

Literature Review

The effects of propylene glycol addition in the combination of calcium hydroxide and propolis on compressive and flexural strength

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ABSTRACT

Background: The mechanical properties of pulp-capping materials may affect their resistance to fracture during placement of a final restorative material or while supporting an overlying restoration over time when the mastication process is carried out. The combination of calcium hydroxide and propolis as a pulp capping material has the weakness on mechanical properties so it is necessary to add another material to improve its mechanical properties. Propylene glycol is used as a vehicle because it improves its handling property. **Purpose:** To explain how the compressive and flexural strength of the pulp capping material in the combination of calcium hydroxide, propolis, and propylene glycol compared without adding of propylene glycol. **Methods:** The study used 4 treatment groups with each group consisting of 7 replications. Control group is a combination of calcium hydroxide-propolis with a ratio of 1: 1.5, group 1 is a combination of calcium hydroxide-propolis-propylene glycol 20% with a ratio of 1:1.5:0.375, group 2 is a combination of calcium hydroxide-propolis-propylene glycol 30% with a ratio of 1:1.5:0.375, and group 3 is a combination of calcium hydroxide-propolis-propylene glycol 30% with a ratio of 1:1.5:0.375. Materials were mixed according to comparison and printed on a cylindrical mold with the size of 4 mm x 6 mm and a block mold with the size 65 mmx10 mmx6 mm. Then, the compressive and flexural strength was tested using an Autograph test instrument. **Results:** The statistical analysis was performed with ANOVA and Tukey HSD's post-hoc test. There were statistically significant differences on compressive and flexural strength between groups ($P < 0.05$). **Conclusion:** Addition of propylene glycol in the combination of calcium hydroxide and propolis have an influence of increasing of compressive and flexural strength, especially in the addition of 40% propylene glycol.

Keywords: compressive strength, flexural strength, calcium hydroxide, propolis, propylene glycol

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INTRODUCTION

Pulp capping is a treatment procedure using pulp capping material placed on the exposed pulp or on a thin layer of dentin that still exists to stimulate hard-tissue barrier.¹ However, both of the placing process of restoration materials on the top of the pulp capping material and the mastication process will involve the mechanical strength of the pulp capping material so that the pulp capping material must be able to withstand the processes. Thus, pulp capping material will not be damaged and will continually maintain tooth vitality as well as form reparative dentin.

Therefore, a pulp capping material made from combination of calcium hydroxide and propolis in order to increase its anti-bacterial and anti-inflammatory properties without causing toxicity. Nevertheless, the combination of these two materials still has a weakness that is not good mechanical properties. A study by Widjiastuti et al.² shows

that the combination of calcium hydroxide and propolis in a ratio of 1: 1.5 has a strength of 0.54 MPa. This means that the compressive strength of the combination of these two materials is lower than that of the combination of calcium hydroxide and aquadest.

As a result, the mechanical properties of this pulp capping combination of calcium hydroxide and propolis may be improved by adding propylene glycol. Propylene glycol is known to be able to increase the hydration reaction that leads to the formation of hydrates, makes the particle combination of these materials smaller, and also functions as an accelerator. Besides, propylene glycol is able to form intermolecular bonds and increase bond strength in dentin.³ Propylene glycol also does not inhibit the release of active components of calcium ions needed for the formation of reparative dentin.⁴

Moreover, the mechanical properties of the pulp capping material are needed since during mastication there is an

occlusal load on the material.³ The mechanical properties of the material can be evaluated by measuring compressive and flexural strengths. The compressive strength of the pulp capping material influences the condensation of the restoration above the pulp capping material, while the flexural strength is related to the resistance of the material to the deformation. Flexural strength is needed to be able to withstand the mastication pressure.

Based on the background above, it is known that the effects of the addition of propylene glycol to the pulp capping material made of calcium hydroxide combined with propolis on the compressive and flexural strengths still have been questioned. Hence, this study aims to reveal the effects of the addition of propylene glycol to the pulp capping material made of calcium hydroxide combined with propolis on the compressive and flexural strengths. The results of this study then are expected to improve mechanical properties in the combination of these materials.

MATERIALS AND METHODS

This study is an experimental laboratory research with a post test control group design. This study used cylindrical material samples with a size of 4x6mm for compressive strength testing and beam material samples with a size of 65x10x6 mm. Each of those then was divided into 4 treatment groups, namely control group with a combination of calcium hydroxide and propolis; Group 1 with a combination of calcium hydroxide, 11% propolis extract, and 20% propylene glycol; Group 2 with a combination of calcium hydroxide, 11% propolis extract, and 30% propylene glycol; and Group 3 with a combination of calcium hydroxide, 11% propolis extract, and 40% propylene glycol.

