

# Preparation and Swelling Behavior of L-Lactide Interpenetrating Networks

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## Abstract

Novel triblock copolymers of poly (L-lactide)-poly (ethylene glycol)-sebacate-poly (ethylene glycol)-poly (L-lactide) were synthesized by Ring-Opening Polymerization of different ratios of L-lactide with other three pre-prepared poly (ethylene glycol)-sebacate-poly (ethylene glycol) polymers, coded A, B and C which had different poly (ethyleneglycol) chain lengths. The copolymers were characterized by FTIR and <sup>1</sup>H NMR spectroscopy, which indicated that the reaction of ROP took place and led to producing nine triblock copolymers having new different lactide chain lengths (n = 10, 25 and 50), AL<sub>10</sub>, AL<sub>25</sub>, AL<sub>50</sub>, BL<sub>10</sub>, BL<sub>25</sub>, BL<sub>50</sub>, CL<sub>10</sub>, CL<sub>25</sub>, and CL<sub>50</sub>. Nine polymer networks were also prepared from copolymers with sodium alginate S<sub>1</sub> - S<sub>9</sub>, and finally mixed with a solution of hydroxyl ethyl cellulose to form SH<sub>1</sub> - SH<sub>9</sub>.

## Keywords

Lactide Copolymers, Ring-Opening Polymerization, Interpenetrating Polymer Networks (IPN's), Swelling Ratio

## 1. Introduction

In polymer chemistry, Ring-Opening Polymerization (ROP) is a form of chain-growth polymerization. At the terminal end of a polymer chain, it acts as a reactive center where further cyclic monomers can react by opening its ring system and form a long polymer chain. The propagation center can be radical, anionic or cationic. Some cyclic monomers such as lactide and glycolide can be polymerized to high molecular weight polymers by using metal catalysts. ROP continues to be the most versatile method of major groups of biopolymers, particularly when they are required in quantity. Commonly, the monomers in ROP are rings irrespective of ring size, but the reason why polymerization takes place can vary. Rings composed of 3 - 8 atoms may polymerize

due to the loss of enthalpy associated with the loss of ring strain as oxiranes, lactones, lactams, and lactide [1] [2] [3] [4]. ROP is important to produce some industrial polymers like polyethylene oxide and polysiloxanes. The activation of initiating and propagating alcohols by strong nonionic bases can be utilized to enable the polymerization of strained cyclic ester monomers, such as lactide. The (1,8-diazabicyclo[5.4.0] undec-7-ene) DBU, (7-methyl-1,5,7-triazabicyclo[4.4.0]dec-5-ene) MTBD and TBD (general structure of a phosphazene base) are highly active catalysts for this ROP process, providing Degree of Polymerization DP 500 PLAs within a few minutes at room temperature and 1 mol % catalysts loadings [5] [6] [7]. Haitao Qian, *et al.* [8] used DBU, a known, effective, and convenient organ catalyst, for the ring opening polymerization of cyclic esters, to synthesize random copolymers of lactide and glycolide. In contrast, the application of DBU to catalyze the ROP resulted in polymers with predictable molecular weights and narrow PDIs (<1.09).

End-group fidelity of the polymerization can be determined by both <sup>1</sup>H NMR and Gel-Permeation Chromatography (GPC) technique [9]. Polymerizations mediated by DBU also showed a linear increase of molecular weight with monomer conversion. DBU is a strictly hindered amidine that has demonstrated usefulness in a variety of different reactions.

## 2. Experiment

### 2.1. Materials

Chemicals used in this study were supplied from different sources, polyethylene glycol (average M. wt. 2000, 4000 and 10000) were supplied from BDH Co., sebacoyl chloride and glutaraldehyde were supplied from Fluka Co., 1,2-dichloro ethane (EDC) was supplied from Lab Co., triethylamine was supplied from RDH Co., L-lactide was supplied from BDH Co., Diazabicyclo [5.4.0] undec-7-ene (DBU), diethyl ether and hydroxy ethyl cellulose were supplied from Sigma-Aldrich Co., sodium alginate was supplied from HIMEDIA Co., all materials were used without any purification.

