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Research Report

Potential of 5% tamarind extract gel as an etching agent: tensile strength and scanning electron microscope (SEM) evaluation

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ABSTRACT

Background: *Acid etching is a stage in obtaining bonds between composites and enamel. The application of acid to the enamel surface, however, can cause dissolution of hydroxyapatite and demineralisation of the enamel surface. Phosphoric acid, a strong acid, is an etching material that can reduce enamel hardness. Excessively reducing hardness can interfere with attachment to the restorative material. One medicinal plant that can be used as an alternative material in acid etching is tamarind.* ***Purpose:*** *This study aims to determine the effect of 5% tamarind extract gel on the tensile strength of composite resins.* ***Methods:*** *This is an experimental research study with a post-test-only control-group design. The study used 14 mandibular incisors. The labial part of the incisor was prepared using a diamond fissure bur with a diameter of 4 mm and a depth of 2 mm. The control group was then etched with 37% phosphoric acid gel, while the experimental group was etched with 5% tamarind extract gel. Bonding resins and micro-hybrid composite resins were applied, based on the manufacturers' instructions. Next, a tensile strength test and seeing formation resin tags by scanning electron microscope (SEM) were performed. Data were analysed using an independent t-test ($p < 0.05$).* ***Results:*** *The average tensile strength of composite resins in the group etched with 5% tamarind extract gel was the same as in the 37% phosphoric acid group ($p > 0.05$). SEM images also show that enamel etched with 5% tamarind extract gel produced a tag similar to that etched with 37% phosphoric acid gel.* ***Conclusion:*** *5% tamarind extract as an etching material can generate tensile strength of composite resin and trigger formation of resin tags in the same way as 37% phosphoric acid.*

Keywords: *acid etching; composite resin; tamarind extract; tensile strength*

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INTRODUCTION

Composite resin is an anterior and posterior restoration material often chosen because it has good aesthetics, a similar colour to natural teeth and good mechanical strength so that it can withstand mastication.[1–3] This material is micromechanically adhesive on the surface of enamel. One of the stages in obtaining bonding between composite and enamel is acid etching. The etching process starts with removing the enamel by as much as 10 µm, forming a microlayer as deep as 5–50 µm, removing surface contaminants and smear layers and then producing micro-irregularity/micro-porosity in prismatic enamel surfaces to provide micromechanical retention for resins.[4,5] Next, the application of acid to the enamel surface causes dissolution of hydroxyapatite and the enamel surface becomes demineralised. If resin bonding material is applied to the etched surface, it will flow to fill the irregular surface and form a resin tag; there will also be a mechanical bond between the composite resin and the enamel. The acid etching material most often used is 37% phosphoric acid.[6,7]

Phosphoric acid is a strong acid that can reduce enamel hardness.[8,9] Excessive reduction of hardness can interfere

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with the attachment of the restorative material.[9] The length of the tag produced from etching using phosphoric acid does not contribute to the strength of adhesion.[10] In addition, phosphoric acid causes pulp irritation.[8,11] These side effects can be minimised by using alternative ingredients from medicinal plants. Medicinal plants have advantages: as natural basic ingredients they are considered safer and have less serious side effects.[12] One medicinal plant considered as an alternative is tamarind (*Tamarindus indica*). Ripe tamarind flesh contains organic acids, such as citric acid, lactic acid, malic acid and tartaric acid,[12] which are classified as chelation agents, similar to ethylenediaminetetraacetic acid (EDTA) and phosphoric acid.[2] Tamarind extract at a concentration of 5% as an irrigation agent is able to dissolve calcium in root canal dentine.[13] The toxicity of 5% tamarind extract in root canal irrigation is lower than 3% hydrogen peroxide.[14] An ingredient that can remove and bind metal ions, such as calcium, can be used as an etching material,[2] therefore 5% tamarind extract has the potential to be used as an etching material.

To determine the ability of a material and to maintain its attachment under a received load, a tensile strength test can be performed. Measurement of the bond between the dental tissue and the restoration material can be carried out to assess the ability of the restoration material to remain in place.[15] Hence, this study aims to determine the potential of 5% tamarind extract as an alternative agent for acid etching, measuring the tensile strength of composite resins and descriptively seeing the formation of resin tags using a scanning electron microscope (SEM).

MATERIALS AND METHODS

This study has been approved by the Ethics Commission of the Medical Faculty of Jember University, Number 1138 / H25.1.11 / KE / 2017. This is an experimental research study with a post-test-only control-group design. This study used 14 lower mandibular incisors, which were single root and caries free, with no fracture and no abrasion on the buccal surface.

