



Indonesian Dental Association

Journal of Indonesian Dental Association

<http://jurnal.pdgi.or.id/index.php/jida>  
ISSN: 2621-6183 (Print); ISSN: 2621-6175 (Online)



Research Article

# Effect of Red Betel Leaf Infusion (*Piper crocatum*) on Physical and Mechanical Properties of Nanohybrid Composite Resins

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## KEYWORDS

discoloration, hardness,  
nanohybrid composite resin, red  
betel leaf infusion

## ABSTRACT

**Introduction:** Nanohybrid resin composite as a dental restorative material has physical properties (water absorption and color stability) and mechanical properties (hardness). Red betel infusion as a mouthwash, contains tannins and color pigment that will lead to discoloration when accumulated within the resin matrix. Water absorption is one of the supporting factors for color change. The weak acids in red betel leaf resulted breakdown of resin matrix bonds and a decrease in hardness. **Objective:** To analyze the effect on hardness and color change of nanohybrid resin composite after immersion in red betel leaf infusion. **Methods:** Samples of nanohybrid composite resin were shaped cylindrical with a diameter of 10 mm and a height of 2 mm. The samples were formed using a stainless-steel mold. Samples were immersed in red betel leaf infusion for 30 minutes then immersed in artificial saliva for 23 hours and 30 minutes. Samples for control group were immersed in artificial saliva for 24 hours. The immersion conducted for 12 and 15 days. The water absorption was measured using analytical balance, the color change was measured using the VITA Easyshade V, and hardness using a Vickers Microhardness Tester. **Results:** One Way ANOVA resulted for the water absorption  $p$ -value 0.268 ( $p > 0,05$ ),  $\Delta E$   $p$ -value 0.000 ( $p < 0,05$ ),  $\Delta L$   $p$ -value 0.444 ( $p > 0,05$ ),  $\Delta C$   $p$ -value 0.001 ( $p < 0,05$ ),  $\Delta H$   $p$ -value 0.012 ( $p < 0,05$ ), and hardness  $p$ -value 0.000 ( $p < 0,05$ ). Post Hoc Tukey resulted, significant differences in  $\Delta E$   $p$ -value 0,001 ( $p < 0,05$ ),  $\Delta C$   $p$ -value 0,002 ( $p < 0,05$ ),  $\Delta H$   $p$ -value 0,014 ( $p < 0,05$ ) and hardness  $p$ -value 0,000 ( $p < 0,05$ ) between immersion of 12 and 15 days compared to the control group. **Conclusion:** Red betel leaf infusion decreases the hardness of nanohybrid resin composite and overall color changed after 30 minutes immersion for 12 and 15 days.

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DOI: 10.32793/jida.v7i2.1147

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## INTRODUCTION

Dental plaque on the tooth surface is an etiological agent for caries and periodontal disease.<sup>1</sup> There are chemical and mechanical procedures for plaque management. Mouthwash as a chemical agent for plaque management, is used only as an extension and not as a replacement for mechanical procedure.<sup>2</sup> Mouthwash such as chlorhexidine has long term side effect. Herbal mouthwash is an alternative to mouthwash with fewer side effect. The natural ingredients should contain antimicrobial compounds for plaque management.

Red betel (*Piper crocatum*) in Indonesia is used as natural medicine to treat hypertension, lower blood sugar, and eliminating bad breath.<sup>3</sup> The chemical compound in red betel possess antimicrobial, antifungal, and anti-inflammatory properties of which are needed in the mouthwash composition. Red betel leaf is infused to be used as a mouthwash. The infusion results in a brown color due to the presence of tannins and color pigments in red betel leaves.<sup>4</sup>

