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# Hydrolysis of green nanocomposites of poly(lactic acid) (PLA), chitosan (CS) and polyethylene glycol (PEG) in acid solution

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**Abstract:** Green nanocomposites based on poly(lactic acid), chitosan, and polyethylene glycol (PLA/CS/PEG) were prepared by the solution method. The content of PEG was 2–10 wt.% compared with the weight of PLA. The characterization and morphology of the nanocomposites before and after hydrolysis in acid solution were determined by Fourier transform infrared (FTIR) spectroscopy, differential scanning calorimetry and scanning electron microscopy (SEM). The hydrolysis of PLA/CS/PEG nanocomposites in acid solution for different times was also investigated. The shift of C=O, CH<sub>3</sub> groups in FTIR spectra of PLA/CS/PEG nanocomposites before and after hydrolysis was clearly observed. The SEM images of the nanocomposites indicate that PEG plays a role in improving the interaction between PLA and CS, resulting in limiting the permeability of acid solution into the structure of the nanocomposites in the presence of PEG. The obtained results after 28 days of testing in the acid solution show that the PLA/CS/PEG8 nanocomposite (containing 8 wt.% of PEG) had the lowest weight loss with the highest regression coefficient ( $R^2=0.9614$ ).

**Keywords:** chitosan; hydrolysis; nanocomposite; poly(lactic acid); weight loss.

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## 1 Introduction

Biodegradable polymers include natural polymers and synthetic polymers. The natural biodegradable polymers are starch, cellulose, chitin, chitosan (CS), gelatin, etc. The biodegradable synthetic polymers are polyester, i.e. poly(lactic acid) (PLA), poly glycolic acid, polyhydroxy alcanoat, polyamides, polyurethanes, polyvinyl acetate, polyacrylate, etc. [1]. Among the thermoplastic polymers, PLA is the most studied because of its advantages like some thermoplastic polymers (polyethylene, polypropylene, polyethylene terephthalate) as tensile strength, large modules and thermal stability [2]. In addition, PLA also has combustion resistance and anti-ultraviolet radiation stability [3], and especially is biodegradable.

CS is one of the natural resource polymers, which is present in the shells of insects and marine crustaceans, etc. CS and its derivatives have been used in many different areas [4–6]. In biomedical and pharmaceutical fields, CS is a good candidate for regenerating bone tissue and as a drug carrier [7, 8]. According to studies of Inez et al. [9], nasal and oral drugs are more easily transported after combination with CS. CS has high antibacterial activity, is safe for humans and has antimicrobial activity, depending on its molecular weights [10, 11].

The PLA/CS nanocomposite is expected to form new biomaterial exhibiting combinations of good properties of a component polymer that could not be obtained by individual polymers. The nanocomposite is promised to achieve better biodegradability, biocompatibility, elongation and antibacterial activity, improvement of water repellency of CS and increases in thermal stability of PLA [12–16]. Compatibilizers such as poly(ethylene oxide), poly(caprolactone) and poly(ethylene glycol) (PEG) have been used to enhance dispersibility and compatibility between CS and PLA. In this study, the characteristics of green nanocomposites of PLA/CS with and without PEG were studied. The hydrolysis of PLA/CS nanocomposites in hydrochloric 0.1 N acid solution was also evaluated and discussed.

## 2 Materials and methods

### 2.1 Materials

PLA was purchased from NatureWorks LLC (USA). CS and PEG were obtained from Sigma-Aldrich (USA). Chloroform and hydrochloric acid were of analytical reagent grade and used without further purification were provided by Guangdong Guanghua Chemical Factory Co. (China).

### 2.2 Preparation of PLA/CS nanocomposites

The solution method was applied for preparation of PLA/CS nanocomposites. PLA (1.5 wt.%) was dissolved in chloroform to form a fine solution. CS (at a concentration of 20 wt.% in comparison with PLA weight) was dissolved in acetic acid 1% solution (v/v) at room temperature under magnetic stirring. PEG as a compatibilizer was added to the PLA solution at different contents (0 wt.%, 2 wt.%, 4 wt.%, 6 wt.%, 8 wt.%, 10 wt.% PEG compared with PLA weight) that were abbreviated as PLA/CS; PLA/CS/PEG2, PLA/CS/PEG4, PLA/CS/PEG6, PLA/CS/PEG8, and PLA/CS/PEG10, respectively. The PLA/CS composites were obtained by solvent casting on Petri dishes, kept at room temperature for 24 h to evaporate the solvent and then dried in a vacuum oven at 40°C for 8 h.

