The Influence of Zalacca Short-Fiber Addition with Random Orientation on the Characteristics of Low-density Polyethylene Matriced Composites

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Abstract. The aim of this research is to study the influence of zalacca fiber addition on physical, chemical, and mechanical characteristics of LDPE matriced composites fabricated by hot-pressing method. The fibers were short, random-oriented and varied as 10, 20, 30, 40 and 50 wt. %. The examinations included density testing, XRD, TGA-DTA, tensile and flexural testing, SEM and DMA. There was a reduction in composite density due to fiber loading. The zalacca fiber addition did not change degree of crystallinity of composites significantly. There was a bit decrease in thermal stability caused by fiber addition. The composite tensile strength and modulus of elasticity were enhanced with addition of zalacca fibers up to 40 wt. %. Tensile strength and elastic modulus declined due to further addition of fibers. At 40 wt. % fiber loading, there were several fiber fractures indicating that the applied load was borne effectively by the fibers after transferred from LDPE matrix. The fiber loading induced a meaningful improvement of flexural strength and modulus. There was a raise in storage and viscous modulus of composites due to fiber addition. However, fiber loading decreased loss factor, thereby reducing their damping capacity.

Keywords: zalacca fibers, LDPE-matriced composites, random orientation, hot press

1. Introduction
There is a requirement to substitute metallic materials with polymer matriced composites due to several benefits, such as high ratio of stiffness and strength to weight, lower density, higher corrosion resistance, thermal and electrical resistance, and lower cost of fabrication and recycling [1, 2]. The environmental issue endorses the utilization of biocomposites in which at least one of their components are biodegradable, like natural fibers.

Natural fibers consist of animal, plant and mineral fibers [3]. Among natural fibers, plants fibers have the highest availability and species. Jute [4], kenaf [5], coir [6], sisal [1], sugarcane bagasse [7], banana [8], and wood fibers [9] are the most utilized due to their high supply, variety and cheap cost. However, the application of plant fibers in industries are limited due to their poor compatibility with some polymer matrices [10]. Essential factors are involved in obtaining composites with optimal...
mechanical properties: mechanical properties of fibers and matrix, adhesivity between fibers and matrix [11], also fibers volume fraction, length, orientation [12] and thermal stability [13].

Zalacca edulis is an indigenous plant of Indonesia, Malaysia and Southeast Asia. Its nice fruit is harvested to eat. To maintain the quality and quantity of the crop, several of its midribs are cut twice a year after harvest. Commonly, the cut zalacca midribs are banished as waste or compost. Like sugarcane bagasse as waste [14], fibers can be extracted from the midribs. Like other lignocellulosic fibers, zalacca fiber is comprised of cellulose, hemicellulose and lignin as the main components [11]. Cellulose is the fiber part consist of amorphous and crystalline structure. It has high strength [15], resistance to oxidation and strong alkali. However, it is hydrolyzed by acids or sugar solution.

Hemicellulose has highly hydrophilic properties and amorphous structure. It is easily dissolved in alkalis and hydrolyzed by acids [11]. Lignin is hydrophobic and amorphous. It provides rigidity to the plant [16]. It is easily condensed and oxidized by phenol [17] and soluble in acetic acid [18]. Its characteristics is nearly similar to thermoplastic polymers, having melting point of 170°C and glass-transition temperature of ±90°C. Based on the study of Raharjo and co-workers [19], zalacca fiber contains 42.54 % cellulose so that it has potency as reinforcement for composites. In addition, zalacca can be classified as a secondary plant in which the fibers extracted from its cut midribs are by-products or residues [20].

Low-density polyethylene (LDPE) is thermoplastic polymer with relatively low melting point and hardness. Its toughness, impact properties and resistance to chemicals like alkalis, salts and acids are excellent [21]. Its low manufacturing cost is attributed to its low melting point, but its application is limited caused by its low strength and elastic modulus. Like polypropylene as thermoplastic [22], LDPE is considered as environmentally friendly due to its recyclability as recycled LDPE [23]. Also, wastes of LDPE can be converted as fuel [24], industrial raw materials [25] and building materials [26]. One of several methods in strengthening and stiffening LDPE is using fibers, especially zalacca fibers, as reinforcement. However, the influence of zalacca fibers loading on physical, thermal, morphological and mechanical properties of LDPE matrixed composites has not been investigated yet.

