Improving Thermal and Mechanical Properties of Injection Moulded Kenaf Fibre-reinforced Polyhydroxybutyrate Composites through Fibre Surface Treatment

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Ecofriendly biodegradable composites were developed to mitigate the impact they have on the environment. Composites made from completely biodegradable materials were prepared using kenaf fibres and polyhydroxybutyrate (PHB). The composites were mixed using a twin screw extruder and injection moulded into dumbbell-shaped specimens. The composites were subjected to thermal analysis *via* differential scanning calorimetry (DSC) and mechanical testing (tensile and flexural tests). The addition of alkali-treated kenaf fibre reduced the crystallinity of PHB (up to 6% reduction), making it more ductile. The rheological behaviour of PHB was modified with the addition of kenaf fibres. Additionally, the flexural modulus was improved by up to 11% with the addition of kenaf fibres, for both the treated and untreated kenaf fibres. However, it was also observed that to improve fibre matrix adhesion, an alkali treatment alone is insufficient, as shown by the 16% reduction of tensile strength in composites with 20% fibre loading.

Keywords: Biodegradable; Polyhydroxybutyrate; Rheology

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INTRODUCTION

The need for more energy-efficient vehicles has increased the use of polymer composites in the transportation industry. Polymer composites are used to produce parts such as car bumpers and interior furnishings (Holbery and Houston 2006). Composites have high strength, high stiffness, and low density, which allows the fabrication of relatively lighter parts but with similar or superior strength compared to more traditional materials (Saba *et al.* 2016). However, because polymer composites are usually comprised of two or more components, it is cost-intensive to separate the components for recycling purposes (Pickering *et al.* 2016). Currently, the most popular method for disposing of polymer composites is *via* landfill and incineration, and both methods impact the environment in a significant way. The main issue is that the traditional polymers used as a matrix in composites, such as polyethylene and polypropylene, are not biodegradable. In addition, the production of these polymers is dependent on petroleum, a non-renewable resource.

To solve this problem, a better, alternative composite can be produced by substituting the components in the composites with materials that have lower environmental impact. These materials are often referred to as green composites and are usually made from natural fibres as the reinforcement and bioplastics as the matrix. Bioplastic, such as polyhydroxybutyrate (PHB), is derived from renewable sources and is biodegradable (Poirier *et al.* 1992). The ability to naturally degrade does come with some trade-offs. For PHB, it is extremely brittle, which limits its applications. Improvements can be made by introducing reinforcements in the form of natural fibres or by blending with other types of polymers or plasticisers (Yusoff *et al.* 2016; Joyyi *et al.* 2017).

Kenaf is the cheapest and most commercially available of the natural fibres. Several researchers have found that kenaf bast fibres have great promise as a reinforcement component in polymer composites. However, untreated kenaf fibres have poor interfacial adhesion with the matrix, producing composites with subpar mechanical properties as well as a wetting problem at higher fibre content (El-Shekeil et al. 2012; Park et al. 2015; Wang et al. 2016). The interfacial adhesion can be improved via fibre surface treatment. Surface treatment can be in the form of physical treatment, such as heating, or chemical treatment, for example an alkali treatment. Asim et al. (2017) reported that the overall mechanical properties of kenaf and pineapple leaf fibre-reinforced phenolic formaldehyde composite are improved by silane treatment. Additionally, the product is suitable for applications in the automotive industry and building structures (Asim et al. 2017). Ariawan et al. (2017a) investigated the wettability of kenaf fibres through surface energy using the Washburn contact angle principle and found that the alkaline treatment improved the wettability of the fibres, with fibres that soaked for 3 h exhibiting the highest interlaminar shear strength value. They also reported increased fibre-matrix contact area on the treated kenaf fibres through atomic force microscopy. In another paper, it is reported that composites fabricated using alkali treated fibres exhibit better durability in terms of flexural properties after the composite is subjected to 12 months of a natural weathering environment (Ariawan et al. 2017b). Orue et al. (2016) reported that the mechanical properties of sisal fibre and polylactic acid (PLA) composites are improved by alkaline and silane fibre surface treatment. It was suggested that the improved fibre-surface interaction between the treated fibres and PLA was responsible for the increases in tensile strength.

