The effect of low viscous coir fiber resin composite immersion time on composition dissolution

Yongki Hadinata Wijaya, Twi Agnita Cevanti*, Diana Soesilo*, Linda Rochyani**

**Conservative Dentistry Departement, Faculty of Dentistry, Universitas Hang Tuah, Surabaya*

Correspondence: twi.agnita@hangtuah.ac.id

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ABSTRACT

Background: *E glass fiber dental is a fiber that is often used in dentistry. The availability of E glass fiber in Indonesia is still very limited. Various types of non-dental glass fiber materials are found in the market as engineering materials with relatively cheap prices so that they are expected to be used as an alternative to E glass fiber dental, which one of is coconut fiber. The composition of non-dental glass fiber is almost the same as that of dental E glass fiber. The composition affects the mechanical properties and chemical properties of the fiber. Glass fiber compositions such as* Na_2O *and* K_2O *will increase water resistance.*

Method: *This study was conducted using a post test only control group design. The material used in this study was a composite of coconut coir fiber. The subjects of the coco fiber composite were divided into 4 groups for solubility test based on ISO 4049. The results obtained were analyzed using one-way ANOVA (p = 0.000). The results showed the lowest mean component solubility (%) was in the coconut fiber composite group A (0.476±0.03) and the highest yield was in the coco coir fiber composite D (0.6±0.01).*

Results: *The results of the one-way ANOVA test showed a significant difference in the coco fiber composite in the solubility of the components* ($p<0.05$)*.*

Conclusion: *The low composition of* Na_2O *and* K_2O *as well as CaO and MgO could reduce the solubility properties of the components of the coconut fiber composite.*

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INTRODUCTION

Microleakage can be caused by various factors that result in gaps between the tooth structure and the restorative material. Internal contractions that occur during polymerization can damage the marginal seal of the composite resin that has bonded to the cavity wall, resulting in the formation of an interfacial gap.[1] During the polymerization process, the resin material will experience shrinkage. Therefore, resin materials require a matrix and filler material to reduce the shrinkage process. Clinically, these materials are bonded to the cavity walls in such a way that their deformation is limited under stress. Several techniques have been used to minimize the problem of micro-gaps, one of which is the use of an elastic layer between the dentin and the composite. This elastic layer is known as a base, liner or intermediate restorative material.[2]

One of the materials that is often used as an intermediate restorative material is flowable composite resin. Flowable composite resin is composite resin that have low viscosity and high flowability so that it can adapt better microstructurally and macrostructurally to the bottom and walls of the cavity. With high flowability, apart from being able to better coat every part of the cavity, it can also act as a stress breaker in the restoration interfacial. The main advantage of all low-viscosity composites is that they can act as a stress-absorbing layer, i.e., absorb the stresses of the polymerization contraction of the composite resin.

The use of fiber as a filler in composite resins has several functions including reducing polymerization contraction, continuing the load received on the matrix, providing thermal stability and chemical changes, increasing the strength, stiffness and resistance of the material to fracture.[3]

The environmental conditions of the oral cavity can affect the properties of dental restorative materials. Especially the water content present in the saliva, where the water absorbed by the polymer matrix can lead to the release of bonds between the matrix-filler or even hydrolytic degradation of the filler. Water can be trapped during polymerization in microgels between polymer chains. In addition, water can stick around the cross-linked chain or another possibility is trapped in the micropores. Water ingress is affected by the density of the polymer chains and the potential for hydrogen bonding and polar interactions. The degree of degradation depends on a complex function of the physical and chemical characteristics of the resin, filler, and coupling agent.[4]

The components of soluble resin-based materials are monomers, co-monomers, fillers, activators, polymerization inhibitors, and various other degradation products such as formaldehyde and methacrylic acid. Water solubility is related to the absorption of water itself, for example because the material has a component that has a high solubility so that it dissolves at a fast rate and leaves a gap that makes it easier for liquids from the surrounding environment to enter it. The absorption and solubility of liquids into resins has both advantages and deleterious consequences. Dimensional changes caused by water absorption can compensate for premature polymerization contractions. Hygroscopic expansion can reduce shrinkage due to polymerization. However, many disadvantages are given due to the absorption of this water. For example, stretching between matrix chains can weaken the bonds between monomers, and incoming water can trap unreacted monomers and dissolve them into a liquid environment.[5]

