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The Mechanical Properties of Sisal/PMMA and Sisal/Carbon/PMMA Biomedical Composites

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Abstract. Sisal, carbon, and poly-methyl methacrylate (PMMA) are the component materials that have been developed for the biomedical composite. However, characterization of the mechanical properties of the composites affected by some modified treatments is still opened for discussion. Sisal/poly-methyl methacrylate (PMMA) and sisal/carbon/PMMA composites with 30% fiber content and 6 mm fiber length were manufactured using a cold press molding at room temperature for about 60 min curing time. Tensile and bending properties of the composites were investigated by the influence of alkalization, the addition of maleic-anhydride-grafted polypropylene (MAPP) and hybridization of sisal and carbon fibers. The results indicated that the addition of MAPP (3, 5 and 10 wt. %) increases the tensile and flexural strengths of sisal/PMMA composites which are higher than the composites reinforced with alkali-treated and untreated sisal fibers. The addition of 5 % MAPP resulted in more effective improvement in mechanical properties compared to the effect of alkalization. However, a significant enhancement of tensile properties was shown by the hybridization effect of sisal and carbon with a ratio of 1:1 and 1:2 in sisal/carbon/PMMA composites. Scanning electron microscopy (SEM) of tensile fracture surfaces confirmed the presence of a functional relationship between the high mechanical strength of the composites with excellent adhesion between sisal fiber and PMMA by introducing 5% MAPP. Relatively homogeneous fiber dispersion in the matrix either sisal fibers or mixed sisal and carbon fibers within the PMMA matrix with sisal/carbon ratio of 1:2 have also contributed to the improvement of the mechanical strength. The use of alkali-treated sisal and HNO_3-treated carbon fibers had promoted a remarkable increase in tensile strength of the sisal/carbon/PMMA hybrid composites.

Introduction

Sisal (*Agave sisalana*) fiber, carbon fiber, and poly-methyl-methacrylate (PMMA) are useful materials for biomedical applications because their properties are compatible with human tissue [1-3]. Sisal fiber is mostly available in tropical countries and is one of the commercial natural fibers having antimicrobial properties. It has been reported that sisal extract can effectively suppress the microbial growth [2], antihelmintic, and also anti-inflammatory [4-6]. PMMA is a non-crystalline synthetic polymer of methyl methacrylate used in orthopedic as bone cement to affix implants and to remodel lost bone. This material, however, is unbeneficial in operational used at high temperature because it destroys the surrounding area [2]. Diamond, diamond-like carbon, graphene, and carbon nanotube are the allotrope of carbon that is currently used as biomedical materials. Carbon can be fabricated as carbon nanoparticles and carbon nanotubes, which are usually used as fillers for composite materials [7]. It is well known that carbon has superior properties. Due to their (sisal fiber, PMMA, and carbon fiber) excellent compatibility with human tissue, they have been selected as the constituents for the biomedical composites in this research.

There are two categories of human tissue, i.e., hard tissues (bone and tooth) and soft tissues (skin, blood vessels, cartilage, and ligament), in which hard tissues have higher tensile strength and modulus of elasticity than the soft ones [3]. Xu et al. [8] have reported short sisal fiber/PMMA composites with 2 mm fiber length and various fiber loading up to 10% for dental application. Introducing 2.5% untreated-sisal fiber into the PMMA matrix decreased flexural strength from 56 MPa to 47 MPa. Further increase the fiber loading gradually increases the flexural strength both for untreated and alkali-treated sisal. Based on the trend of those results, the flexural strength of sisal/PMMA composite with fiber content higher than 10% would possibly continuously enhance mechanical properties. Further research is needed to verify the pattern. Besides, the mechanical properties of PMMA composites filled with nanosize natural powders, pomegranate peels (PPP) and seed powder of dates Ajwa (SPDA) in relatively low concentration (0-2 wt.%) [9], have been investigated and resulted in very high mechanical properties.

