Effect of vibration duration of high ultrasound applied to bio-composite while gelatinized on its properties

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ABSTRACT

This article reports effect of vibration duration of high ultrasound applied to bio-composite while gelatinized on its properties. The bio-composite consists of mixing of both the tapioca starch based bioplastic and oil palm empty fruit bunch (OPEFB) fibers with high volume fraction. Gelatinization of the bio-composite sample was poured into a rectangular glass mold placed then in an ultrasonic bath with 40 kHz, and 250 watt in different duration for 0, 15, 30, 60 min respectively. The results show that vibration during gelatinization has changed the characterisation of the bio-composite. SEM photograph displayed different fracture surface of tensile sample. For vibration duration of 60 min, tensile strength (TM), and tensile modulus (TM) was improved to 64.4, 277.4%, respectively, meanwhile strain was decreased to 35.1% in comparison without vibration. Fourier Transform Infrared Spectroscopy (FTIR), and XRD diffraction of the bio-composite has changed due to various vibration duration. Moisture absorption of the vibrated bio-composite was lower than that of the untreated one.

1. Introduction

A big attention of researchers for replacement synthetic plastic with biodegradable bioplastic has been increasing to reduce energy consumption as well as to save environment in all fields worldwide. Our world provided us much more resources of food polysaccharide with different characterization in physical and mechanical properties. The food polysaccharide is a main basic material for producing of starch base bioplastic. The natural resources for bioplastic are abundantly available, biodegradable, cheap, and renewable. However, this bioplastic has low mechanical properties, and absorbs high moisture. Various attempts have been made previously to improve the mechanical properties and the resistance to water vapor [1,2]. Last work reported that mixing nanofibers as well as nanoparticle in starch base bioplastic improved the mechanical properties and the resistance in water vapor absorption [3]. This improvement is due to the fact that existing of the nanoparticle in the bioplastic hinders movement of molecule chains of the biopolymer as well as water molecules to diffuse in the matrix [4].

An ultrasonic bath is an equipment operated using ultrasound (usually from 20–400 kHz). The ultrasound treated on starch has changed its characterization [5–7]. The ultrasound can affect starch dispersions and damage the starch granules [8]. High power of

ultrasonic is able to reduce the molecular weight of amylose and amylopectin due to breakage of C-C bonds, which leads to a decrease in viscosity [9,10,7]. Furthermore, swollen starch granules and aggregates can be solubilized, including "ghost" granules which remain even after the full gelatinization of starch dispersion [6]. Last study reported that ultrasound treatment was an effective way in increases solubility of starch in water, thus increasing tensile strength, and moisture resistance of the starch bioplastic [8]. Ultrasound treatment provides facilitates of the disintegration of starch granules and the formation of homogenous starch solution [5]. High-frequency vibrations treated maize starch dispersion have improved mechanical properties and lower water vapor permeability of the film [8].

Mechanical properties of bio-composite reinforced by high volume content of fibers could decrease. This is due to the fact that some the fibers was not glued effectively by the matrix, and not dispersed homogeneously in the matrix [11,12]. Because of high volume content of fibers, agglomeration is possible to exist in the composite matrix. Agglomeration of the fibers results decrease in mechanical properties and increase in moisture absorption of the bio-composite [13]. It could be believed that agglomeration of natural fibers in matrix of biopolymer due to high volume content could be disentangled. Vibration with high frequency has evidenced result that the aggregation of a substance can be destroyed or solubilized [6]. Last finding was reported that the

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effects of ultrasonic treatment on starch properties provided holes or channels formed both on the surface and inside the granules [14]. As a result, the specific area of starch particles increased, which may lead to an increase in the reaction efficiency [15].

