R.I.S. Asri[1], B. Sunendar[2], I. Dwiandhono[1], A. Harmaji[3]

Properties of Metal Oxide and Pineapple Fiber Reinforced Dental Composite Resin

[1]Faculty of Dentistry, Universitas Jenderal Soedirman, Purwokerto, Indonesia, rahmaphrodite@gmail.com
[2]Department of Engineering Physics, Institut Teknologi Bandung, Bandung, Indonesia
[3]Advanced Materials Processing Laboratory, Institut Teknologi Bandung, Bandung, Indonesia

Indonesia is among the world's top pineapple-producing nations, making pineapple leaf fiber waste widely available there. This study aimed to utilize the pineapple leaf fiber (Ananas comosus (L.) Merr) to increase the mechanical properties of metal oxide-based direct dental restoration applications material. The sample consisted of four composite groups with the addition of 0-5% fiber. All composite samples were then tested for hardness, flexural strength, and Scanning Electron Microscope (SEM). The results of the composite hardness test without the addition of fiber were 30.31 VHN. With the addition of 1%, 2.5%, and 5%, the composite has a hardness value of 31.13 VHN, 34.02 VHN, and 27.22 VHN, respectively. The results of the three-point bending test showed that the flexural strength of the sample without the addition of fiber was 1.6 MPa, while the addition of 1%, 2.5%, and 5% fiber resulted in the flexural strength of 2.1 MPa, 2.3 MPa, and 1.8 MPa, respectively. The SEM results show a homogeneous particle dispersion morphology, with various agglomerations and gaps. Composites with the addition of 1% and 2.5% fiber have a narrower gap than without the addition of fiber. This explains the increase in the hardness and flexural strength of the composite.

Keywords: pineapple leaf fiber, dental composite resin, hardness, flexural strength, SEM.

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Introduction

The level of oral health can be used as a benchmark or indicator of a person's health. At present, there are still many oral health problems suffered by the societies. According to the Data and Information Center of the Indonesian Ministry of Health, the Indonesian population had dental and oral problems by 23% in 2007 and increased to 26% in 2013. One of the biggest problems of oral health to date is regarding dental caries. Caries is a damage to the teeth due to destructive factors, namely bacteria and the presence of carbohydrate fermentation from daily food which results in demineralization of the hard tissues of the teeth, followed by damage to the organic tissue, causing cavities in the teeth [1]. The prevalence of caries in Indonesia is quite high.

According to Basic Health Research Results Report (Riskesdas) data in 2013, Indonesia's Dental Caries Index (DMF-T) index was 4.6, which means that the Indonesian population's tooth decay was 460 teeth per 100 people. The action to repair the damaged teeth is restoration. Composites are the most popular material for direct restoration, but until now Indonesia has not been able to process composite raw materials efficiently, so they must be imported from other countries. This causes the composite price to be relatively expensive. Science and technology that continues to develop rapidly can used to create new materials with natural resources owned by Indonesia for dental applications. These natural resources include alumina (Al_2O_3) and zirconia (ZrO_2), which categorized as ceramic material. Based on the description above, this study was conducted to synthesize alumina-zirconia-carbonate apatite filler with the addition of pineapple leaf fiber by 1%, 2.5%, and 5%. The mechanical properties in the form of hardness and flexural strength and morphological characteristics were conducted. The

hardness of composite was tested using the Vickers microhardness while the flexural strength was obtained using the three point bending test. Morphological characteristics were seen by testing the morphology of the scanning electron microscope (SEM). This research was expected to produce fiber-reinforced composites with better mechanical properties than without addition of fiber. Dental restoration is a dental procedure that aims to restore the shape, function and aesthetics of the teeth [2]. Based on how it works, restoration can be done by direct restoration and non-direct restoration. Direct restorations are repairment of teeth that are placed directly into the prepared cavity whereas non-direct restorations are restorations of tooth structure that carried out in the laboratory [3]. Direct restoration is the action that is most often done because it is considered practical and can be done in one visit. Commonly used direct restorative materials include composite resins, glass ionomer cements, and amalgams. Today, direct restoration using composite resin is the treatment of choice [4]. Composite resin is a tooth-colored restorative material that is usually applied to anterior and posterior teeth, which is composed of a resin matrix and filler particles mixed with a coupling agent as an adhesive for filler particles into the matrix [5].