Except for application in dentistry, high-performance composites are required to replace traditional prostheses such as those made of wood and aluminum. Some research groups have developed natural fiber composites reinforced with rattan, ramie, kenaf for external prosthesis application included for socket prosthesis [10-13]. Ramie reinforced epoxy [10] composite showed higher mechanical properties in comparison with rattan reinforced epoxy [11], but lower than pineapple fiber reinforced epoxy [12]. Hybrid composites containing woven kenaf, glass, silk, helenca stockinette, and polyvinyl alcohol (PVA) [13] exhibited much lower mechanical properties than ramie and rattan reinforced composites. In the case of material used for internal prosthesis, it should be highly corrosion and abrasion resistants, and high biological suitability.

Investigation on sisal/PMMA composite seems to be relatively scarce, especially research which used MAPP as a coupling agent in PMMA composites. The presence of interaction between the PMMA and MAPP phases has been shown by introducing organoclay [14], suggesting that MAPP was working as a crucial role in the PMMA. In current preliminary research for biomedical composites, improving the tensile and bending properties of sisal/PMMA and sisal/carbon/PMMA composites were investigated by applying alkali-treatment on sisal fibers, the addition of a MAPP coupling agent and hybridization of sisal and carbon. Enhancement of the mechanical properties was discussed by SEM and optical analyses results of the fracture surface morphologies.

Experimental Methods

Materials. Sisal fiber with a tensile strength of 706.00 MPa and modulus of 16.80 GPa was purchased from Balittas-Malang, Indonesia. Toray 1700sc 120000-50C carbon fiber with the tensile strength of 4.9 GPa and modulus of 250 GPa was supplied from Hobbyrover, China. While PMMA (ISO 1567 Type II Class I) for use in Dentistry was purchased from Dental Jaya, Indonesia and MAPP (Mw ~9100) supplied by Sigma Aldrich were used as the matrix material and a coupling agent, respectively.

Treatments of Sisal and Carbon Fibers. Untreated sisal fibers were washed in flushing water and dried in an oven at 70°C for 30 min. Alkalization of sisal fibers was carried out by soaking the fibers into 6% NaOH at room temperature for 4 h and neutralized with 1% CH_3COOH to avoid the remaining non-cellulosic components, and then finally the fiber washed in flushing water. The untreated and alkali-treated sisal were both subsequently chopped into 6 mm length. Treatment of carbon fibers was carried out under the optimized conditions: i.e., by immersing the fiber in nitric acid (HNO_3) solution with 68.3% concentration at room temperature for 48 h, and then dried in an oven at 80°C for 6 h.

Fabrication Techniques. There are presently two kinds of composites; sisal/PMMA and sisal/carbon/PMMA hybrid composites. The composites were fabricated by applying a hand lay-up method using a cold press molding at room temperature for approximately 60 min curing time. Sisal/PMMA composites with 30% fiber loading were distinguished in three types: i.e., untreated-sisal/PMMA, alkali-treated sisal/PMMA and untreated sisal/PMMA with MAPP addition (3, 5 and 10%). Sisal/carbon/PMMA hybrid composites were prepared in three kinds of sisal-to-carbon ratios; 2:1, 1:1 and 1:2, and 20% fiber content (kenaf and carbon fibers) due to manual fabrication for hybridization of sisal and carbon fibers with 30% fiber loading was complicated; the fibers tended to agglomerate. In a similar case, our previous research has reported that no significant difference in tensile strength of kenaf/glass/polypropylene composites with 20% and 30% fiber loadings was shown [15]. For comparison, alkali treated-sisal/PMMA with 20% fiber content was fabricated as well.

Mechanical Testing and Morphological Characterization. Tensile testing specimens were prepared according to ASTM D638-01 and conducted with a universal testing machine (UTM) having a maximum load of 2 kN and at a cross-head speed of 5 mm/min. ASTM D790-02 was adopted for a three-point bending test carried out using a UTM (J.T.M. Technology Co., Ltd) having a maximum load of 1 kN and at a cross-head speed 3.3 mm/min. For each case, eight (8) composite specimens were prepared. In this research, tensile and bending tests were carried out on sisal/PMMA composites while on sisal/carbon/PMMA composites were only subjected to tensile testing because it was just for comparison and understanding the hybridization effect on the enhancement of the mechanical properties compared to alkalization and impact of adding MAPP.

