A Comparative Study on the Desiccant Effect of Polypropylene and Polylactic Acid Composites Reinforced with Different Lignocellulosic Fibres

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Awareness of the sustainability of the environment has inspired many researchers to explore the benefits of renewable resources like plant-based fibre, which then introduces the topic of the output of green products. The potential of polypropylene (PP) and polylactic acid (PLA) based composites with two types of natural fibres, palm oil empty fruit bunch (EFB) and microcrystalline cellulose (MCC), as a moisture absorbent or desiccant was investigated. PP and PLA composites with various contents of EFB and MCC fibres were prepared using an internal mixer followed by hot pressing. To evaluate their use in certain applications, moisture uptake was measured through water absorption testing. Experimental results indicated that moisture uptake influenced by fibre loading, size of fibre and type of matrix. PLA/MCC composites exhibit considerable moisture uptake compared to other composite samples. Qualitative moisture uptake measurement was also carried out by packing the composites and green chillies in plastic bags and storing them at ambient humidity and temperature. The efficiency of the composites as a desiccant was in agreement with the results of the water test. Therefore, the developed composites have the potential to be used as a desiccant.

Keywords: moisture content; oil palm fibres, microcrystalline cellulose, composite, characterization

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1. Introduction

The shelf life or storage period of food, particularly inside packaging, is affected by the moisture content. This is because the presence of moisture will induce the growth of bacteria or mould which degrades the food quality [1, 2]. Without proper moisture control, food with a high transpiration rate, such as vegetables and fruit, usually spoils easily [3]. Furthermore, the non-perforated packaging film, which is not water vapour- or gas-permeable, will lead to water condensation. This condition not only accelerates the growth of bacteria but also makes the appearance less attractive. Malaysia and other Asian countries possess climates with high temperatures and high humidity levels. These conditions could speed up the food spoilage process, especially of vegetables and fruit. As a consequence, these foods are rendered unacceptable for human consumption, leading to food loss. Sachets, film, trays and pads are the most common moisture absorbers or desiccants added into plastic packaging to increase the shelf life of food by controlling the humidity in the food packaging. Sachets which consist of desiccant materials such as silica gel, dry sorbitol, xylitol, sodium chloride, potassium chloride, and calcium chloride are available in the form of powder, granules, and beads. However, the use of this desiccant type can trigger consumer safety issues as the desiccants may be unintentionally consumed by youngsters. Another drawback of sachets is that they are normally more suitable for dried food [4].

The issue of environment sustainability has led researchers into developing a polymer composites-based natural fibre. To date, extensive research on the properties of thermoplastic materials i.e., polypropylene (PP) and polylactic acid (PLA), reinforced with various types of natural or lignocellulosic fibres, has been reported. Empty fruit bunches [5], kenaf [6], rice husk [7] and cellulose [8, 9] are among the natural fibres that have been studied in PP and PLA. The review on polymer-composite based natural fibre and its application is summarized in Table 1 [10–15]. As the Table shows, most researchers focused on improving the mechanical property of composite-based natural fibre, including tensile strength, tensile modulus, impact strength and fracture. The influence of fibre loading, fibre treatment, processing method and environmental conditions, are common factors that are considered during mechanical characterization.

The developed composites (shown in Table 1) could be applied in various industries. One sector that has applied polymer-based lignocellulosic composites is the automotive industry [13–15]. The purpose of using these developed materials is to reduce the weight and cost of vehicles, along with the lifecycle environmental impact and marketing advantages. Many car manufacturers, in Europe and Asia, have used polymer-based lignocellulosic composites in car interior body components. For example, Ford has used PP/kenaf composites in their Mondeo model and PP/flax composites in floor trays [13, 14]. Meanwhile, Toyota RAUM made a spare tire cover from PLA/kenaf composites [14]. In addition to the automotive industry, polymer-based lignocellulosic composites are also used for non-load bearing or semi-structural applications, such as packaging, furniture, construction, home and personal care [15]. Some products are already available in the marketplace. Jacob Winter (Satzung, Germany) sell cases for musical instruments made of plastic (PP and PLA) reinforced with natural fibres (flax and kenaf). In 2018, the NEC corporation, along with UNITIKA LTD, released a mobile phone casing made using PLA/kenaf composites [13].

