human premolars were thoroughly cleaned of debris. The roots were centrally embedded in self-curing epoxy resin blocks up to 1 mm below the cementoenamel junction, leaving the entire coronal portion exposed for treatment. After the resin had set, the buccal enamel surfaces were flattened using 600-grit abrasive paper under running water to create a standardized bonding area. The enamel surfaces were etched for 15 seconds, rinsed, and dried following standard adhesive protocols. Subsequently, the samples were restored using a bonding agent (Hexabond, Indonesia) and microhybrid composite resin (Master Fill A2, Biodinamica). A 1 mm stainless-steel wire was embedded in the final composite layer to facilitate tensile testing.[6,11,19]

To simulate oral conditions, the samples were incubated at 37 °C for 24 hours prior to evaluation. The tensile bond strength was tested using a universal testing machine (Hydraulic UTM, WAW 300D, Ying Baixin Co., Ltd. Jinan, China) at a crosshead speed of 33 mm/min until debonding occurred.[3,19] Tensile bond strength was calculated in megapascals (MPa). Data distribution and variance homogeneity were evaluated using the Shapiro–Wilk and Levene's tests, respectively. Due to the non-normal distribution of the data, intergroup comparisons were analyzed using the non-parametric Kruskal–Wallis test.[20]

RESULTS

Based on the results of the tensile bond strength test, the average tensile bond strength values were obtained for the treatment groups etched with *Averrhoa bilimbi L.* extract gel at concentrations of 2.5%, 5%, and 7.5%, as well as for the control group etched with 37% phosphoric acid.



	Averrhoa bilimbi L. 2.5%	*Averrhoa bilimbi* L. 5%	*Averrhoa bilimbi* L. 7.5%	Phosphoric Acid 37%
N	7	7	7	7
Mean ± SD (MPa)	6.0457 ± 3.0123	6.1714 ± 1.3942	6.7086 ± 1.7104	7.5600 ± 3.8764
Min (MPa)	1.57	4.36	4.33	4.95
Max (MPa)	11.51	8.25	8.76	15.40

Error bars represent standard deviation (±SD).

Figure 1. Mean tensile bond strength by group

Based on Table 1, the group treated with Averrhoa bilimbi L. extract gel at a concentration of 2.5% showed the lowest mean tensile bond strength value, while the highest tensile bond strength value was observed in the group treated with 37% phosphoric acid.

To assess the normality of the data distribution among the treatment groups, a Shapiro-Wilk test was conducted. The analysis revealed that while the Averrhoa bilimbi L. extract gel groups exhibited a normal data distribution ($p > 0.05$), the control group showed a non-normal distribution ($p=0.017$). Consequently, data transformation was necessary. Following data transformation, the 37% phosphoric acid control group still demonstrated a non-normal distribution ($p=0.042$). Although Levene's test confirmed the homogeneity of data variances (p=0.348), the persistent non-normal distribution necessitated the use of the non-parametric Kruskal-Wallis test.

Table 2. Kruskal-wallis test results

Treatment	N	Tensile Bond Strength (MPa)	
		Mean ± SD	*P value*
Averrhoa bilimbi L. 2.5%	7	6.0457 ± 3.0123	0.874
Averrhoa bilimbi L. 5%	7	6.1714 ± 1.3942	
Averrhoa bilimbi L. 7.5%	7	6.7086 ± 1.7104	
Phosphoric Acid 37%	7	7.5600 ± 3.8764	

The Kruskal-Wallis test was used to compare the mean tensile bond strengths across all four groups (Table 2). The statistical analysis yielded a p-value of 0.874. Since this value is greater than the significance level of 0.05, there was no statistically significant difference in the tensile bond strength among the groups treated with various concentrations of Averrhoa bilimbi L. extract gel and the 37% phosphoric acid control group.

DISCUSSION

This study was conducted to determine the effect of Averrhoa bilimbi L. extract gel as a natural enamel etching agent on the tensile bond strength of composite resin restorations. The descriptive statistics revealed that the control group etched with 37% phosphoric acid exhibited the highest mean tensile bond strength. Among the experimental groups, an increase in tensile bond strength was observed with increasing concentrations of the Averrhoa bilimbi L. extract gel, peaking at the 7.5% concentration. However, statistical analysis confirmed that these differences were not statistically significant (p=0.874), suggesting that the natural extract performed comparably to the conventional chemical etchant. The concentrations used in this study (2.5%, 5%, and 7.5%) were based on prior evidence demonstrating optimal efficacy of similar organic acids at 5%. These findings are consistent with those of Wulandari et al., who reported that a 5% tamarind extract gel produced a mean tensile bond strength not significantly different from that of 37% phosphoric acid.[17]

