

Effects of filler volume of nanosisal in compressive strength of composite resin

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ABSTRACT

Background: One of the composite resin composition is inorganic filler. The production of inorganic filler materials was highly dependent on non-degradable, and nonrenewable fossil fuels. Therefore, natural fibers can be used as substitute for inorganic fillers. One that can be developed is sisal. **Purpose:** This study aimed to determine the effects of nanosisal filler volume on compressive strength of composite resin. **Methods:** In this study, composite resins with nano-sized sisal as filler were manufactured and labeled as nanosisal composites. This research processed sisal fibers into nano size and mixed them with Bis-GMA, UDMA, TEGDMA, Champhorquinone (Sigma Aldrich). Nanofiller composite (Z350 XT, 3M, ESPE) was utilized as a control. The 20 samples utilized were divided into 4 groups (each group containing five samples): Group A contained nanosisal composite of 60% filler volume, group B, nanosisal composite of 65% filler volume, group C, nanosisal composite of 70% filler volume and group D, nanofiller composite (Z350 XT, 3M, ESPE). Samples were 2 mm in diameter and 6 mm in height. The sample was tested for compressive strength using a universal testing machine (UTM). Data was analyzed by means of a Kruskal Wallis procedure. **Results:** The mean of the compressive strength of the nanosisal composite 60% was 16.80 MPa; the nanosisal composite 65% was 10.80 MPa, the nanosisal composite 70% was 7.20 MPa and the nanofiller composite was 7.40 MPa. There was a significant difference in data analysis ($p = 0.033$; $p < 0.05$). **Conclusion:** In this study, the filler volume of nanosisal influenced the compressive strength of a composite resin and the nanosisal filler volume was recommended at 60%.

Keywords: nanosisal; composite resin; compressive strength; nanofiller

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INTRODUCTION

Composite resin is one of the most commonly used dental fill materials since it has high aesthetic attractiveness compared to other dental fill materials.¹ The mechanical properties of composite resin are known to be influenced by filler volume. Therefore, the higher the volume of filler, the greater the hardness, stiffness, strength, and resistance against fracture.² Based on the amount of fill material used,

composite resin can be classified into traditional composite resin, micro filter composite resin, hybrid composite resin, and nanofil composite resin. Nanofil composite resin possesses high aesthetic value since it is easy to polish and produces a shiny dental filling.^{3,4}

Composite resin consists of matrix, inorganic filler and a coupling agent. The matrix contained in composite resins plays a role in forming its physiology. Inorganic filler is a reinforcing material dispersed within the matrix.

A coupling agent plays a role in combining the matrix and inorganic filler. In addition to these three components, there are others present, namely: activator, pigment, initiator and ultraviolet absorbent.³ The greater the filler volume used in the composite resin, the greater the mechanical strength.⁵ The volume of filler in the composite resin material is usually between 60–70%.⁶

Filler used in composite resin is derived from inorganic materials such as glass, quartz, and silica since these materials are strong, hard and stable.⁷ Nevertheless, inorganic materials, still demonstrate several weaknesses. First, inorganic materials are produced through energy process heavily dependent on fossil fuels. As a result, the emulsion of pollutants produced by the production of inorganic materials is so high that it is prejudicial to the environment and, by extension, health. Second, inorganic materials are also non-degradable, non-renewable and non-recyclable.^{8,9}

Therefore, natural fibers are expected to become substitutes for inorganic materials due to their high compressive strength, low weight, and environmental-friendly relation.¹⁰ One such natural fiber that can be developed is nanosisal since its hard fibers are produced from sisal plants (*Agave sisalana*) which are easily cultivated. However, the use of sisal fibers is still limited to marine and agricultural fields, usually being used as rope, yarn, carpets and handicrafts.¹¹

The research reported here focused on the use of nanosisal fibers in manufacturing composite resin by mixing the resin matrix with the nanosisal filler without the use of a coupling agent due to their being organic materials. Thus, both can bind without coupling agent. The bond between the nano-sisal and the resin matrix can be considered to be a chemical bond of OH group.¹² This study aimed to analyze the effects of the volume of nanosisal fibers used as filler in composite resin on its compressive strength.

MATERIALS AND METHODS

Sisal fibers used in this research were obtained from the Indonesian Crops and Fiber Research Institute (Balittas), Malang, Indonesia (Figure 1). They were cut into pieces weighing up to three grams. The fibers were then scoured (alkalized) by soaking in NaOH solution at 100°C for two hours while being agitated with a magnetic stirrer. This treatment was repeated three times. The fibers were then filtered and washed in aquadest, before being bleached using a mixture solution of NaOH, H₂O₂ and aquadest. The bleaching process was carried out at 80°C for two hours while agitated with a magnetic stirrer. The bleaching process then was repeated four times. After each stage of the bleaching process, the fibers were filtered and washed in aquadest.