Some previous works have reported using of ultrasound of ultrasonic bath for preparation of biodegradable composite [8,16,17,18,19]. However, no last reports explained study of various ultrasonic duration applied to the OPEFB fibers reinforced starch based bio-composite while gelatinized. This is important to get effective duration of ultrasound for the sample preparation in order to obtain the best results of its properties. In the present paper, we investigate effect of various vibration duration while sample gelatinized on properties of bio-composites reinforced by extraordinary volume content of 75% OPEFB. The high volume fraction of the fibers has been incorporated in a thermoplastic cassava starch matrix to increase the mechanical properties as well as the hydrophobic character of starch based bio-composite. So, the main originality of the research is that various vibration duration with high frequency was applied to 75% high fibers content of OPEFB starch bio-composites while gelatinized. The bio-composites have been prepared and characterized by tensile testing, moisture absorption, X-ray diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), and SEM fracture surface.

2. Materials and experiment

2.1. Materials

The oil palm empty fruit bunch were obtained from West Pasaman, Indonesia. Tapioca starch was purchased from local market in Padang under the "Cap Gunung Agung" type. Sodium Hydroxide (NaOH) technical grade was purchased from Bratachem Company, Padang, Indonesia. Distilled water was supplied from the Mechanic and Metallurgy Laboratory of Andalas University.

2.2. Experiment

2.2.1. Extraction the OPEFB fibers

OPEFB fibers were extracted from oil palm empty fruit bunch cooked during 6 h. The extracted fibers were cleaned and cut about 1 cm long, then they were mixed into solution of 25% NaOH (from the weight of the dried fibers) for making pulping with a high-pressure reactor. This compound in the reactor was heated at 170 °C and 8–9 bars respectively for 2 h. Finally, we produce soft OPEFB fiber in form of pulp with pH of about 12. Then, it was neutralized with distilled water until pH 7. The OPEFB fiber was blended in wet condition using blender for 30 min. The pulp was cast in a pulp paper into screen printing, and then dried until becoming a paper. Diameter of the treated OPEFB fiber was about 6 μm.

2.2.2. Measuring chemical compositions

Chemical composition of OPEFB fibers before and after being pulp was tested to determine change of content of cellulose, hemicellulose, and lignin in the fiber by using ASTM 1104–56, Technical Association of the Pulp and Paper Industry (TAPPI) standard T9M-54, and TAPPI T13M-54 respectively. Dried OPEFB fibers of 5 g was tested for measuring the holocellulose content, and then, 2 g of those was verified of cellulose content. Meanwhile disparity between holocellulose and cellulose was lignin content in OPEFB fibers.

2.2.3. Preparation of bio-composites

Weight of the dried OPEFB fiber was 7.5 g (75% based on starch weight). Then it was mixed with 100 ml distilled water, 2 ml glycerol, and 10 g starch respectively. The compound was stirred using a magnetic stirrer at about 500 rpm for 5 min and heated at the 65 °C for 30 min until gelatinized. The gelatinized bio-composite was poured into a rectangular glass mold that was then placed in an ultrasonic bath

filled by distilled water. In this work, the ultrasonic bath (model number PS-70AL China) was operated at fixed frequency of 40 kHz, and 250 watt average ultrasonic power. Duration during vibration was varied at 15, 30, 60 min respectively. The water in the bath was replaced every 15 min for keeping temperature lower than 40 °C. The sample was dried in a drying oven (Memert Germany, Model UN 55) at 50 °C for 20 h.

2.2.4. Moisture absorption

A Piece of the bio-composite with dimension of 1×1.5 cm was dried in the drying oven at 50 °C until constant weight. After that, the sample was stored in a moisture chamber with high relative humidity (99%). Measurement method of the moisture absorption was conducted by weighing the sample every 30 min for span of 180 min. To get precise data, there were three different samples for each similar ultrasonic duration. Then, the samples was stored for 17 h in the high humid chamber, after that, it was taken out from the chamber to be weighted by using precision balance of 0.1 mg (Kenko). Calculation of the moisture absorption uses the equation below:

Moisture absorption $= (w_h - w_0)/w_0$

where w_h is final weight and w_0 initial weight of sample.