Although 37% phosphoric acid remains the gold standard for creating enamel microporosities, its highly acidic nature (pH approximately 1) and aggressive demineralization profile pose clinical challenges. Excessive etching can deplete the inorganic mineral content to an excessive depth, exposing a vulnerable collagen network that is susceptible to enzymatic degradation by matrix metalloproteinases (MMPs), ultimately compromising the longevity of the restoration.[21-23] Furthermore, aggressive acid application may increase dentin permeability, leading to pulpal inflammation and postoperative hypersensitivity.[24]

In contrast, the mechanism by which *Averrhoa bilimbi L.* acts as an effective etchant lies in its unique phytochemical composition. Despite having a natural pH of 2-3, it is rich in organic weak acids, particularly citric, oxalic, and lactic acids. These organic acids function as natural chelating agents. When applied to the enamel surface, the acidic pH increases the concentration of hydrogen ions, which interact with the phosphate groups in the hydroxyapatite crystals. This interaction facilitates a diffusion process, releasing calcium and phosphate ions and forming soluble compounds such as calcium oxalate and calcium citrate.[25] This selective demineralization process adequately removes the smear layer and generates sufficient microporosities for the penetration of the bonding agent and composite resin.[26] Recent studies by Asmah et al. demonstrated that Averrhoa bilimbi L. extract effectively removes the smear layer and opens dentinal tubules, confirming its demineralizing ability.[16]

These findings (Averrhoa bilimbi L. extract gel at concentrations of 2.5%, 5%, and 7.5%) are also consistent with a previous study investigating natural plant extracts as alternative etchants. Wulandari et al. found that a 5% tamarind extract gel, which shares a similar organic acid profile (citric and lactic acids), produced a mean tensile bond strength that was not significantly different from that of 37% phosphoric acid.[17] Natural etchants mimic the selective etching approach favored in contemporary developments of universal adhesives, creating stable adhesive interfaces without the aggressive mineral depletion associated with conventional phosphoric acid, which may reduce MMP-induced degradation.[27,28] Similarly, research on natural citric acid-derived solutions reported that they effectively modified the smear layer and produced stable adhesive interfaces.[29]

However, it is crucial to discuss the clinical relevance of the obtained values. The generally accepted clinical standard for tensile bond strength of composite resin is approximately 14 to 30 MPa.[30] The overall mean values obtained in this study (6.05 - 7.56 MPa) fall below this standard. This highlights the distinction between statistical similarity and clinical performance, which can be heavily influenced by the testing methodology.

The limitations of this study primarily stem from the use of a macroscopic tensile test. As observed during testing, the manual placement of the stainless-steel wire in some samples resulted in imprecise positioning within the composite resin. This led to uneven load distribution and premature cohesive failures within the composite rather than true adhesive failures at the tooth-resin interface.[31] This methodological constraint explains why even the gold-standard 37% phosphoric acid yielded values (7.56 MPa) significantly below the typical 14-30 MPa clinical standard. Previous methodological assessments have highlighted that conventional tensile testing can be highly sensitive to specimen geometry and stress concentrations.[3,31]

Therefore, future research should consider employing the microtensile bond strength (μTBS) test, which provides a more representative depiction of clinical conditions and detects microscopic variations in adhesion more accurately.[16,32] Furthermore, the exact pH was measured using litmus paper instead of a digital pH meter due to the gel's viscosity. Another limitation is the absence of Scanning Electron Microscopy (SEM) analysis, which would have provided valuable visual confirmation of the enamel surface morphology and the depth of the microporosities following the etching procedure.

Beyond its efficacy in creating microporosities, Averrhoa bilimbi L. offers potential biological benefits. A recent study by our research team demonstrated that its extract possesses significant antibacterial properties against cariogenic bacteria such as Lactobacillus acidophilus.[33] Although this specific antibacterial activity was not evaluated in the present study, its potential dual role in cavity disinfection warrants further investigation, as it could reduce the risk of secondary caries.

CONCLUSION

The use of Averrhoa bilimbi L. extract gel as an etching agent showed no significant difference in the tensile bond strength of composite resin compared to 37% phosphoric acid. The extract gel demonstrated promising potential as a natural and biocompatible alternative that warrants further investigation, including studies involving enamel morphology (SEM), microtensile bond strength (μTBS), bond durability, aging simulation, and clinical evaluation.

The implication of this study is that *Averrhoa bilimbi L.* extract has the potential to serve as an eco-friendly and biocompatible alternative to conventional chemical etchants in adhesive dentistry, potentially reducing postoperative sensitivity while maintaining adequate restoration retention.

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