Composite resins based on filler particle size are classified into macrofilled, microfilled, hybrid, and microhybrid composites. Along with the development of nanotechnology, currently composites with nano-sized filler materials have been created. The development of nanotechnology is related to the physical, chemical, and biological properties of the structure and components of materials at the nanometer scale (10^{-9} m) [6].

The addition of nanoparticles aims to improve the material in various properties such as mechanical strength, thermal change, water absorption, optical properties, aesthetics, wear resistance, and others. The use of dental polymer-based nanocomposites is often used in the field of modern dentistry [7]. Alumina is an opaque and heat-resistant material with good biocompatibility, is bioinert and has excellent hardness, strength, and wear resistance [8]. The addition of alumina as a filler in composites can improve mechanical properties [9]. In addition, stable composite materials can be obtained from mixing alumina and zirconia. During the last few decades, zirconia has been developed as a non-metallic dental material that has an effect on increasing biocompatibility, aesthetics, and mechanical strength [10]. The addition of carbonate apatite as a filler can also increase the hardness of the composite. This material contains three main components, namely calcium, phosphate, and carbonate. Calcium phosphate compounds are inorganic materials that are widely used in medical applications such as bone implants, remineralization and repair of early caries because they are bioactive and biocompatible. The combination of alumina, zirconia, and carbonate apatite is expected to produce fillers with hardness that meet the standards as direct restoration materials. The bond between the matrix and the filler in Composite can be increased by addition of coupling agent.

One of the coupling agent is chitosan, which with its mechanical, thermal, antibacterial, and biological properties has been successfully applied in various types of composites [11]. Pineapple leaves are part of the pineapple plant that must be dismantled every two to three harvests [12]. The utilization of pineapple leaf waste so far is used as an adsorbent, raw material for paper, textiles, rope, and composite reinforcement. Pineapple leaves must be extracted first to become fiber to be applied in its utilization. Pineapple leaf fiber is a natural fiber that has high strength properties, abundant sources, and relatively inexpensive. The main mechanical properties of pineapple leaf fiber lies in the content of -cellulose. Some of the fiber content of pineapple leaves include -cellulose by 81.27%, hemicellulose by 12.31%, and lignin by 3.46% [13]. Based on these discussions above, study aimed to synthesize fillers in the form of alumina-zirconia-carbonate apatite and pineapple leaf fiber Merr) as matrix reinforcement consisting of UDMA, TEGDMA, and DMAEMA for direct dental restoration applications, characterized by its comprresive and flexural strength, as well as morphological analysis by Scanning Electon Microscope.

I. Methodology

1.1. Extraction of Pineapple Leaf Fiber

Pineapple leaf fiber (Figure 1) was powdered to obtain a weight of 75 grams then transferred into a glass beaker and added a mixture of 1 L of 3.5% HNO_3 solution and 10 mg $NaNO_2$ which was heated on a hotplate stirrer at 90°C for 2 hours then filtered until the pH became neutral. It was mixed with 750 mL of a solution containing 2% NaOH and 10 grams of 2% Na_2SO_3 then heated on a hotplate stirrer at 50°C for 1 hour and then filtered until the pH became neutral.

The filter results were bleached with 500 mL of 1.75% NaOCl solution at 90°C for 30 minutes and filtered again until the pH became neutral. Cellulose was dissolved in 500 mL of 45% H_2SO_4 at 45°C for 45 minutes then filtered until the pH became neutral. Cooling is done by adding 25 mL of aqua dm and left for 1 night until a suspension is formed then homogenized using ultrasonic homogenizer for 15 minutes and then centrifuged at 10000 rpm for 20 minutes until the pH became neutral.