This study was aimed in characterizing LDPE matrixed composites reinforced by random-oriented short zalacca fibers. The study includes physical, thermal, morphological and mechanical properties of the composites.

2. Materials and Methods

2.1. Materials

Zalacca fibers were extracted from the cut midribs of 3-years-aged plants from some plantation in Sleman, Special Region of Yogyakarta Province, Indonesia, as shown in Figure 1 (a). The midribs, Figure 1 (b), were retted by soaking them in distilled water for 14 days. After separated from their husks, the fibers were then washed using distilled water and dried in open air for 2 days. Afterwards, they were heated in a ventilated oven at 70°C for 2 h and then cut into approximately 40 mm lengths and stored in sealed containers in which silica gel is added.

LDPE pellets used were Petlin LD C150Y having density and melting temperature of 924 kg.m⁻³ and 145-165°C, respectively. It was produced by Petling Sdn. Bhd., Malaysia. The 99.8 % anhydrous methanol used in determination of composite density, was purchased from Merck.

2.2. Fibers preparation

The fibers were cut into ±10 mm length then sieved through a 40-mesh strainer to obtain uniform length. They were then saved in containers with silica gel.

2.3. Preparation of composites

To fabricate random-orientated short fiber composites, LDPE was crushed into powder using a grinding mill, before sieved through an 80-mesh strainer and then stored in plastic containers. A digital scale having capacity and accuracy of 500 g and 10⁻² g, respectively, was used to weight LDPE
powder and zalacca fibers to obtain the specific composition and then mixed in an electric-powered mixer for 1 h. It was done to assure the macro-homogeneity of LDPE-fibers mixture and random orientation of the fibers. The mixture was manually fed into the dies. A hydraulic press machine was used for hot-pressing process at 5 MPa pressure, 115°C temperature and 5 minutes holding time. After cooled by air, the as-processed composites were then machined into the dimensions of specimens.

2.4. Density of composites

Theoretically, density of the composites can be calculated using Equation (1) [27]. Data of the matrix density entered into the equation were density of the hot-pressed LDPE matrix of Petlin LD C150Y. Zalacca fiber density was obtained from the density of untreated fiber.

\[
\rho_c = \frac{1}{\left(\frac{W_f}{\rho_f}\right) + \left(\frac{W_m}{\rho_m}\right)}
\]

in which \(W_f\) and \(W_m\) represents weight of fibers and matrix, while \(\rho_f\) and \(\rho_m\) are density of fibers and matrix, respectively.

Density of composites was measured according to ASTM D1037 standard, using Archimedes principle. Anhydrous methanol 99.8 % having density of 791 kg/m³ at 25°C was used due to its lower density than LDPE matrix. Measurement of composite density was done with variation of 0, 10, 20, 30, 40 and 50 wt. % fiber addition.

2.5. X-ray diffraction

XRD analysis was carried out to observe influence of fiber loading on degree of crystallization of LDPE matrix. X-ray diffractograms were obtained by a Philips PW 1800 X-ray diffractometer using CuKα radiation with voltage and current used were 30 kV and 17.5 mA, respectively. Scanning was performed in a range of 20 from 5 to 35° at a rate of 2°/min. Degree of crystallization was calculated by dividing the crystalline by the total portion of crystalline and amorphous phase.

2.6. Thermogravimetric and differential thermal analysis

Thermal stability of the composites was determined by TGA-DTA, using a Linseis Type STA PT 1600 TGA-DTA analyzer equipped with nitrogen as purge gas. Approximately 20 mg of samples were grinded and put through a 60-meshed strainer, then put in a sample pan. Heating was done at 10°C/min.

Figure 1. Zalacca: (a) plants; (b) midrib and fibers
heating rate from room temperature to 600°C, meanwhile the samples were simultaneously weighed. Analysis was done on LDPE-zalacca fibers composites compared to neat LDPE.

