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Enzymatic Hydrolysis of Polylactic Acid Fiber

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Abstract This study investigated the optimization of the enzymatic processing conditions for polylactic acid (PLA) fibers using enzymes consisting of lipases originating from different sources. The hydrolytic activity was evaluated taking into consideration the pH, temperature, enzyme concentration, and treatment time. The structural change of the PLA fibers was measured in the optimal treatment conditions. PLA fiber hydrolysis by lipases was maximized for lipase from *Aspergillus niger* at 40 °C for 60 min at pH 7.5 with 60% (owf) concentration, for lipase from *Candida cylindracea* at 40 °C for 120 min at pH 8.0 with 70% (owf) concentration, and for lipase from *Candida rugosa* at 45 °C for 120 min at pH 8.0 with 70% (owf) concentration. There was a change in protein absorbance of the treatment solution before and after all lipase treatments. The analyses of the chemical structure change and structural properties of the PLA due to lipase treatment was confirmed by tensile strength, differential scanning calorimetry, wide-angle X-ray scattering diffractometry, Fourier transform infrared spectroscopy, and scanning electron microscopy.

Keywords Polylactic acid · PLA · Fiber · Hydrolysis · Lipase

Introduction

In the twenty-first century, the textile industry has focused on "green technology." Since using chemicals is harmful and causes considerable pollution, there is a growing demand in the textile industry to switch over to environmentally friendly processing from chemical processing. Applications of green technology for textile industry include biomass and eco-processing. Polylactic acid (PLA) has received considerable attention as a biomass material in the textile industry; it is a linear aliphatic thermoplastic polyester made up of lactic acid (2-hydroxy propionic acid) building blocks or derived from 100% renewable sources such

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as corn [1, 2]. PLA has been highlighted because it is highly marketable due to the low unit cost of raw materials, it is available from agricultural renewable resources, it is biodegradable, and its mechanical properties such as density, glass transition temperature, tensile strength, and Young's modulus are similar to those of polyethylene terephthalate (PET) or nylon [3, 4].

Due to these properties, PLA has the potential to replace conventional petrochemical-based polymers [5]. Most studies on PLA fibers focused on plastics, sheets, or films replacing PET or PTT in the industry [1, 2, 6]. With the increasing interest in the environment, the usage of PLA fibers has expanded to the textile industry field such as clothing materials and household care products. Therefore, various types of PLA fibers such as nonwoven, woven, and knit fabrics have been manufactured and developed in advanced countries [3, 7]. In particular, since the physical properties of PLA fibers are similar to those of PET, they have received attention in the textile industry as an alternative to PET fibers.

To apply PLA fibers in the field of textile industry, suitable postprocessing is needed. Applicable processing for PLA fibers are chemical processing, plasma processing, or enzymatic processing. In general, in many fields of industry, chemical processing has been done; however, chemical processing methods can damage the fibers by decreasing the fiber strength or elasticity and also cause water pollution [8]. On the contrary, plasma processing and enzymatic processing does not cause environmental pollution and minimizes fiber damage to maintain the fiber's benefit. Especially, enzyme processing has been proposed and introduced to natural, synthetic, and recycled fibers, and it has been proven to be effective and environmentally friendly [9–14]. Moreover, hydrolysis by enzymes normally occurs only on the fiber surface without affecting the fiber's chemical structure and, thus, can maintain the fiber's properties [15].

Therefore, to investigate the enzymatic hydrolysis of PLA fibers, three types of commercial ester hydrolases originating from different sources were chosen. Lipase from *Aspergillus niger*, *Candida cylindracea*, and *Candida rugosa* are known as effective enzymes for hydrolysis of ester bonds on polyesters, therefore, those three enzymes are selected for this study. To determine the hydrolytic effect on PLA fibers, the number of carboxyl groups and pH change in the enzyme solution were measured. The optimal conditions for enzyme hydrolysis were measured taking into consideration the pH levels, temperature, treatment time, and enzyme concentration. The mechanical properties of the enzyme-treated PLA fibers were measured by tensile strength, differential scanning calorimetry (DSC), wide-angle X-ray scattering (WAXS) diffractometry, and Fourier transform infrared spectroscopy (FT-IR). Finally, the surface morphologies of the PLA fibers were observed by scanning electron microscopy (SEM).

Experimental Design

Materials

One hundred percent PLA nonwoven supplied from Toray Industries, Inc. was used for the experiment (Table 1). Three lipases originating from different sources, lipase from *A. niger* (LAN), lipase from *C. cylindracea*, and lipase from *C. rugosa* were used without further purification (Table 2). Phosphate buffer (pKa=7.2, Sigma Chemicals Co., U.S.A) solution was used as buffers for lipases. Sodium hydroxide (Duksan Pure Chemicals, Korea) was used as an alkaline agent. A 0.1-N sodium hydroxide solution (Junsei Chemicals, Japan), thymolphtalein (TPH, ACS reagent, Aldrich Chemicals Co., U.S.A) as an indicator, and 95% ethanol (Duksan Pure Chemicals, Korea) were used for titration method. Bradford

Table 1 Characteristics of PLA nonwoven

Purity (%)	Thickness (mm)	Weight (g/m^2)	Manufacturing method
PLA 100	0.126	30	Spunbond

reagent (Sigma Chemicals Co., U.S.A) was used for protein assay. All chemicals were used without further purification.