1.2. Synthesis of Alumina and Zirconia

A total of 4.510 grams of $ZrCl_4$ was dissolved into 250 mL of aqua dm and stirred using a magnetic stirrer for 10 minutes. 5.303 grams of $Al(NO_3)_2$ was added to the $ZrCl_4$ solution and stirred using a magnetic stirrer for 10 minutes. The solution is heated using an oven until a crust appears and was smoothed with a mortar and pastle. It was then calcined using a furnace at a temperature of 900°C for 2 hours.

1.3. Synthesis of Carbonate Apatite

A total of 2,362 grams of $Ca(NO_3)_2.4H_2O$ was dissolved in 100 mL of aqua dm and stirred using a magnetic stirrer to form a solution. 0.792 grams of $(NH_4)_2.HPO_4$ was dissolved in 100 mL aqua dm and stirred using a magnetic stirrer to form a solution. 0.504 grams of $NaHCO_3$ was dissolved in 100 mL of aqua dm and stirred using a magnetic stirrer to form solution. The resulting calcium nitrate tetrahydrate $(Ca(NO_3)_2.4H_2O)$, diammonium hydrogen phosphate

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Fig. 1. Pineapple Leaf Fiber (a) original and (b) powdered.

$((NH_4)_2.HPO_4)$, and sodium hydrogen carbonate $(NaHCO_3)$ solution was added with 25% titrated ammonia and until it reached a pH of 9-11. All solutions were stored in sealed container for 24 hour. The precipitate is separated from the solution by centrifugation and then homogenized with an ultrasonic homogenizer. The resulting precipitate is put in the oven for 10 minutes at a temperature of 50-80°C to form a paste. The paste formed was placed in a combustion boat and calcined at a temperature of 700°C for 2 hours. The cooled precipitate were grounded with a mortar and pestle

1.4. Synthesis of Chitosan

A total of 2 mL acetic acid was put in a measuring cup containing 98 mL of aqua dm while stirring using a magnetic stirrer. 2 grams of chitosan powder was dissolved in each measuring cup until homogeneous.

1.5. Synthesis of composite

Fiber with various composition according to Table 1 was mixed into alumina zirconia and carbonate apatite with 1:1 ratio and chitosan in petri dish. The mixture was stirred using magnetic stirrer until homogenous. Matrix with total weight of 0.3g consists of UDMA 17% wt, TEGDMA 6% wt, DMAEMA 6% wt, HEMA 10% wt, and Camphorquinone 1% wt were mixed homogenously.

Table 1.
Variations of Pineapple Leaf Fiber (PLF) in composite

No	Code	Pineapple Leaf Fiber (%)
1	PLF-0	0
2	PLF-1	1
3	PLF-2.5	2.5
4	PLF-5	5

Matrix and Filler were mixed and transferred into a mold with diameter of 6 mm and a height of 3 mm for hardness tests (Figure 2a), and 20 mm long, 4 mm wide, and 2 mm thick for flexural strength tests (Figure 2b). Both were conformed to American Dental Association (ADA) standards. The composite was mixed layer by layer every 1 mm into the mold after which it was leveled using a plastic instrument. The composite was irradiated with

the LCU for 40 seconds every 1-2 mm of the composite layer to fill the mold. It was then released from the mould and tested its hardness, flexural strength, and SEM.

II. Result and discussions

2.1. Hardness Test Result

Hardness tests were carried out on 4 groups of composites with each group consisting of 8 samples. Each sample was tested at 3 points on the sample surface. The load used is 100 grams for 15 seconds. Vickers hardness values are generated in units of Vickers Hardness Number (VHN). The average results of the hardness test values for the synthesized composite samples can be seen in Figure 3.