Currently, extensive studies on the material properties of the biocomposite from plant fibres and PHB are relatively limited. In this work, the influence of the fibre surface treatment on the mechanical, rheological, and thermal properties of kenaf-reinforced PHB composites was investigated. The fracture surface of the composite was also studied *via* scanning electron microscopy (SEM).

EXPERIMENTAL

Materials

The PHB pellets were supplied by Tianan Biologic Materials (Ningbo, China). Kenaf fibre bales were supplied by the National Kenaf and Tobacco Board (Kota Bharu, Malaysia) and processed into 3-mm short fibres by the Malaysian Agricultural Research and Development Institute (Serdang, Malaysia). Sodium hydroxide pellets were supplied by Merck (Kenilworth, NJ, USA).

Preparation of composites

Two types of fibres were used to prepare two different composites: untreated and alkali-treated kenaf. The composites were first mixed using a Brabender Ketse 20/40 (Duisburg, Germany) twin screw extruder with a processing temperature range from 180 to 190 °C and screw speed at 80 RPM. The extruded composites were cut into pellets using

a Pelletizer (Brabender, Duisburg, Germany) and fed into a BOY 55M injection moulding machine (DrBOY, Neustadt-Fernthal, Germany) and moulded dumbbell-shaped specimens according to ASTM D638 (2010). The barrel temperature zones were set at 180 °C, 180 °C, and 185 °C from back to front. The nozzle temperature was set at 185 °C. The screw speed was maintained at 50 RPM. The injection pressure was set at 45 to 55 bar and 50 s holding time. The untreated fibre-reinforced composite was designated as UT, and the alkali-treated kenaf-reinforced composite was designated as CT. These acronyms are followed by a number referring to its fibre weight fraction (5%, 10%, or 20%).

Methods

Thermogravimetric analysis (TGA)

The thermal stability of the composites was studied using a Perkin Elmer TGA 6 (Waltham, USA) thermogravimetric analyser. For each sample, 5 to 7 mg of the fibre was placed in ceramic pans and heated from 50 to 900 °C under nitrogen (flow rate: 20 mL/min) atmosphere at a heating rate of 10 °C/min.

Differential scanning calorimetry (DSC)

A Perkin Elmer DSC6 (Waltham, USA) was used to study the phase transitions in the composites. Approximately 5 mg of specimen was used for the analysis. A typical DSC run would consist of three steps. The first step involved heating from 50 °C to 200 °C. Then, the sample was cooled to 50 °C and held for at least 1 min. The final step involved heating from 50 °C to 200 °C. All of these steps were performed under controlled conditions, with a heating and cooling rate of 10 °C/min. The first step was taken as a precaution to remove the thermal history of the materials. The melting temperature (T_m) and melting enthalpy (ΔH_m) were determined from the second heating run.

Rheology

The rheological behaviour of the composites under oscillatory shear flow was investigated using an Anton Paar MCR 301 rheometer (Anton Paar, Graz, Austria) with a CTD450 attachment. A parallel plate with a 25-mm diameter and 1-mm gap was used. A dynamic frequency sweep of the sample was conducted with 1% strain amplitude and an angular frequency ranging from 0.5 to 500 rad/s at 180 °C.

Tensile properties

Tensile tests were performed using an Instron 5569 Universal Testing Machine (Canton, NJ, USA) equipped with a 50 kN load cell and a mechanical extensometer (Instron, Canton, NJ, USA) at a cross-head speed of 10 mm/min⁻¹. The specimen dimension and shape was in accordance with ASTM D638 (2010). The tests were performed at room temperature and the extensometer jaw grip was set at 50 mm. A minimum of seven specimens per batch were tested to obtain the best reproducible results. For each batch of specimen, the average values of Young's modulus (*E*), tensile strength, and tensile strain were calculated by the Bluehill software (Instron, version 2.24, Canton, NJ, USA) from the stress over strain curve obtained. The *E* was calculated at 0.5% strain.

Flexural properties

Flexural tests were performed using an Instron 5569 universal testing machine (Canton, NJ, USA). The distance between the support spans (L) were 50 mm and the cross-

head speed was 1.31 mm/min. The speed of the cross-head motion (R) was calculated by using Eq. 1,

$$R = \frac{ZL^2}{6d} \tag{1}$$

where L and d are the specimens that support span (mm) and depth (mm), respectively, and Z is the rate of straining equal to 0.01. The test was conducted at room temperature. The specimen shape and dimension was in accordance with ASTM D790 (2010). Five specimens per batch were tested and the averages were recorded. The flexural modulus and strength were calculated by the Bluehill® software (Instron, version 2.24, Canton, NJ, USA) from the flexural curve obtained.