Methods

Alkaline Treatment

PLA nonwovens were treated with alkaline with a liquor ratio of 100:1 at 150 rpm using a shaking water bath (BS-21, Jeio Tech., Korea). All samples were treated at various conditions, temperature of 30–90 °C, and alkaline concentration of 5–20% for 30 min.

Weight Loss

The weight loss of PLA nonwovens was evaluated by measuring the dry weight of the nonwovens. The samples were dried in a conventional dry oven at 105 °C for 90 min and then weighed in a closed weighing bottle after being cooled in an automatic desiccator. The percentage weight loss was calculated as follows:

$$\text{weight loss } (\%) = \frac{W_1 - W_2}{W_1} \times 100$$

where W_1 and W_2 are the dry weights of the nonwovens before and after treatment, respectively.

Enzyme Treatment

All enzymatic treatment on PLA nonwovens were performed in 50 mM of phosphate buffer solution, using a liquor ratio of 50:1. Each nonwoven sample was cut to a dimension of 20×20 cm. Depending on different pH levels, temperatures, treatment times, and enzyme concentrations, PLA nonwovens were treated by three different lipases at 150 rpm using a shaking water bath (BS-21, Jeio Tech., Korea). Following treatment, the residual enzyme of

Table 2 Enzyme properties

Enzymes	Abbrev.	Source	Activity	Form	Manufacturer
Lipase (EC 3.1.1.3)	LAN	*A. niger*	200 U/g[a]	Powder	Fluka
Lipase (EC 3.1.1.3)	LCC	*C. cylindracea*	2 U/mg[b]	Powder	Fluka
Lipase (EC 3.1.1.3)	LCR	*C. rugosa*	700 U/mg[c]	Powder	Sigma

[a] One unit corresponds to the amount of enzyme which hydrolyzes 1 μmol of acetic acid per minute

[b] One unit corresponds to the amount of enzyme which liberates 1 μmol of oleic acid per minute

[c] One unit corresponds to the amount of enzyme which hydrolyzes 1 μmol of fatty acid from a triglyceride per minute

the samples was inactivated by heating them at 90 °C for 10 min. After inactivation, the samples were thoroughly washed with water at a liquor ratio of 100:1 and then dried at room temperature.

Hydrolytic Activity

The hydrolytic activity of the enzyme was measured using the titration method [11, 13]. A PLA sample weighing approximately 0.1 g was prepared. All specimens are placed into 20-mL bial bottles, and 8 mL of buffer solution was added. Depending on the pH level, temperature, treatment time, and enzyme concentration, enzymatic treatment was performed. After enzymatic treatment, 20 mL of ethanol and 4 drops of 0.9% TPH were added to the solution. In addition, 0.1 N sodium hydroxide was added until the solution color turned light blue. We then recorded the volume of 0.1 N sodium hydroxide used in the test. Each test was repeated five times.

Enzyme Protein Absorbance and Change of pH

The change of enzyme protein absorption was evaluated according to the Bradford assay [14] using UV–vis spectroscopy (M-3,000, Scinco Co. Ltd., Korea) compared with the before and after enzyme treatment. The pH change of the residual enzyme solution was measured by pH meter (pH meter, Jeio Tech., Korea) compared with the before and after enzyme treatment.

Tensile Properties

The tensile strength of the enzyme-treated PLA nonwovens was determined using a Universal Testing Machine (H 100 KS, Hounsfield Test Equipment Ltd., UK) by the strip method according to KS K 0860. An average of five test runs has been reported.

Structure Analysis

A structural change of untreated and enzyme-treated PLA nonwovens was evaluated using DSC, WAXS, and FT-IR analysis. Thermal properties of PLA nonwovens were examined by means of differential scanning calorimetry (DSC, DSC Q1000, TA Instrument, USA) at a heating rate of 20 °C min^{-1} over the temperature range of 50 °C to up to 300 °C. The procedure was repeated two times. The melting temperature (T_m) was obtained from the DSC curves of the second heating cycle. The crystallinity of PLA nonwovens was measured by wide-angle X-ray scattering diffractometry (WAXS, Bruker, Germany) under the following operating conditions: 40 kV and 45 mA at λ, 1.5406 Å. The relative intensity was recorded in the scattering range (2θ) of 0–40° in steps of 0.02°. The infrared spectra of PLA nonwovens were measured by Fourier transform infrared spectroscopy (FT-IR, Spectrum One, Perkin Elmer, USA) taken on KBr pellets. All samples were analyzed in the range of 0–4,000 cm^{-1} at a resolution of 4 cm^{-1}.

Scanning Electron Microscopy

The surface of the alkaline and lipase-treated PLA nonwovens was analyzed using scanning electron microscopy (SEM, S-4,800, HITACHI, Japan) after the samples were plated with platinum.