Scanning electron microscopy (SEM, VEGA 3 TESCAN, and HITACHI SU-3500) was performed to examine the morphology of the tensile fracture surfaces. The SEM results characterized alteration of the mechanical values. An optical microscope was also used to observe the fiber distribution in the matrix from a cross-section view.

Results and Discussion

Fiber Surface Morphology. SEM micrographs of sisal fiber before and after alkali-treatment (Fig.1) show the difference in surface morphology. Surface contaminants and hemicelluloses covering the fiber surface (Fig.1a) were removed by alkalization in 6% NaOH for 4 h, and it made the surface clean (Fig.1b) [15] and more cellulose fiber exposes to the surface. It is also exhibited that the fiber has started to fibrillate (Fig.1b, see arrows). It is possible that the fibers were partially fibrillated, confirming the elimination of hemicellulose. On the other hand, SEM images of carbon fibers (Fig.2) show cleaner fiber surface after HNO_3 treatment for 48 h (Fig.2b) compared to the untreated one (Fig.2a), indicating that the treatment procedure is effective.

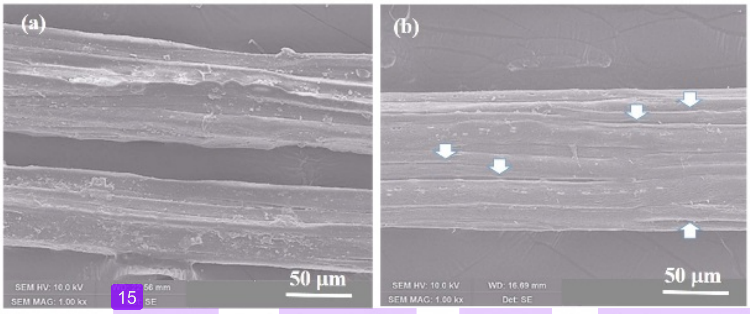

Fig.1. SEM images of sisal fibers before (a) and after (b) alkali-treatment

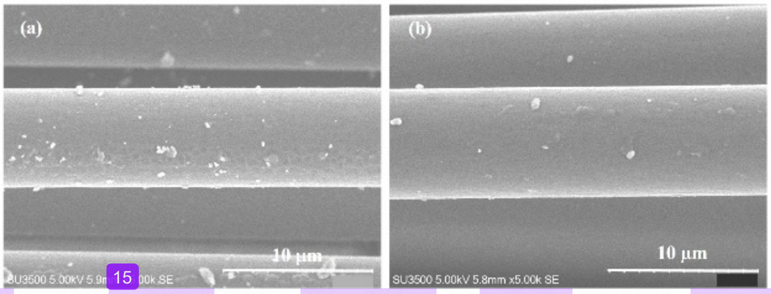

Fig.2. SEM images of carbon fibers before (a) and after (b) HNO_3 treatment

Tensile and Flexural Strength of the Composites

Sisal/PMMA Composite. Tensile strength and modulus of sisal/PMMA composites (Fig.3a) indicated that an increase of tensile strength of alkali-treated-sisal/PMMA is caused by the change in fiber surface morphology (Fig.1b). Hemicellulose may completely remove after alkalization, but residual lignin as the fiber binder in between the sub-fibers remain, making the fibers in slightly hydrophobic and compatible with hydrophobic matrix resulting in higher tensile strength of the composite than that of untreated-sisal/MAPP.

The addition of MAPP in the untreated-sisal/PMMA composites resulted in improving the tensile strength of sisal/PMMA composites, especially in 5% MAPP. As a result, 5% concentration of MAPP is an optimal value which is consistent with our earlier study of MAPP in the sisal/polypropylene (PP) composites [16]. It can be noted as an optimum concentration of MAPP for the PMMA composite, suggesting MAPP has a substantial effect on the effectiveness of the surface modification due to a molecular weight of MAPP. MAPP had also been confirmed to improve fiber-matrix interface bonding in the oil palm fiber reinforced composite. Adding MAPP in between 5-10% did not increase the mechanical properties.