After the bleaching process, the sisal fibers were processed by means of an ultrasonic machine (Cole-Parmer Ultrasonic Processor, Model CP 505). The sisal fibers



Figure 1. Sisal fibers

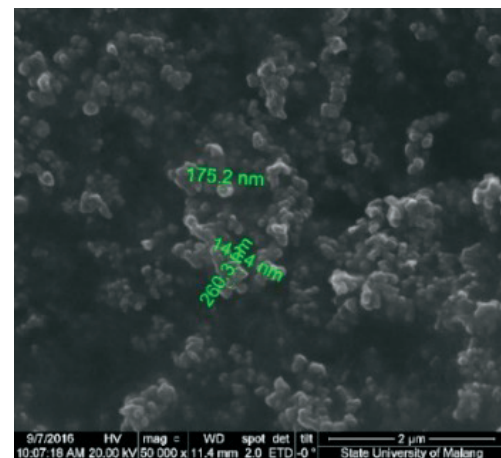


Figure 2. SEM analysis of the nanosisal fiber (the marked image) with a magnification of 50,000.

obtained were filtered with fritted glass filter No 1 to remove residual aggregate and then dried in a freeze drier (Flex-Dry™ μP Microprocessor Control, FTS Systems, Inc., USA) in order to obtain solid nanosisal fibers (Figure 2). The solid sisal fibers were subsequently observed by means of a scanning electron microscopy (SEM) to determine its size. SEM analysis showed that the size of the sisal fibers was 143.4–260.3 nm. (Figure 2). Solid nanosisal fibers was obtained.

At the next stage, the solid nanosisal fibers were weighed by a digital balance and separated into samples weighing 0.003 grams (60% filler volume), 0.005 grams (65% filler volume), and 0.007 grams (70% filler volume). Each sample was mixed with 0.5 grams of Bis-GMA (bisphenol A glycerolate dimethacrylate, Sigma Aldrich), 0.02 ml of TEGDMA (triethylene glycol dimethacrylate, Sigma Aldrich), 0.02 grams of UDMA (diurethane dimethacrylate, Sigma Aldrich) and 0.09 grams of champorquinone (Sigma Aldrich) to obtain a nanosisal composite dough. This was then placed into a cylindrical-shaped mold 6 mm in height and 2 mm in diameter in accordance with ISO 4049 guidelines.¹³ The materials were mixed on a glass plate using a stainless steel spatula. Thereafter, the samples were

radiated with a visible light cure (LED curing unit, LY-B200, S&D Dental International Co., Ltd, Shanghai, China) for 40 seconds until they set. The nanosisal composite resin samples were then divided into three groups of varying concentrations, namely: A (60%), B (65%), and C (70%).

On the other hand, the nanofiller composite (Z350 XT, 3M ESPE) which was to act as the control (group D) was extracted from the tube with plastic instruments, inserted into a mould, and radiated with a visible light cure for 40 seconds until setting. The four groups were then marked at the center of the samples to be tested for their compressive strength by using a universal testing machine (Mettler Toledo AL 204). Each mould was placed in the centre of the UTM with the 1000 N load being applied on top of the material with the vertical printout position at 1 mm/min. The data obtained was in the form of N unit which was then converted into MPa using the following formula:¹⁴

$$R_c = \frac{F}{A}$$

Note:

Rc : Compressive strength (MPa)

F : Maximum force (N)

A : width of sample base area (1r2) (mm²)

The score of the compressive strength obtained then was analyzed by non-parametric tests, namely; Kruskal Wallis and Mann-Whitney tests.

RESULTS

The results showed that the average value of compressive strength in the nanosisal composite resins group A was 16.80 Mpa, in group B 10.80 Mpa, in group C 7.20 Mpa and in group D 7.40 Mpa. The normality of the resulting data was then tested by means of a Saphiro Wilk test, the results of which demonstrated the distribution of the data to be normal. The homogeneity of the data was then tested using a Levene test whose results indicated that the variance of the data was not homogeneous. As a result, a Kruskal Wallis procedure was carried out.

Table 1 show that the largest mean value of compressive strength was found in the group A nanosisal composite resins. Therefore, the optimum nanosisal filler volume recommended by this research was one of 60%. Furthermore, Table 2 demonstrates a p value of < 0.033 (p < 0.05). This confirms that there was a filler volume effect on the compressive strength of the nanosisal composite resins.

In addition, Table 3 shows the nanosisal composite resins group A have significant differences in compressive strength compared of the nanosisal composite resins C and D groups with p value (p < 0.05). However, there were no significant differences between the nano-sisal composite resins when comparing group A with group B, group B with group C and group D and group C with group D.