From Table 1, most of these composites exhibited high modulus properties mainly at higher levels of fibre content. However, the main flaw of polymer-based natural fibre composites is that the composites are susceptible to moisture. The high water absorption of natural fibre-plastic composites is due to the hydrophilic behaviour of natural fibre. Generally, the absorption of moisture or water in polymer composites will cause a deterioration in their mechanical properties, such as tensile properties [16, 17] and fatigue [18], which is undesirable in most engineering applications. Nevertheless, the hydrophilic behaviour of natural fibre might be exploited and used as a moisture absorber or food desiccator. Therefore, the objective of this research is to evaluate the potential of thermoplastic-based natural fibre composites as moisture absorbent materials. This behaviour was determined by measuring the water absorption properties of polypropylene (PP) and polylactic acid (PLA) reinforced with EFB fibres and MCC fibres at different levels of loading. A qualitative observation was also made in plastic packaging with small green chillies added, and the developed composites.

2. Experimental setup

2.1. Material

Polypropylene (PP) with a density of 0.9 g/cm^3 and polylactic acid (PLA) with a density of 0.998 g/cm^3 were purchased from Lotte Chemical Titan (M) Sdn. Bhd and Unic Technology (Suzhou) Ltd., China, respectively. White and odourless microcrystalline cellulose (MCC) with a size of 20 µm and density of 0.6 g/cm^3 was supplied by Sigma-Aldrich Company, whilst an empty fruit bunch (EFB) was obtained from Malaysian Palm Oil Board (MPOB), Bangi Selangor. The chemical composition of EFB is shown in Table 2 (obtained from Abdul Khalil et al. [19], their EFB came from the same source as this present study). No coupling agent was used in this study. A food grade commercial desiccant-oxygen absorber was purchased from Bake Well Supplies Sdn. Bhd., Gombak, Selangor. The sachet contains the active ingredient of iron-based materials.

2.2. Sample preparation

The EFB was dried under the sun for about one day and crushed into a smaller fibre size. Two EFB fibre sizes were used: length of above 250 μm and below 250 μm. In this study, the PP and PLA composites were prepared through a melt-blending technique using an Internal Mixer, from the Haake PolyLab System. The compositions of the composites are shown in Table 3. Prior to mixing, all raw materials (i.e., PP and PLA pellets, MCC and EFB fibres) were dehumidified in an oven at 80 °C for 7 hours. Compounding temperatures were set in the range 170-180 °C with a rotor speed of 50 rpm for approximately 20 mins. Thermoplastic pellets (PP and PLA) were fed into the mixing chamber for approximately 2 mins, followed by the fibres. The compounded composite was then hot-pressed for 8 mins with a Xihua (XH-406B) Tablet Press Machine under a pressure of 150 kg/cm^2 at 200 °C. Moulded samples were cut into dimensions of 2cm x 2cm.

2.3. Testing and characterization

The samples were immersed in distilled water at room temperature for 6, 12, 24, 48, 72, 96, 120 and 144 hours. All samples were weighed before and after the immersion period, and the percentage of water absorption (WA) of a

Table 1. The review on polymer-composite based natural fibre and its application

Reference	Types of Fiber	Type of plastic	Topic in the review
[10]	EFB	PP etc	Manufacturing method, tensile properties, flexural properties
[11]	Cotton, hemp, kenaf, Cellulose, lyocell, etc	PLA	Mechanical properties
[12]	kenaf, Cotton, ramie, jute, hemp, lyocell, bamboo, Cellulose fibers, abaca, etc	PLA	Chemical composition of green fibers, Mechanical properties of green fibers, PLA: A sustainable polymer, Processing of PLA green composites, Mechanical characterization of PLA-based green composites, Tensile and compressive strength, Flexural and impact strength, Applications of green composites
[13]	Bast, hemp, kenaf, jute, sisal, coconut, coir, flax, hemp, wood, wood flour, etc	PP etc	Natural Fibre-Reinforced Bio-Composites (Bio-polymeric Mattix, Natural Fibres) Applications (Industrial Applications of Natural Fibre Composites in the Automotive Sector, Applications in other Sectors than Automotive)
[5]	jute, hemp, kenaf, and cotton, EFB kenaf, cotton, ramie hemp, lyocell, bamboo, etc	PP, PLA, etc	Natural fiber reinforced composites (NFPCs), General characteristis of NFPCs, Mechanical, Flame Retardant, Biodegradability, Energy Absorption and Tribology Properties of NFPCs, Water absorption characteristics of the NFPCs Viscoelastic and Relaxation behavior of the NFPCs, NFPC application (Natural fiber composites applications in the interior car, The natural fiber applications in the industry)
[15]	PALF, recycled wood fibre, EFB, cellulose, flax, maple hard-wood fibre, sweet sorghum, bamboo, ramie, lyocell, jute, etc	PLA, etc	Major classes of degradable polymers (Renewable resources based-biopolymers, Biodegradable synthetic ploymers) Natural fibre for biocomposites (Lignocellulosic fibre, Surface treatment, Manufacturing/ processing aspects of biocomposites). Biocomposites, Environmental and economic impact of bicomposites, Future scope of biocomposites