### 2.3 Characterization

Fourier transform infrared (FTIR) spectra of PLA/CS/PEG nanocomposites were recorded on a Nicolet/Nexus 670 spectrometer (USA) at room temperature by 16 scans with 4 cm<sup>-1</sup> resolution in the wave number range from 400 cm<sup>-1</sup> to 4000 cm<sup>-1</sup>.

Field emission scanning electron microscopy (FE-SEM) of the PLA/CS/PEG nanocomposites coated with platinum was conducted using an S-4800 FE-SEM instrument (Hitachi, Japan).

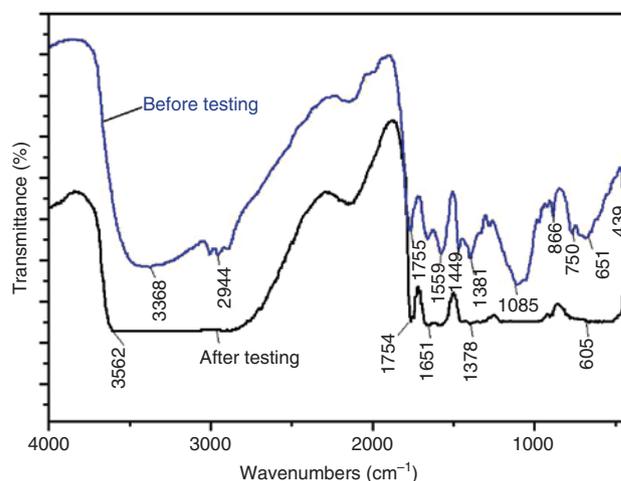
Thermal properties were studied using a DSC-60 thermogravimetric analyzer (Shimadzu Co.) under argon atmosphere, from room temperature to 400°C, at a heating rate of 10°C/min.

Determination of weight loss of the samples in acid solution is based on the weight change after hydrolysis by the formula:  $m = [(m_b - m_s) / m_b] \cdot 100\%$ , in which  $m$  is loss weight of the sample (%),  $m_b$  is initial sample weight (g), and  $m_s$  is loss weight of the sample after hydrolysis (g).

## 3 Results and discussion

### 3.1 FTIR spectroscopy spectra of PLA/CS/PEG nanocomposites before and after hydrolysis in acid solution

Figure 1 shows FTIR spectroscopy spectra of the PLA/CS/PEG8 nanocomposite before and after 28 testing days in HCl 0.1 N acid solution. In the spectrum of the PLA/CS/PEG8 nanocomposite before testing, it is clear that the broad



**Figure 1:** Fourier transform infrared (FTIR) spectroscopy of the poly(lactic acid) (PLA), chitosan (CS), and polyethylene glycol (PEG), (PLA/CS/PEG8, containing 8 wt.% of PEG) nanocomposite before and after testing 28 days in acid solution.

band at 3368 cm<sup>-1</sup> corresponds to the -NH<sub>2</sub> and -OH groups, the peak at 2944 cm<sup>-1</sup> can be attributed to -CH stretching, the absorption band at 1754 cm<sup>-1</sup> is due to C=O stretching, and the bending vibrations of the N-H are at 1559 cm<sup>-1</sup>. Additionally, the bending vibrations of the -CH<sub>2</sub>, -NH<sub>2</sub> groups are observed at 1381 cm<sup>-1</sup> and 1559 cm<sup>-1</sup>, respectively.

