

THE APPLICATION OF RICE STRAW-BASED PLA/NR BIOCOMPOSITE AS GREEN PLANTING BAGS

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Abstract

This study's primary objective was to observe the growth of marigold seedlings in biodegradable planting bags made from polylactic acid (PLA), natural rubber (NR), and rice straw (RS) film, as well as the biodegradation of the bags during planting. The flexible PLA/NR/RS film was produced using a PLA/NR blend ratio of 70:30 percent by weight (wt%) as a matrix and filled with RS at 3, 5, and 10 wt%. All PLA/NR/RS biocomposite films were produced via an extrusion casting method. To examine the growth efficiency of marigold seedlings in biodegradable planting bags, marigold seedlings were transferred into PLA/NR/RS biocomposite bags. After being planted for 90 days, the stalk diameter and dry mass of the marigold seedlings grown in the PLA/NR/RS biocomposite bags tended to increase with increasing RS content. The biodegradability of all PLA/NR/RS biocomposite bags was measured by how much weight each bag lost after marigold seedlings were planted in it. The amount of rice straw in PLA/NR/RS biocomposite bags was found to affect how much weight they lost. Maximum weight loss was observed when the film contained 10 wt% RS fiber, which corresponded to the lowest PLA molecular weight assessed by gel permeation chromatography (GPC).

Keywords: Biocomposite; Biodegradable Planting Bags; Natural Rubber; Poly (Lactic Acid); Rice Straw

Introduction

Plastics in agriculture have helped farmers improve crop quality and increase food production. Recently, in 2021, the global plastic use in agriculture was estimated at 15.6 million tons, or four percent of the estimated global plastic production of 390.7 million tons (Plastic Europe, 2022). The most frequently used plastics in agriculture are non-biodegradable

and fossil-based plastics, namely polyethylene (PE), polypropylene (PP), and ethylene-vinyl acetate copolymer (EVA) (Plastic Europe, 2022) PE in the form of flexible film or sheet is used to protect crops in a range of applications, including greenhouse roofing, mulching, planting bags, and others. A PE planting bag is used to contain the plant from

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seedling to maturity before it is permanently transplanted into the soil. The PE planting bag cannot be planted with the plant since it inhibits root development and contaminates the soil. Consequently, it will be removed prior to the transplanting process. The growth in demand for cultivated seedlings has led to an increase in the use of PE planting bags, which will eventually contribute to plastic waste accumulation. Due to the significant amount of organic material that adheres to the waste PE films, it is difficult to recycle them. Several studies have been conducted to develop bio-based and biodegradable planting bags as one of the potential solutions to the plastic pollution problem (Bilck *et al.*, 2014; Huzaisham and Marsi, 2020; Shaari *et al.*, 2020; Khamsao *et al.*, 2021).

In our previous work, we have successfully produced biocomposite films from polylactic acid (PLA), natural rubber (NR), and rice straw (RS) using the cast film extrusion process (Pongputthipat *et al.*, 2022). NR, one of Thailand's most important crops, and PLA were chosen because they come from renewable resources and are biodegradable. NR has been reported to improved flexibility and toughness of PLA in numerous research studies (Jaratrotkamjorn, *et al.*, 2012; Mohammad, *et al.*, 2016; Sookprasert, *et al.*, 2017; Fekete *et al.*, 2021). Adding NR to PLA not only improves its toughness but also reduces the overall production costs of the final material, as NR is significantly less expensive. Also, it has been reported that the presence of hydrophobic NR in PLA does not affect the hydrolysis of PLA (Buys, *et al.*, 2018; Rosli, *et al.*, 2018), which is the limiting step of its biodegradation (Husárová, 2014). RS, an abundant agricultural waste produced in rice fields, has been studied for its performance as a bio-filler for PLA, and it has been discovered to improve the biodegradability of PLA (Sakai, *et al.*, 2011; Wu, *et al.*, 2013; Xie, *et al.*, 2022). The optimum ratio of PLA to NR that we found was 70:30 percent by weight (wt%), and the amount of RS can be varied from 3-10 wt% of the total amount of the PLA/NR blend matrix (Pongputthipat *et al.*, 2022).

In this work, we aim to observe the growth of marigold seedlings in biodegradable planting bags made from PLA/NR/RS film compared with those planted in conventional PE.

Materials and Methods

Materials

Poly (lactic acid) (PLA, 4043D) was purchased from NatureWorks LLC. Natural rubber (STR 5L)

was obtained from Natural Art and Technology Co., Ltd. Dimethylsulphoxide (DMSO) (purity=99.90%, AR grade), hexane (purity=99.00%, AR grade), methyl alcohol (purity=99.00%, AR grade), and chloroform (purity=99.80%, HPLC grade), were supplied by RCI Labscan. Rice straw was supplied by a local farm in Pak Thong Chai district, Nakhon Ratchasima, Thailand. Potting soil was supplied by SUT Farm, Suranaree University of Technology, Nakhon Ratchasima.