Table 2. Chemical composition of Empty Fruit Bunch (EFB) fibres [19]]

Fibre	Extractive(%)	Holocellulose(%)	Cellulose(%)	Lignin(%)	Ash(%)
EFB	3.21	80.09	50.49	17.84	3.4

Table 3. Formulation of composites

Polymer matrix	EFB (wt%)		MCC(wt%)
	$\geq 250\ \mu m$	$\leq 250\ \mu m$	
PP	50,70	50,70	50,70
PLA	-	-	70

sample was computed using equation (1).

$$W_A(\%) = (W_f - W_i)/W_i \times 100 \quad (1)$$

where W_i = initial and W_f = final weight, respectively

The structure of the samples before and after immersion in water was determined using a Perkin Elmer Spectrum-100 FTIR machine. All the measurements were done within the wavenumber range of 500 - 4000 cm^{-1}. The morphology of composite samples before and after water immersion was examined using scanning electron microscopy (SEM) (JOEL- JSM 6300F). The surfaces of the specimens were sputter-coated with a gold layer to eliminate electron charging.

For the qualitative testing, 3 green chillies of approximately the same size, and the PP/EFB and PLA/MCC composite samples were placed inside a non-perforated plastic container. A control sample (without desiccant) and a sample inserted with the commercial desiccant were also prepared to compare their moisture absorbency with the developed composites. All plastic containers were placed in an ambient temperature and the moisture uptake was observed until the chillies became spoiled.

3. Result discussions

3.1. Moisture absorption

The percentage of the moisture absorption of the PP/MCC, PP/EFB, and PLA/MCC composites at different immersion periods and fibre contents was determined, and results are depicted in Fig. 1. For the first 6 hours, all the composites show a slow moisture uptake rate (less than 12 %) and this increased linearly after 6 hours up to 24 hours. Nevertheless, an extension of the time beyond 48 hours resulted in a constant absorption rate value, indicating that the composites had reached saturation condition. Overall, all samples exhibited high moisture absorption rates of more than 10 %. Indeed, this observation is attributed to the nature hydrophilic behaviour of MCC and EFB fibre, which consists of hydroxyl groups within the fibre. Accordingly, the formation of hydrogen bonds with the water molecules existed. The findings in this study are in accordance with previous studies on natural fibre-reinforced thermoplastic polymers [20–22].

By comparing the effect of fibre size, the moisture absorption rate of composites with EFB fibre >250 μm was greater than those of other composites. The absorption rate rose considerably at all the measured hourly points. Similar to other lignocellulosic material, the major components of EFB are lignin, hemicellulose and cellulose [19]. According to Mokhothu and John [23] hemicellulose and cellulose is hydrophilic in nature which associated it to a polar OH group. In this work, no treatment was carried out on the EFB fibres. Hence, it can be expected that the fibre of longer length and higher fibre loadings would contain a higher amount of hemicellulose and cellulose, which promotes better fibre-moisture interaction.

Obviously the type of fibre also influences the moisture absorption behaviour in the PP composites. For the same fibre fraction (at 50wt %), the MCC fibre showed a lesser absorption rate than the EFB fibres. The trend was unchanged even at higher fibre loading (70wt %). It is known that a common process to produce the MCC fibre is by means of acid hydrolysis [24] and non-acidic [25] treatment, which targets the removal of the lignin and hemicellulose components. As a result, the absence or reduction of these components, particularly the hydrophilic hemicellulose [26], would lead to a decline in the moisture absorption affinity of the MCC. It should be noted that the moisture absorption of the PP/MCC and PLA/MCC composites differed significantly, primarily at MCC loading of 70wt %. As seen in Fig. 1, even though the initial absorption rates in both composites was low, after the immersion time was prolonged for more than 24 hours, a tremendous absorption rate of the PLA/MCC sample was evident, compared to the PP/MCC composites.