### 2.2. Instruments

FTIR-Jasco 4200 supplied by JASCO Company was used for recording I.R spectra of the new polymers as KBr disc from (400 - 4000) cm<sup>-1</sup>. The <sup>1</sup>H NMR was recorded using Bruker 400 MHz spectrometer. Type INSPECT S50 supplied by FEI Company. Polymers molecular weights and molecular weight distributions ( $M_w/M_n$ ) were determined using a Waters 1515 gel permeation chromatography (GPC); DMF was used as the eluent at a flow rate of 1.0 ml/min and calibrated with polymethyl methacrylate standard

### 2.3. Preparation of Polymers

#### 2.3.1. Preparation of Poly (Ethylene Glycol)-Sebacate-Poly (Ethylene Glycol) (A)

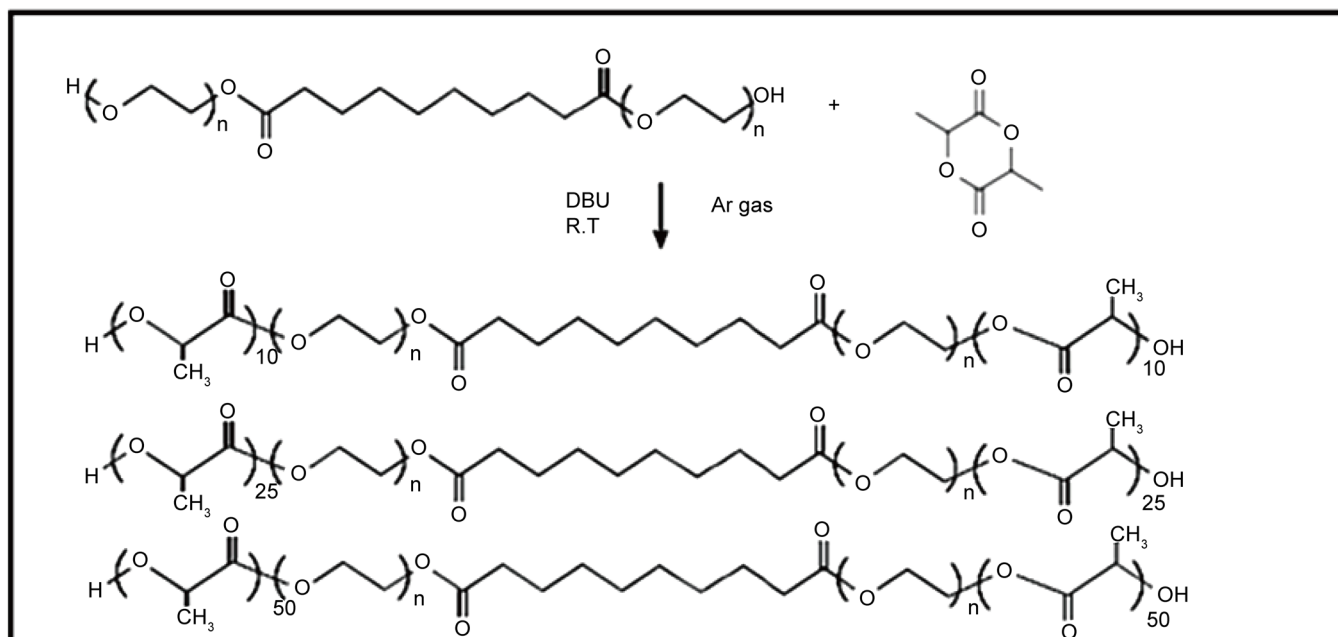
Poly (ethylene glycol), PEG, (M. Wt. 2000) (40 g, 0.02 moles), (2.79 ml, 0.02 moles) of



The same procedure was carried out to prepare the polymers AL<sub>25</sub>, AL<sub>50</sub>, BL<sub>10</sub>, BL<sub>25</sub>, BL<sub>50</sub>, CL<sub>10</sub>, CL<sub>25</sub>, and CL<sub>50</sub>. The chemical equations can be represented by the **Scheme 2** and **Table 2** shows the quantities of reactants used in the preparation of the polymers, AL<sub>25</sub>, AL<sub>50</sub>, BL<sub>10</sub>, BL<sub>25</sub>, BL<sub>50</sub>, CL<sub>10</sub>, CL<sub>25</sub>, and CL<sub>50</sub>.

### 2.3.3. Preparation of Network S<sub>1</sub>

0.4 g of AL<sub>10</sub>, 0.4 g of sodium alginate (Na-alg.) were dissolved in 5 ml distilled water (solution A), 0.1 g of calcium chloride was dissolved in 5 ml distilled water (solution B). Solution A was added drop wise to solution B to form polymeric beads. These beads were collected and washed with water two times and dried to obtain S<sub>1</sub>. The same procedure was carried out to prepare networks S<sub>2</sub> - S<sub>9</sub> [12].