Field emission scanning electron microscopy (FE-SEM)

The fractured surface was examined using a Hitachi Field Emission Scanning Electron Microscope model SU8220 (Krefeld, Germany).

RESULTS AND DISCUSSION

TGA

The thermal degradation behaviour of the composites and the influence of fibre surface treatment were studied using TGA. The TGA curve of PHB and the treated kenaf composites are provided in Fig. 1.



Fig. 1. TGA curve for PHB and treated kenaf composites

The PHB exhibited a single step degradation between 240 and 320 °C. The sharp incline suggested that the degradation rapidly occurred. In contrast, the composite displayed two-step degradation. The first step was similar to pure PHB, containing a sharp incline, occurring between 230 and 320 °C, and it was attributed to the degradation of the matrix. The second step was much slower and occurred between 320 and 600 °C. The second transition was more prominent in the samples with a higher fibre content and was attributed to degradation of kenaf fibres. The onset temperature (T_{onset}), $T_{50\%}$, and derivative peak temperature (DT_p) were calculated from the curve and tabulated in Table 1.

Sample	T _{onset} (°C)	<i>T</i> _{50%} (°C)	DT _p (°C)	
PHB	298.1	310.0	314.2	
UT5	286.2	297.3	300.8	
UT10	288.2	296.7	299.5	
UT20	279.6	288.0	289.7	
CT5	284.3	296.4	298.6	
CT10	279.8	292.2	295.0	
CT20	273.5	283.7	284.6	
PHB – polyhydroxybutyrate; UT – untreated fibre composite; CT – alkali treated fibre composite				

Table 1. Onset Temperature (T_{onset}), $T_{50\%}$, and Derivative Peak Temperature (DT_p) for Kenaf-reinforced Composites

In general, kenaf-reinforced PHB composites, regardless of the treatment, had a lower thermal stability compared to pure PHB. For example, the T_{onset} for PHB, UT20, and CT20 were 298.1 °C, 279.6 °C, and 273.5 °C, respectively. A lower onset temperature indicated that the material began to degrade at a lower temperature. A similar trend was observed for $T_{50\%}$ and DT_p . It was observed that the thermal stability of the composite decreased with increasing kenaf fibre content, for both the untreated and treated fibres. In addition, it was observed that the thermal stability of untreated kenaf-reinforced composites were higher than their treated counterparts. It was shown that the alkali treatment stripped the lignin from the outer layer of the fibers (Hassan *et. al.* 2018). It was possible that there was an interaction between the exposed cellulose portion of the treated kenaf fibres and the PHB at elevated temperature, which led to a lower thermal stability.

DSC



Fig. 2. DSC thermogram for PHB and treated kenaf composites

Information obtained through DSC would improve the understanding of the phase changes (*e.g.*, melting) that the composite went through in the temperature range studied. The mechanical properties of the composite were influenced by the molecular structure of the matrix (crystalline structure, orientation of amorphous regions), especially the degree of crystallinity. The DSC thermogram for the PHB and treated kenaf composites is provided in Fig. 2.

Important parameters that were studied, such as melting temperatures (T_m) , enthalpy heat of melting (ΔH_m) , and the degree of crystallinity (X_c) , for the composites are given in Table 1. The X_c was calculated using the following equation,

$$X_c = \frac{\Delta H_m}{\Delta H_m^*} \times 100 \tag{2}$$

where $\Delta H_{\rm m}^*$ is the enthalpy heat of fusion for an 'ideally' fully crystalline PHB, taken as 146 J/g (Barham *et al.* 1984). The values provided in the table have been normalized according to the actual PHB content in the composite.