2.2.5. Tensile test

Tensile strength of the bio-composite samples was measured by using equipment Com-Ten testing machine of 95T Series with speed and temperature during testing were 3 mm/min and room temperature, respectively (3 mm/min, RT). There were five samples for each vibration duration. Standard of the sample for tensile testing was ASTM D638 type I. TS was obtained by dividing maximum force of the tested sample and average cross section area of initial sample. Before conducting the tensile test, in order to obtain average cross section area of the initial specimen accurately, there were ten different location points of thickness and width of the film measured by using dial indicator micrometer of 0.001 mm. TM was automatically delivered by the tensile equipment from slope between elasticity stress and strain. Tensile strain of the sample was measured of a fracture strain.

2.2.6. SEM observation

Hitachi 3400 N series was a kind of SEM equipment used for observation fracture surface of OPEFB paper. Voltage while observation of the surface of tested sample was 15 kV.

2.2.7. X-Ray diffraction

X-ray diffraction (XRD – PANalytical Xpert PRO) measurements were performed by using CuKα radiation (λ = 0.1542 nm) at a scan from 10 to 30°. The operation voltage and current were 40 kV and 35 mA respectively.

2.2.8. FTIR

FTIR characterization was performed by using "The Perkin Elmer Frontier". The aim of FTIR characterization was to determine functional group of bio-composites (starch + OPEFB fiber). The dried samples were formed in sheet film. All samples were scanned at a frequency range of 4000–600 cm^{-1}.

3. Results and discussions

3.1. SEM fracture surface

Fig. 1 shows some SEM photographs of fracture surface of OPEFB fibers composite after tensile testing. Fig. 1an exhibits the fracture of sample without ultrasound, meanwhile Fig. 1b, c, d with ultrasound for 15, 30, 60 min respectively. In figure, it can be seen that there was significant difference between each pictures. As shown in Fig. 1a that bio-composite without vibration displayed fracture surface with a big
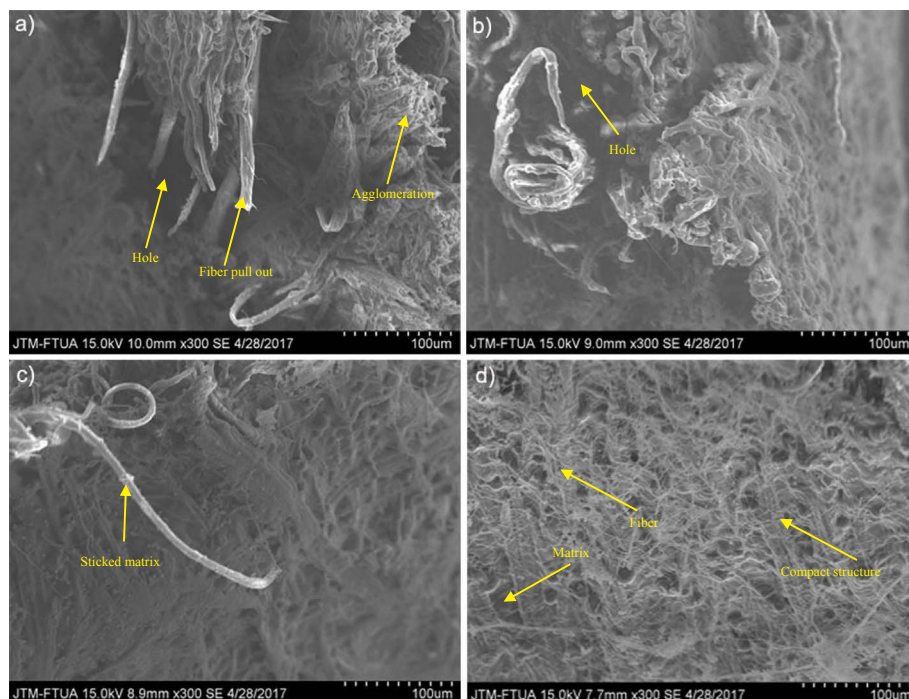
Fig. 1. SEM photograph of tensile fracture surface of studied bio-composite without (a) and with ultrasound duration for 15 min (b), 30 min (c), and 60 min (d) respectively.