Afterwards, propolis extract was made with solid *Apis mellifera* as much as 1000 grams, which were cut into small pieces with a size $\pm 1/2 - 1$ cm, then put into an extractor, and added with 1000 ml of 96% ethanol in a closed container and mixed until all propolis submerged in ethanol solution. It was then shaken with a shaker for 2x24 hours. After that, maceration was stopped and filtered. From the filtering results, clear liquid was derived from propolis. The clear liquid then was evaporated with a vacuum evaporator at a temperature of 50°C-60°C. Next, thick and brownish propolis extract liquid with a concentration of 100% known

as pure propolis was obtained. 11% propolis solution then was made by diluting 100% propolis solution with sterile aquades. The dilution was performed with the following comparison formula between concentration and volume as follows:

$$M1 \times V1 = M2 \times V2$$

M indicates concentration and V indicates volume, for both solutions with different concentrations.

Model mold used to measure compressive strength was acrylic molds with a diameter of 4 x 6 mm according to ISO 9917-1: 2007 (see Figure 1), and molds with a size of 65 mmx10 mmx6 mm to measure flexural strength (Figure 2).

To measure compressive strength, a combination of calcium hydroxide and propolis was made by mixing calcium hydroxide powder and propolis extract in a ratio of 1:1.5. Thus, 0.125 grams of calcium hydroxide powder was mixed with 0.1875 ml of propolis extract. Next, a combination of calcium hydroxide, propolis and 20% propylene glycol was made with a ratio of 1:1.5:0.375. Hence, 0.125 grams of calcium hydroxide powder was mixed with 0.1875 ml of propolis extract and 0.047 ml of 20% propylene glycol. After that, a combination of calcium hydroxide, propolis, and 30% propylene glycol then was made with a ratio of 1: 1.5: 0.375. Therefore, 0.125 grams of calcium hydroxide powder was mixed with 0.1875 ml of propolis extract and 0.047 ml of 30% propylene glycol. And, a combination of calcium hydroxide, propolis and 40% propylene glycol was made with a ratio of 1:1.5: 0.375. As a result, 0.125 grams of calcium hydroxide powder was mixed with 0,1875 ml of propolis extract and 0.047 ml of 40% propylene glycol.

The combination of calcium hydroxide and propolis was made by mixing calcium hydroxide powder and propolis extract in a ratio of 1:1.5. Thus, 3 grams of calcium hydroxide powder was mixed with 4.5 ml of propolis extract. Next, a combination of calcium hydroxide, propolis, and 20% propylene glycol was made with a ratio of 1:1.5:0.375. Hence, 3 grams of calcium hydroxide powder was mixed with 4.5 ml of propolis extract and 1.125 ml of 20% propylene glycol. Afterwards, a combination of calcium hydroxide, propolis and 30% propylene glycol was made with a ratio of 1:1.5:0.375. Therefore, 3 grams of calcium hydroxide powder was mixed with 4.5 ml of propolis extract and 1.125 ml of 30% propylene glycol.

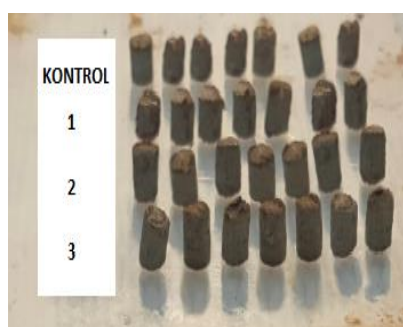


Figure 1. Samples for Compressive.

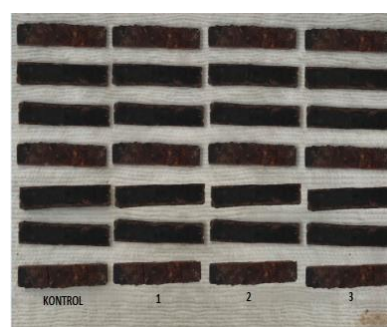


Figure 2. Samples for Flexural.

And, a combination of calcium hydroxide, propolis and 40% propylene glycol was made with a ratio of 1:1.5:0.375.⁵ Consequently, 3 grams of calcium hydroxide powder was mixed with 4.5 ml of propolis extract and 1.125 ml of 40% propylene glycol.