**Scheme 2.** Chemical equations of preparation of triblock copolymers AL<sub>10</sub>, AL<sub>25</sub>, and AL<sub>50</sub> from polymer A with L-Lactide (PEG = 2000).

**Table 2.** The composition of prepared block polymers AL<sub>10</sub>, AL<sub>25</sub>, AL<sub>50</sub>, BL<sub>10</sub>, BL<sub>25</sub>, BL<sub>50</sub>, CL<sub>10</sub>, CL<sub>25</sub>, and CL<sub>50</sub>.

Tri block polymers	M. Wt. of PEG	Polymers A, B and C (g)	A, B, and C (mole)	Wt. of L-lactide (g)	L-lactide (mole)	DBU ( $\mu$ l)
AL <sub>10</sub>	2000	4.168	0.001	1.44	0.01	55
AL <sub>25</sub>	2000	4.168	0.001	3.603	0.001	77
AL <sub>50</sub>	2000	2.084	0.0005	3.603	0.025	112
BL <sub>10</sub>	4000	8.166	0.001	1.44	0.01	90
BL <sub>25</sub>	4000	8.166	0.001	3.603	0.025	116
BL <sub>50</sub>	4000	4.083	0.0005	3.603	0.025	76
CL <sub>10</sub>	10000	20.17	0.0005	1.44	0.005	212
CL <sub>25</sub>	10000	20.17	0.0005	3.603	0.0125	234
CL <sub>50</sub>	10000	10.08	0.0005	3.603	0.025	135

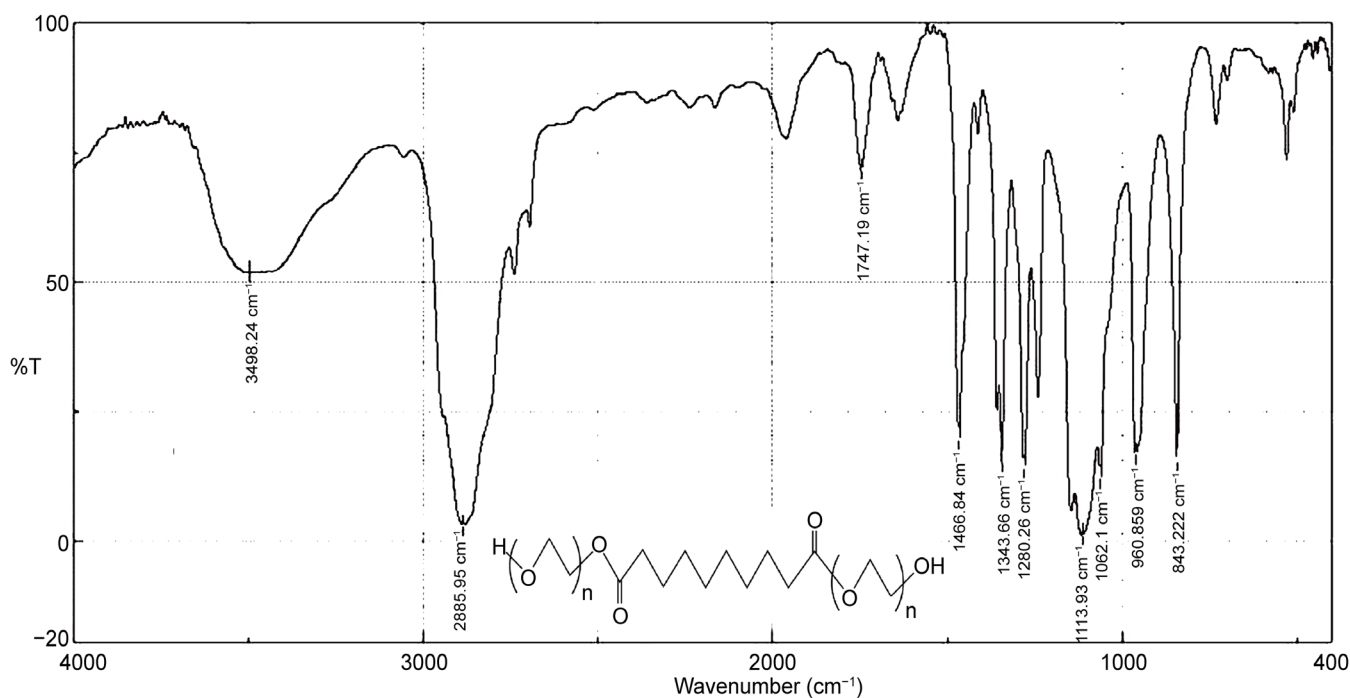
### 2.3.4. Preparation of Fibers IPN's SH<sub>1</sub> - SH<sub>9</sub>