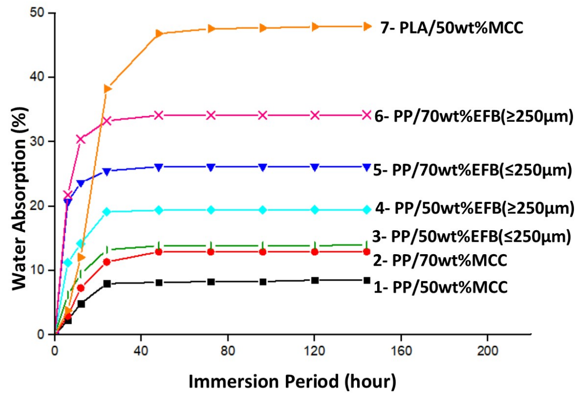


Fig. 1. Moisture absorption of PP and PLA composites.

Interestingly, the absorption rate of the PLA/ MCC sample was also greater than that of the sample of PP/EFB with a fibre size of >250 μm. This indicates that the PLA is more susceptible to moisture than PP as it also contains hydroxyl groups. Although the hydrophilicity of MCC could be lower than the EFB fibre (owing to the MCC having lower hemicellulose content), a contradictory result was obtained, as observed in Fig. 1. Thus, it is clear that the type of thermoplastic matrix is also one major factor that affects the moisture absorption behaviour in the lignocellulosic-based polymer composite. It can be emphasised again that the hydrophilic nature of both fibre and PLA induced a better compatibility between these components and enhanced the moisture absorption capacity of the fabricated composites. From Fig. 1, factors that affect the moisture absorption in thermoplastic composite reinforced with natural or lignocellulosic fibres can be outlined as follows: (i)fibre size (ii)fibre loading (iii)type of fibre and (iv)type of thermoplastic.

3.2. Morphology analysis with SEM

A comparison of the morphology of samples before and after water immersion is shown in Fig. 2. For PP/70wt%EFB composites, the EFB fibres adhere relatively well to the PP matrix (Fig. 2a). Nevertheless, after immersion, the characteristics of the morphology of composites differ slightly. Fig. 2b clearly shows that detachment of the EFB fibres from the PP surfaces was evident, implying that the absorbed moisture had weakened the physical bonding at the filler/matrix interface. A closer view of the EFB fibre (Fig. 2c) also found the existence of voids and similar features of the fibre were reported by Jawaid et al. [27]. In contrast, at saturation (Fig. 2d) the voids disappeared and the fibre surface turned smooth. The reason behind this observation might be that the absorbed moisture had occupied these voids, and upon saturation the cell walls of the fibre swelled, leading to an expansion of the composites thickness [28].

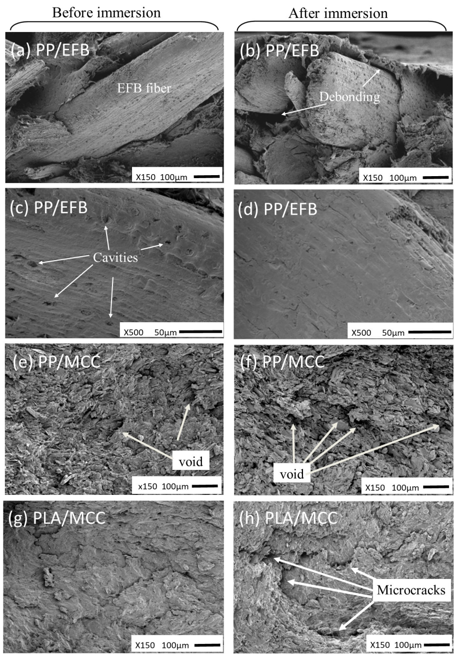


Fig. 2. SEM morphology of sample before and after immersion for samples of PP/70wt%EFB, PP/70wt%MCC and PLA/70wt%MCC.

Figs. 2e and 2f show that the morphologies of PP/70wt%MCC composites, before and after immersion, respectively, did not vary considerably. Before immersion (Fig. 2e), the sample exhibited a rough surface, and the existence of voids were seen on the matrix surface. These morphology features are associated with the high loadings of the MCC fibre in the sample. After immersion, the surface of the sample became rougher with relatively high void contents (Fig. 2f). For the PLA/70wt%MCC composites, despite the high loadings of the MCC fibre, the PLA/MCC composite surface appeared smoother (Fig. 2g) than the PP/70wt%MCC composite sample (Fig. 2e). No voids were observed; however, micro-crack formations were evident and their openings appeared to be large after immersion in water (Fig. 2h).