The results of the Vickers hardness test in Figure 3 shown the average values of different hardness in each group. Groups of PLF-0, PLF-1, PLF-2.5, and PLF-5 each had an average hardness value of 30.31, 31.13, 34.02, and 27.22 VHN. The highest hardness value was found in PLF-2.5 of 34.02 VHN while the lowest value was found in PLF-5 of 27.22 VHN. The difference in hardness values is influenced by the addition of different concentrations of Fiber. The addition of fiber concentration as a composite filler can increase the hardness value given the right amount. The synthesized fiber in this study used the acid hydrolysis method, in which the method was able to remove the amorphous structure of cellulose and leave only the crystalline structure. The crystal structure of fiber will increase the adhesion to the matrix because it facilitates mechanical interlocking and bonding reactions due to the presence of hydroxyl groups on the resin [14]. The increase in adhesion resulted in an increase in the mechanical properties of the composite. The results of the hardness value in this study increased with the addition of 1% and 2.5% fiber compared to the group without the addition of fiber. In addition to the concentration of nanocellulose, the hardness value of the nanocomposite in this study was also influenced by the presence of alumina-zircona. Alumina is used to maintain the tetragonal zirconia phase so that it can increase the hardness, strength, and inhibit the crack propagation of the composite [15]. The value of composite hardness in this study increased when given the addition of 1% and 2.5%
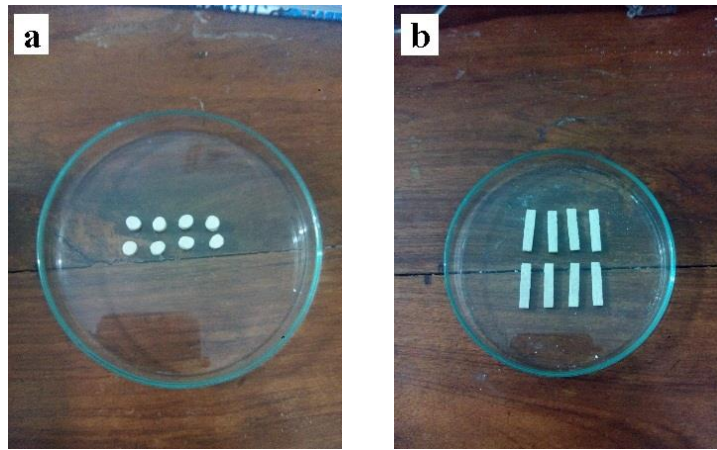
Fig. 2. Test Sample for (a) hardness and (b) flexural measurement

then decreased when given the addition of 5%. The results of this study explain that the addition of fiber as a composite filler has an optimum limit of only up to 2.5%. The exvessive amount of Fiber resulted in the aggregation of nanocrystals into the matrix during the sample hardening process [16]. This resulted in a decrease in the hardness value of the composite because the mixing between the matrix and filler became uneven.
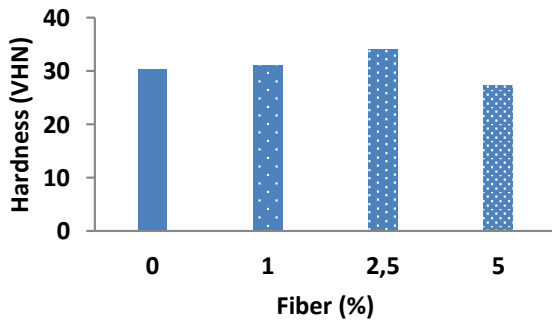

Fig. 3. Hardness Test Result.

2.2. Flexural Test Result

Flexural tests were carried out on 4 groups of composites with each group consisting of 8 samples. The results of the average flexural strength test values for synthesized composite samples can be seen in Figure 4. The results of the flexural strength test in Figure 9 show different values between each group. Groups of PLF-0, PLF-1, PLF-2.5, and PLF-5 each had an average flexural strength value of 1.6, 2.1, 2.3, and 1.8 MPa, respectively. The addition of 1% and 2.5% Fiber to the composite increase the Flexural strength compared to the control group. The increase in the value of flexural strength occurs due to the reinforcing effect of crystalline cellulose and the strong bond between itself and the resin matrix [17]. The value of flexural strength in this study decreased when given the addition of 5% Fiber. The decrease was due to the aggregation of cellulose crystals into the resin, resulting in an uneven distribution between the matrix and filler. Aggregation that occurs will destroy the resin matrix microstructure so that the mechanical properties of composites will decrease. The tensile strength of pineapple leaf fiber is 413-1627 MPa [18], explained the PLF addition increase the flexural strength of composite.
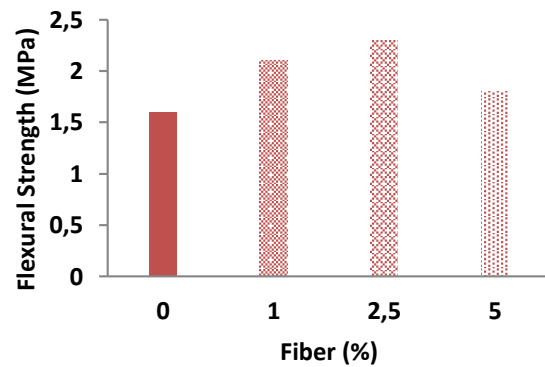