0.4 g of hydroxyethylcellulose (HEC), 377  $\mu$ l glutaraldehyde and 0.25 ml of concentration of sulfuric acid were heated with continuous stirring at 60°C - 65°C for one hour. Then the crosslinked product solution was cooled. The cold solution of crosslinked HEC was mixed with 0.4 g of S<sub>1</sub> in an ice bath with continuous stirring for half an hour. The product was then dried in vacuum oven at 25°C, ground and pressed as discs. The same procedure was carried out to prepare of fiber IPN's SH<sub>2</sub> - SH<sub>9</sub>.

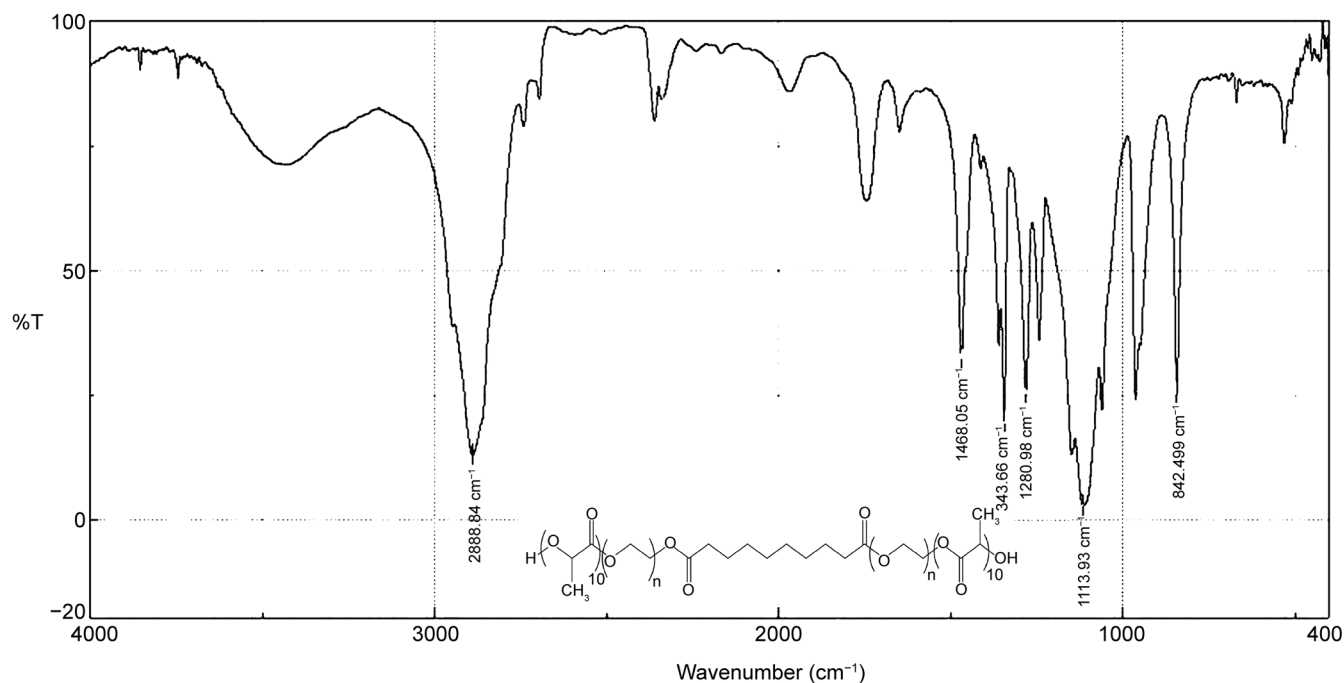
## 3. Results and Discussion

The FTIR spectra of (A, B, and C) polymers with the characteristic absorption bands were intense due to the associated hydroxyl groups (3434, 3498, and 3447  $\text{cm}^{-1}$ ) and bands at (2883, 2885, and 2887  $\text{cm}^{-1}$ ) for (CH<sub>2</sub>) of PEG repeated units. Representative I.R. spectrum is shown in **Figure 1**. The intensity of the hydroxyl group band was decreased with increasing PEG molecular weight, the same observation was made by Jalal and Al-Lami [13]. The appearance of the stretching band of (C=O) at 1725  $\text{cm}^{-1}$  as a result of esterification reaction of PEG with SC show less intensity due to the loss ratio of SC comparing to the molecular weight of PEG.