porosity and agglomeration. In contrast, fracture sample vibrated reveals a decrease in amount of porosities and agglomeration as increasing vibration duration. Even, as seen in Fig. 1d, the fibers were seemingly dispersed homogeneously. Here, ultrasound of ultrasonic bath for 40 kHz, 250 watt could provide better quality of the studied composite.

3.2. Chemical compositions in ST fibers

Cellulose, hemicellulose, lignin of untreated OPEFB fiber was about 74.90, 22.29, 8.97% respectively, meanwhile treated ones was 78.52, 9.34, 7.76%, respectively. Hence, hemicellulose and lignin content after treatment were reduced significantly. In this case, cellulose in the OPEFB fibers used for reinforcement in cassava starch matrix was more dominant.

3.3. FTIR spectra

Change in the chemical structures of studied bio-composite molecules may be observed by FT-IR spectroscopy. Fig. 2 displays FTIR spectra of all studied samples without (No UB) and with vibration treatment for duration 0, 15 (UB 15), 30 (UB 30), 60 (UB 60) min respectively. In the spectrum of untreated sample, the bands of $3304\ cm^{-1}$ and $2929\ cm^{-1}$ correspond to OH and CH stretching, respectively. Meanwhile the bands at $1647\ cm^{-1}$ correspond to the scissoring of two O-H bonds of water molecules. Other important band in FT-IR spectra is vibration stretching for $1348\ cm^{-1}$ (C-O stretching of C-O-C group in the aryl–alkyl ether), and $1015\ cm^{-1}$ (C-O stretching of C-O-C group in the anhydroglucose ring), respectively [20,21]. As seen in figure, appearance of FT-IR spectra with and without ultrasound have similar profiles indicating that the ultrasonic vibration duration do not affect significantly form of its functional groups. All FT-IR spectra show similarly a broad peak in region of $3200–3800\ cm^{-1}$ that corresponds to the O-H stretching. It is noted that, in the spectra of the bio-composites, different wave number of treated and treated specimen could be distinguished. The untreated one appeared wave number at approximately $3304\ cm^{-1}$, and the other treated one for vibration duration of 15, 30, 60 min was 3301, 3297, $3292\ cm^{-1}$ respectively. Changes on this wave number could be due to the formation of good
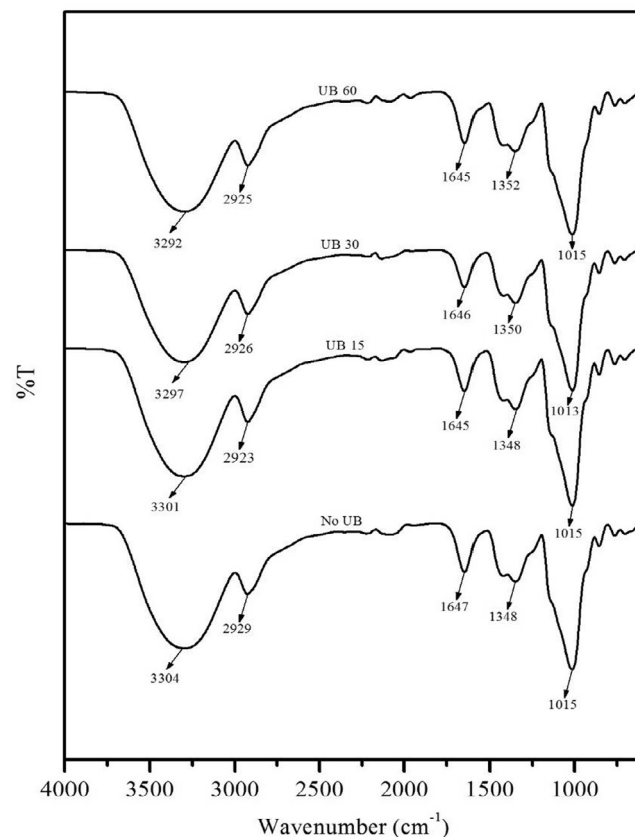