2.7. Tensile test of composites

Static mechanical properties, like tensile strength, modulus of elasticity and strain at fracture of composites, were determined by tensile tests, in accordance with ASTM 638-02 standard. Testing was done using a JTM-UTS210 universal testing machine equipped with a 2000 kg-load cell and its cross-head speed was set at 5 mm/min. Composite specimens had 50 mm gauge length and both of their ends were gripped by the clamps on upper and lower crosshead of the testing machine. For each fiber loading variation, five specimens were tested. Tensile strength was calculated by dividing the maximum applied force by the cross-sectional area of specimen, as shown in Equation (2). Samples being fractured outside the gauge length were not considered into analysis.

\[ \sigma_u = \frac{F_{\text{max}}}{A} \]  

in which \( A \), \( F_{\text{max}} \), dan \( \sigma_u \) refer cross-sectional area of a specimen, maximum applied force and tensile strength, respectively. Cross-sectional area of a specimen was calculated by equation (3).

\[ A = wt \]  

in which \( t \) and \( w \) are specimen thickness and width of narrow section, respectively. Strain at fracture was determined by dividing elongation by initial length of specimens, while modulus of elasticity was calculated by dividing the stress by the strain in linear region of the stress-strain curve.

2.8. Scanning electron microscopy

Failures in tensile specimens of the composites were observed by SEM using a FEI Type Inspect S50 instruments. Samples were settled on a silver-painted holder after gold-coated using low-vacuum sputtering process then put into the SEM chamber.

2.9. Flexural test of composites

Flexural strength and modulus of elasticity in bending as flexural characteristics of the composites were determined with flexural test using three-point bending method according to ASTM D790-02 standard, which was carried out using the same equipment for tensile testing. Flexural strength and modulus were calculated using Equations (4) and (5), respectively.

\[ \sigma_f = \frac{3Fl}{2bd^2} \]  

\[ E_B = \frac{l^3m}{4bd^3} \]  

in which \( \sigma_f \), \( E_B \), b, d, F, L, and m express flexural strength, modulus of elasticity in bending, width of beam tested, depth of beam tested, applied force, span of two supports, and slope of the tangent to the initial straight-line portion of the load-deflection curve, respectively. Specimen was laid on two supports having a 52 mm-span. Loading nose speed was determined using equation (6).

\[ R = \frac{zl^2}{6d} \]  

in which \( R \) and \( Z \) represent rate of crosshead motion and rate of straining of the outer fibers, respectively. Actual crosshead speed should not deviate by more than ±10%.

2.10. Dynamic mechanical analysis of composites

Visco-elasticity characteristic of materials was determined with dynamic mechanical analysis (DMA) using sinusoidal strain then the stress as response was measured [28]. Visco-elastic materials, like composites, show a combination of elastic and viscous properties, represented by storage and loss
modulus and being proportional to energy stored and dissipated by the cycle, respectively [29]. Ratio between loss and storage modulus is named as loss factor, generally known as damping, and is represented in equation (7).

\[
\tan \alpha = \frac{E''}{E'}
\]

in which \(E'\) and \(E''\) express storage and loss modulus, respectively.

DMA was performed with dynamic bending method at a frequency range of 0–100 Hz at room temperature. A Mettler Toledo DMA SDTA861E was the equipment used.

3. Results and Discussion

3.1. Density of composites

The theoretical and experimental density of LDPE-zalaca fiber composites are shown in Figure 2. The theoretical density is calculated using equation (1) with LDPE matrix and zalacca fiber density are 920.9 and 600.9 kg/m³ [30], respectively. This theoretical density of LDPE is in the range of 910-925 kg/m³ for the LDPE density according to Holbery and Houston [31]. Composite density decreases with the addition of the fibers due to lower fiber density than that of LDPE matrix.

Measurement of composite density is performed with maximum fiber loading of 50 wt. % due to the imperfect composites obtained with the higher fiber addition. The increasing of fiber content from 10 - 50 wt. % lowers their average density to 857.78 - 728.80 kg/m³. It designates that their density is decreased by 6.85 - 20.86%.

Compared to the theoretical density, the average measured density of the composite has the slightly lower value. It is due to the absence of void in theoretical calculation. There are data of measured density being higher than that of the theoretical one is caused by the change of fibers dimension after hot-pressing process.