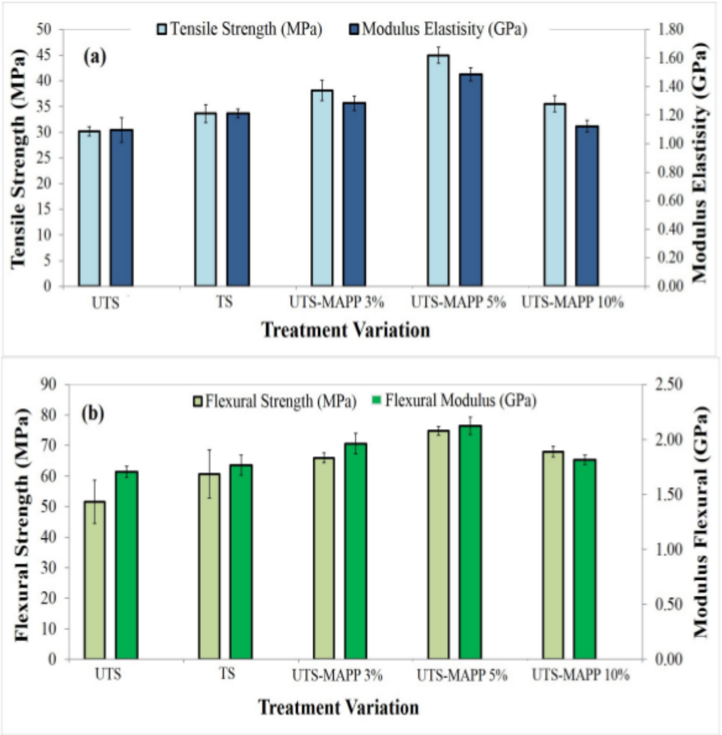

Fig.3. Tensile (a) and bending (b) properties of sisal/PMMA composites versus treatment variation of sisal fiber. UTS: untreated sisal and TS: treated sisal

A similar trend is demonstrated on the flexural strength and modulus (Fig.3b). Compared to the results reported by Xu et al. [8], the flexural strength of untreated-sisal/PMMA at 30% fiber content (51.52 ± 7.08 MPa) (present study) is consistent with that at 10% fiber content (~ 51.50 MPa) [8]. However, for the treated-sisal/PMMA, the flexural strength resulted in this study (60.70 ± 7.87 MPa) is higher than that by Xu et al. [8] (~55.00 MPa), and much higher flexural strength reached by untreated-sisal/PMMA with the addition of 5% MAPP (74.87 ± 1.40 MPa). Except for the fiber content, the differences in parameters between this study and Xu's study are the fiber length and fiber treatment. These are the key parameters that caused higher flexural strength in this study. On the contrary, flexural modulus resulted in this study is lower than that reported by Xu et al. [8], which leads to being lower in brittleness. This result is corresponding to the absence of failure, but just cracking occurred in all bending specimens. Thus, the present composite could be recommended for dental application.

Sisal/Carbon/PMMA Hybrid Composites. The tensile strength of sisal/carbon/PMMA as a function of treated-sisal/carbon ratio (Fig. 4) exhibited that the higher the volume fraction of carbon yields higher tensile strength of the composite. This result is closely related to the significantly higher tensile strength of carbon fiber than that of sisal fiber as described in the previous section. The effect of HNO_3-treatment on the carbon fiber surface leads to excellent fiber-matrix interfacial bonding. It is also indicated that using alkali-treated sisal in the hybrid composites increased overall tensile properties compared to the results shown in Fig.3.