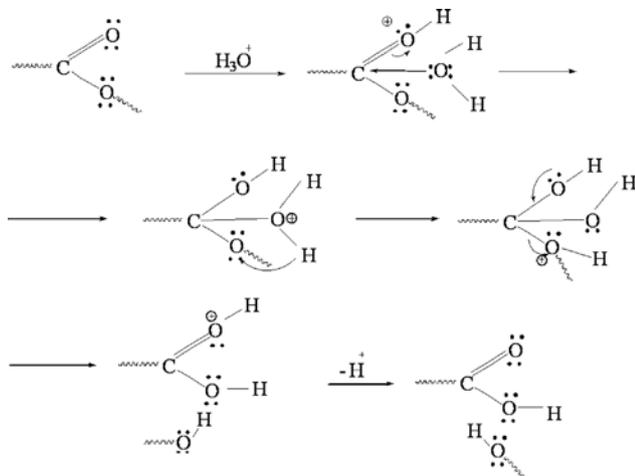
The shift of wave number of the above groups can be observed for the PLA/CS/PEG8 nanocomposite after testing in comparison with the original sample. The wave number of the -OH group shifted from 3368 cm<sup>-1</sup> to 3562 cm<sup>-1</sup> with an expanded peak, and the NH<sub>2</sub> group shifted from 1559 cm<sup>-1</sup> to 1651 cm<sup>-1</sup> with more weak intensity. Similarly, the C=O group vibration moves from 1755 cm<sup>-1</sup> to 1753 cm<sup>-1</sup>. This can be explained by the fact that PLA may be hydrolyzed in acid solution to break ester linkages. This leads to short segments of PLA, and LA oligomers were separated from the nanocomposite. The vibrations of the functional groups in the PLA/CS/PEG8 nanocomposite before and after 28 testing days in acid solution are shown in Table 1.

### 3.2 Hydrolysis of PLA/CS/PEG nanocomposites in acid solution

The hydrolysis process of the PLA/CS/PEG nanocomposite in acid solution is mainly due to the hydrolysis of the PLA by direct influence of water and temperature. The hydrolysis mechanism of PLA in acid solution (HCl 0.1 N) is shown in Figure 2.

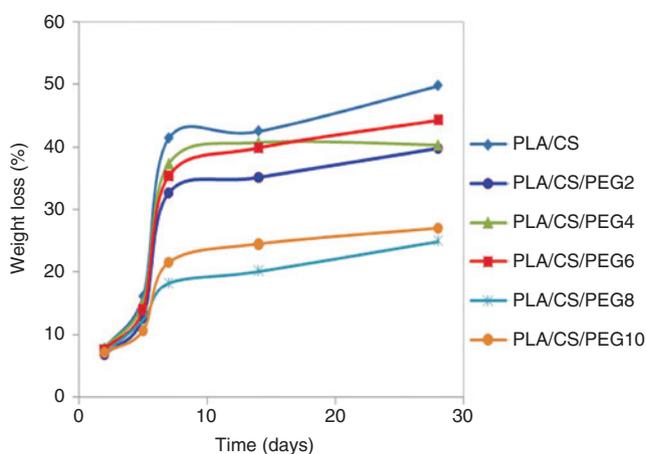
**Table 1:** The characteristic absorption peaks of poly(lactic acid) (PLA), chitosan (CS), and polyethylene glycol (PEG), (PLA/CS/PEG8, containing 8 wt.% of PEG) nanocomposite before and after 28 testing days in acid solution (HCl 0.1 N).

Sample	Wavenumber (cm <sup>-1</sup> )	
	PLA/CS/PEG8 before testing	PLA/CS/PEG8 after 28 testing days
V <sub>C=O</sub>	1755	1753
V <sub>CH</sub>	2878	–
V <sub>-NH<sub>2</sub></sub>	1559	1651
V <sub>C-O-C</sub>	1086	–
δ <sub>CH<sub>3</sub></sub>	1381	1377
δ <sub>CH<sub>2</sub></sub>	750	–
V <sub>OH</sub>	3368	3562



**Figure 2:** Hydrolysis mechanism of poly(lactic acid) (PLA) in acid solution (HCl 0.1 N).

Figure 3 presents weight loss of the PLA/CS/PEG nanocomposites vs. testing time in HCl 0.1 N acid. Obviously, the weight loss of PLA/CS/PEG nanocomposites with different contents of PEG is lower than that of the PLA/CS nanocomposite after 2 days, 5 days, 7 days, 14 days, and 28 days of hydrolysis in acid solution. This can be explained by the presence of PEG, which improves the dispersion and adhesion between CS and PLA phases and leads to decreased numbers of defects and holes in the PLA/CS/PEG nanocomposites compared with the PLA/CS nanocomposite. Thus, acid solution is more difficult to permeate into the PLA/CS/PEG nanocomposites and the hydrolysis of PLA in the nanocomposites is reduced. Among the tested samples, the PLA/CS nanocomposite containing 8 wt.% of PEG had weight loss lower than the others for the same hydrolysis time in acid solution.