Table 2. Melting Temperature (T_m), Enthalpy of Fusion (ΔH_m), and Degree of Crystallinity (X_c) for Kenaf-reinforced PHB Composites

Sample	T _m (°C)	Δ <i>H</i> _m (%)	X _c (%)
PHB	175.1	73.9	50.6
UT5	166.1	78.3	56.4
UT10	169.0	71.2	54.2
UT20	169.0	63.3	54.2
CT5	172.7	61.9	44.7
CT10	172.3	63.5	48.4
CT20	169.4	60.2	51.5

The thermogram obtained from DSC showed a single endothermic peak, which was attributed to the melting of PHB. As shown in the table, the addition of kenaf fibres slightly reduced the melting temperature of PHB. For example, the T_m for neat PHB and UT5 were 175.1 and 166.1 °C, respectively. Melting temperature is influenced by the intermolecular forces and the packing of the crystals. If the crystal is closely packed, there will be more intermolecular forces holding the crystals together. These minor bonds need to be broken to melt, hence a higher T_m value. However, the presence of the kenaf fibres in the composite disrupted the packing of the crystals, reducing the intermolecular bonds and thus lowering the T_m . In contrast, when the fibre weight fraction was increased, no clear trend regarding the T_m was observed regardless of the fibre type used.

The introduction of untreated kenaf fibres into PHB increased its X_c . For example, the X_c for neat PHB and UT5 was 50.6% and 56.4%, respectively. It has been reported that fibres can promote nucleation along the fibre matrix interface resulting in increased X_c (Yang *et al.* 2014). However, increasing the fibre weight fraction of heat-treated fibres from 5% to 20% caused the X_c to decrease from 54.7% to 50.8%. In addition, introducing alkali-treated kenaf fibres caused the X_c to decrease. For example, X_c for CT10 was 48.4%, which was lower than neat PHB (50.6%). Lower X_c indicated that the composite had a higher amount of amorphous region that could mean it was more ductile. This increased ductility could have been beneficial for its mechanical properties because neat PHB is brittle.

Rheology

The flow of composite melt is important and can be influenced by factors such as temperature, pressure, screw speed, filler content, and die size. Information from rheology can reflect the internal structure and processability of the melt. Flow behaviour is important during compounding and injection moulding, especially to understand and optimise the mould filling process. Information, such as the storage modulus (*G*'), loss modulus (*G*''), and complex viscosity (η^*), were obtained from a dynamic frequency sweep of the composite sample at constant amplitude and temperature. Figure 3 illustrates the complex viscosity curve as a function of angular frequency for the kenaf-reinforced PHB composites for both the treated and untreated fibres and different fibre loadings.



Fig. 3. Complex viscosity-angular frequency behaviour of untreated and treated kenaf PHB composites at three different fibre loadings

From Fig. 3, it can be observed that for neat PHB, UT5, and CT5, the complex viscosity (η^*) slightly increased with increasing angular frequency (ω). However, above 10 rad/s⁻¹ the melts exhibited Newtonian fluid behaviour. For UT10 and CT10, the melts exhibited Newtonian fluid behaviour throughout the tested ω range. However, for UT20 and CT20, the composite melts displayed a shear thinning behaviour, where the η^* decreased with increasing ω . These behaviours indicate the transformation from solid-like behaviour at lower ω to fluid-like behaviour at higher ω (Salleh *et al.* 2014). At higher fibre loading, the presence of fibres affected the normal melt flow and hindered the mobility of the polymer chain. In addition, the fibre-fibre collision compounded this effect. However, at higher frequency, sufficient force was subjected to the fibres, aligning the fibres with the flow direction and greatly improving its flowability. It was also observed that increasing the fibre content increased the η^* . Composites reinforced with treated kenaf fibres also exhibited higher η^* for all fibre loadings, an indication of the fibre and matrix having improved interaction. Stronger interaction between the fibre and matrix obstruct the flow of the matrix, hence increasing the viscosity (Chun *et al.* 2015).

The storage and loss modulus for neat PHB and all the composites are illustrated in Figs. 4 and 5.



Fig. 4. Storage modulus-angular frequency behaviour of untreated and treated kenaf PHB composites at three different fibre loadings

Both the storage modulus (G') and loss modulus (G") for neat PHB and all the composites increased with increasing ω . The G' of the composite melted at a lower ω and was heavily influenced by the kenaf fibre content. The composite melt exhibited a plateau at a lower ω and the magnitude of the plateau increased with increasing kenaf fibre content. The increase was attributed to the increasing rigidity of the melt due to the addition of fibres. The G" also displayed a similar trend; however, the plateau was only visible in composites with 20% fibre loading. At lower fibre loading (5% and 10%), the melt behaved as a Maxwell fluid, a typical behaviour for thermoplastic melts. For both G' and G", the CT composites exhibited higher values.