It should be noted that the effect of hybridization between sisal fiber and carbon in the hybrid composite at treated-sisal/carbon ratio of 1:2 could significantly improve around 40% tensile strength compared to the tensile strength of treated-sisal/PMMA composite. According to the results in Fig.3, the alkalization and addition of MAPP improve the tensile strength about 33% and 10%, respectively, and for flexural strength about 31% and 15%, respectively. Therefore, the hybridization technique, in this case, is an effective way of enhancing mechanical properties.

Based on the tensile strength and modulus of both sisal/PMMA and sisal/carbon/PMMA composites resulted in the current study yield relatively lower value compared with the composites of ramie/epoxy [10] and rattan/epoxy [11] which have been developed for socket prosthesis application. So, the present composites might be suitable for internal prosthesis application because all the composite components are biologically suitable. Further study may be required.

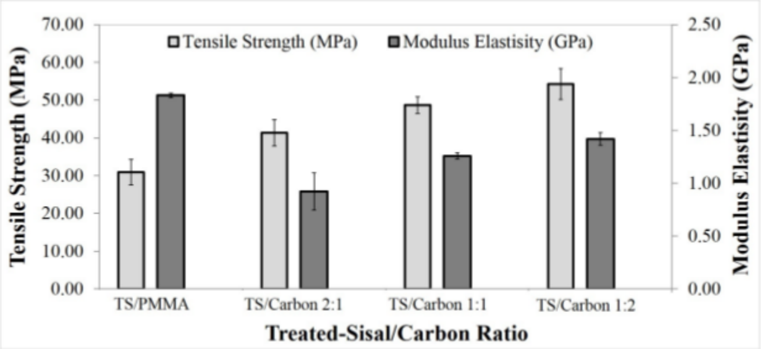

Fig.4. Effect of treated-sisal/carbon ratio on tensile strength and modulus of sisal/carbon/PMMA hybrid composite in comparison with a treated-sisal/PMMA composite

Morphological Characterization

The changes in the mechanical strength depicted in Fig.3 due to alkalization and the addition of MAPP are considered to correspond to the compatibility between the fiber and the matrix, and also fiber distribution within the matrix. SEM images (Fig.5) reveal the related morphologies of tensile fracture surface for each specimen with the difference in tensile strength and modulus (Fig.3a). In untreated-sisal/PMMA composite, the fibers were relatively not tightly bonded with the matrix (Fig. 5a), so that some fiber pullout, debonding, and voids were observed. Unlike the appearance of the morphology in Fig.5b, the structure of composite seems to be denser due to alkali-treated sisal fibers made better interfacial bonding with the matrix compared to the use of untreated-fibers. In addition, the effect of adding MAPP with different concentration showed apparent differences in morphology. A suitable concentration of 5% MAPP, in this case, made a matching interaction between MAPP and hydroxyl group formed at the fiber surface, leading to better stress transfer from the matrix to the fiber and improve compatibility between the fiber and the matrix (Fig.5d). Inversely, the composite with < 5% MAPP leads to weakening in fiber-matrix interfacial bonding, which also caused the formation of more voids (Fig.5c). The addition of > 5% MAPP had altered the stress transferred from the matrix to the fiber resulting in reduced tensile strength of the composite (Fig.5e). Optical microscopy on the composite specimen with 5% MAPP also revealed more homogeneous fiber distribution in the matrix than the others, which contributed to improving the flexural strength.