Results and Discussion

Alkaline Treatment

Figure 1 shows the weight loss of alkaline-treated PLA nonwovens depending on different alkaline concentrations and temperatures. The weight of all specimens decreased as the treatment temperature increased. The weight loss of PLA nonwovens sharply increased as the alkaline concentration increased to 5%, 10%, 15%, and 20% for 30 min of treatment time. In general, alkaline hydrolysis of PET fiber occurred at an alkaline concentration of 10–20% at 75–80 °C for 30 min; however, under this condition, PLA nonwovens hydrolyzed perfectly. Thus, we can infer that PLA fiber hydrolyzes easier than PET fiber. As shown in Fig. 1, as the alkaline concentration and temperature increase, the weight loss of PLA nonwovens increases. Under low conditions, 5% alkaline concentration, 30–60 °C temperature, there was a small loss in weight for PLA nonwovens. Therefore, alternative mild processing methods are needed for PLA fibers such as enzymatic processing.

Hydrolytic Activity

Effect of pH, Temperature, Treatment Time, and Enzyme Concentration on the Hydrolytic Activity

The enzyme shows the highest activity at a certain pH and temperature [10]. Thus, finding the optimum pH and temperature depending on the substrate was needed. Figures 2–4 indicate the hydrolytic activity on PLA fibers as processing conditions were varied over pH values of 7.0–9.0, temperatures of 30–60 °C, treatment times of 10–300 min, and enzyme concentrations of 1–200%. The hydrolytic activity figured out the amount of carboxyl hydrolyzed from the ester group, and it was presented by NaOH consumption. Figure 2 shows the effect of pH and temperature on the hydrolytic activity of three different lipases for PLA fibers in the pH range of 7.0–9.0 and temperature range of 30–60 °C. All experiments were done with 100% (owf) lipase concentrations for 60 min. From Fig. 2, LCC (b) had the highest value of NaOH consumption comparing the relative hydrolytic

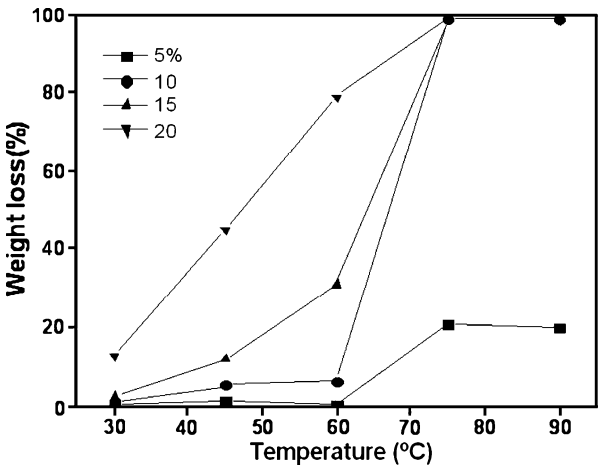

Fig. 1 Weight loss of alkaline-treated PLA nonwovens depending on different alkaline concentrations and temperatures for 30 min. Alkaline concentration, 5–20%

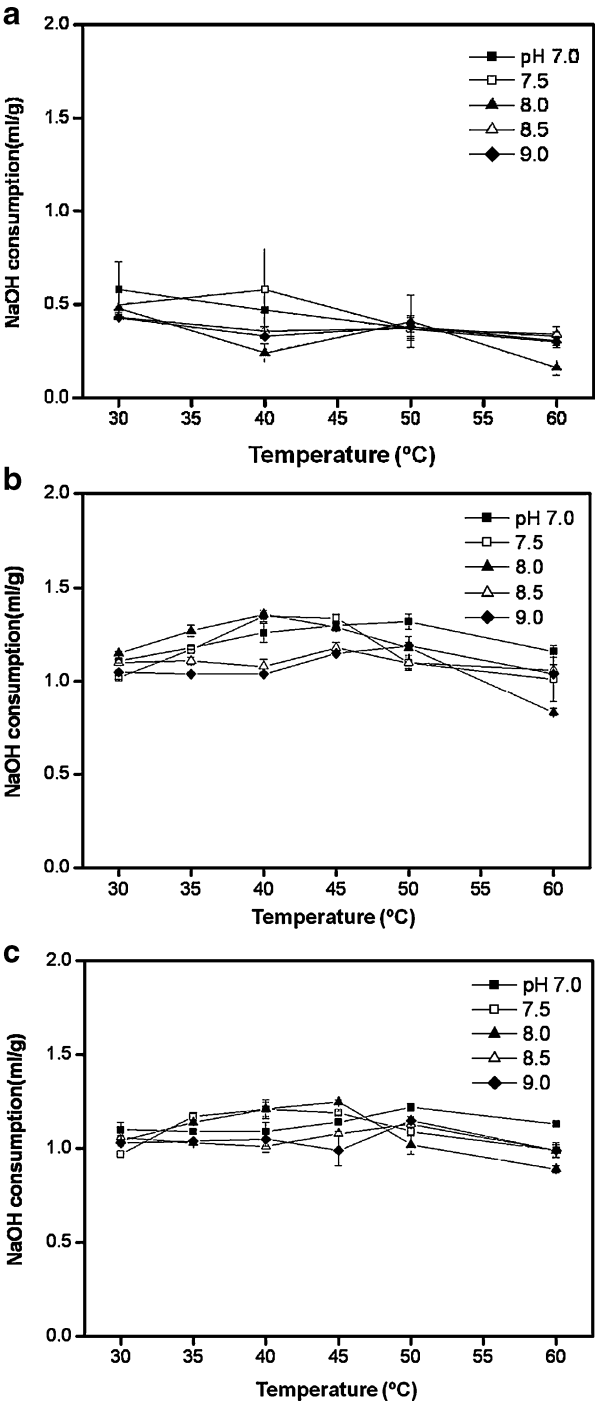

Fig. 2 NaOH consumption of lipase-treated PLA nonwovens depending on different pH levels and temperatures with 100% (owf) of lipases for 60 min. **a** LAN, **b** LCC, **c** LCR