3.2. X-ray diffraction analysis

The diffractograms resulted from XRD analysis of neat LDPE and LDPE-zalacca fibers composites are indicated in Figure 3. Meanwhile the obtained degree of crystallinity is shown in table 1.
Table 1. Degree of crystallinity of LDPE-zalacca fibers composites

| Composites         | 20° for (110) | 20° for (200) | Degree of Crystallinity |
|--------------------|---------------|---------------|-------------------------|
| Neat LDPE          | 21.35         | 23.53         | 0.47                    |
| LDPE-10 wt.% ZF    | 21.41         | 23.55         | 0.46                    |
| LDPE-20 wt.% ZF    | 21.515        | 23.69         | 0.48                    |
| LDPE-30 wt.% ZF    | 21.49         | 23.69         | 0.31                    |
| LDPE-40 wt.% ZF    | 21.41         | 23.45         | 0.33                    |
| LDPE-50 wt.% ZF    | 21.47         | 23.57         | 0.44                    |

Figure 3. X-ray diffractometer of LDPE-zalacca fibers composites

There are three prominent features in the neat LDPE and LDPE-zalacca fiber spectra, namely, a broad halo and two peaks. Amorphous phase of the LDPE matrix is depicted by the halo and the crystalline phase is indicated by two peaks representing the plane of <110> and <200>. Figure 3 reveals that the sharpest peak of all curves is acquired by neat LDPE. The lower crystalline peaks are due to the addition of zalacca fiber causing superposition of LDPE and fiber spectrum [19]. Degree of crystallinity is determined by dividing the crystalline portion of the area under two crystalline peaks by total area under two crystalline peaks and a broad halo. Addition of zalacca fibers does not significantly alter the degree of crystallinity as the fibers do not induce the polymer molecules to be straightened and occupy their positions in the crystal lattice.

3.3. Thermogravimetric-differential thermal analysis

The results of thermogravimetric and differential thermal analysis can be expressed as TGA and DTA curves, as shown in Figures 4 (a) and (b), respectively. Figure 4 (a) indicates that there is a slightly decrease in the weight of LDPE-zalacca fibers composite all along heating from room temperature to 200°C, compared to neat LDPE. It is caused by the elimination of water content from the fibers around 100°C. The weight reduction also takes place at higher temperatures up to 350°C due to the hemicellulose and cellulose decomposition in zalacca fibers [18]. This is confirmed by the portion of DTA curve having reverse peaks, indicating that endothermic reactions occur in relation to the evaporation of water, and decomposition of hemicellulose and cellulose, Figure 4 (b). This case resembles to the study of Arrakhiz and co-workers [32] for doum fiber-reinforced LDPE composites.
Compared to neat LDPE, zalacca fiber content slightly lowers the thermal stability of composites, as shown in Figure 4 (a). Meanwhile, the DTA curve, Figure 4 (b), also indicates that the fiber addition shifts a bit the curve to the left.

3.4. Tensile testing of composites

The tensile strength, modulus of elasticity and strain of fracture resulted from the tensile testing of the composites are shown in Figures 5 (a), (b) and (c), respectively. The average of obtained tensile strength, elastic modulus and strain of fracture of neat LDPE are 8.85 MPa, 35.55 MPa and 746.0 %, respectively. Meanwhile, the average tensile strength, elastic modulus and fracture strain of zalacca fiber are 182.12 MPa, 3.36 GPa and 5.58 % [33], respectively. The average of interfacial shear strength between zalacca fiber and LDPE matrix is 12.58 MPa [30].

Figure 4. (a) Thermogravimetry analysis curve; (b) Differential thermal analysis curve

Figure 5. (a) Tensile strength; (b) elastic modulus; and (c) strain at fracture of LDPE-zalacca fibers composites
Addition of zalacca fibers to the composites enhances their tensile strength and elastic modulus compared with those of neat LDPE, as shown in Figures 5 (a) and (b). It is caused by higher tensile strength and elastic modulus of zalacca fibers [33] than that of LDPE matrix, as a result of their high cellulose content [19] which exceeds that of coir [6] and wheat straw fiber [34]. The fiber content of 10% does not increase the tensile strength or even decreases it so that the tensile strength of composites is lower than that of the matrix. It is caused by the poverty of load transferred from LDPE matrix to the fibers due to the insufficiency of fiber content. It is appropriate to the study of Gibson [27]. The addition of 10 wt. % zalacca fibers significantly raises the elastic modulus caused by zalacca fibers having higher stiffness. The higher σ_u and E of the composites with fiber content of 20 and 30 wt. % are caused by the larger load transferred to the fibers so that they can partly bear the load, as well as the LDPE matrix. The fiber content of 40 wt. % results in the maximum value of σ_u and E. This is the condition in which maximum load can be transferred from the matrix and defended by the fibers due to the sufficiency of interfacial adhesion between the fibers and LDPE matrix. Further fiber loading causes imperfect bonds between the matrix and fibers in such a way that the external load cannot be transferred adequately. This is proved by the SEM examination (Figure 6).