Composite resin has become one of the preferred dental restorative materials due to its biocompatibility, high compressive strength, and a wide range of shades to achieve a good aesthetics. Composite resin has physical properties such as water absorption and color stability. One of its mechanical properties is hardness. The filler in composite resins minimize polymerization shrinkage, reduce thermal and contraction coefficients, strengthen restoration, and control viscosity. Nanohybrid composite resins were formed by micro-sized fillers (0,1– 2  $\mu\text{m}$ ) nano-sized fillers (<100nm).<sup>5</sup> Polymer bonds of composite resins are unstable and easily degraded by acidic pH then formed microporosity. Diffusion process of liquid from the environment outside materials into the composite resins.<sup>6</sup> Water absorption is one of the supporting factors for color change, the tannins and color pigments that were accumulated between matrix resin bonds cause a color change of composite resin.<sup>7</sup> The weak acids in red betel infusion which used as a mouthwash can lead a result breakdown of resin matrix bonds and a decrease in hardness of composite resins.<sup>8,9</sup>

## MATERIALS AND METHODS

This study is a laboratory experiments with post-test with control group study design. The research was carried out at the Dental Material Testing Center & Center of Research (DMTcore), Faculty of Dentistry, Universitas Trisakti. The sample of this study is Filtek Z250 XT A3 shade LOT 9776658 (3M ESPE) nanohybrid composite resin. There are 3 sample groups and each group consist of 10 samples.

Nanohybrid composite resin (Filtek Z250 XT 3M ESPE) was shaped in cylindrical stainless-steel mold with 10 mm in diameter and 2 mm in height. The samples

were light cured using LED light curing for 20 second and then removed from the mold.<sup>10</sup>

Thirty samples of nanohybrid composite resins were divided into three groups: 12 days immersion group, 15 days immersion group, and control group. The treatment groups were immersed into 10mL of red betel leaf infusion with the pH range 5,39 - 6,21 for 30 minutes per day with rinsing intervals per day for 12 and 15 days. The control group was immersed in artificial saliva for 15 days. The cycling method is used in this study, meaning that the red betel leaf infusion and artificial saliva are replaced every day. Immersion for 30 minutes per day with rinsing interval indicated a usage of red betels leaf infusion for 30 seconds every day for a month. The intervals rising is carried out to illustrate that the maximum use of mouthwash is 2 weeks. Each group is tested for mass, color, and hardness measurement after 12 and 15 days of immersion stimulated as 12 and 15 months of usage. All samples of this study were immersed in artificial saliva for 24 hours to reduce the amount of residual monomer from the polymerization process.

Mass measurement is used to determine the water absorption of resin composite. The mass measurement is conducted using the Analytical Balance, Fujitsu FS-AR210. Each sample were placed inside the analytical balance after pressing the button. The stable number will show after couple of seconds.

The color of composite resin can be tested using the spectrophotometer Vita Easyshade V. The tip of the spectrophotometer was placed in the center of the sample and pressed the button of the spectrophotometer to analyze the sample color. The spectrophotometer displayed four color measurement E (total color deviation), L (lightness/value), C (chroma), and H (hue). The total color deviation is processed from lightness, chroma, and hue using the CIE L\*a\*b (CIELAB) system.

Hardness measurement is conducted using the Vickers Microhardness Tester HMV- G31DT, Shimadzu, Tokyo, Japan. A 100-gram diamond indenter pressing on the sample for 15 seconds will form square-shaped indentation. The sample divided into three indentation areas. The unit of this measurement is VHN (Vickers Hardness Number). The hardness number of each sample is calculated as the average VHN value obtained from three indentation.

The statistical analysis for this study were the Shapiro-Wilk normality test ( $N < 50$ ) and Levene's homogeneity test. The overall significance differences of the sample groups were tested using parametric test with the One Way ANOVA. The Post Hoc Tukey test was to find out the differences of each group that showed significant differences in the One Way ANOVA. This statistical analysis was carried out using IBM SPSS Statistics for MacBook Version 27 (IBM, New York, USA).

## RESULTS

Mass measurement for water absorption was observed and averaged after 12 and 15 days of immersion (Figure 1). The mean of color measurement after immersion shown in color green for parameters E, color yellow for parameters L, color blue for parameters C, and color red for parameters H (Figure 2). The results of hardness measurements on 12 and 15 days of immersion showed differences (Figure 3).