From Figs. 2e-h, the slight change in morphology of both composite samples after immersion demonstrate the interaction of PP and PLA composites with moisture or water molecules. When comparing the surface morphology at saturation (Figs. 2f and 2h), the deformation of the PLA matrix is quite visible compared to the PP matrix via the formation of larger micro-crack openings. This observation is an additional indicator that the PLA has a higher sensitivity to moisture than the PP, which is associated with the highest moisture absorption rate (Fig. 1). It can be expected that the presence of micro-cracks revealed the inner regions of the composites to the moisture, which in turn accelerated the diffusion of moisture into these regions. According to Samat et al. [25], water molecules were able to diffuse into the polymer matrix during the fatigue testing in water. The absorbed water caused swelling of the microfibril of the craze structure and, at high absorbance of water fraction, led to brittle failure. Although they used different polymeric materials i.e., polyvinyl chloride, and the fatigue testing was carried out until the sample fractured, it can be expected at saturation that a similar phenomenon may occur i.e., the moisture is absorbed and promotes a deformation polymer matrix.

3.3. Fourier Transform Infrared (FTIR) Spectra

The FTIR spectra of neat PP and PP composites samples are presented in Fig. 3. The PP existence of a matrix is validated through the formation of several prominent bands of 2950 cm^{-1}, 2915 cm^{-1} and 2846 cm^{-1} , which correspond to CH stretching. The band 1457 cm^{-1} is related to the CH_2 bending vibration, whilst band 1375 cm^{-1} refers to stretching of CH_3 [29]. After the addition of EFB fibres, the emergence of new broad peaks between 3600-3000 cm^{-1} was noticed (Figs. 3b-c). This range of peaks was assigned to the OH stretching vibrations and hydrogen bonds of hydroxyl groups of cellulosic materials.

The presence of hemicellulose and lignin in EFB fibre is located at a peak of 1725 cm^{-1}. This band is attributed to the C=O stretching in the acetyl and uronic ester groups of the hemicelluloses or the ester carbonyl groups in the p-coumaric units of the lignin [30]. Interestingly, this band was no longer present in spectra of PP/MCC (Fig. 3d). It is known that MCC is produced through the chemical treatment of fibre. Hence the disappearance of this band indicates the removal or decrease of hemicellulose and lignin from the fibre.

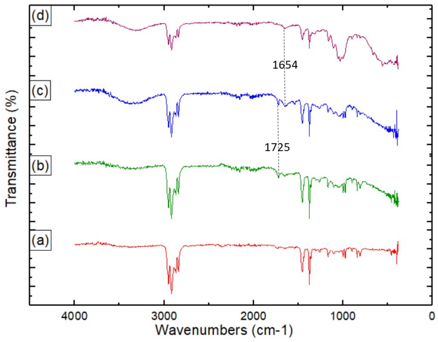


Fig. 3. FTIR spectra of (a) neat PP and composite samples; PP/EFB (b) before immersion (c) after immersion and (d) PP/MCC after immersion.

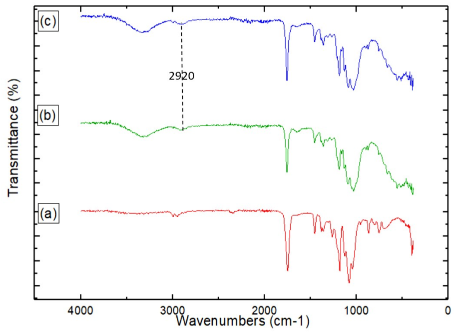


Fig. 4. FTIR Spectra of neat PLA (a) and PLA/MCC (b)before immersion and (c)after immersion.

Several changes occurred after the immersion of the PP/EFB in water. The intense peak at 1654 cm^{-1} implies the interaction of the cellulose of the EFB with the free water. This band was also observed in the PP/MCC composites. The interaction of EFB with water molecules was also attributed to the broader 3600 - 3000 cm^{-1} peaks in Fig. 3c. The changes of these peaks were associated with numerous OH groups of the EFB fibre. However, for the PP/MCC samples, the intensity of their 3600 - 3000 cm^{-1} peaks was lower than the PP/EFB samples. Indeed, this spectral profile correlates well with the low moisture absorption rate of this sample compared to the PP/EFB sample. As mentioned previously, the removal/absence of hemicellulose in MCC had reduced the hydrophilicity behaviour of the cellulose.