The fiber content of 10 wt. % decreases the strain of fracture of the matrix drastically, Figure 5 (c). It is caused by the existence of little fibers, which plays role as defects due to their inability to receive the transferred load. The higher fiber of the composites loading results in their lower strain of fracture. Nevertheless, the next fiber addition does not significantly change it due to the lower zalacca fibers strain of fracture [33] than that of LDPE [31]. In terms of the fiber as a load supporter, fracture of the composites is mainly caused by the breakage of fibers. Contrasted to the study of Agunsoye and co-workers [35] using coconut shell particles as reinforcements for LDPE matrix, the zalacca fiber reinforced composites are significantly superior. The LDPE panels has a higher σ_u and E without the coconut shell particles addition. Their tensile strength is inversely proportional to the addition of particles. Meanwhile, the content of 5 wt. % coconut shell particles results in the drop of its elastic modulus, but the higher addition proportionally increases it. This is because of the shorter coconut shell particles than the critical length. Therefore, the particles cannot support the transferred load from the matrix. This case is also similar to the study on mechanical properties of LDPE-matriced composites reinforced by date palm wood powder by AlMaadeed and co-workers [36].

The tensile strength of zalacca fiber reinforced LDPE composites is higher than that of doum fiber reinforced composites [32], which shows a decline after the fiber loading. It is due to the irregular dimension and shape of doum fibers having average 1380 µm length and 138 µm diameter. The lower length than its critical value causes the limited load beared by the fibers.

The mechanical properties of LDPE-zalacca fiber composites are proportionate to those of LDPE-corn husk fiber composites [37]. The corn husk fiber reinforced composite is slightly stronger and attains its highest strength at 5 wt. % fiber content. Meanwhile, the elastic modulus of zalacca fiber-reinforced composites is higher and achieves its peak at 40 wt. % fiber addition.

3.5. Scanning electron microscopy
The SEM analysis indicates the fracture characteristics of composites with a fiber content of 10, 20, 40 and 50 wt. %. The images are obtained from the fractured specimens after tensile testing.

Figure 6 (a) for LDPE composite with 10 wt. % fiber content, shows several fiber pull-outs and a little amount of fibers. Fiber pull-outs are defined by fiber-printed holes on the matrix. It signifies that the fibers play no convincing role in reinforcing the composites as they do not well retard the movement of the matrix due to the external applied load. This is proved by the low value of composite tensile strength, as shown in Figure 5 (a). Else, the presence of fibers is similar to defects in LDPE matrix which reduce its tensile strength.

In LDPE-zalacca fiber composite with a fiber loading of 20 wt. %, as shown in Figure 6 (b), there are some fiber pull-out and fractures. These reveal that the fibers make significant contribution in bearing the load delivered by LDPE matrix in addition to restricting the matrix strain. The fiber pull-outs refer that the interfacial adhesion between matrix and fibers is not so strong that it breaks up
before the fibers can effectively detain the load. Fiber fractures show that the load is hold back by the fibers until they break. It indicates that the reinforcement is so adequate that the load borne is larger than that of the composite with 10 wt. % fiber content. Therefore, the strength increases, as shown in Figure 5 (a).

Figure 6 (c) shows the fracture of composite with a fiber loading of 40 wt. %. There are almost no fiber pull-outs but several fiber fractures are existing. Fiber fractures indicate that almost all load is borne by the fibers after being transmitted by the matrix. Nearly no fiber pull-outs indicate that the external load has been effectively transferred from matrix to the fibers. Hence, its tensile strength and elastic modulus are highest of all the composites, as shown in Figure 5 (a) and (b).

The fracture characteristic of LDPE-zalacca fiber composite with fiber loading of 50 wt. % is indicated in Figure 6 (d). This reveals that there are imperfect bonds between fibers and matrix due to high amount of fibers, thereby causing poor load transfer from LDPE matrix to the fibers. It causes the fibers are not properly bound by the matrix. Therefore, they are unable to bear the load simultaneously. Hence, the tensile strength and modulus of elasticity are smaller than those of the composite with 40 wt. % fiber loading.