Fig. 2. FTIR spectrum of all samples tested with varied duration vibration.

hydrogen bonds between fibers and matrix [22]. It was observed that the bio-composites wave number for 60 min vibration are the largest shifting based on that of No UB specimen. Similar result was also found by last study that ultrasound effect shifted peaks of spectra to lower number [23].
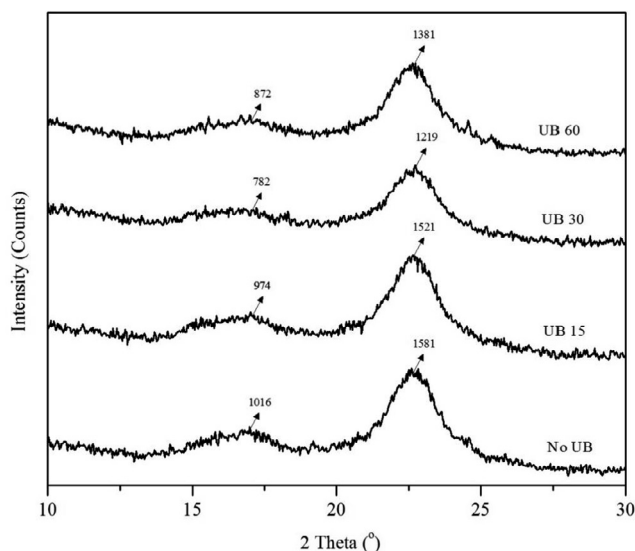
Fig. 3. XRD pattern of all samples tested with varied duration vibration.

Fig. 4. Stress strain curve: (a) no vibration; (b) 15 min; (c) 30 min; (d) 60 min.

3.4. X-ray diffraction

Fig. 3 shows diffraction patterns of studied samples without treatment, and vibrated by 40 kHz, 250 watt, respectively for 0 (No UB), 15 (UB 15), 30 (UB 30), 60 (UB 60) min. X-ray diffraction patterns show two major peaks (at around $2\theta = 16$ and $2\theta = 22$) of diffraction intensity that was believed that the studied OPEFB fibers indicated cellulose I type [24]. The curve for all experimental films with different ultrasound treatment times shows similar appearance at the scattering major angle. The similar finding was also reported by previous study [15]. However, only height of the intensity of both $2\theta = 16$ and $2\theta = 22$ for each sample was not similar as shown in Fig. 3. It was measured that the height for about $2\theta = 16$ was 1016, 974, 782, 872, meanwhile for about $2\theta = 22$ was 1581, 1521, 1219, 1381 respectively for vibration duration of 0, 15, 30, 60 min, respectively. Based on this present data, the shift of the major intensity peak for further ultrasonic duration is probably due to improvement interface bonding among matrix and fiber. Because ultrasonication treatment could accelerate chemical reaction [15]. The longer sonication time provides the samples more compact microstructures that led to changes of characterization of XRD pattern [25]. This is consistent to other data of the present work as shown in Section 3.5 that mechanical properties of each studied sample were different for varied ultrasonication time.