Preparation of PLA/NR/Rice straw biocomposite

The rice straw (RS) was washed with water and then dried in a hot air oven at 70°C for 12 h. The RS were then ground and sieved with a 200-mesh screen to obtain RS fibers in the range of 53-106 µm long. Before compounding, PLA pellets and RS fibers were dried at 70°C for 4 h in a hot air oven. The biocomposites were prepared using an internal mixer (Haake Rheomix, PolyLab OS System) at a temperature of 170°C with a rotor speed of 60 rpm for 10 min. The compositions in percentage by weight (wt%) of PLA/NR/RS biocomposites are shown in Table 1.

Film preparation

Before film casting, the granulated samples were dried at 70°C for 4 h. The neat PLA and the PLA/NR and PLA/NR/RS films were prepared by a single screw extrusion (Betol, BC 32) casting through a slit die. The barrel temperature profile of 45/165/180/190°C for zones 1-4 and the slit die zone of 190°C were applied. The screw speed and chill roll speed were 40 and 6 rpm, respectively. The films were produced at a chill roll temperature of 60°C

Tensile test

Tensile tests of neat PLA, PLA/NR (70:30), and biocomposite films were performed according to ASTM D882 using a universal testing machine (Instron, 5565) with a load cell of 5 kN and a crosshead speed of 12.5 mm/min. Both machine (MD) and transverse (TD) directions of extruded films were cut for the test.

Table 1. Designation and compositions of PLA/NR/RS biocomposites

Designation	PLA (wt%)	NR (wt%)	RS (wt%)
neat PLA	100	-	-
PLA/NR (70:30)	70	30	-
PLA/NR/RS (3%)	67.90	29.10	3
PLA/NR/RS (5%)	66.50	28.50	5
PLA/NR/RS (10%)	63.00	27.00	10

Planting of the marigold seedling in biodegradable planting bags

To prepare the planting bags, the neat PLA, PLA/NR (70:30), and all PLA/NR/RS biocomposite films were cut to a size of 12 cm wide and 17 cm long and sealed using a heat-sealing machine, obtaining the 10-cm-wide and 12-cm-long planting bags. Marigold seedlings that were about 1 month old and bought from a local garden in Nakhon Ratchasima were planted in high-density polyethylene (HDPE), PLA, PLA/NR (70:30), and PLA/NR/RS bags, with 5 repetitions for each bag type. Each planted bag was then transplanted into plastic plant pots (58×20×20 cm) filled with soil for 90 days and watered daily with 5 liters per plant pot. The diameters of marigold stalks were measured using a vernier caliper. The total mass of dried marigold was calculated by drying it at 60°C for 24 h.

Biodegradability of biodegradable planting bags

The planting bags after a 90-day planting were washed with distilled water, dried in an oven at 40°C for 24 h, and cooled in a desiccator. The weight loss of the planting bag was calculated using Equation 1.

$$\% \text{Weight loss} = [(W_i - W_t)/W_i] \times 100 \quad (1)$$

where W_i is the initial weight of the sample and W_t is the weight of the sample after soil burial for a specified time (Mohammad, *et al.*, 2016).

Morphological Characterization

The surface of extruded cast films was studied using an optical microscope (OM, Leica, DM4M). using reflected and transmitted light modes.

Morphological properties of the surfaces of samples cut from neat PLA, PLA/NR (70:30), and all biocomposite bags before and after use for marigold planting were observed using a field emission scanning electron microscope (ZEISS, AURIGA). The rubber phase was stained with osmium tetroxide for 24 hours to be able to distinguish the phases. An acceleration voltage of 3 kV was used to collect FE-SEM images of the samples. The surface of the specimen was coated with carbon before the examination.

Molecular weight characterization

To determine the molecular weight of PLA in neat PLA, PLA/NR (70:30), and PLA/NR/RS biocomposite bags before and after marigold planting for 90 days, the bags were cut and dissolved in DMSO. The solution was poured into the separatory funnel, and after that, hexene was added. The separatory funnel is then closed with a stopper and shaken vigorously. The DMSO layer was