activity of LCR and LAN. That is, LCC has the highest hydrolytic activity among the three lipases. Also, considering the active pH and temperature of lipases, each of the three lipase optimal hydrolysis conditions are as follows: LAN showed the largest NaOH consumption at a pH level of 7.5 at 40 °C, and LCC and LCR had the largest value of NaOH consumption at a pH level of 8.0 at 40 °C and 8.0 at 45 °C, respectively. At other pH levels, the hydrolytic activity decreased rapidly because the enzyme protein structure was denatured [11]. In addition, at other temperatures, the enzyme activity either diminished or ceased entirely [11].

Figure 3 shows the effect of treatment time on the hydrolytic activity of lipases on PLA fibers in the time range of 10–300 min at pH values of 7.5 at 40 °C for LAN, 8.0 at 40 °C for LCC, and 8.0 at 45 °C for LCR with an enzyme concentration of 100% (owf). The hydrolytic activity of LAN showed a linear increase until 60 min had elapsed, then, 60 min later, it reached a plateau. The hydrolytic activity of LCC and LCR showed some increase until 120 min; however, after this the hydrolytic activity decreased. Until the optimum reaction time, the reaction rate of lipases increase linearly because the reaction occurs proportionally between the enzyme and substrate; after the optimum time, there is no more reaction. In addition, in the case of LCC and LCR, some enzymatic reaction decreased as the treatment time increased because denatured enzyme protein in the solution in effect disturb the enzyme reaction [11], so the hydrolytic activity of LCC and LCR showed first an increase and then a decrease. Following the results, the degree of hydrolytic activity depending on the treatment time was less than the pH and temperature because the reaction time was less affected than the pH and temperature with the substrate [16]. Therefore, we concluded experimental treatment times of 60 min for LAN and 120 min for LCC and LCR are reasonable.

Figure 4 shows the effect of enzyme concentration on the hydrolytic activity of lipases on PLA fibers in the concentration range of 1–200% (owf) at pH 7.5 at 40 °C for 60 min for LAN, 8.0 at 40 °C for 120 min for LCC, and 8.0 at 45 °C for 120 min for LCR. The hydrolytic activity showed a linear increase as the enzyme concentration increased, and some enzymes solidified over certain concentrations. LAN increased and showed a plateau at over 60% (owf) because enzyme protein and substrate made equilibrium state of enzymatic reaction, and increased again over 100% (owf); however, it coagulated at 100% (owf). The hydrolytic activity of LCC increased linearly; however, it coagulated at over 70% (owf). LCR had the first activation equilibrium at

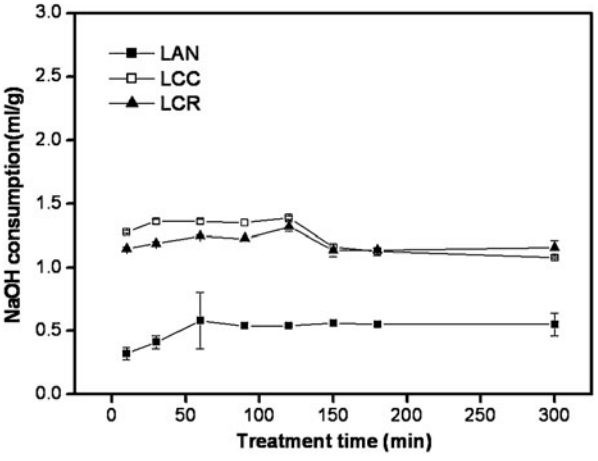

Fig. 3 NaOH consumption of lipase-treated PLA nonwovens depending on treatment time [treatment conditions: pH of 7.5 at 40 °C for LAN, pH of 8.0 at 40 °C for LCC, pH of 8.0 at 45 °C for LCR with lipase concentrations of 100% (owf)]

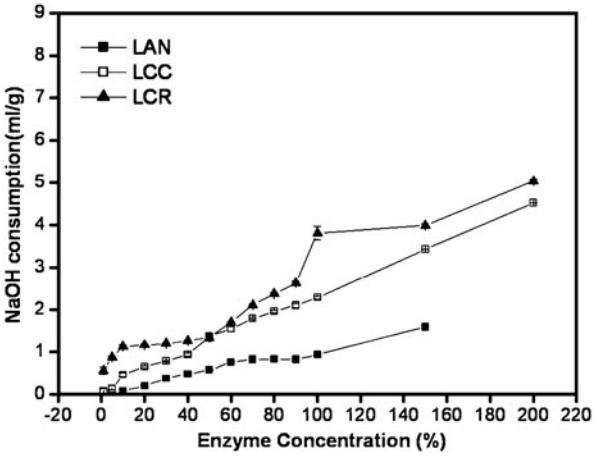

Fig. 4 NaOH consumption of lipase-treated PLA nonwovens depending on enzyme concentration (treatment conditions: pH of 7.5 at 40 °C, 60 min for LAN; pH of 8.0 at 40 °C, 120 min for LCC; pH of 8.0 at 45 °C, 120 min for LCR)

10% (owf), and the second was observed at 70% (owf); however, it also coagulated at a concentration of 70% (owf). Overall, hydrolytic activity depending on enzyme concentration is not stable because the enzyme reacts to the substrate and made equilibrium state; however, some of the denatured enzyme protein produced coagulation in the solution when excess enzyme was used [11]. All things considered, in this study, we discretized the enzyme concentration at 60% (owf) for LAN and 70% (owf) for LCC and LCR before solidification.