The oral environment where Fiber Reinforced Composite (FRC) is applied contains water that comes from saliva and other fluids. When the composite resin is exposed to water, an adverse effect occurs. Water can destroy the polymer-fiber matrix bonds and can affect cytotoxicity because it will cause the components that make up FRC to be released and dissolve. rtengren stated that the concentration of released composite constituents in water will increase in proportion to the immersion time. The solubility of composite resins used in dentistry is of great clinical importance. Incomplete polymerization and non-conversion of monomers can result in resin loss and may affect the biological compatibility of the materials. Water absorption and solubility can also trigger chemical and physical processes resulting in a destructive effect on the composite resin structure. Hydrolytic degradation will destroy the chemical bonds of the resin or softening will occur through the action of water plasticity. This destruction can have an impact on the clinical appearance and mechanical properties. Water has an important role in the process of releasing substances, so this study aims to assess the water absorption and solubility of restorative materials, as well as identify components that are eluted during storage in water.[6,18,19]

Based on this thought, the researcher wanted to conduct research on the solubility ability of synthetic fiber flowable composite resins with a fiber matrix volume fraction of 50:50 % by volume weight. On the measurement of hygroscopic expansion can be started 15 minutes after polymerization occurs, generally the resin takes 7 days to reach equilibrium and about 4 days to show the greatest expansion.[7,8,19]

RESEARCH METHOD

Coconut Coir Fiber Extraction Procedure

Preparing 2000 grams of fresh coconut leaf fiber. Coconut coir fibers were washed and then dried indoors at room temperature (32-35°C), not exposed to sunlight for approximately five days until completely dry. Dried coconut coir fiber was then mashed with a blender until it becomes a fine powder / simplicia. After that, the coconut coir fiber extract was made by maceration method using 96% ethanol as solvent. Coconut coir fiber simplicia was soaked in 96% ethanol and left for 1x24 hours. The maceration process was carried out for 3 days and every 1x24 hours the filtrate was taken using a Buchner funnel and a vacuum pump. The filtrate that had been collected then concentrated using a rotary evaporator to obtain the final extract.

Figure 1. Coconut coir fiber

Manufacture of Flowable Coconut Coir Composite with 50% Volume Filler

The methacrylate-based resin mixture was manipulated consisting of: an organic matrix containing 49.5% by weight BisGMA (0.5 gram) and 49.5% by weight of TEGDMA comonomer (0.02 ml) in a photoinitiator mode system with 0.02 grams of DGEBA as a silane, 1 mol% (0.5wt%/0.09gram) camphorquinone, 2 mol% dimethylaminoethyl methacrylate (0.5 wt%) (DMAEMA/EDMAB), and 0.1% mole of butylated hydroxytoluene (BHT) were added as initiators and co-initiators. Semi-solid coco fiber was weighed with a digital balance of 0.003 grams (50%) then mixed with a resin mixture.

Coconut coir fiber was placed on a surface that has been treated with a silane coupling agent before being inserted into the resin. After that, it was manually inserted into the resin and weighed with an analytical balance. All components were weighted on an analytical balance and mixed in a high speed mixer. Various fiber mass fractions were added to the vial for mixing with the matrix and the total mass fraction of the filler was 50% by weight.

The matrix and filler were mixed with a magnetic stirrer for about 24 hours. The initiator and co-initiator (CQ and EDMAB/DMAEMA) were added to the monomer solution after covering the entire bottle with aluminum foil to prevent hardening/curing due to ambient light. After thorough mixing with the initiator and solution, the solution with the expected viscosity could be added carefully to the mold according to the test mold. Then, the sample was photo-cured for 40 seconds. Samples were stored in sterile distilled water at 37°C for 24 hours

Manufacturing of coconut fiber flowable composite samples:

After the composite resin was ready, proceed with making molds according to the solubility test requirements. Coir fiber flowable composite resin was inserted into the mold according to the test to be carried out using a syringe every 2mm and then irradiated with visible light cured. The direction of irradiation was perpendicular to the surface of the sample and the irradiation distance of 1 sheet of celluloid tape was above the material to avoid the oxygen-inhibited superficial layer. After setting the mold, the sample was removed from the mould.

Solubility test procedure

The treatment of the samples was in accordance with the International Organization for Standardization (ISO) 4049 to calculate the water absorption and solubility of composite resin materials. After making the sample, the sample was stored in a desiccator containing silica gel and put into an incubator with a temperature of ± 37^{o}C for 24 hours. After 24 hours, the samples were removed and stored in another desiccator containing silica gel and put in an incubator at a temperature of ± 23 ^{o}C for 1 hour. After 1 hour, the sample was removed from the desiccator and weighed until a constant mass (3 times weighing) was obtained, namely m1 (which is the average weight). The sample was then immersed in distilled water and put into an incubator with a temperature of ± 37^{o}C for 7 days, then the sample was removed and then cleaned and dried by aerating the sample in the air for 15 seconds. After 1 minute after being removed from the distilled water, weighing was carried out 3 times and the average weight was marked as m^2.