**Figure 3:** Weight loss of poly(lactic acid), chitosan, and polyethylene glycol (PLA/CS/PEG) nanocomposites vs. testing time in HCl 0.1 N solution.

**Table 2:** The regression equation between the weight loss (Y-%) of the samples and the testing time (X-days) in acid solution.

Sample	Regression equation	R <sup>2</sup>
PLA/CS	$Y = -0.1179X^2 + 5.0619X - 0.1747$	0.8462
PLA/CS/PEG2	$Y = 13.464 \ln(X) - 2.5185$	0.8781
PLA/CS/PEG4	$Y = -0.1242X^2 + 4.949X - 1.2591$	0.8926
PLA/CS/PEG6	$Y = -0.13X^2 + 5.3223X - 3.0684$	0.913
PLA/CS/PEG8	$Y = 6.8378 \ln(X) + 2.5579$	0.9614
PLA/CS/PEG10	$Y = -0.0579X^2 + 2.4846X + 2.587$	0.9013

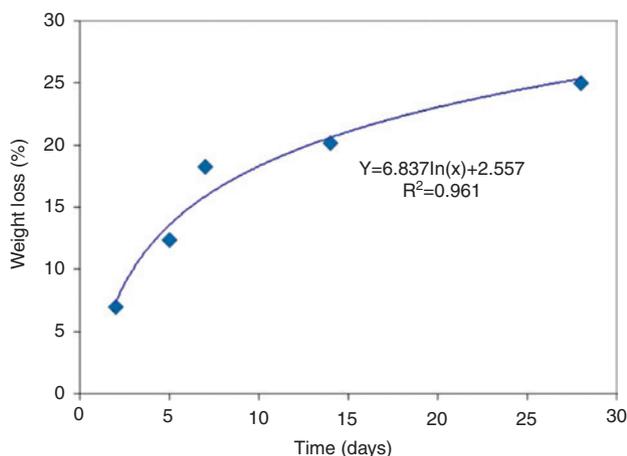
CS, Chitosan; PEG, polyethylene glycol; PLA, poly(lactic acid).

According to the data obtained from Figure 3, the regression equations reflecting the relationship between the weight loss of the samples and the testing time in acid solutions is presented in Table 2.

It can be clearly seen from Table 2 that all the obtained equations are suitable quadratic curves, with regression coefficients ranging from 0.8462 to 0.9614. The highest regression coefficient from the regression equations, reflecting weight loss of PLA from PLA/CS/PEG8 nanocomposites hydrolyzed in acid solution, is 0.9614 (Figure 4) and the regression equation is  $Y = 6.8378 \ln(X) + 2.5579$ .