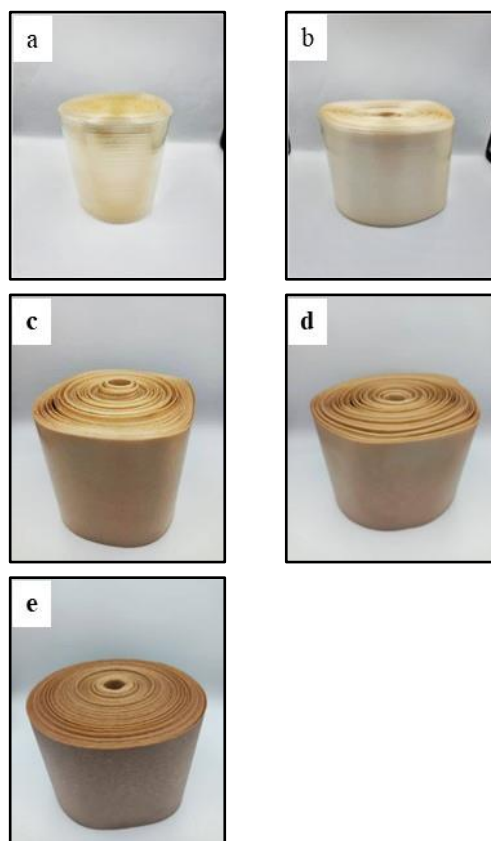


Figure 1. Photographs of the films: (a) neat PLA; (b) PLA/NR (70:30); (c)-(e) the PLA/NR/RS biocomposites with different RS content; 3%, 5%, and 10%, respectively.

drained out once the mixture split into two separate layers and was precipitated and washed with methyl alcohol. The obtained PLA was dried in a hot air oven for 24 hours at 40°C. The average molecular weight and polydispersity index (PDI) of the PLA from each film were determined using gel permeation chromatography (GPC; Waters Alliance e2695) with chloroform as a solvent and a flow rate of 0.5 ml/min. A calibration curve was generated from a polystyrene (PS) standard (Waters Corporation, USA).

Results and Discussion

Physical characteristics of the films

The visual appearance of neat PLA film was transparent and slightly yellow, as seen in Figure 1(a), while the PLA/NR (70:30) film showed an opaque light golden brown color, as seen in Figure 1(b). All PLA/NR/RS biocomposite films (as seen in Figures 1(c)-(e)) exhibited an opaque light brown color.

The optical micrographs of neat PLA, PLA/NR (70:30), and all of the PLA/NR/RS biocomposite films in reflected and transmitted light modes are shown in Figure 2. Under reflected mode OM, the surfaces of neat PLA and PLA/NR (70:30) blend films were rather smooth (Figure. 2(a) and (c)). On the other hand, the surfaces of PLA/NR/RS (3%) and PLA/NR/RS (5%) biocomposite films appeared rough (Figure 2(e) and 2(g)). The surface of the PLA/NR/RS (10%) biocomposite film (Figure. 2(i)) exhibited roughness with visible pores. In transmission light mode, it is clear that rice straw was evenly distributed in the PLA/NR/RS biocomposite films (Figures 2(f), (h), and (j)).

The width and thickness of neat PLA, PLA/NR (70:30) and the biocomposite films are shown in Table 2. The width of the films ranged from 113.33

mm to 124.05 mm. The thickness of PLA/NR/RS biocomposite films tended to increase with an increase in RS content.

Tensile properties

Tensile stress-strain curves along the machine direction (MD) of the neat PLA, and PLA/NR (70:30) and PLA/NR/RS biocomposite films are shown in Figure 3(a). Brittle fractures were seen in PLA. The incorporation of 30% NR into the PLA matrix converted the brittle fracture of PLA into a ductile fracture with the formation of a neck while stretching. The addition of rice straw fiber up to 5 %wt into the PLA/NR (70:30) blend still shows ductile fracture. However, when the rice straw fiber content was increased to 10% wt, the composite reverted to brittle behavior. In the transverse

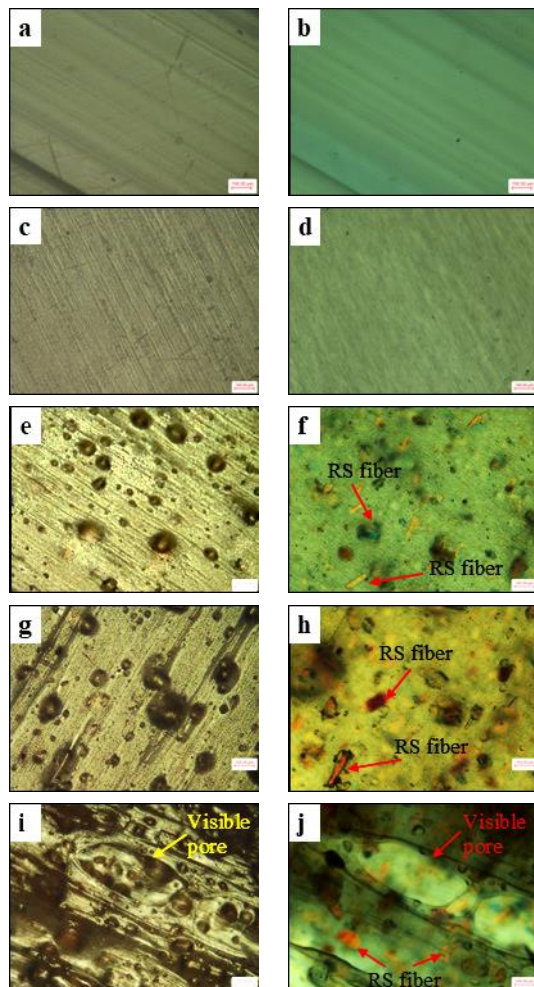
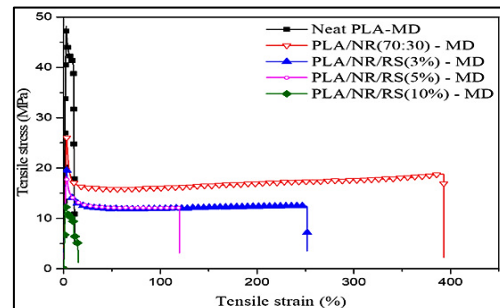