Table 1. The mean value of compressive strength of the nanosisal composite resins

	Group	n	Mean (MPa)
Compressive strength	A	5	16.80
	B	5	10.80
	C	5	7.20
	D	5	7.40
Total		20	

Group A: nanosisal composite 60% filler

Group B: nanosisal composite 65% filler

Group C: nanosisal composite 70% filler

Group D (control): nanofiller composite Z350 XT

Table 2. Results of the Kruskal Wallis Test

Compressive strength test	
Chi-Square	8.707
Df	3
Asymp. Sig.	.033

Table 3 Results of the *Mann-Whitney* Test

Variable	Variable			
	A	B	C	D
A	–	0.076	0.009	0.028
B	–	–	0.295	0.347
C	–	–	–	0.917
D	–	–	–	–

Group A: nanosisal composite 60% filler

Group B: nanosisal composite 65% filler

Group C: nanosisal composite 70% filler

Group D (control): nanofiller composite Z350 XT

DISCUSSION

The nanosisal composite resin in group A had greater compressive strength than the nanofiller composite group D. This is because the nanosisal composite resin forms a stronger bond between the nanosisal fiber and resin matrix due to a chemical bond of the OH group.¹² Based on the observation results of the chemical structures of the resin matrix and nanosisal fibers, the H atoms present in the resin matrix bind to the O atoms present in nanosisal fiber. As a result, a new OH group is formed. Moreover, mechanical bonds are also formed between the resin matrix and nanosisal fiber due to the roughness of the surface of the sisal fibers. The initial process of nanosisal composite manufacture greatly influences the mechanical properties of the composite resins.¹⁵

In this research, scouring including alkalization treatment was conducted. The sisal fibers themselves contain a group of hydroxyl groups that can form hydrogen bonds which can affect the dimensional stability of natural

fibers. This will then lead to a weak bond between the resin and the matrix.¹⁵ According to previous research, alkalization can break the hydrogen bonds in the tissue structure, thereby increasing the surface roughness of the sisal fibers. This, in turn, results in the stronger bonds of the resin matrix and the sisal fiber. In other words, alkalization treatment has been shown to increase the mechanical strength of natural fibers.¹⁶

The imbalance of the coupling agents on the Z350 XT composite resins may also lower the compressive strength of the composite resins. The coupling agent used in Z350 XT composite resins is silane which is an adhesion promoter containing two different reactive functions that can react and combine with variations of organic and inorganic materials. Silane is used to strengthen the bonds of different materials. The hydrolysable functional group will react with the hydroxyl group on the surface of the inorganic substrate to form a siloxane (Si-O-Si) bond. Organic groups that cannot be hydrolyzed together with a double bond C = C can polymerize with a double-bonded composite resin monomer.¹⁷

A balance between the number of inorganic substrate hydroxyl groups and the hydrolysable functional groups in the silane must exist. If the amount of silane contained in the Z350 XT composite resin is imbalanced, the compressive strength distribution from the matrix to the filler will not proceed properly and composite resin will then be easily broken.¹⁸ Nanosisal composite resins used in this research were prepared without the use of coupling agents because the resin matrixes and nanosisal fibers are organic materials. Thus, both can bond chemically without the presence of a coupling agent.¹² In the pre-elementary study, resin matrix, silane (coupling agent), photoionization, and nano-sisal fibers were mixed. Unfortunately, the composite resins did not harden after being radiated with a visible light cure for 40 seconds.

Based on the data obtained, the nanosisal composite resins at group A demonstrated the highest compressive strength. Previous research comparing hybrid composite and filler composite consisting of areca fibers at concentrations of 50% and 60% showed that the filler composite at a concentration of 60% had a lower water absorption capacity than the filler composite at a concentration of 50%.¹⁹ This may be due to the higher compatibility between hydrophilic fibers and composite matrixes at a concentration of 60%.²⁰

In contrast, this research indicates that the higher the volume of the nanosisal volume used in composite resins, the smaller the compressive strength. The results of this research were supported by that of a previous investigation examining palm fibers as composite fillers at volumes varying from 30% to 70%. This showed that the compressive strength of the palm fibers increased as the filler volume expanded to 60% and decreased when the filler volume was 70%.²¹ This could be explained by the compressive strength decreasing as filler volume increases since, at the higher volume, many fiber ends will experience

fibrous discohension within the resin matrix leading to initial cracking on the composite resins.²² Increasing the concentration of sisal/palm hybrid fibers resulted in a reduction of tensile strength and tear strength. Palm fibers can, therefore, be seen to have similar characteristics as sisal fibers.²³

Table 3 showed no significant difference in the compressive strength of nanosisal composite of groups A and B since the structure of nanosisal composite resins began to crack if the filler volume was slightly higher than 60%. This was the finding of a previous study on palm fibers.²¹ Similarly, there was also no significant difference in the compressive strength of nano-sisal composite resins at the volumes of B and C groups since the probability of the number of cracks occurring did not vary much.²¹ These results may be caused by the fibrous discohension in the number of fiber ends of both nanosisal composites not differing greatly.²² Finally, it can be concluded that nanosisal filler volume has an effect on the compressive strength of the nanosisal composite resins. The recommended filler volume of the nanosisal composite resin is 60%.

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