3.5. Tensile Strength, tensile modulus, strain

Fig. 4 displays individual performance of sample in form of stress-strain curve for various vibration duration. It is shown in figure that various duration at high frequency has changed significantly value of TS, TM, and strain of the starch based plastic reinforced by high volume content of the OPEFB fibers. Significantly, value of TS and TM improved, and strain of the bio-composite decreased, in this case, the sample become strong and brittle due to vibration of an ultrasonic bath equipment applied to the gelatin samples. In the beginning, for this cases, stress-strain curve improved strongly up to 30 min duration; then it will reach a saturation point for further shaking. Average TS, TM, and strain of bio-composite for various shaking time were shown in Fig. 5. As seen in figure, average TS, TM, and strain without shaking were 15.45, 296.50 MPa, 16.91%, respectively. After increasing shaking time of 15, 30, 60 min, average TS shown in Fig. 5a was measured for 20.80, 24.78, 25.40 MPa, respectively. There was 64.40% increment of TS value for 60 min shaking in comparison without vibration. However, a slight improvement of TS was observed for 30 up to 60 min, in this case,
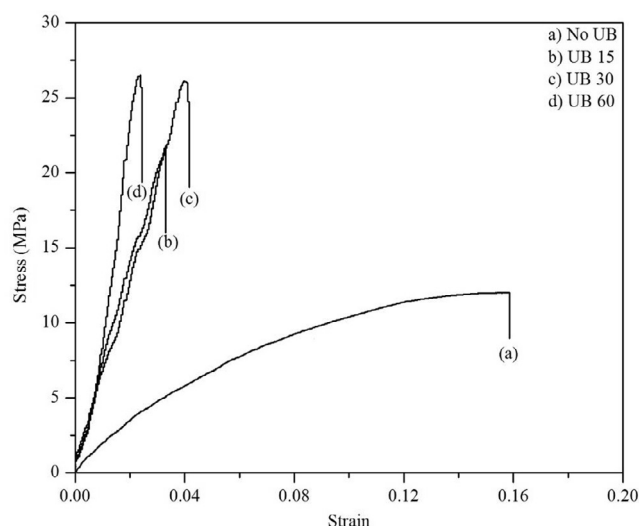
there was only 2.5% change of TS among duration. Meanwhile, TM for 15, 30, 60 min vibrated bio-composites was 685.00, 906.20, 1119.00 MPa, respectively (Fig. 5b). It was recorded that there was significant improvement of TM from 0 up to 30 min; then TM increased slightly for further vibration. Hence, there was 277.40% improvement of TM of 60 min vibrated sample in comparison without vibration. These results are in agreement with last finding [6]. Fig. 5c shows fracture tensile strains of bio-composites with different shaking time. As shown in figure, fracture strain decreased as increases in duration of shaking, although it increased at shaking for 30 min. High decreases in the strain was observed for shaking time from 0 up to 15 min; then slight reduction was recorded for 60 min in comparison to 15 min. As shown in figure, strain value without vibration was 16.91%, and it lowered into maximum value of 10.97% on 60 min duration of shaking (35.13% decreasing). According to last studies that TS and TM value of bio-composites without vibration were lower than vibrated one [8,22]. In these cases, vibration have changed mechanical properties of the bio-composites. Due to fixed vibration with high frequency, all substances of bio-composites could be vibrated with high kinetic energy to be able to move. Therefore the kinetic energy provides possibility of OPEFB fibers to separate individually as well as distribute homogeneously in matrix. This good dispersion may be also evidenced by shifting the FTIR peak in region of 3200–3800 cm^{-1} as shown in Fig. 2. As reported earlier, specimen become compact due to vibration with high energy [26]. Furthermore, due to this high vibration, porosities in the matrix as well as clearance between matrix and fibers were reduced. Therefore, increases in the mechanical properties of bio-composites may probably be explained by the reinforcement effect from the homogeneously dispersed OPEFB fillers in the tapioca starch matrix, and the strong hydrogen bonding interaction between the fibers and matrix [27,28].