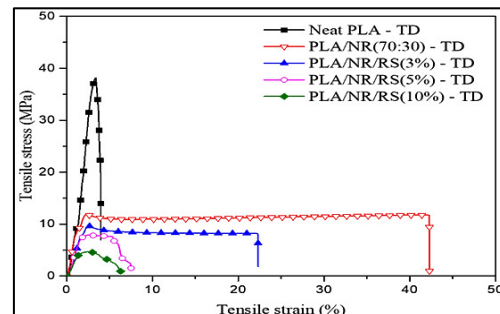
Figure 2. Optical micrographs of the film surface: the neat PLA (a, b); PLA/NR (70:30) (c, d); and the PLA/NR/RS biocomposites with different RS content: 3% (e, f); 5% (g, h); and 10% (i, j). (magnification: 10x)

Table 2. Width and thickness of neat PLA, PLA/NR (70:30), and PLA/NR/RS biocomposite films

Materials	Width (mm)	Thickness (mm)
neat PLA	113.33±1.02	0.205±0.036
PLA/NR (70:30)	123.87±0.34	0.185±0.027
PLA/NR/RS (3%)	124.05±0.26	0.224±0.025
PLA/NR/RS (5%)	119.78±0.03	0.225 ±0.017
PLA/NR/RS (10%)	114.23±0.13	0.271±0.035



(a)



(b)

Figure 3. Stress-strain curves of neat PLA, PLA/NR (70:30) and PLA/NR/RS biocomposite films; Machine direction (a), Transvers direction

direction (TD) (Figure 3(b)), the result shows a similar trend as in the machine direction. The comparison of tensile properties of neat PLA, PLA/NR (70:30), and PLA/NR/RS films in MD and TD is shown in Figures 4-6. In the machine direction, the biocomposites with 3 and 5 wt% RS showed a higher modulus than those of neat PLA and the blend (Figure 4). The PLA/NR/RS biocomposites displayed lower tensile strength and elongation at break than the PLA/NR blend, and these properties deteriorated with increasing RS content (Figures 5 and 6). This could be a result of the poor compatibility between the hydrophilic RS and the hydrophobic PLA/NR blend (Alias, *et al.*, 2021).

Morphological properties

FE-SEM micrographs of the tensile fracture surface of neat PLA, PLA/NR (70:30), and PLA/NR/RS biocomposite films are shown in Figures 7(a)-7(e). Figure 7(a) displays the brittle and smooth fracture surface morphology observed in the neat PLA film, while Figure 7(b) reveals phase separation in the PLA/NR (70:30) blends, with numerous NR droplets dispersed within the PLA matrix. The morphologies of the tensile fracture surface in PLA/NR/RS biocomposites, illustrated in Figures 7(c)-(e), revealed the presence of visible gaps between the RS and matrix. Additionally, the FE-SEM micrographs of the PLA/NR/RS (10%) biocomposites exhibit holes that result from the RS fibers being pulled out of the PLA matrix. These observations indicate poor interfacial adhesion between the RS fiber and matrix. These findings confirm a decrease in the tensile properties of the biocomposite films.

The growth efficiency of marigold seedlings in biodegradable planting bags

The average stalk diameter was used as an indicator of the growth efficiency of marigold seedlings in biodegradable planting bags compared to conventional HDPE planting bags. In Figure 8(a), it can be observed that the diameter of the marigold stalk grown in the neat PLA, PLA/NR (70:30) blend, and all PLA/NR/RS biocomposite bags tended to be higher than that of the marigold grown in an HDPE bag. This could be because PLA and PLA/NR blends, as well as all PLA/NR/RS composites, can absorb moisture, which is essential for plant growth.