In all of the experiments described below, the highest hydrolytic activity for LAN was maximized at 60% (owf) enzyme concentration at 40 °C for 60 min under pH 7.5. LCC and LCR were maximized at 70% (owf) enzyme concentration at 40 °C and 45 °C, respectively, for 120 min under pH 8.0.

Change in Protein Absorbance

Figure 5 indicates the change in protein absorbance before and after enzymatic treatment to confirm the enzyme usage in the enzymatic treatment. This means that the larger the change

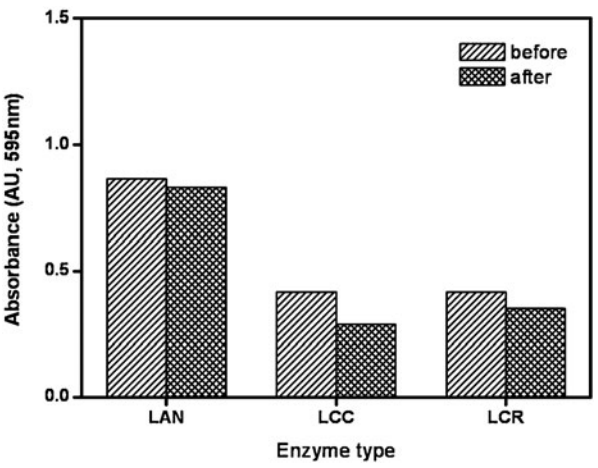

Fig. 5 Absorbance of PLA nonwovens before and after lipase treatment [treatment conditions: LAN, pH of 7.5, temperature of 40 °C, treatment time of 60 min, and LAN concentration of 60% (owf); LCC, pH of 8.0, temperature of 40 °C, treatment time of 120 min, and LCC concentration of 70% (owf): LCR, pH of 8.0, temperature of 45 °C, treatment time of 120 min, and LAN concentration of 70% (owf)]

in protein absorption, the more is the amount of enzymes used for the hydrolysis of PLA fibers. LCC had the largest decrease in absorption after enzyme treatment, which confirmed that the enzyme protein was used in the hydrolysis. LCC and LCR were also confirmed. This result agrees with Figs. 3 and 4. Based on these results, we can conclude that LCC has the largest activity of the three lipases, followed by LCR and LAN.

Change of pH in Solution

Generally, lactic acid is created by hydrolysis of PLA fibers. In this study, we assumed that there was a shift in pH forward to the acid position after enzymatic treatment because of hydrolysis of PLA fiber due to lactic acid. Figure 6 shows the change in pH in the enzyme solution. As shown in the results, LAN, LCC, and LCR treatments were all observed to show a pH shift to acid, so the creation of lactic acid due to hydrolysis was confirmed. For LCC and LCR, the highest level of hydrolytic activity changed from pH 8.0 to 5.7; for LAN, which had the lowest level of hydrolytic activity, the pH changed from 7.5 to 6.1, which confirms that it had the smallest amount of lactic acid. This result was related to the hydrolytic activity. In Figs. 2, 3, and 4, the hydrolytic activity, NaOH consumption of LCC, and LCR showed similar and higher than that of LAN. Therefore, we could assume that the greater the amount of lactic acid produced, the greater the amount of NaOH consumed.

Structure Analysis of PLA Nonwovens

Figure 7 depicts the DSC curves for PLA nonwovens untreated and treated by the three lipases under optimum hydrolysis conditions. In all of these curves, for both the untreated and lipase-treated PLA nonwovens, an endothermic peak was observed at about 160 °C. From Fig. 7 and Table 3, the melting point and heat flow of lipase-treated PLA nonwovens were similar, and ΔH showed no change due to enzymatic treatment. Based on the results, compared to the untreated value, there was no significant change in crystallinity by enzymatic treatment. These results concur with previous works [17, 18].