Fig. 4. Flexural Test Result

2.3. Scanning Electron Microscope Test Result

The process to determine the characteristics of SEM requires a sample with good electrical conductivity because it uses electron scans to produce images. The sample under study is a material that has a low electrical conductivity, so the sample needs to be coated with a conductive metallic coating. SEM testing was carried out on 4 samples of composite with the grain size average was presented in Table 2.

Table 2.
Grain size average of resulting composites

No	Code	Grain size (μm)
1	PLF-0	26.6
2	PLF-1	14.2
3	PLF-2.5	7.52
4	PLF-5	13.25

The SEM image of PLF-0 in Figure 5 show that there is a homogeneous picture between the particles in the synthesized composite consisting of a composite resin matrix and filler in the form of alumina-zirconia and carbonate apatite. In addition, there is a gap.

The results of the SEM test for PLF-1 in Figure 6 show a picture of a homogeneous dispersion between the particles in the synthesized composite consisting of a composite resin matrix and alumina-zirconia filler, carbonate apatite, and 1% Fiber.
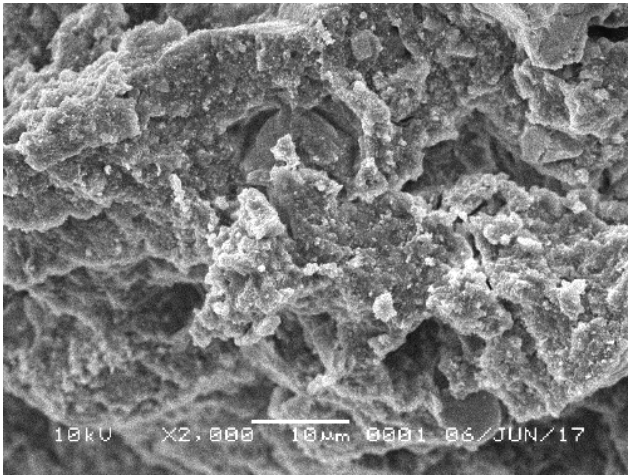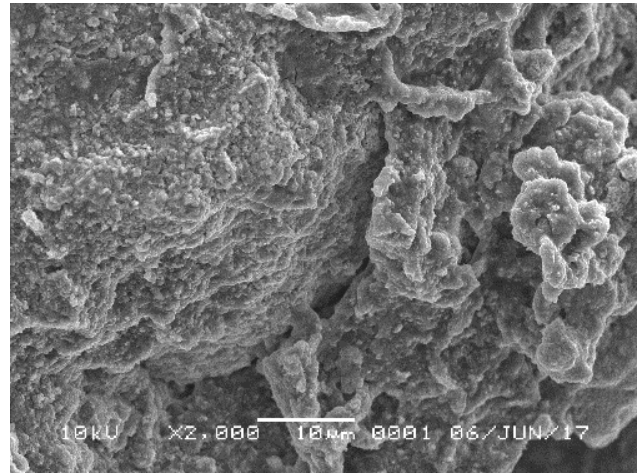
Fig. 5. SEM Images of PLF-0 (Magnification 2000X).
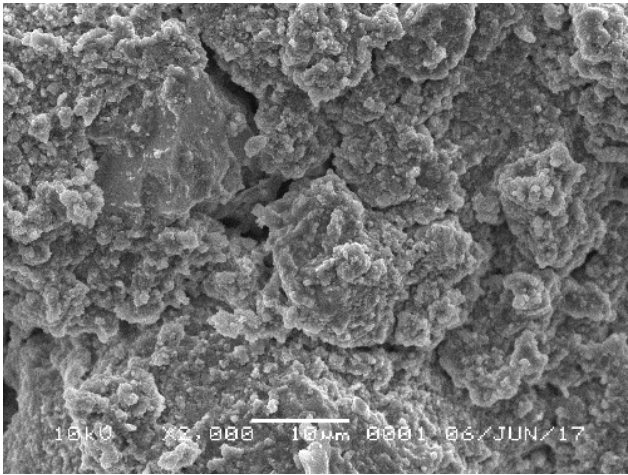