After planting for 90 days, the dry masses of the marigolds that were cultivated in HDPE, neat PLA, and PLA/NR (70:30) bags were only slightly different. On the other hand, the dry mass of the marigold seedlings grown in the PLA/NR/RS biocomposite bags was significantly higher than that

of those planted in HDPE bags and increased with increasing RS content in the biocomposites bags, as seen in Figure 8(b). The nutrients, including potassium (K), phosphorous (P), and nitrogen (N) in rice straw (Sarkar *et al.*, 2017), may help the growth of marigold seedlings.

Biodegradability of biodegradable planting bags

The biodegradability of neat PLA, PLA/NR (70:30), and PLA/NR/RS biocomposite planting bags was evaluated by measuring the percent (%) weight loss of the planting bags after 90 days of marigold planting. Figure 9 shows the percent weight loss of HDPE, neat PLA,

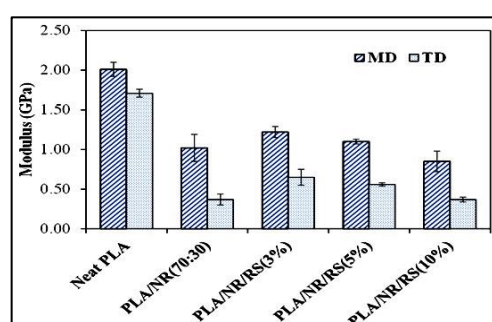


Figure 4. Modulus of neat PLA, PLA/ NR (70:30) and PLA/NR/RS biocomposite films

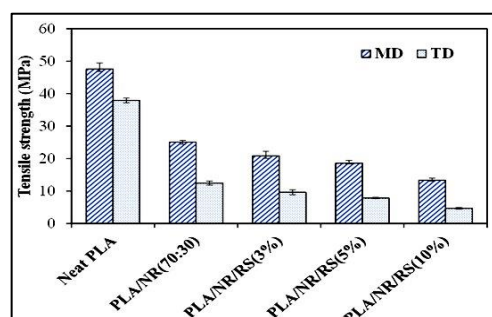


Figure 5. Tensile strength of neat PLA, PLA/ NR (70:30) and PLA/NR/RS films

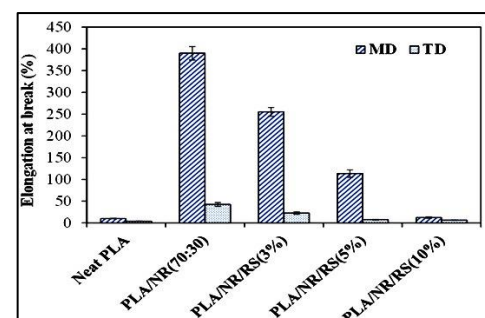


Figure 6. Elongation at break of neat PLA, PLA/NR (70:30) and PLA/NR/RS biocomposite films