![Figure 6](image)

**Figure 6.** Scanning electron microscopy of fractured LDPE matriced composites with the addition of: (a) 10 wt. %; (b) 20 wt. %; (c) 40 wt. %; and (d) 50 wt. %

3.6. Flexural testing
Figures 7 (a) and (b) show the flexural strength and modulus of LDPE-zalacca fiber, respectively. These histograms indicate that the $\sigma_f$ and $E_B$ increases almost proportionally with the fiber content up to 40 wt. % before decreasing. This is correlated to increased amount of the fibers in LDPE matrix that retard the matrix strain and support it in detaining bending load. Like in tensile testing, the load is transmitted to fibers by the matrix.

Zalacca fiber reinforced composites have some advantage over LDPE-doum fiber composites [32] in terms of the flexural modulus, in which at 20 wt. % fiber content, they have higher flexural modulus than the maximum value of the doum fibers reinforced composites. After 20 wt % fiber addition, the flexural modulus of LDPE-doum fibers composites tends to decline, while $E_B$ of LDPE-zalacca fibers composites consistently increases. This confirms that LDPE-zalacca fibers composites have higher stiffness than that of doum fibers.

The flexural strength of LDPE-zalacca fiber composites is higher than that of bio-epoxy reinforced by powdered coconut shells, rice husks and walnut shells [38]. It is because the powdered reinforcements render the load transferred ineffectively. LDPE-zalacca fiber composites perform more than polyester-matriced composites reinforced by the midribs of coconut palm leaves [39]. A fiber content of 10 wt. % is required in inducing maximum flexural strength in those composites, while a fiber addition of 40 wt. % is requested for zalacca fiber composites due to LDPE matrix having tough and ductile characteristics.
3.7. Dynamic mechanical analysis

The DMA results of LDPE-zalacca fiber composites are storage or elastic modulus \( (E') \), loss or viscous modulus \( (E'') \) and loss factor \( (\tan \delta) \), as shown in Figures 8 (a), (b) and (c), respectively. From those curves, the composites have natural frequency received at ±40 Hz. Figures 8 (a) and (b) indicates that the storage and loss modulus raise nearly proportional with the fiber content, especially in lower frequency. Meanwhile, the loss modulus and loss factor increase with higher frequency, as revealed in Figures 8 (b) and (c), whereas the storage modulus decreases. It means that in general, viscous properties of composites are prominent in high frequency. Especially for a frequency of 0 Hz, the storage modulus matches its flexural modulus (Figure 7), due to the dynamic bending method of the test. The loss factor is decreased with the addition of fibers, as indicated in Figure 8 (c). This is attributed to stiffer composites resulted from higher fiber content, so that energy from the applied load is stored. It is confirmed by the study of Etuati and co-workers [40] on dynamic mechanical analysis of short hemp reinforced PP composites, and Pearson and Naguib [41] on DMA of polyurethane reinforced with alumina, Kevlar, and PBO short fiber. Undissipated energy will induce excessive vibrations, bringing on fatigue fracture [40]. However, the reduction in damping is not significant for fiber content of 10-40 wt. %, as indicated in Figure 8 (c).

![Figure 8. Dynamic mechanical analysis results of LDPE-zalacca fibers composites: (a) storage modulus; (b) viscous modulus; and (c) loss factor](image)

4. Conclusion

There is a significant decrease of the composite density caused by the addition of zalacca fibers due to the lower density of fiber than that of LDPE matrix. On the contrary, there is insignificant change in the LDPE matrix crystallinity after the fiber addition. Addition of zalacca fibers reduces the thermal stability of LDPE-zalacca fiber composites due to the lower decomposition temperature of zalacca fibers compared to that of LDPE. Reinforcement is effective up to 40 wt. % fiber content inducing higher tensile strength and elastic modulus. Higher addition of zalacca fibers causes incomplete bonds between LDPE matrix and the fibers. The similar case is obtained for the flexural characteristics, such as flexural strength and modulus. Storage and viscous modulus are increased, but loss factor declines...
with the fiber addition. In general, zalacca fiber addition improves mechanical characteristics of LDPE-zalacca fibers composites.

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