In the case of the PLA/MCC composites (Fig. 4), the FTIR spectra here also exhibit profile characteristics comparable to the PP composites, with the incorporation of MCC and water immersion; there was the existence of a peak of 3600 – 3000 cm^{-1}. Moreover, the intensity of these broad bands also increased after immersion in water. The existence of a new band at 2920 cm^{-1} after the addition of MCC corresponds to CH_2 stretching, which is indicative of interaction between the cellulose and hemicellulose of the MCC with the free moisture and water [31].

3.4. Observation of Qualitative testing

In this testing all specimens were stored at room temperature until the chillies spoiled. The observation during the testing is summarised in Table 4. It is clear that the PLA/MCC was able to control the excess moisture which developed inside the package. Apparently this condition suppressed the growth of mould or microbial matter, and delayed the chilli spoilage through a lower formation of water condensation. As a result, the chillies in this package remained green for up to 11 days and only turned to light/bright orange at 14 days, indicating the chilli had started to ripen. Mahajan et al. [3] reported that the accumulation of water condensation on the film decreased in mushroom packages containing moisture absorber. In contrast, the chillies in the other packages showed deterioration associated with the formation of severe or greater water condensation on the package surface, and microbial growth on the stalk and chilli surface, particularly after 11 days. Presumably the formation of water condensation accelerated the growth of microbial matter. Signs of decay were also noticed with the ripening and colour changes.

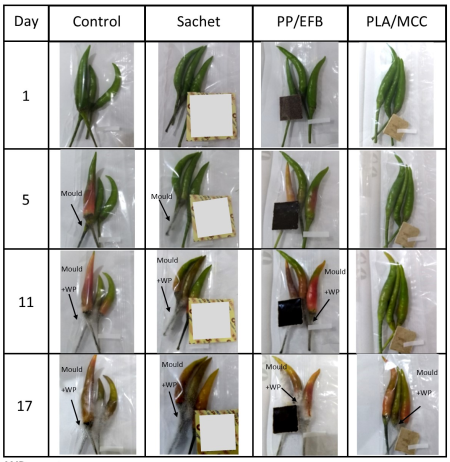
Interestingly, the observation in this testing is consistent with the moisture absorbent results discussed earlier. Fig. 5 compares all the samples on the 1st and after the 17th day, by which time most samples were severely spoiled by the growth of mould, excluding the sample that contained PLA/MCC. This observation signifies that the developed material, particularly the PLA/MCC composites, could bring about improved preservation of chillies or other foodstuffs.

4. Conclusions

The incorporation of a higher content of EFB and MCC fibres enhanced the moisture absorption property of the PP and PLA composites. This behaviour is also indicated in the FTIR analysis which was influenced by the chemical structure of the EFB and MCC fibres. The untreated longer EFB fibre in the PP composites showed greater moisture uptake percentage than the PP/MCC composites. Opposing results were obtained in the PLA composites; the MCC made the PLA more hydrophilic compared to PP. The synergistic effect of the high content of hygroscopic fibre and the hydrophilic matrix is evident through physi-

Table 4. Changes on chilies samples during the qualitative testing

Date	0 day				5 day				11 day				17 day			
Sample	C1	C2	S1	S2	C1	C2	S1	S2	C1	C2	S1	S2	C1	C2	S1	S2
Green chili	√	√	√	√	√	√	√	√		√		√				√
Red chili					√		√		√	√	√		√	√	√	√
Mould (on the stalk)					√	√	√		√	√	√	√	√	√	√	√
Water vapour condensation									√	√	√		√	√	√	√
Decay sign									√	√	√		√	√	√	√
Chili Spoilage													√	√	√	



WP—water vapour

Fig. 5. Plastic packaging contains of chillies and incorporated with PP and PLA composites.

cal and SEM observation of the formation of swelling and cracking respectively, predominantly at the point of saturation. Qualitative analysis/observation also indicated better moisture uptake behaviour within PLA/MCC than within PP composites, as it allowed control of fungal growth and in turn extended the storability of the chillies. These results indicate the potential of polymer-based natural fibre as a moisture absorbent or desiccator to prolong food shelf-life.

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