### 3.3 Morphology of PLA/CS/PEG nanocomposites after hydrolysis in acid solution

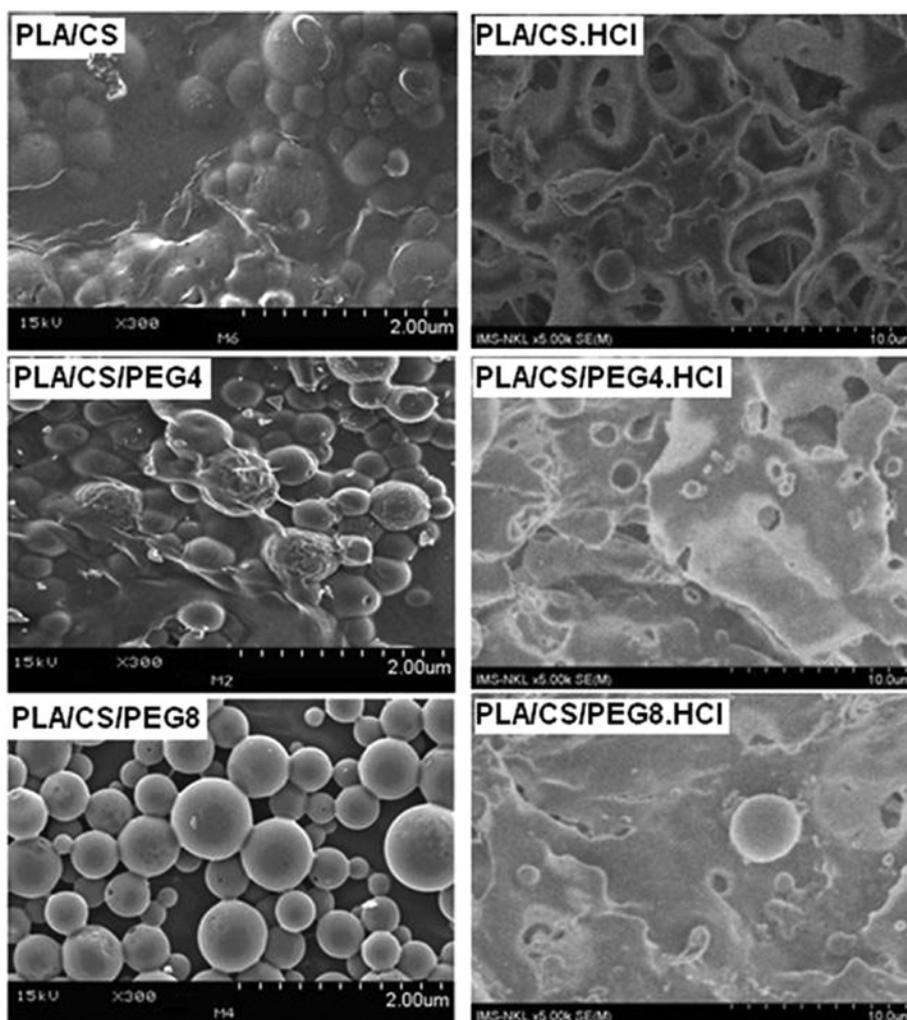
Structure of PLA/CS/PEG nanocomposites has an important influence on their hydrolysis process in different solutions. The tight structure of PLA/CS/PEG has more



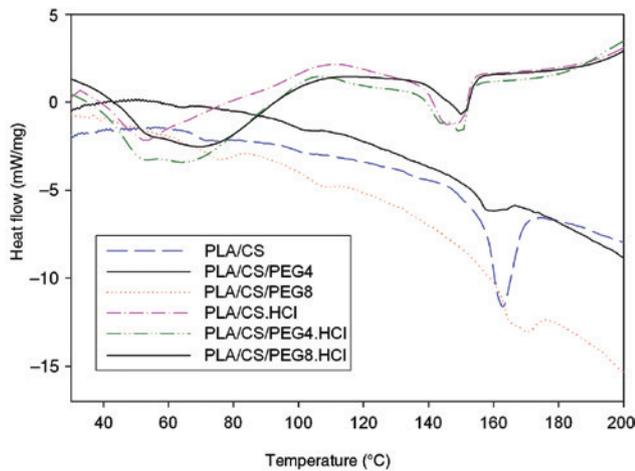
**Figure 4:** Weight loss of poly(lactic acid) (PLA), chitosan (CS), and polyethylene glycol (PEG), (PLA/CS/PEG8, containing 8 wt.% of PEG) nanocomposite vs. testing time in acid solution HCl 0.1 N.

little holes and defects, which results in limitation of permeability of acid solution into the structure of nanocomposites and PLA in nanocomposites is difficult to be hydrolyzed. It can be clearly seen from the SEM images that the surfaces of the PLA/CS, PLA/CS/PEG4 and PLA/CS/PEG8 nanocomposites were destroyed after 28 testing days in the acid solution (Figure 5).

In acid solution, the PLA/CS nanocomposite was hydrolyzed faster than the PLA/CS/PEG nanocomposite, although PLA phases in both nanocomposites were hydrolyzed to form the dark holes, as seen in Figure 5. PEG enhances structural morphology of the PLA/CS nanocomposite by improving the compatibility and adhesion between PLA and CS phases [17]. After hydrolysis, the number and size of holes and defects of the PLA/CS nanocomposite (PLA/CS.HCl image) are higher than those



**Figure 5:** Scanning electron microscopy (SEM) images of poly(lactic acid) (PLA)/chitosan (CS), PLA/CS/polyethylene glycol (PEG)4, PLA/CS/PEG8 before and after 28 testing days in the acid solution.