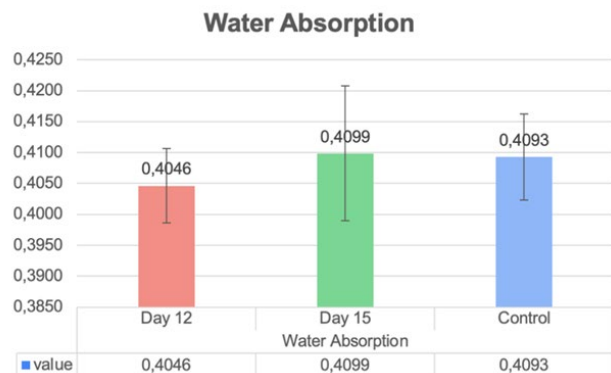


Figure 1. Mean water absorption (gram) of nanohybrid composite resin.

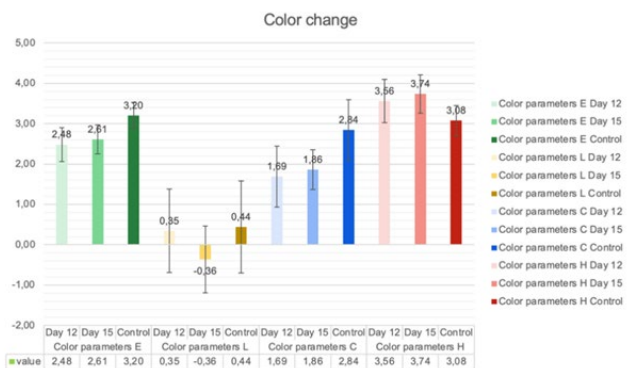


Figure 2. Mean color measurement of nanohybrid composite resin.

The One-Way ANOVA test is used in this study to compare the 12-days immersion, 15- days immersion and control group. There were significant differences for color parameters E, C, H, and hardness of nanohybrid composite resins. (Table 1).

The difference from each group of color and hardness measurements were tested using Post Hoc Tukey test. From Post Hoc Tukey test, there were significant difference between day 12 and 15 with control for color parameters E, C, and hardness (Table 2 and 3). Significant difference shown between day 15 with control and day 12 with day 15 for color parameters H (Table 2).

## Hardness

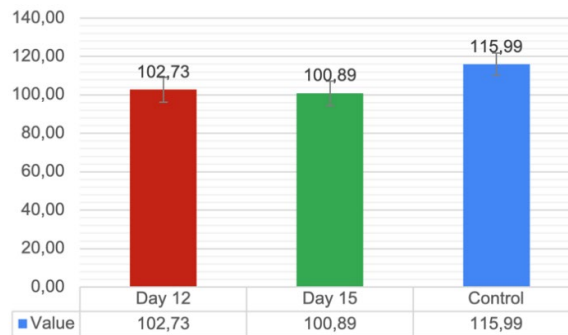


Figure 3. Mean hardness of nanohybrid composite resin (VHN).

Table 1. ANOVA test for water absorption, color measurement and hardness.

Parameter	p
Water absorption	0.268
Color parameters E	0.000*
Color parameters L	0.444
Color parameters C	0.001*
Color parameters H	0.012*
Hardness	0.000*

\*p>0.05 (statistically significant difference)

Table 2. Post Hoc Tukey test for color measurement of nanohybrid composite resin

		p
Color Parameters E	Day 12 - Control	0,001*
	Day 15 - Control	0,001*
	Day 12 - Day 15	1.000
Color parameters C	Day 12 - Control	0,002*
	Day 15 - Control	0,002*
	Day 12 - Day 15	0.836
Color parameters H	Day 12 - Control	0.816
	Day 15 - Control	0,014*
	Day 12 - Day 15	0.014*

Table 3. Post Hoc Tukey test for hardness of nanohybrid composite resin

Parameter	p
Day 12 - Control	0.000*
Day 15 - Control	0.000*
Day 12 - Day 15	0.787

\*p>0.05 (statistically significant difference)

## DISCUSSION

Red betel leaf as an herbal mouthwash has antimicrobial and anti-inflammatory agents. Red betel leaf contains tannins and pigment which will be dissolved during infusion process.<sup>11</sup> The pH level of red betel leaf infusion is 6 which is in weak acid category. The pigments and acid in this infusion could affect the dental restorative material.