Tamarind extract was made from 300 g of ripe tamarind meat that had been separated from the seeds; 1 L of distilled water was added and the mixture stirred until homogeneous. The mixture was then centrifuged (Centrifuge Hermle Z-306, Germany) for 15 minutes, filtered and dried to produce extract in the form of crystals. The tamarind crystal extract then was stored in a freezer (-3°C) to keep it stable. Tamarind extract gel at a concentration of 5% was prepared by mixing 5 g of carboxymethyl cellulose sodium (CMC-Na) powder (Yanxing, China) and 100 mL of sterile distilled water. It was then stirred in a porcelain cup until it reached the phase of homogeneous gel. Then, 5 g of tamarind crystal extract was added and stirred until completely dissolved. The gel was stored in a glass jar. The pH of this gel had to be 4.

Subsequently, dental samples were prepared. First, tooth samples were cut at the cemento-enamel junction using a carborundum disc (Zhengzhou Shengxin Medical Instrument Co., China), so that the crown and root were separated. Second, cylindrical moulds (made of PVC) were prepared to fix the sample. The moulds were filled with investment material (Super Gips Dental Plaster, Indonesia). Third, the samples were placed on the surface of the moulds (precisely in the middle), with the labial part facing upwards. Fourth, the labial part was prepared using a diamond fissure bur (Baistra, China) with a diameter of 4 mm and a depth of 2 mm.

Fifth, the enamel surface of each sample was washed with distilled water and dried with air spray. Sixth, the samples in the control group were etched with as much as 0.1 ml of 37% phosphoric acid gel (Magnumdental, USA), while those in the experimental group were etched with as much as 0.1 ml of 5% tamarind extract gel, for 25 seconds. The etching materials were applied until the cavities were fully filled. Seventh, the cavities were washed with distilled water for 20 seconds and dried using air spray. Eighth, as much as 0.1 ml of bonding resin (Master Bond Biodinamica, Brazil) was dripped on a micro-brush and applied to the etched surfaces.[16] Ninth, the cavities were dried with light air pressure and then irradiated for 20 seconds (according to factory instructions). Tenth, the cavities were filled with a micro-hybrid composite resin (Master Fill A2 Biodinamica, Brazil) using layer-by-layer plastic filling instruments, where each layer was condensed with a cement stopper and then shined for 40 seconds (according to the manufacturer's instructions).

Eleventh, an acrylic ring (4 mm in diameter and 2 mm in height) was attached to each cavity that had been restored, using double-sided tape. Twelfth, the ring was filled with a micro-hybrid composite resin and shined. Thirteenth, polymethyl-methacrylate (PMMA) with a diameter of 10 mm and a height of 20 mm was glued on top of the acrylic ring with self-cured acrylic (Figure 1).[16] After completion,

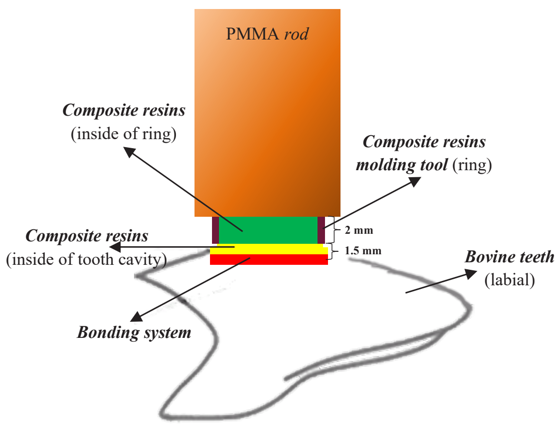

Figure 1. Shape of sample specimen for tensile strength test (side view).

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the sample was stored at room temperature (20–25°C) for 24 hours. Fourteenth, the samples were tested for tensile strength using a universal testing machine (Shimadzu, Japan). The result that appears on the universal testing machine monitor indicates the tensile strength of the composite resin, i.e. the force required to break it away from the tooth surface, in newton units (N) and in megapascal units (MPa). The research data were analysed statistically by independent t-test using SPSS Statistics for Windows, Version 20 ($p < 0.05$) (IBM, USA).

After the tensile strength test was performed, the tooth sample was removed from the investment material and cleaned of the remnants of the investment material. The tooth then was cut using a carborundum disc to obtain a sample 5 mm in length, 3 mm in width and 2 mm in thickness. After that, photographs were taken by SEM (Hitachi, Japan).

RESULTS

The data in Table 1 show the tensile strength of composite resins in the group etched with 5% tamarind extract gel as well as in the 37% phosphoric acid group. The results of the independent t-test show a value of $p = 0.175$ ($p > 0.05$), which means there is no significant difference between two groups. SEM images in the two groups can be seen in Figure 2, descriptively showing the presence of resin bonding that fills the tag/micro-porosity formed from etching using 5% tamarind acid extract and 37% phosphoric acid.