Subsequently, both of the combination of calcium hydroxide-propolis and the combination of calcium hydroxide-propolis-propylene glycol were put into molds. The bottom of the molds used a flat area of acrylic. Those combinations of materials then were put into the molds with a plastic filling instrument and a cement stopper. The dough was overloaded from the molds and then pressed on a flat plane made of acrylic on it.



Figure 3. Compressive strength measurement.



Figure 4. Flexural strength measurement machine.



Figure 5. The numbers listed on the universal measurement machine.

After the combination materials had hardened, they were removed from the molds and then evaluated physically. Uneven or distorted samples were not used as samples. The combinations of these materials then were allowed to stand for 48 hours at 37° C.⁶

First, each sample was placed in the center of the pressing device with the vertical axis of the sample perpendicular to the plane. Second, the universal testing machine was turned on, and then the pressure section moved slowly with a pressure of 1kN and a speed of 1mm / min pressing until it broke (ISO 9917-1: 2007). Third, after the sample was destroyed, the numbers listed on the device were recorded (see Figure 3). Fourth, the numbers listed in kgF were converted in Newton and then divided by cross-sectional area so that the compressive strength was obtained in Mega Pascal units as the following formula:

$$C = 4P / \pi D^2$$

Note:

C = Compressive strength value (in Mega Pascal)

P = Maximum force applied to the sample when the sample is destroyed (in Newton); D = Sample diameter (in millimeters)

Flexural strength measurement was conducted with Universal Testing Machine. First, each sample was given a center line as an emphasis point. Second, each sample was supported at both ends with a support distance of 50 mm. Third, pressing was given in the middle until the material was broken (see Figure 4). Fourth, the tool then showed the load value (see Figure 5). Fifth, the flexural strength was calculated with the following formula:

$$S = 3IP / 2bd^2$$

Note:

S = flexural strength (N / mm²); I = Supporting distance (mm)

P = load (N); b = test rod width (mm); d = test rod thickness (mm)

Subsequently, the research data were grouped and tabulated statistically with Shapiro-Wilk normality test. Homogeneity test then was performed with Levene test. Afterwards, difference test was carried out with one-way ANOVA to evaluate differences in mean values. Post hoc test then was conducted with Tukey HSD to compare among the samples.

RESULTS

The result data of compressive strength measurement using the Universal Testing Machine in Mega Pascal (MPa) units can be seen in Table 1. Based on this table, it can be seen that the compressive strength increased in the group with the

Table 1. The number of samples together with the mean and standard deviation values of compressive strength

Groups	N	Mean	SD
Control	7	1.13	0.39
1	7	1.35	0.26
2	7	1.67	0.15
3	7	2.18	0.42

Note:

Control Group: Ca(OH)₂ + 11% Propolis Extract

Group 1: Ca(OH)₂ + 11% Propolis Extract + 20% Propylene Glycol

Group 2: Ca(OH)₂ + 11% Propolis Extract + 30% Propylene Glycol

Group 3: Ca(OH)₂ + 11% Propolis Extract + 40% Propylene Glycol

addition of propylene glycol (Group 1,2,3) compared to the control group (without the addition of propylene glycol).

Next, the results of the normality test using Shapiro Wilk on compressive strength showed normal distribution in all groups ($p > 0.05$). After that, the results of the homogeneity test using Levene test indicated homogeneous data in all groups ($p > 0.05$). And, the results of the difference test using one-way ANOVA then revealed significant differences in the compressive strength between all treatment groups ($p < 0.05$). This can be seen in Table 2.

Furthermore, to evaluate which pair of groups had significant differences, Tukey HSD test was performed. The test results can be seen in Table 3. Based on Table 3, it can be concluded that there were significant differences between the control group and Group 2, as well as between the control group and Group 3.

The results of flexural strength measurement using the Universal Testing Machine in Mega Pascal (MPa) units can be seen in Table 4. Based on this table, it can be seen that flexural strength increased in the groups with the addition of propylene glycol (Groups 1,2,3) compared to the control group (without the addition of propylene glycol).

The results of the normality test using Shapiro Wilk on flexural strength showed a normal distribution in all groups ($p > 0.05$). Afterwards, the results of the homogeneity test using Levene test indicated homogeneous data in all groups ($p > 0.05$). And, the results of the difference test using one-way ANOVA then revealed significant differences in the compressive strength between treatment groups ($p < 0.05$). This can be seen in Table 5.

Moreover, to find out which pair of groups had significant differences, the Tukey HSD test was carried out. The test results can be seen in Table 6. Based on Table 6, it can be concluded that there were significant differences between the control group and Group 2 as well as between the control group and Group 3.