Composite resins as used as a dental restoration is classified by the filler size distribution and composition. Nanohybrid composite consist of micro-sized (0,1 – 2 µm) and nano-sized (<100nm) filler.<sup>5,12</sup> Nanohybrid composite resin has physical properties (water absorption and color) and mechanical properties (hardness). The water absorption occurs because the molecular diameter of water (0.16 nm) is smaller than the distance between the polymer chains, resulting diffusion of water into the polymer chains.<sup>13</sup> The water absorption is a factor for the color changes and decrease in hardness of composite resins.

Based on the statistical analysis there were significant differences in color parameter E (total color deviation), C (chroma), H (hue). The color change of nanohybrid composite resins occurs on the day 12 and 15 but cannot be seen clinically. Color intensity of the sample group shown significant differences where the intensity of control group is higher than the intensity of 12 and 15 days of immersion with red betel leaf infusion groups. Based on the color parameter hue of 12 and 15 days of immersion shown a color change than the control group, the immersion group is redder than the control group.

The infusion of red betel leaf has brown color because the tannins and the pigments of red betel dissolved in water during the boiling process.<sup>3</sup> The tannin content in red betel leaf is 0.25% and has brown color pigment.<sup>14</sup> Discoloration of composite is caused by the extrinsic and intrinsic factors.<sup>15</sup> Pigmented and acid mouthwash is one

of the extrinsic factors where the pigment of the infusion are accumulated between resin polymer chain and caused discoloration of composite resins.<sup>7</sup> The pigments diffused into composite resin's matrix during water absorption and would be accumulated between the matrix bonds. The weak acid of red betel leaf infusion led to degradation of the polymer bonds, therefore facilitates the diffusion process of pigmented infusion into the composite resins.<sup>6</sup> This study is in line with previous study where the pigments and staining agent of black tea (*Camellia sinensis*) diffused into composite resins and caused a discoloration of nanofiller and nanohybrid composite resins.<sup>16</sup>

Hardness of composite resins is an important mechanical property and valuable parameter comparison with the tooth structure. Hardness defined by degree of surface deformation of a material.<sup>5</sup> Hardness number is a parameter of a material ability to abrasive forces, especially from chewing and toothbrushing activities.<sup>17</sup> Composite resin's hardness are affected by the degradation of composite resin.<sup>18</sup> The mastication pressure can be measured using the Vickers Microhardness Tester. This study measured the hardness of nanohybrid composite resins after immersion in red betel leaf infusion after 12 and 15 days. Based on the statistical analysis, there were significant difference between the immersion in red betel leaf infusion groups and control group (immersed in artificial saliva). The 12- and 15-days immersion in red betel leaf infusion have lower hardness than the control group. The decrease in hardness has effects on wear resistance, fatigue of materials, and could lead to failed composite resins restoration.<sup>19</sup>

The weak acid that contained in the red betel leaf infusion and water absorption affect the hardness of composite resins.<sup>6</sup> The reaction between H<sup>+</sup> ions of weak acids and OH<sup>-</sup> ions inside resin matrix caused unstable chemical bonds between filler and matrix of the composite resins.<sup>20</sup> The fillers of composite resins were released and the hardness of composite resins was decreased because of this process.<sup>6</sup> The hardness of the resin composite will decreases when the composite its saturation point.<sup>21</sup> Therefore, the duration of immersion affects the hardness level of composite resins.<sup>22</sup>

Water absorption occurs because of the diffusion process. Acid is one of the factors that exacerbated high absorption of water in composite resins. The bond of the matrix and filler of the composite resins will be damaged because of the acid pH. Matrix becomes detached and form microcracks and micro-voids resulting the decreases of composite resin's hardness.<sup>23,24</sup> The more micro-voids formed, the higher color change will occur because the pigment will be accumulated in the microvoids.<sup>25</sup>

## CONCLUSION

Red betel leaf decreases the hardness of nanohybrid resin composite and overall color changed after 30 minutes immersion for 12 and 15 days.

## ACKNOWLEDGMENTS

The authors would like to thank the Faculty of Dentistry, Universitas Trisakti, for the support of this study.

## CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest.

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