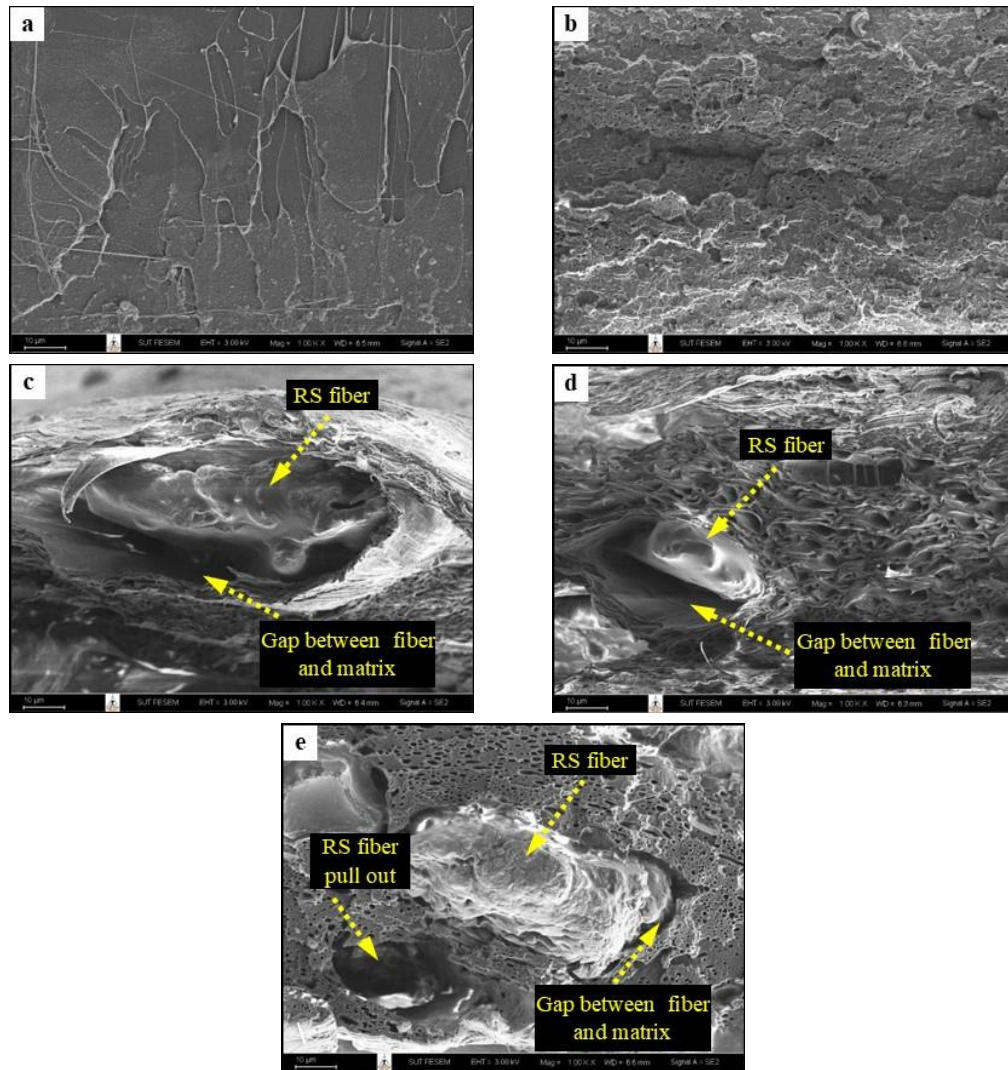


Figure 7. FE-SEM micrographs at $1000\times$ magnification of the tensile fracture surface of neat PLA (a), PLA/NR (70:30) (b), PLA/NR/RS (3%) (c), PLA/NR/RS (5%) (d) and PLA/NR/RS(10%) films (e)

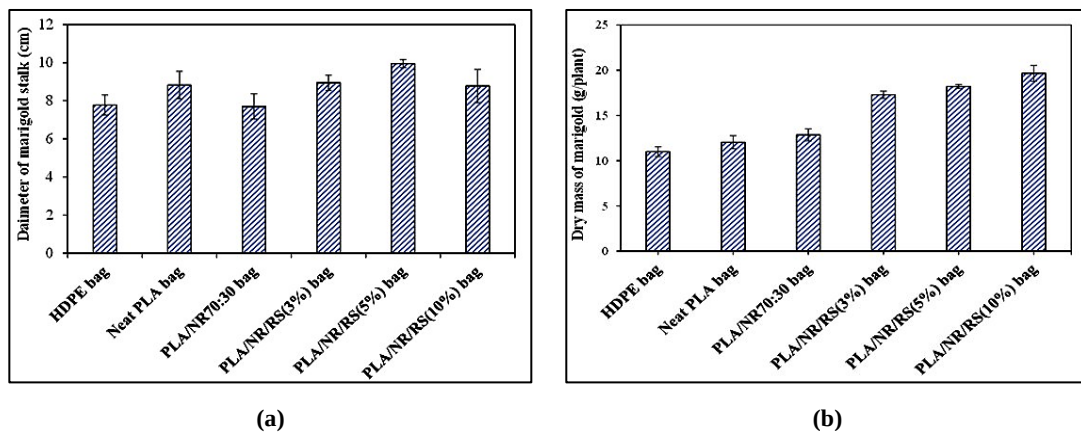


Figure 8. (a) diameter of marigold stalk, (b) dry mass of marigold after planting for 90 days

PLA/NR (70:30), and PLA/NR/RS biocomposite planting bags. The HDPE planting bag exhibited the lowest weight loss of 0.39 percent. The high crystallinity and backbone chains of HDPE are built entirely from C-C single bonds, which are not readily hydrolysable. A PLA/NR (70:30) planting bag has a higher weight loss than a neat PLA bag. For all PLA/NR/RS biocomposite bags, the degree of weight loss increased with rice straw fiber content. The PLA/NR/RS (10%) showed the highest weight loss of 19.56%. The degradation rate of PLA seemed to increase with the incorporation of NR and rice straw fiber. The hydrophilic nature of RS could increase the water absorption rate of the composite, leading to a higher hydrolytic degradation rate (Sakai *et al.*, 2011).

Figure 10 shows the visual appearance of planting bags after 90 days of planting marigold seedlings. The neat PLA bag showed a reduction in transparency and had become brittle. The neat PLA, PLA/NR (70:30), and PLA/NR/RS biocomposites bags became fragile, while the HDPE bags showed no signs of degradation. Generally, the biodegradation of polyester depends on chemical structures and especially on the hydrolysis of the

ester bond in the main chain, which favors the formation of monomers and oligomers that are easily absorbed by microorganisms (Siracusa, 2019).