Table 1. Mean tensile strength score of composite resins

Groups	*n*	Mean (MPa)	SD
5% tamarind extract	7	5.442	0.07
37% phosphoric acid	7	5.376	0.09

n: number of samples; SD: standard deviation.

DISCUSSION

Restoration with a composite resin requires etching on tooth enamel, which aims to clean the smear layer as well as to produce micro-porosity on the surface of the tooth and the formation of tags. The application of the bonding material will fill the tags, forming a resin tag. This results in a mechanical interlocking bond between the composite resin and the dental tissue.[6]

The results of this study show that the samples in the group etched with 5% tamarind extract generated an average tensile strength value as large as samples in the group etched with 37% phosphoric acid. This is presumably because the tamarind extract contains several organic acids, such as citric acid, lactic acid, tartaric acid, acetic acid and malic acid, which, although they are weak acids, have almost the same effect as phosphoric acid (a strong acid).[2,12] As a strong acid, phosphoric acid releases more hydrogen ions (full ionisation) than a weak acid (half ionisation).[17,18] As more hydrogen ions are released, demineralisation of active teeth occurs, so that the microhardness of the enamel is reduced.[19] The decrease in microhardness influences the attachment of the restorative material to the hard tissue of the tooth.[20,21]

SEM photographs were taken to show the teeth after application of 5% tamarind extract and 37% phosphoric acid. In teeth etched with 5% tamarind extract, radiopaque lines as a result of the formation of resin tags are apparent (Figure 2); these also appear in the teeth etched with 37% phosphoric acid. This proves that 5% tamarind extract has the same potential for etching as 37% phosphoric acid. The organic acid content in tamarind extract is a chelating agent similar to EDTA and phosphoric acid, which can change the ratio of calcium/phosphate (Ca/P) to hydroxyapatite.[13] The application of acids on the enamel surface causes demineralisation and hydroxyapatite dissolution, leading to micro-irregularity/micro-porosity on the surface of the prismatic enamel, providing micromechanical retention for the resin.[4]

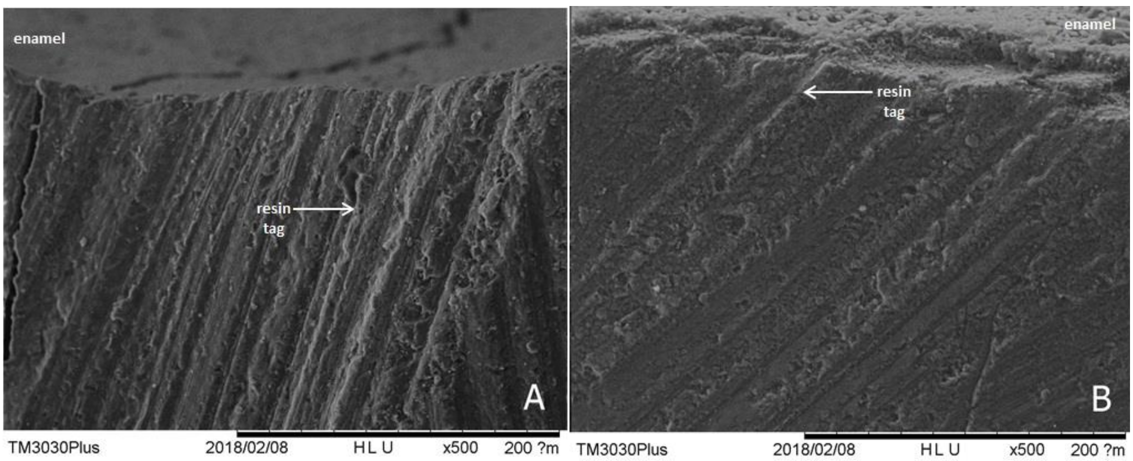

Figure 2. Formation of resin tags (arrows) after teeth were etched with (A) 5% tamarind extract and (B) 37% phosphoric acid (500x magnification SEM).

In addition, the statistical analysis shows that there is no significant difference between the average tensile strength of composite resins after etching with 5% tamarind extract and after etching with 37% phosphoric acid. This indicates that the demineralisation caused by the two materials on the surface of teeth is similar. Although the organic acid content in tamarind is weak, this weak acid still can release hydrogen ions. These ions will accumulate and stimulate further demineralisation,[21] with an insignificant difference in the tensile strength of composite resins between 37% phosphoric acid and 5% tamarind extract. In conclusion, 5% tamarind extract as an etching agent can generate tensile strength of composite resins and trigger the formation of resin tags to the same extent as 37% phosphoric acid.

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