Fig. 6. SEM Images of PLF-1 (Magnification 2000X)

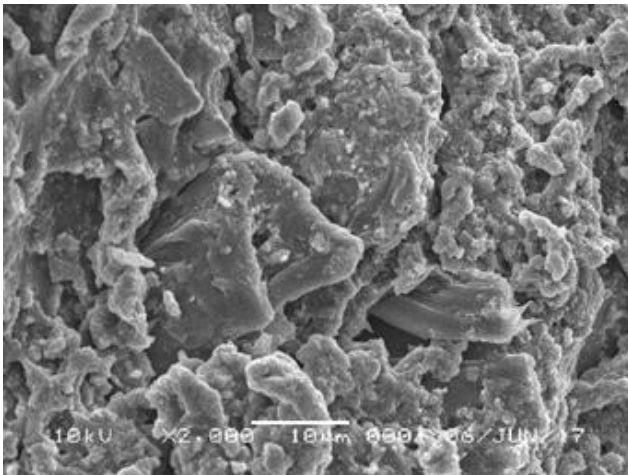**Fig. 7.** SEM Images of PLF-2.5 (Magnification 2000X).

Fig. 8. SEM Images of PLF-5 (Magnification 2000X).

The results of the SEM test for PLF-2.5 in Figure 7 show a picture of a homogeneous dispersion between the particles in the synthesized composite consisting of a composite resin matrix and alumina-zirconia filler, carbonate apatite, and 2.5% Fiber.

The results of the SEM test for PLF-2.5 in Figure 8 show a picture of a homogeneous dispersion between the particles in the synthesized composite consisting of a composite resin matrix and alumina-zirconia filler, carbonate apatite, and 5% Fiber.

The difference in the results of the SEM image lies in the homogeneous image of the particles, gaps, and agglomeration. PLF-1 gave a homogeneous picture of composite particles and there were several gaps. A homogeneous picture shows that the dispersion between the matrix and filler occurs well and evenly. PLF-2.5 generates a morphological with narrower gaps than PLF-1. This proves that the addition of Fiber has an effect on the density. More addition of fiber that will cover the gap. The SEM results of PLF-2.5 showed the presence of agglomeration or clumping of filler particles. The agglomeration could be due to the excessive amount of Fiber concentration. Fiber should bind to the polymer matrix, but due to excess, it will bond with each other and form agglomeration. PLF-5 shows more agglomerate than PLF-1 and PLF-2.5 indicates that the addition of Fiber exceeds the optimum limit. Several studies have shown that the optimum amount of Fiber as a composite filler is achieved in a low amount, which is less than 5%. The higher the concentration of nanocellulose added, the less the dispersion of the matrix and filler [19]. This is in line with research conducted by Talari et al. [20] which states that the addition of 5% nanocellulose to PMMA-based composites shows the presence of many bubbles and the distribution of cracks. The increase in filler volume and the formation of bubbles is the starting point for cracking. A composite needs to be given a coupling agent to increase the bond between the matrix and the filler. Results in Table 2 shown that increase of PLF have will decrease the grain size of matrix up to 2.5% nanocellulose addition, which explains the best mechanical properties in PLF-2.5 since finer grain size resulted in improved mechanical properties. However, the latter will decrease along with subsequent addition of PLF which tends to agglomerate the material.