Figure 11 shows FE-SEM micrographs of the surfaces of samples cut from HDPE, neat PLA, PLA/NR (70:30), and biocomposite planting bags before and after marigold planting for 90 days. Before marigold planting, the neat PLA and PLA/NR (70:30) bags exhibited a rather smooth surface. The PLA/NR/RS biocomposite bags displayed a heterogeneous phase surface with holes dispersed on their surfaces. The hole size and phase separation tended to increase with increasing RS content. After marigold planting for 90 days, the neat PLA planting bag exhibited surface roughness, holes, cracks, and small pores. All PLA/NR/RS biocomposite bags exhibited large cracks, holes, cavities, and surface irregularities on their planting bag surfaces. The surface of the neat PLA, PLA/NR (70:30), and all PLA/NR/RS biocomposite planting bags was destroyed by moisture and attacked by soil microorganisms, while the surface of the HDPE bag before and after marigold planting was not significantly changed.

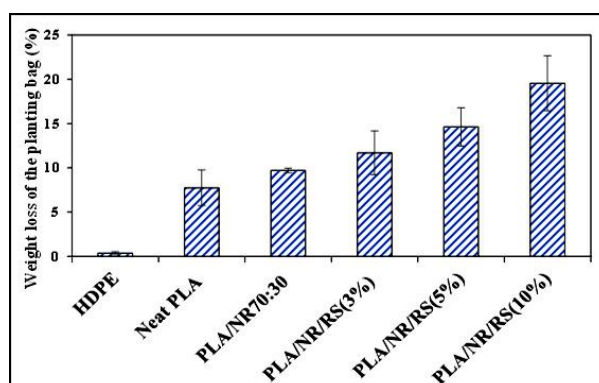


Figure 9. The percent weight loss of the neat PLA, PLA/NR(70:30), and PLA/NR/RS biocomposite bags after use for marigold planting for 90 days



Figure 10. The visual appearance of planting bags after 90 days of planting marigold seedlings