Figure 8 shows the WAXS patterns of untreated and lipase-treated PLA nonwovens under each optimum hydrolysis conditions. Two crystalline reflections were observed in the

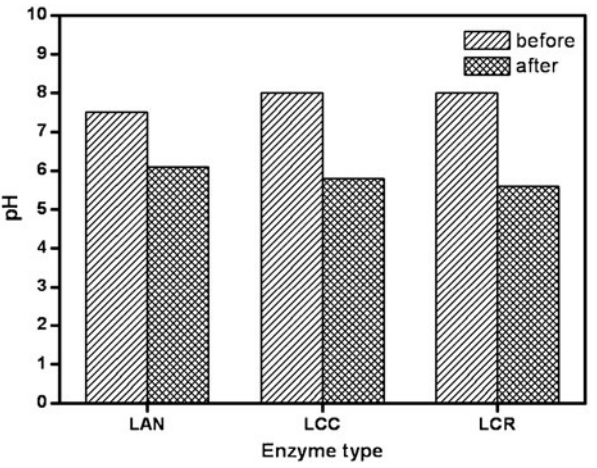

Fig. 6 The pH change of PLA nonwovens before and after lipase treatment [treatment conditions: LAN, pH of 7.5, temperature of 40 °C, treatment time of 60 min, and LAN concentration of 60% (owf); LCC, pH of 8.0, temperature of 40 °C, treatment time of 120 min, and LCC concentration of 70% (owf); LCR, pH of 8.0, temperature of 45 °C, treatment time of 120 min, and LAN concentration of 70% (owf)]

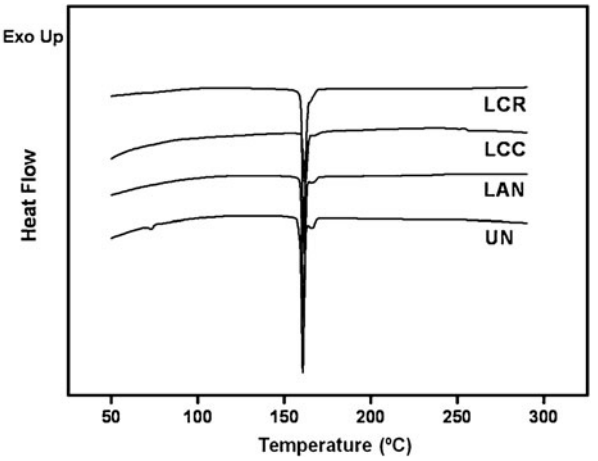

Fig. 7 DSC curves of untreated and lipase-treated PLA nonwovens [treatment conditions: LAN, pH of 7.5, temperature of 40 °C, treatment time of 60 min, and LAN concentration of 60% (owf); LCC, pH of 8.0, temperature of 40 °C, treatment time of 120 min, and LCC concentration of 70% (owf); LCR, pH of 8.0, temperature of 45 °C, treatment time of 120 min, and LAN concentration of 70% (owf)]

2θ range of 5°–40°. The crystal peak of the untreated sample was localized at 2θ=16°, 18° and it corresponded with general PLA fiber [19]. Also, lipase-treated samples had the same crystal peak. Among the three lipases, LAN and LCC-treated PLA fibers showed an increase in the peak intensity, and LCR showed a similar pattern in the untreated sample. In addition, there was no peak shift. From the results of DSC, based on the results of ΔH, all samples were similar, the results of the WAXS of LAN and LCC thought measurement error and fiber condition. Therefore, there was no change in the crystalline structure due to lipase treatment.

Figure 9 shows the FT-IR spectra of untreated and lipase-treated PLA nonwovens under optimal hydrolysis conditions. The FT-IR spectra of untreated PLA nonwovens show a peak assigned C=O stretching band at 1,750 cm^{-1} indicating the carbonyl group, and the FT-IR spectra of the PLA fiber exhibits broad peaks at 2,800–3,000 cm^{-1} assigned to the aliphatic group, which corresponds with previous work [20]. The transmittance (percent) decreased as the C=O combination hydrolyzed. The structural change did not change, but the intensity decreased a little.

Tensile Strength of PLA Nonwovens

Figure 10 provides the tensile strength of alkaline and lipase-treated PLA nonwovens under optimum hydrolysis conditions. In this study, PLA nonwovens manufactured with the spunbond method were used. Due to its characteristics, the tensile strength in the machine direction was stronger than that in the cross-machine direction. The tensile strength for the lipase-treated samples decreased slightly against the machine direction, but very few decreased with the cross direction. LCC-treated samples showed the largest loss of tensile

Table 3 Melting point and heat flow of lipase-treated PLA nonwoven

	Untreated	LAN	LCC	LCR
Tm (°C)	160.7	161.1	162.2	161.4
ΔH (J/g)	47.9	47.8	47.5	48.1

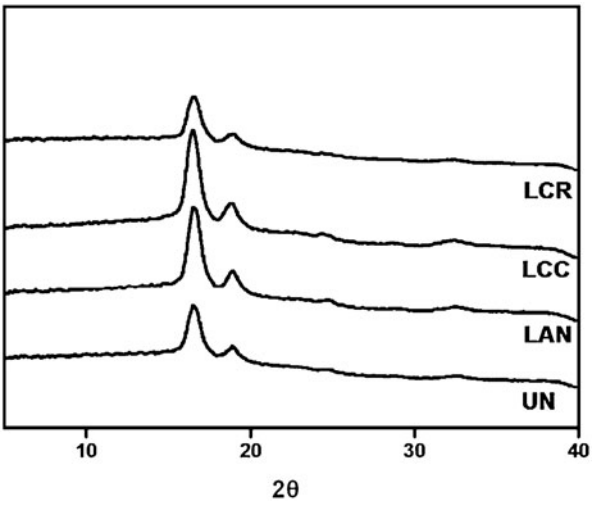

Fig. 8 WAXS of untreated and lipase-treated PLA nonwovens [treatment conditions: LAN, pH of 7.5 temperature of 40 °C treatment time of 60 min and LAN concentration of 60% (owf); LCC, pH of 8.0 temperature of 40 °C treatment time of 120 min and LCC concentration of 70% (owf); LCR, pH of 8.0 temperature of 45 °C treatment time of 120 min and LAN concentration of 70% (owf)]

strength at about 16%. This result can be related to the pH change experiment (Fig. 6) for LCC, and then LCR and LAN, respectively. In addition, when comparing the alkaline-treated sample with the lipase-treated sample, the lipase-treated sample showed only a 16% loss of tensile strength, whereas a 30% loss of tensile strength in the alkaline-treated sample.