The bond formed by the dissolution of chitosan has a positively charged amine group and the presence of acetic acid can cause chitosan to have bioadhesive properties so that it can bind to negatively charged fillers. Chitosan with low viscosity can produce a good bond between the filler and matrix in a composite, resulting in a homogeneous composite mixture and only a small amount of agglomeration is formed [21]. A good composite material not only depends on composition but also the size and form of individual components [22]. The SEM morphology shows that the gaps are narrower when given

2.5% Fiber compared to 1%. Addition of 5% Fiber has formed several agglomerations which tends to reformed the gaps. In the mechanical properties of flexural strength, a wide gap will actually result in a change in the modulus of elasticity and easily cause cracks to propagate.

Conclusions

Based on the research that has been done, the conclusions that can be drawn are as follows. Addition of pineapple leaf (Ananas comosus (L.) Merr) fiber and alumina-zirconia-carbonate apatite as filler were successfully synthesized. The hardness values of alumina-zirconia-carbonate apatite nanocomposite with the addition of 0%, 1%, 2.5%, 5% pineapple leaf fiber resulting in 30.31 VHN, 31.13 VHN, 34.02 VHN, and 27.22 VHN respectively. Addition of 1% and 2.5% fiber had a higher hardness value than the group without the addition and 5% fiber.The flexural strength of composite with the addition of 0%, 1%, 2.5%, 5% resulting in 1.6 MPa, 2.1 MPa, 2.3 MPa, and 1.8 MPa, respectively. The addition of 1% and 2.5% nanocellulose had a higher flexural strength value than the group without addition and 5% fiber. The morphology of composite with the addition of 1% and 2.5% pineapple leaf fiber showed more homogeneous particle and narrower gaps. Meanwhile, composite without addition of fiber shows agglomeration of particles and 5% fiber addition shows wider gaps and agglomerations.

Rahma Isnani Surya Asri – Bachelor of Dentistry;
Bambang Sunendar – Professor of Advanced Materials Processing Laboratory;
Irfan Dwiandhono – Medical Doctor;
Andrie Harmaji –Master of Material Science and Engineering.

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Р.І.С. Аспі[1], Б. Сунендар[2], І. Двяндхоно, А. Хармаджі[3]

Властивості стоматологічної композитної смоли, армованої оксидом металу та ананасовим волокном

[1]*Факультет стоматології, університет Джендлерал Сьодірман, Пурвокерто, Індонезія rahmaphrodite@gmail.com*
[2]*Кафедра інженерної фізики, Технологічний інститут Бандунга, Бандунг, Індонезія*
[3] *Лабораторія вдосконаленої обробки матеріалів, Інститут технологій Бандунга, Бандунг, Індонезія*

Індонезія є одним з найбільших у світі виробників ананасів, тому відходи листя ананаса там широко доступні. Метою дослідження є демонстрація використання волокна листя ананаса (Ananas comosus (L.) Merr) задля підвищення механічних властивостей матеріалу стоматологічного застосування на основі оксиду металу. Зразок складався з чотирьох композиційних груп з додаванням 0-5% клітковини. Далі усі композитні зразки перевіряли на твердість, міцність на вигин і за допомогою скануючої електронної мікроскопіх (SEM). Результати випробування на твердість композиту без додавання фібри склали 30,31 VHN. З додаванням фібри 1%, 2,5% і 5% твердість композиту становить 31,13 VHN, 34,02 VHN і 27,22 VHN, відповідно. Результати випробування на триточковий згин показали, що міцність на вигин зразка без додавання волокна становила 1,6 МПа, тоді як додавання 1%, 2,5% та 5% волокна призвело до міцності на вигин 2,1 МПа, 2,3 МПа та 1,8 Мпа, відповідно. Результати SEM демонструють однорідну морфологію дисперсії частинок з різними агломераціями та проміжками. Композити з додаванням 1% і 2,5% фібри мають більш вузьку щілину, ніж без додавання фібри. Цим пояснюється підвищення твердості та міцності композиту на вигин.
Ключові слова: волокно листя ананаса, стоматологічна композитна смола, твердість, міцність на згин, СЕМ.