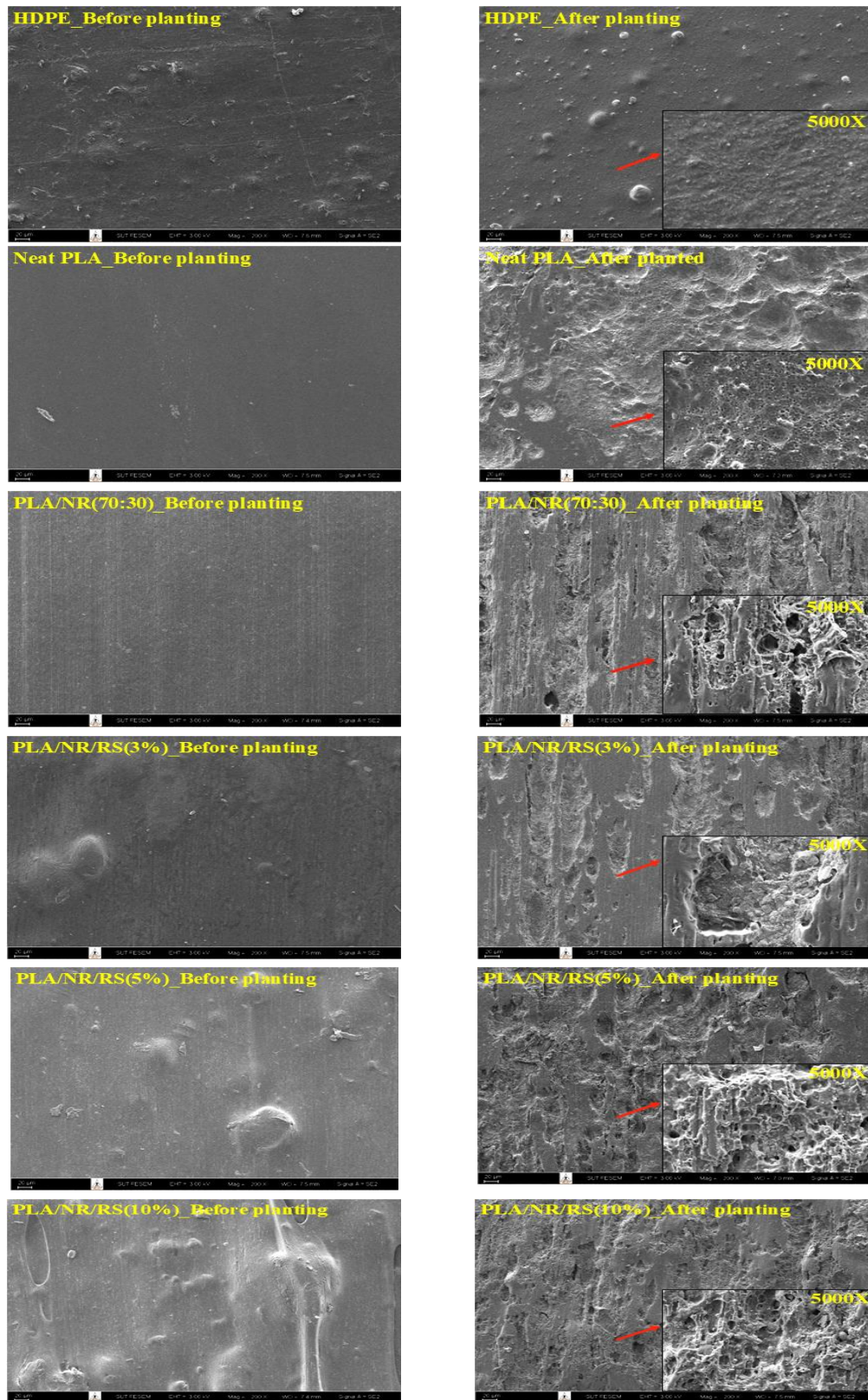


Figure 11. FE-SEM micrographs at 200x of the surfaces of samples cut from the planting bag before and after marigold planting

Table 3. The number-average molecular weights ($\bar{M}_n$), the weight-average molecular weights ($\bar{M}_w$), and the polydispersity index (PDI) of the PLA extracted from neat PLA, PLA/NR (70:30), and PLA/NR/RS bicomposites bags before and after marigold planting

Materials	Before marigold planting			After marigold planting		
	$\bar{M}_n$ (Da)	$\bar{M}_w$ (Da)	PDI	$\bar{M}_n$ (Da)	$\bar{M}_w$ (Da)	PDI
neat PLA bag	54,143	172,778	3.19	51,864	140,553	2.71
PLA/NR (70:30) bag	49,345	127,952	2.59	47,100	116,616	2.48
PLA/NR/RS (3%) bag	47,044	109,430	2.32	44,397	106,565	2.40
PLA/NR/RS (5%) bag	44,641	101,386	2.27	41,352	90,416	2.19
PLA/NR/RS (10%) bag	41,558	96,796	2.33	33,896	80,231	2.37

Molecular weight and polydispersity index

Table 3 shows the number-average molecular weight ($\bar{M}_n$), the weight-average molecular weight ($\bar{M}_w$), and the polydispersity index (PDI) of the PLA extracted from neat PLA, PLA/NR (70:30) and PLA/NR/RS biocomposite bags before and after marigold planting for 90 days. Before planting, the results show that $\bar{M}_n$ and $\bar{M}_w$ of the PLA in the blend and composites decreased when compared to those of neat PLA. This indicated that PLA was degraded after melt mixing. The decrease in molecular weight of PLA at high temperatures during processing in the molten state has been reported by Speranza et.al (Speranza, *et al.*, 2014). Additionally, the $\bar{M}_n$ and $\bar{M}_w$ of PLA dropped with the addition of rice straw. Both before and after marigold planting, this impact was seen in the biocomposites planting bag. This is a result of the hydroxyl groups in rice straw, which can encourage PLA's hydrolysis. The molecular weight of PLA decreased further as the quantity of rice straw increased. Rice straw is naturally hydrophilic, so it should help soil water penetrate the planting bag's surface and accelerate the hydrolysis rate. Moreover, lignocellulosic materials are more easily attacked by microorganisms (Yaacob, *et al.*, 2016). This obtained result corresponds to an increase in weight loss of the PLA/NR/RS composites as rice straw content increases.

Conclusions

The growth efficiency of marigold seedlings in the PLA/NR/RS biocomposite planting bags was compared with that of those planted in neat PLA, PLA/NR (70: 30) blend, and HDPE bags. After being planted for 90 days, the stalk diameter and dry mass of the marigold seedlings grown in the PLA/NR/RS biocomposite bags tended to increase with an increase of RS in the biocomposite. The weight loss of the PLA/NR/RS biocomposite bags with respect to time was recorded as a measure of degradation. It was observed that the % weight loss

of PLA/NR/RS biocomposite bags tended to increase with increasing rice straw content. A better growth efficiency of marigold seedlings planted in PLA/NR/RS biocomposite bags compared to that of those planted in neat PLA, PLA/NR blend, and HDPE bags might be due to the high water absorption and nutrition in rice straw. After 90 days of marigold planting, the molecular weight of PLA extracted from biocomposite planting bags decreased more than PLA extracted from neat PLA and PLA/NR (70:30) planting bags. All PLA/NR/RS biocomposite planting bags shows more environmental friendliness than that neat PLA, PLA/NR (70:30), and HDPE planting bags.

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