Scanning Electron Microscopy

Figure 11 shows the SEM micrographs of PLA nonwovens before and after lipase treatment: (a) untreated, (b) alkaline-treated, and (c–e) lipase-treated PLA nonwovens. A comparison of Fig. 11b with Fig. 11a shows that many pores are formed by alkaline treatment under the condition of 10% alkaline concentration at 60 °C for 30 min. The SEM micrographs only show tiny cracks and fibrils on the surface of the PLA fiber due to LCC

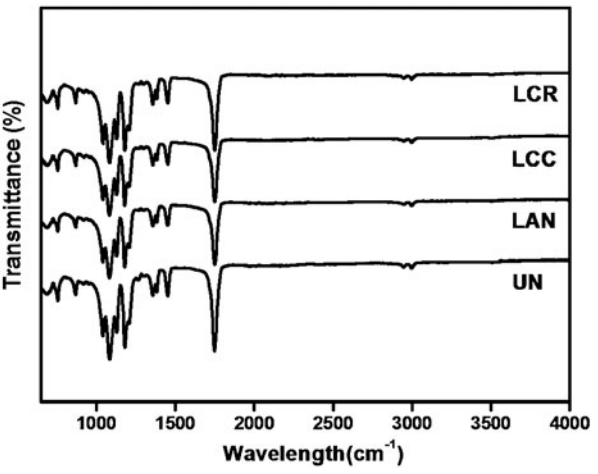

Fig. 9 FT-IR spectra of lipase-treated PLA nonwovens [treatment conditions: LAN, pH of 7.5, temperature of 40 °C, treatment time of 60 min, and LAN concentration of 60% (owf); LCC, pH of 8.0, temperature of 40 °C, treatment time of 120 min, and LCC concentration of 70% (owf); LCR, pH of 8.0, temperature of 45 °C, treatment time of 120 min, and LAN concentration of 70% (owf)]

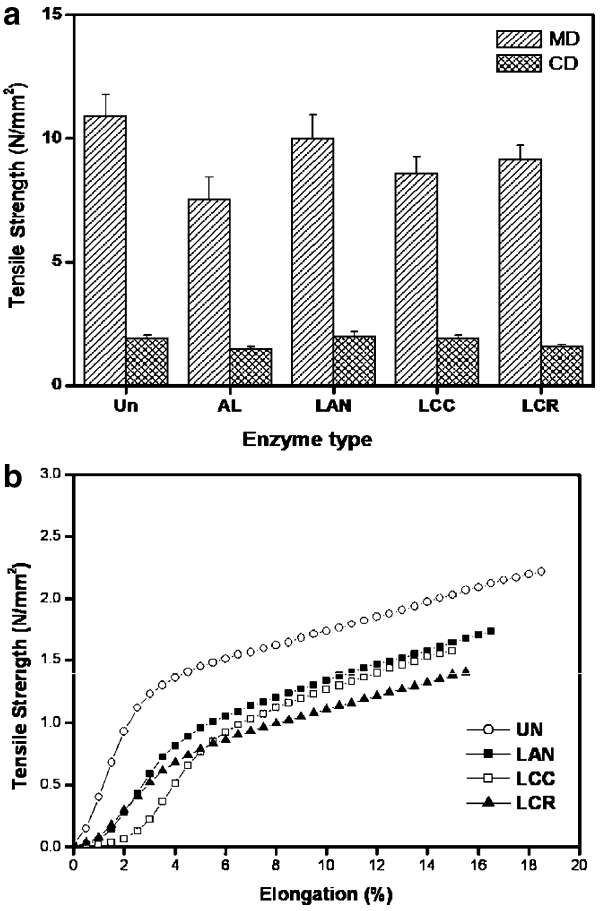

Fig. 10 **a** Tensile strength and **b** strain–stress curves of lipase-treated PLA nonwovens [treatment conditions: AL, 10% of alkaline concentration at 60 °C for 30 min; LAN, pH of 7.5, temperature of 40 °C, treatment time of 60 min, and LAN concentration of 60% (owf); LCC, pH of 8.0, temperature of 40 °C, treatment time of 120 min, and LCC concentration of 70% (owf); LCR, pH of 8.0, temperature of 45 °C, treatment time of 120 min, and LAN concentration of 70% (owf)]

and LCR treatment (Fig. 11d, e), which had the largest hydrolysis; however, there was no surface change with the LAN treatment (Fig. 11c). Furthermore, all lipase treatment indicated less change compared with the alkaline treatment.