3.6. Moisture absorption

High moisture absorption is a big problem to bio-composite based on starch and reinforced by natural fiber due to hydrophilic nature. Therefore, improvement of water resistance is important for development of bio-composite. In this case, high relative humidity for testing the water resistance was used. Moisture absorption ability was observed on untreated and treated samples. In case of treated samples, the various vibration duration were carried out to study the effects of such treatments on bio-composite moisture absorption ability. Fig. 6 shows average value of moisture absorption ability of samples treated by various vibration duration at the constant frequency of 40 kHz, 250 watt respectively. For span of 3 h, moisture absorption was
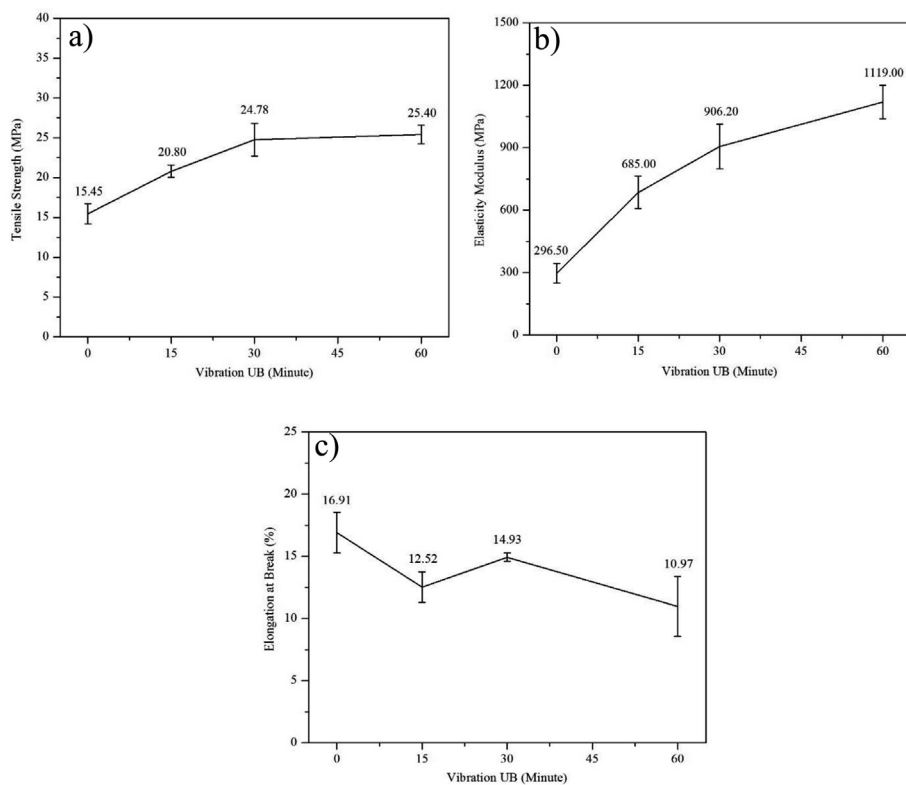
Fig. 5. Comparison of mechanical properties of all tested samples as function of duration vibration for (a) tensile strength; (b) elasticity modulus; (c) elongation at break.
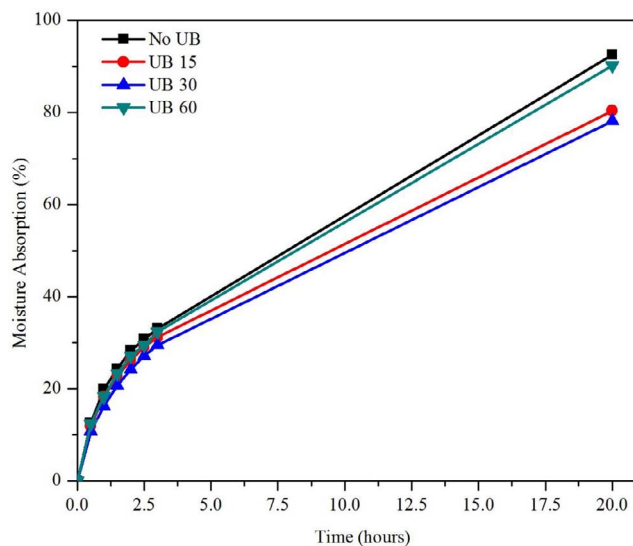