DISCUSSION

The results show that the compressive and flexural strengths in the groups with the combination of calcium hydroxide, propolis, and propylene glycol are better than the group without the addition of propylene glycol. Besides, the

Table 2. Difference test results (one-way ANOVA) on compressive strength

	Sum of Squares	Df	Mean Square	F	Sig.
Between Groups	4.371	3	1.457	13.953	.000
Within Groups	2.506	24	0.104		
Total	6.877	27			

Table 3. Tukey HSD test results on compressive strength

Groups	Mean Difference	Std. Error	Sig.
Control Group and Group 1	0.223	0.173	0.578
Control Group and Group 2	0.539	0.173	0.022*
Control Group and Group 3	1.052	0.173	0.000*
Group 1 and Group 2	0.316	0.173	0.283
Group 1 and Group 3	0.829	0.173	0.000*
Group 2 and Group 3	0.513	0.173	0.032*

Table 4. The number of samples together with the mean and standard deviation values of flexural strength

Groups	N	Mean	SD
Control	7	0.34	0.27
1	7	0.81	0.48
2	7	1.02	0.45
3	7	1.30	0.48

Table 5. The results of the difference test (one-way ANOVA) on flexural strength

	Sum of Squares	Df	Mean Square	F	Sig.
Between Groups	3.465	3	1.155	6.343	.003
Within Groups	4.370	24	0.182		
Total	7.834	27			

Table 6. The results of Tukey HSD test on flexural strength

Groups	Mean Difference	Std. Error	Sig.
Control Group and Group 1	0.467	0.228	0.199
Control Group and Group 2	0.680	0.228	0.031*
Control Group and Group 3	0.963	0.228	0.002*
Group 1 and Group 2	0.213	0.228	0.788
Group 1 and Group 3	0.496	0.228	0.158
Group 2 and Group 3	0.284	0.228	0.606

results also indicates that the pulp capping material with the combination of calcium hydroxide and propolis only has poor mechanical properties. Similarly, a previous study shows that the compressive strength of the combination of calcium hydroxide and propolis in a ratio of 1:1.5 has a lower compressive strength (0.54 MPa) than that of the combination of calcium hydroxide and aquadest (1.24 Mpa).²

Moreover, certain factors affecting the mechanical properties of the pulp capping material actually need to be considered since during mastication there will be an occlusal load on the material.⁴ Compressive strength is one of the factors that influence the hardness of the material.⁷ The compressive strength of the pulp capping material influences the condensation of the restoration above the pulp capping material, thus, it must be able to withstand the pressure from the restoration material. In addition to the compressive strength, the pulp capping material has flexural strength.⁸ Flexural strength is the ability of a material to resist flexural force, which is a combination of compressive, tensile, and shear forces while functioning inside the oral cavity.⁹ Flexural strength is related to material resistance to deformation. Hence, materials with high flexural strength have advantages in cases with extensive restoration and also in cases of minimally invasive treatment options with thin wall thicknesses. Now, flexural strength test is preferred for brittle dental materials, such as cement or composites since the stress distribution is closer in simulating what happens under clinical function.¹⁰ Flexural strength, as a result, is needed by a material to be able to withstand the mastication pressure which can result in permanent deformation such as fracture and crack.¹¹

Furthermore, imperfect setting reactions can reduce compressive and flexural strengths.⁶ In general, the mixing process of calcium hydroxide and propolis requires about 48 - 57 minutes to harden.¹² The pulp capping material which cannot be hardened/ set, therefore, will not have mechanical strength and also cannot be used directly under restoration with resin material since the resin is hydrophilic and will affect its bonding system to teeth.¹³

Moreover, solubility is also considered as an important factor in assessing the clinical resistance of pulp capping materials. The combination of calcium hydroxide and propolis is also sensitive to erosion. This is mainly due to the hydrolysis of the component ingredients. Propolis is known to have the highest solubility compared to Dycal and MTA because of the hydroxyl groups that bind to water so that it affects the mechanical strength that is not good.¹²

The pulp capping material is actually set through the acid-base mechanism. This acid-base reaction, however, can form salt and water. Materials with higher water levels will certainly produce materials with a more dilute consistency, thereby affecting the structure of the material and ultimately reducing the compressive strength and flexural strength.¹⁴

On the other hand, the combination of calcium hydroxide and propolis is known to be able to increase the solubility of calcium hydroxide due to the presence of resin which can reduce the diffusion of water into the cement. The presence

of resin in propolis can also prevent cement maturation thereby increasing solubility.¹⁵