After being weighed, the samples were stored back in a desiccator containing silica gel and put into an incubator at a temperature of ± 37^{o}C for 24 hours. After 24 hours, the samples were removed and stored in another desiccator containing silica gel and put in an incubator at a temperature of ± 23^{o}C for 1 hour. After 1 hour, the sample was removed from the desiccator and weighed 3 times until a constant weight was obtained, which was m3.

The water absorption (Wsp) and solubility (Wso) values in micrograms per cubic millimeter were calculated using the Oysaed and Ruyter formulas, namely Wsp = (m2–m3)/V and Wso = (m1-m3)/V (Wsp: water absorption, Wso: solubility, m1: sample weight after conditioning before immersion (μg), m2: sample weight after soaking in distilled water for 7 days (μg), m3: sample weight after being put into a desiccator a second time (μg), and V: sample volume (mm^3).The data were then analyzed using t test for water absorption value, and Mann-Whitney test for solubility.

The solubility test of the components was carried out by looking at the changes in the sample weight before immersion with the sample weight after immersion and through the conditioning process in a desiccator. The solubility of the components was measured using a formula based on the formula ISO FDIS 4049, 1999 in Wei et al, 2013.

$$M_d(\%) = m_3(t) – m_1 / m_1 \times 100$$

Information:

$Md(\%)$: weight change (%)

m1 : sample weight before immersion in water (μg)

m3 : sample weight after conditioning (μg)

Furthermore, the sample soaked water was tested using X-Ray Fluorescence (XRF) to provide clarification on the soluble component

RESULTS

The average solubility of the components generally showed an increase in all groups with variable fiber composition. The average value of the solubility of the components in the coconut fiber composite can be seen in Table 1.

Normality test using Saphiro Wilk showed a statistical value with p> 0.05. This indicates that the data obtained are normally distributed. The homogeneity test of the data was carried out by Levene's test. The results of the Levene’s test showed p=0.202, the result can be concluded that the data is homogeneous.

One-way ANOVA test was used to determine the effect of composition on component solubility. The results of the ANOVA test can be seen in Table 2 which shows the significance value (p<0.05). Based on the results of statistical tests, it could be concluded that the composition of coco fiber affects the solubility of the components.

Table 1 The mean and standard deviation (s.d) of the solubility of the coconut fiber composite components

Group	Component solubility (%) Mean ± s.d
Coconut coir fiber composite group A	0.476±0.03
Coconut coir fiber composite group B	0.595±0.01
Coconut coir fiber composite group C	0.488±0.03
Coconut coir fiber composite group D	0.600±0.01

Table 2. Summary of statistical results of ANOVA 1 path of solubility of coconut fiber composite components with variable composition and fiber volume

Group	Component solubility F	p
Composition	45,90	0,000

The results of Tukey's post hoc analysis to determine the average difference between each group could be seen in Table 2. To confirm the soluble fiber component, XRF was used. The results of the examination with XFR could be seen in Table 3. The average solubility of the components generally showed an increase in all groups, both the control group (A) and the treatment group (B, C, D). Based on the results of statistical tests, it was found that the composition of coconut fiber affects the solubility of the components. The results of Tukey's post hoc analysis found no significant difference in all groups, both control and treatment groups. This indicates that the chemical bonding that occurs in coir fiber composites is very good.