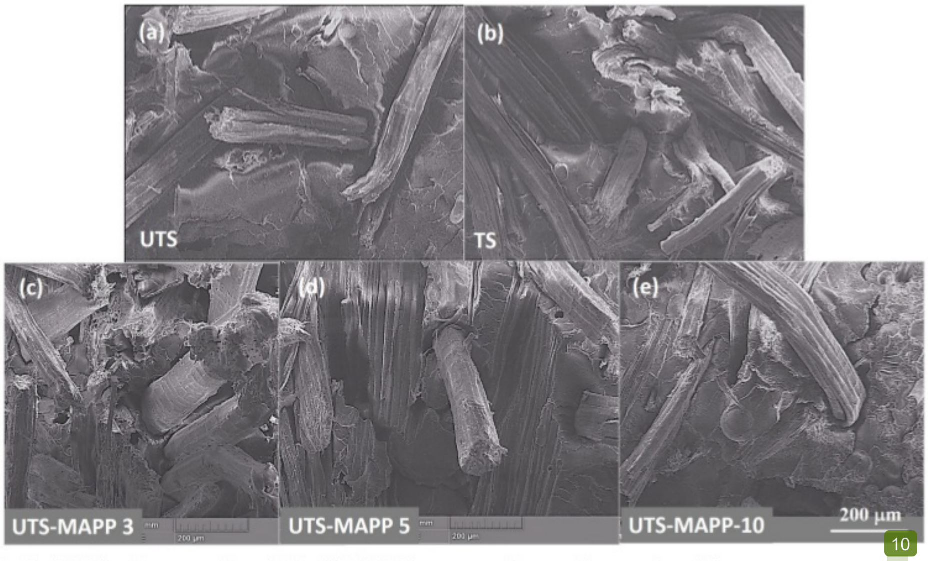

Fig.5. SEM micrographs of sisal/PMMA composites with varying fiber treatments. (a) Untreated-sisal, (b) alkali-treated sisal, (c) untreated-sisal + MAPP 3%, (d) untreated-sisal + MAPP 5% and (e) untreated-sisal + MAPP 10%

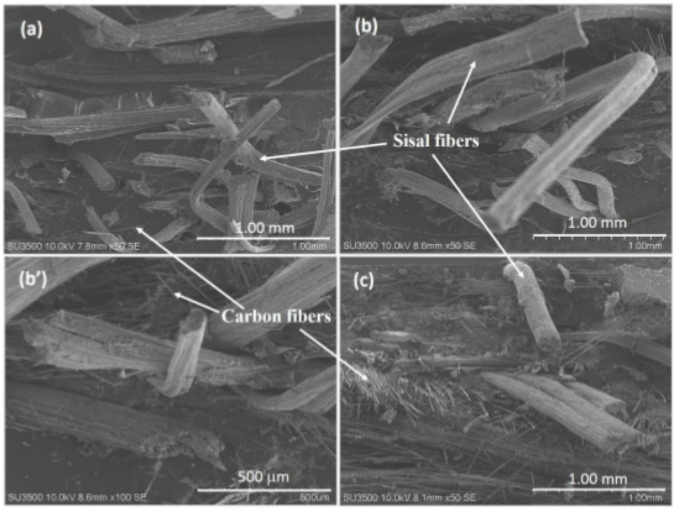

Fig.6. SEM micrographs of sisal/carbon/PMMA hybrid composites with varying ratio of sisal to carbon. (a) 2:1, (b) 1:1, (b') a magnified image of (b), and (c) 1:2

On the other hand, an effect of sisal and carbon fiber hybridization shown in Fig.6 distinctly observed that both kinds of fibers had tightly bonded with the matrix due to the chemical treatments carried out on both sisal fiber and carbon fiber. Thus, higher carbon fiber content leads to higher tensile strength. The opposite effect would happen in case no treatment conducted on the carbon fiber surface. In other words, higher tensile strength is reached by a lower ratio of sisal to carbon. SEM images demonstrated in Fig.6 have a good relationship with tensile properties shown in Fig.4.

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Summary

1. Sisal reinforced PMMA composite and sisal/carbon/PMMA hybrid composite materials with high mechanical strength, and low brittleness have been successfully fabricated using hand lay-up in a cold press molding.
2. Improvement in tensile and bending properties of those composites was predominantly affected by hybridization of sisal and carbon fibers in comparison with alkalization and the addition of MAPP.
3. Tensile strength (54.21 ± 4.07 MPa) and modulus (1.42 ± 0.06 GPa), and flexural strength (74.87 ± 1.40 MPa) and modulus (2.12 ± 0.08 GPa) resulted from the present composites can be considered as biomedical composite material: i.e., useful for dental and alternative internal prosthesis applications.

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