**Figure 6:** Differential scanning calorimetry (DSC) thermograms of poly(lactic acid) (PLA)/chitosan (CS), PLA/CS/polyethylene glycol (PEG)4 and PLA/CS/PEG8 composites before and after 28 testing days in acid solution.

of PLA/CS/PEG nanocomposites (PLA/CS/PEG4.HCl and PLA/CS/PEG8.HCl images). The image of the PLA/CS/PEG8 nanocomposite (8 wt.% of PEG) indicates the best compatibility between PLA and CS phases. This leads to a close and tight structure, which will limit the formation of holes and defects after hydrolysis of the samples in the acid solution. The morphologies of PLA/CS/PEG2, PLA/CS/PEG6 and PLA/CS/PEG10 nanocomposites are similar to the morphology of the PLA/CS/PEG4 nanocomposite.

### 3.4 Thermal behavior of PLA/CS/PEG nanocomposites after hydrolysis in acid solution

The differential scanning calorimetry thermograms of PLA/CS and PLA/CS/PEG nanocomposites before and after 28 testing days in HCl 0.1 N acid solution are displayed in Figure 6. Thermal behaviors such as glass transition

temperature ( $T_g$ ), melting temperature ( $T_m$ ), enthalpy of melting, and degree of crystallinity of the nanocomposites are calculated and listed in Table 3.

The  $T_g$  and  $T_m$  of PLA/CS/PEG nanocomposites after hydrolysis are higher than those of original samples.  $T_g$  and  $T_m$  of the PLA/CS nanocomposite with different contents of PEG are higher than those of the PLA/CS nanocomposite. This proves that using PEG, the PLA and CS phases are compatible because of hydrogen bond and dipole-dipole interactions [17]. The enthalpy of crystallization and melting of PLA/CS/PEG nanocomposites are higher than those of the PLA/CS nanocomposite. This is due to regular dispersion of CS into the PLA matrix leading to rearrangement of the crystal structure of PLA. Especially, the physical interactions formed between the PEG, PLA and CS make the degree of crystallinity of PLA/CS and PLA/CS/PEG nanocomposites significantly increase.

After 28 testing days in acid solution, the crystallinity of PLA/CS/PEG nanocomposites is higher than that of the nanocomposite before testing. This demonstrates that the amorphous parts of the PLA in the nanocomposites were hydrolyzed and the PLA crystal structure was rearranged. The degree of crystallinity of the PLA/CS/PEG8 nanocomposite after hydrolysis in acid solution is highest.

## 4 Conclusions

The FTIR spectra show the shift of characteristic peaks of functional groups in PLA/CS/PEG nanocomposites before and after testing in acid solution. The FE-SEM images of the nanocomposites indicate that the number of holes and defects in the structure of the PLA/CS/PEG nanocomposites after hydrolysis is lower than that of the PLA/CS nanocomposite. The weight loss of PLA/CS/PEG nanocomposites is lower than that of the PLA/CS nanocomposite. Among the tested samples, the weight loss

**Table 3:** Differential scanning calorimetry (DSC) data and the degree of crystallinity ( $\chi_c$ ) of poly(lactic acid)/chitosan/polyethylene glycol (PLA/CS/PEG) nanocomposite before and after 28 testing days in acid solution.

Sample	$T_g$ (°C)		$T_m$ (°C)		$\Delta H_m$ (J/g)		$\chi_c^a$ (%)	
	Initial	28 days	Initial	28 days	Initial	28 days	Initial	28 days
PLA/CS	59.7	61.9	157	162	10.6	18.61	10.8	19.97
PLA/CS/PEG4	64.0	74.2	150	171	11.4	19.36	12.2	20.79
PLA/CS/PEG8	69.6	73.6	151	167	16.4	35.55	17.7	38.18
PLA/CS/PEG10	66.2	63.6	151	165	15.8	26.94	16.9	28.93

<sup>a</sup> $\chi_c$  (%) =  $\Delta H_m \times 100 / \Delta H_m^*$  where  $\Delta H_m^*$  is the heat of fusion for completely crystallized PLA (93.1 J/g).  $T_g$ , the glass transition temperature;  $T_m$ , the melting temperature;  $\Delta H_c$ , the crystallization enthalpy;  $\Delta H_m$ , the enthalpy of melting;  $\chi_c$ , the degree of crystallinity.

CS, Chitosan; PEG, polyethylene glycol; PLA, poly(lactic acid).

of the PLA/CS/PEG8 nanocomposite vs. testing time is suitable to the regression equation  $Y=6.8378\ln(X)+2.5579$  with maximum regression coefficient ( $R^2$ ) of 0.9614.

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## Bionotes



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