Fig. 6. Moisture performance of all tested samples.

measured for each 30 min. As shown in figure, for all cases, samples in a super-drying state will absorb moisture in high capacity at the beginning, then, the moisture absorption acceleration will decrease toward a saturation point noted about 3 h. In figure, it is seen that average of moisture absorption in 3 h interval was 33.06, 31.18, 29.51, 32.34% for sample of No UB, UB 15, UB 30, UB 60 respectively. Hence, resistance of sample in absorbing water improve in increases of vibration duration; except sample of UB 60 still high due to hydrophilic nature. As shown in figure, that vibration has provided a strong effect in decreasing of moisture absorption ability of the samples. It was noted in figure that water molecules still diffuse continuously in interval for 3 up to 20 h resulting swelling on bio-composite. In case of our data, the biggest swelling was on sample without vibration, and the lowest one on sample for UB 30. Hence, comparison of moisture absorption and

Table 1
Moisture gradient (α) of all samples tested.

Treatment	α (°)	Tan α	In Percent (%)	%/Hours
No UB	31	0.60	60	3.52
UB 15	26	0.48	48	2.82
UB 30	26	0.48	48	2.82
UB 60	30	0.57	57	3.35

time or gradient (α) for vibrated sample slightly decreases for further vibration duration. Table 1 displays different gradient for various vibration treatment of sample stored in humid chamber for interval 3 up to 20 h. This α value tends to decrease in increase of vibration duration (31, 26, 26, 30 for No UB, UB 15, UB 30, UB 60 respectively). This is probably due to the fact that the vibrated sample was more compact than that without vibration. In this case, change of water content in sample of No UB, UB 15, UB 30, UB 60 was 3.52, 2.82, 2.82, 3.35% per hour. Based on this result, improving the resistance of vibrated bio-composite to high humidity was affected by change in natural sample behavior into a hydrophobic substance. Water molecules become more difficult to diffuse into the bio-composite due to decreases in dimension of micro porosities in the matrix as evidenced by SEM photographs of the vibrated sample (Fig. 1d). Improvement both interface bonding between matrix and fibers, compatibility, and homogeneity of samples structure also could provide hindering of water molecules to diffuse into the matrix [26].

4. Conclusions

This article reported effect of variety of ultrasound duration of ultrasonic bath applied to gelatinized bio-composite on its properties. This bio-composite consisted of tapioca starch based matrix reinforced by 75% volume content of OPEFB fibers. Based on the results of this study, ultrasound of ultrasonic bath provided improvement of better bio-composite quality in comparison without vibration. The vibrated

samples displayed high compatibility with low amount of porosities, and homogeneously dispersed fibers in matrix as seen in SEM photographs. Both the mechanical properties and resistance of water uptake were better than that of the untreated samples. The bio-composite shows the highest mechanical properties, and become brittle at duration vibration of 60 min. In comparison without vibration, there was improvement of TS, TM, decreasing of strain value of 64.40, 277.40, 35.13% respectively for 60 min shaking. Of FTIR curve, change on the wave number of O-H stretching of vibrated bio-composite was believed due to the formation of hydrogen bonds between fibers and matrix. Intensity of XRD pattern was shifted for various vibration duration. Average moisture absorption in 3 h interval, namely saturation point, was 33.06, 31.18, 29.51, 32.34% for sample of No UB, UB 15, UB 30, UB 60 respectively. From 3 up to 20 h stored in humid chamber, samples evidence swelling after saturation point. Swelling gradient of sample without vibration was high, and it tends to decrease for vibrated sample.

Acknowledgment

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