Fig. 5. Loss modulus-angular frequency behaviour of untreated and treated kenaf PHB composites at three different fibre loadings

Tensile Properties

The tensile properties of the untreated and treated kenaf-reinforced PHB composites were tested. The results (tensile modulus and tensile strength) of kenaf-reinforced PHB composites are illustrated in Fig. 6.



Fig. 6. Young's modulus and tensile strength of kenaf-reinforced PHB composites

The Young's modulus and tensile strength of neat PHB were recorded at 1.95 GPa and 35.6 MPa, respectively. The Young's modulus increased with the addition of treated and untreated kenaf. For example, the Young's modulus for UT10 and CT10 were 2.21 GPa and 2.12 GPa, respectively. This indicated that the incorporation of kenaf fibre into the PHB increased its stiffness.

The effect of fibre content on the Young's modulus was studied. For both the treated and untreated kenaf-reinforced composites, increasing the fibre content increased the Young's modulus. For example, the Young's modulus for CT increased from 1.97 GPa to 2.36 GPa when the fibre content was increased from 5% to 20%. Ibrahim *et al.* (2009) also reported similar observation. The researchers found that increasing the fibre content increased the tensile modulus, up to a certain optimal fibre loading. As the fibre was further increased beyond the optimal value, the modulus decreased due to poor wettability resulting in lower stiffness.

Fibre surface treatment with alkaline media is expected to improve the mechanical properties of the composites. In theory, the surface treatment is expected to produce a cleaner and rougher surface where the matrix and fibre can bind and interlock, producing composites with superior mechanical properties from its untreated counterpart (Fiore *et al.* 2015). However, in this study, it was found that the modulus for composites prepared with treated kenaf fibres had a lower modulus compared to the composites prepared with untreated fibres. The modulus for UT20 and CT20 were 2.68 GPa and 2.36 GPa, respectively. There were several explanations for this observation. Firstly, from the DSC, it was found that the crystallinity of the composites prepared with the treated fibres were lower than the composites prepared with the untreated fibres. Materials with lower crystallinity are more ductile and will record a lower stiffness during tensile testing.

Another possibility was increased fibre breakage during processing. It was found from prior analysis that surface treated kenaf was more crystalline and brittle compared to untreated kenaf. This increased brittleness may have caused the fibre to break more during processing due to the fibre-matrix, fibre-fibre, and fibre-machinery interactions. Increased fibre breakage would result in shorter fibres, which are less efficient at reinforcing the composites due to an increased number of fibre ends (Hassan *et al.* 2011). The treated fibre surface was also rougher, which led to greater fibre-fibre friction.

The tensile strength of the composite was lower than the neat PHB. For example, the tensile strength for neat UT10 and CT10 were 31.37 MPa and 27.44 MPa, respectively, which were lower than the tensile strength of neat PHB (35.6 MPa). Due to the introduction of kenaf fibres, the composites became less ductile as the molecular rearrangement were constrained by the fibres. The notching effect of the fibres is also important because considerable stress concentration is induced in the matrix at the fibre end and matrix flow constrained by adjacent fibres (Takahashi and Choi 1991).



Fig. 7. Fracture surface of UT5 (a) and CT5 (b) captured using FESEM

The effect of fibre loading on the tensile strength of the composite was studied. For both the treated and untreated composites, the tensile strength decreased with increasing fibre weight fraction. For example, the tensile strength for CT decreased from 29.97 MPa to 26.28 MPa as the fibre weight fraction increased from 5% to 20%.

The reduction in tensile strength was attributed to the increasing fibre breakage during processing at higher fibre loading. It could also have been due to a wetting problem at a higher fibre content. The modulus was not affected by these flaws because it was evaluated at very low strain. In the lower strain region of the testing, the flaws discussed above have yet to manifest. The fracture surface of the treated and untreated composites is illustrated in Fig. 7.

It was observed that there was no improvement regarding the fibre matrix adhesion. There was a clear separation between the fibre and the matrix (shown by arrows).