The bond that can affect the mechanical strength in the combination of calcium hydroxide and propolis is Van der Waals bond. Van der Waals bonds are weak bonds that can make the molecules in a material bind tightly, thereby producing a weaker structure and lowering the compressive and flexural strengths.¹⁶

Thus, to overcome the low mechanical properties in the combination of calcium hydroxide and propolis, propylene glycol is added. Propylene glycol can increase consistency when mixing. Besides, propylene glycol can generate bond strength, but reduce setting time. Propylene glycol is also known to be hygroscopic and can reduce the amount of water available in the hydration process so that it can produce hard materials and have good mechanical properties.¹⁷

Propylene glycol at the concentrations of 20%, 30%, and 40% were selected based on a study conducted by Safavi and Nakayama¹⁸ stating that the value of calcium hydroxide released increases in propylene glycol at a concentration of 20% and reaches a peak at a concentration of 40%. Meanwhile, the addition of 50% propylene glycol can make the release of calcium ions (Ca²⁺) decreased.¹⁹ Furthermore, the addition of 20% propylene glycol can increase the bonding of the pulp capping material to dentin.³

Based on the results of this study, the compressive strength and flexural strength are getting better with the addition of 20% propylene glycol, followed by 30% propylene glycol, and 40% propylene glycol. The compressive strength in the group with the combination of calcium hydroxide and propolis was 1.13 MPa, whereas in the groups with the combination of calcium hydroxide, propolis and propylene glycol was 1.35 MPa at the propylene glycol concentration of 20%, 1.67 MPa at the propylene glycol concentration of 30%, and 2.18 MPa at the propylene glycol concentration of 40%. On the other hand, the flexural strength in the group with the combination of calcium hydroxide and propolis was 0.34 MPa, whereas in the groups with the combination of calcium hydroxide, propolis, and propylene glycol was 0.81 MPa at the propylene glycol concentration of 20%, 1.02 MPa at the propylene glycol concentration of 30%, and 1.3 MPa at the propylene glycol concentration of 40%.

Higher propylene glycol concentrations as a vehicle can actually reduce the porosity of the material that has been set. Mechanical strength in cement is influenced by the concentration of the solution added. Adding a high

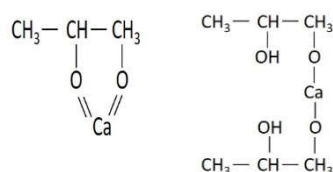


Figure 6. Chemical structure of calcium hydroxide-propolis-propylene glycol.

concentration of propylene glycol (> 50%) to the mixture, consequently, can cause the material to be long-standing, have higher solubility, and form greater porosity thereby reducing the mechanical strength of the material.²⁰ This is supported by a study conducted by Duarte et al.²¹ arguing that in the group added 100% propylene glycol, no setting occurs since there is no hydration in the mixture of these ingredients. Hydration helps stabilize the ions in solution and prevents cations from rejoining the anion.

Actually, in the mixing process of calcium hydroxide, propolis, and propylene glycol, an acid-base reaction occurs. When the reaction begins, water is produced as one of products. At the setting stage, there is loss of the ester group and at the same time it forms a carboxylate band, which shows the formation of calcium salts (CaO) through chelation process with existing calcium ions. Chelation is a type of ionic bond between molecules and metal ions. The chelation process includes the formation of two or more separate bond coordinates between polydentate ligand and single central atom.¹³ The product formed is illustrated in the reactions as seen in Figure 6.

In addition, the combination of calcium hydroxide, propolis, and propylene glycol will form a bond. Calcium hydroxide with strong base properties reacts with propolis and propylene glycol which have properties as weak acids containing at least two hydroxyl groups. In silico, there is actually a probability of ionic bonding between Ca²⁺ and O⁻ ions. As a result, that calcium hydroxide with strong alkaline properties reacts with propolis and propylene glycol with weak acid properties can cause the material harden since propolis and propylene glycol contain phenolic-OH groups with acidic properties. Hence, they can react with alkaline calcium hydroxide. Consequently, the reaction of hydration and hydroxyl groups plays a role in the hardening process of the material also affecting the mechanical strength. In conclusion, the addition of propylene glycol at the pulp capping material made of calcium hydroxide combined with propolis can generate compressive and flexural strengths more than that without the addition of propylene glycol. The best level of *propylene glycol* triggering the best effect on compressive and flexural strengths is 40%.

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