Conclusion

The purpose of this study was to develop an enzymatic processing method for PLA fibers as an environmentally friendly technology. For this purpose, PLA nonwoven and three lipases originating from different sources were chosen and compared with alkaline treatment. The effects of hydrolytic activity depending on the pH, temperature, enzyme concentration, and time on PLA fibers were examined according to the number of carboxyl groups, change in protein absorbance, and pH change in treatment solution. Furthermore, DSC, WAXS, FT-IR, and SEM were used to evaluate the physical and chemical changes due to the hydrolysis of the PLA nonwovens. The conditions for the maximum hydrolysis were pH 7.5, 40 °C, 60 min, and 60% (owf) concentration for LAN; pH 8.0, 40 °C, and 120 min, and 70% (owf) concentration for LCC; and pH 8.0, 45 °C, 120 min, and 70% (owf) concentration for LCR. The gap in protein absorbance due to the LCC treatment had

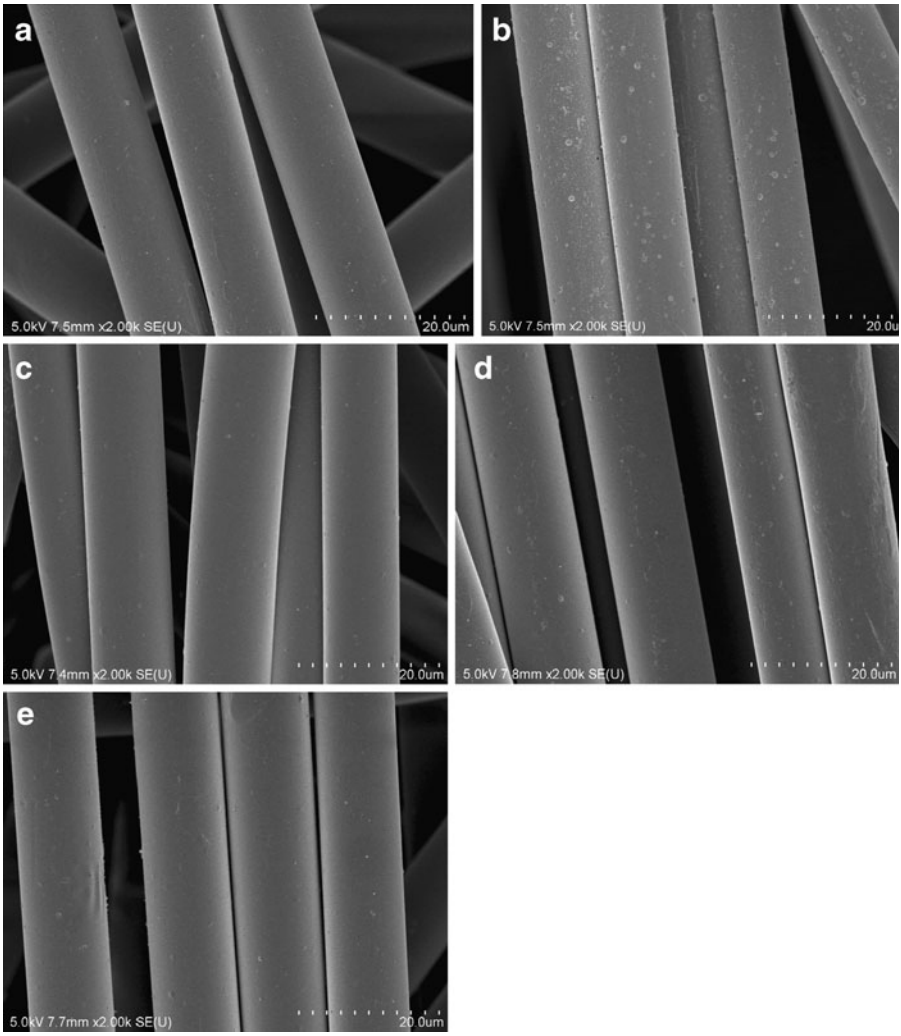

Fig. 11 SEM micrograph of lipase-treated PLA nonwoven. **a** Untreated, **b** alkaline treated, **c** LAN treated, **d** LCC treated, **e** LCR treated [treatment conditions: alkaline, 10% alkaline concentration at 60 °C for 30 min; LAN, pH of 7.5 temperature of 40 °C treatment time of 60 min and LAN concentration of 60% (owf); LCC, pH of 8.0 temperature of 40 °C treatment time of 120 min and LCC concentration of 70% (owf); LCR, pH of 8.0 temperature of 45 °C treatment time of 120 min and LAN concentration of 70% (owf)]

the highest value, followed by LCR and LAN. The measured pH and hydrolytic activity showed the largest change after LCC and LCR treatment. DSC analysis data for the untreated and enzyme-treated PLA nonwovens showed similar trends and had a peak value at 160 °C. WAXS and FT-IR studies showed no change in the crystallinity and molecular structure of the PLA nonwovens due to enzymatic treatment. The surface morphology results for PLA nonwoven showed a few fibrils and cracks after LCC and LCR treatment. This study investigated enzymatic processing on PLA nonwovens in order to determine the optimum hydrolysis conditions for PLA nonwovens without any change in their mechanical or structural properties.

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