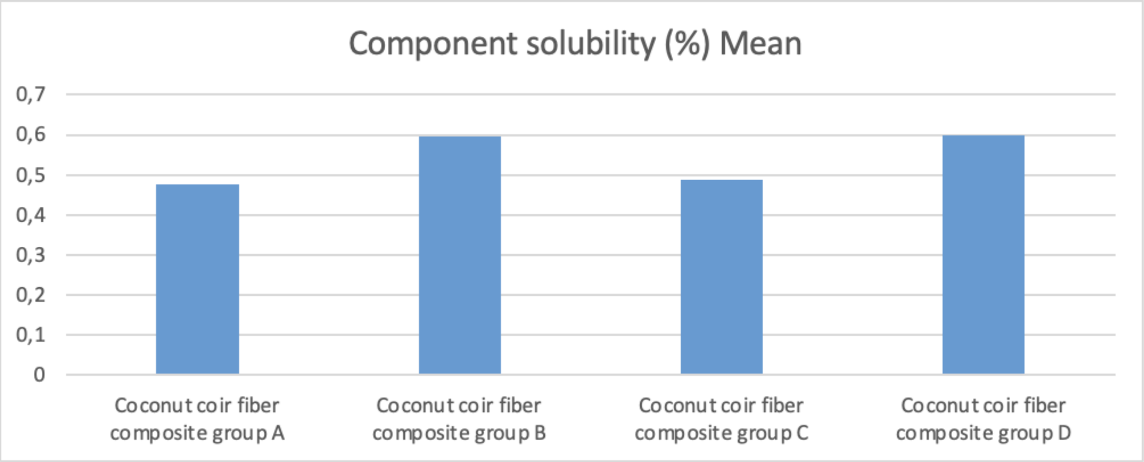

Figure 2. Mean of the solubility of the coconut fiber composite components

Note :
Group A = initial composite weight (gram) of solubility test group before soaking
Group B = composite weight (gram) of solubility test group at immersion for 24 hours
Group C = composite weight (gram) of solubility test group at immersion for 4 days
Group D = composite weight (gram) of solubility test group at immersion for 7 days

Table 3. Summary of Tukey test variable composition of coconut fiber composites

	Coconut coir fiber composite group A	Coconut coir fiber composite group B	Coconut coir fiber composite group C	Coconut coir fiber composite group D
Coconut coir fiber composite group A		-0,057*	0,01	-0,066*
Coconut coir fiber composite group B			0,068*	-0,008
Coconut coir fiber composite group C				-0,076*
Coconut coir fiber composite group D				

DISCUSSION

Chemical properties are one that must be controlled in the manufacture of FRCs by maintaining the stability of the bond strength between components so as to maintain biological stability in the oral cavity. In order for the reinforcing material to bear the load, the reinforcement must have a higher modulus of elasticity. The bond between the matrix and the reinforcing material is critical with respect to the load being passed from the matrix to the fibers. The polyester matrix (BisGMA) has the following properties: density 1.23g/cm^3, Young's Modulus 3.2 GPa, and tensile strength 65 MPa. While coconut fiber has a density of 100-450g/cm^3, Young's Modulus of 4-6 GPa, and tensile strength of 1.15 Mpa. Coconut fiber has a higher young modulus than polyester matrix (BisGMA), so the combination of coconut fiber with BisGMA can meet the requirements of an effective composite.[9,10] The resin matrix and coconut fiber are organic components. Both materials can bond mechanically because they are fellow organic materials. Mechanical bonding or interlocking occurs between the fiber surface and the matrix which has irregular or uneven morphology. The irregularity or unevenness of the fiber surface will produce a fiber-matrix adhesion ability known as lock and key, which is an interlocking bonding mechanism that occurs on two surfaces, namely resin and rough fibers.[11,12]

A natural fiber composite will have good physical properties or strength if it contains little lignin, because lignin is rigid and brittle.[13,14] The chemical composition of coconut fiber consists of cellulose, lignin, pyroligneousacid, gas, charcoal, tar, tannin, and potassium. Lignocellulose is the name used for materials containing lignin, cellulose, and hemicellulose. Hemicellulose and cellulose are polysaccharides that can be broken down into monosaccharides which can then be used as the main ingredient for making biopolymer materials.[15] The chemical structure of cellulose consists of the elements C, O, H which form the molecular formula $(C_6H_{10}O_5)n$ and n is the degree of polymerization, and can be a number of thousands with very tight hydrogen bonds. The degree of polymerization will affect the chain length of a cellulose series. The physical and mechanical properties of a fiber are highly dependent on the degree of polymerization.[15]

The oral environment where fiber composites are applied has water content derived from saliva and other fluids. When the composite resin is exposed to water, adverse effects occur. Water can destroy the fiber-matrix polymer bond and can affect the cytotoxicity properties because it will cause the constituent components of the composite to detach and dissolve. Expansion due to water absorption from oral fluid can relieve polymerization stress, but this property is a slow process, compared to polymerization contraction and stress formation. In hygroscopic expansion measurements that begin 15 minutes after polymerization, it generally takes 7 days for the resin to reach equilibrium and about 4 days to show the greatest expansion.[8,16,17,19]

CONCLUSION

In this study, the results of the coconut fiber composite had almost the same solubility properties in general, with almost the same fiber composition and concentration. This situation could lead to the conclusion that coconut fiber could be an alternative to dental E glass fiber.

CONFLICT OF INTEREST

There is no conflict of interest among authors.

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