Based on the findings, it is suggested that an alkali treatment alone is not enough to improve the fibre matrix adhesion. The addition of compatibilisers or the introduction of functional groups onto the fibre surface is necessary to achieve better fibre-matrix adhesion.

It can also be observed that plastic deformation is absent on the fracture surface, indicating that the material underwent brittle failure. This is supported by the stress *vs.* strain curve for selected specimens illustrated in Fig. 8. After the yield point, there were no plastic deformation. The specimens abruptly breaks into two pieces.



Fig. 8. Stress vs strain diagram for selected specimens

Flexural Properties

The flexural properties of the untreated and treated kenaf-reinforced PHB composites were tested under a three-point bending setup. The flexural properties describe the ability of the material to withstand bending, which is important for load bearing applications. The results (flexural modulus and flexural strength) of kenaf-reinforced PHB composites are illustrated in Fig. 9.



Fig. 9. Flexural modulus and strength of kenaf-reinforced PHB composites

The incorporation of kenaf fibres into PHB improved its flexural modulus. The flexural modulus of neat PHB increased from 2.65 GPa to 2.93 GPa with the addition of only 5% w/w kenaf fibre. The improvement was even higher when the treated kenaf was added at the same weight fraction (F_w) (3.58 GPa). Neat PHB tends to fail catastrophically during flexural testing, with the sample breaking into two big pieces with smaller pieces shattering from the bigger pieces. However, this did not happen with the addition of kenaf fibres. As the fibre content further increased, the flexural modulus also increased. The flexural modulus for CT increased from 3.58 GPa to 4.29 GPa as the F_w value increased from 5% to 20%. The improvement for flexural properties was more noticeable compared to the improvement for tensile properties and was attributed to the nature of the injection moulded fibre-reinforced composites. In injection moulded specimens, the fibre orientation near the mould will align according to the flow direction of the polymer melt (Darlington and McGinley 1975). In contrast, the fibre in the middle section would be perpendicular to the flow direction. These behaviors are illustrated in Fig. 10, where the fibres in the bottom right corner were more aligned to the melt flow while the fibres in the top left corner were perpendicularly oriented. In tensile, these variations in fibre orientation would result in a poorer reinforcement effect, because the strain applied was uniform across the sample. However, for flexural testing, only the outermost layer (top and bottom) experienced the full force of the testing. Because the fibres in these regions were more oriented, the exhibited flexural properties were also higher.

The flexural strength of the neat PHB increased with the incorporation of kenaf fibres, from 54.7 MPa to 58.3 MPa (UT20). For untreated kenaf fibres, increasing the Wf improved the flexural strength of the composite. For example, the flexural strength improved from 54.6 MPa to 58.3 MPa when the F_w increased from 5% to 20%. The UT also exhibited slightly higher flexural strength compared to CT at a similar fibre loading. For example, the flexural strength for UT10 and CT10 were 56.9 MPa and 52.6 MPa, respectively.



1 mm

Fig.10. Fracture surface of UT20

CONCLUSIONS

- 1. The properties of kenaf-reinforced PHB composites were improved by the fibre surface treatment. Composites fabricated with the treated kenaf fibres exhibited improved melt properties and up to 21% improvement in the Young's modulus.
- 2. At 5 and 10% fibre loading, the rheological behaviour of the composites melt was Newtonian. At 20% fibre loading, the melt behaviour was non-Newtonian, exhibiting shear thinning behaviour. This can be attributed to the increased fibre-fibre interaction which interferes with the melt flow.
- 3. Minor reduction in the properties of the composite was observed. Thermal stability was reduced and tensile strength reduced by up to 16% for the treated kenaf composites, indicating poor fibre-matrix interaction.
- 4. Alkali treatment alone is insufficient for improving the fibre-matrix adhesion. Further fibre surface modification such as silane treatment or addition of compatibilisers into the matrix could enhance the properties of the composite.

ACKNOWLEDGMENTS

This work was funded by the Universiti Malaya (Grant No. PG162-2015A) and the Malaysian government through the MyBrain programme.

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Article submitted: September 6, 2018; Peer review completed: November 26, 2018; Revised version received: February 13, 2019; Accepted: February 14, 2019: Published: February 27, 2019.

DOI: 10.15376/biores.14.2.3101-3116