Representative I.R. spectrum of triblock copolymers of PLA-PEG-SC-PEG-PLA (AL<sub>10</sub>) is shown in **Figure 2**. The characteristic absorption bands were intense bands due to the associated hydroxyl groups (3417, 3436, and 3450  $\text{cm}^{-1}$ ). In the block copolymers spectrum, the sharp and intense bands at (1741, 1750, and 1738  $\text{cm}^{-1}$ ), bands at (1280  $\text{cm}^{-1}$ ) for (C-O) groups, and bands at (2882, 2883  $\text{cm}^{-1}$ ) for (CH<sub>2</sub>) of PEG repeated units these bands confirm the presence of the carboxylic ester (C=O) and ether (C-O-C)



**Figure 1.** FTIR spectrum of poly(ethylene glycol)-sebacate-poly(ethylene glycol) (with PEG 2000), polymer (A).

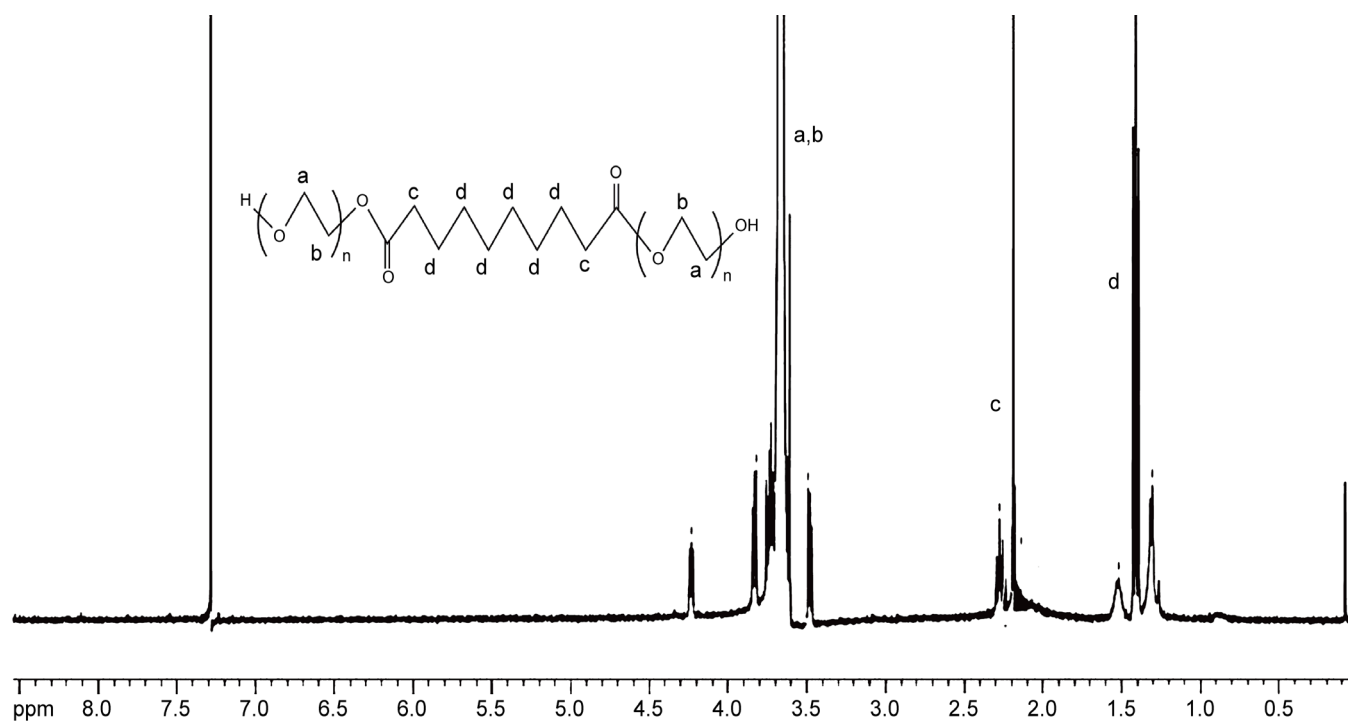


**Figure 2.** FTIR spectrum of triblock copolymer AL<sub>10</sub>.

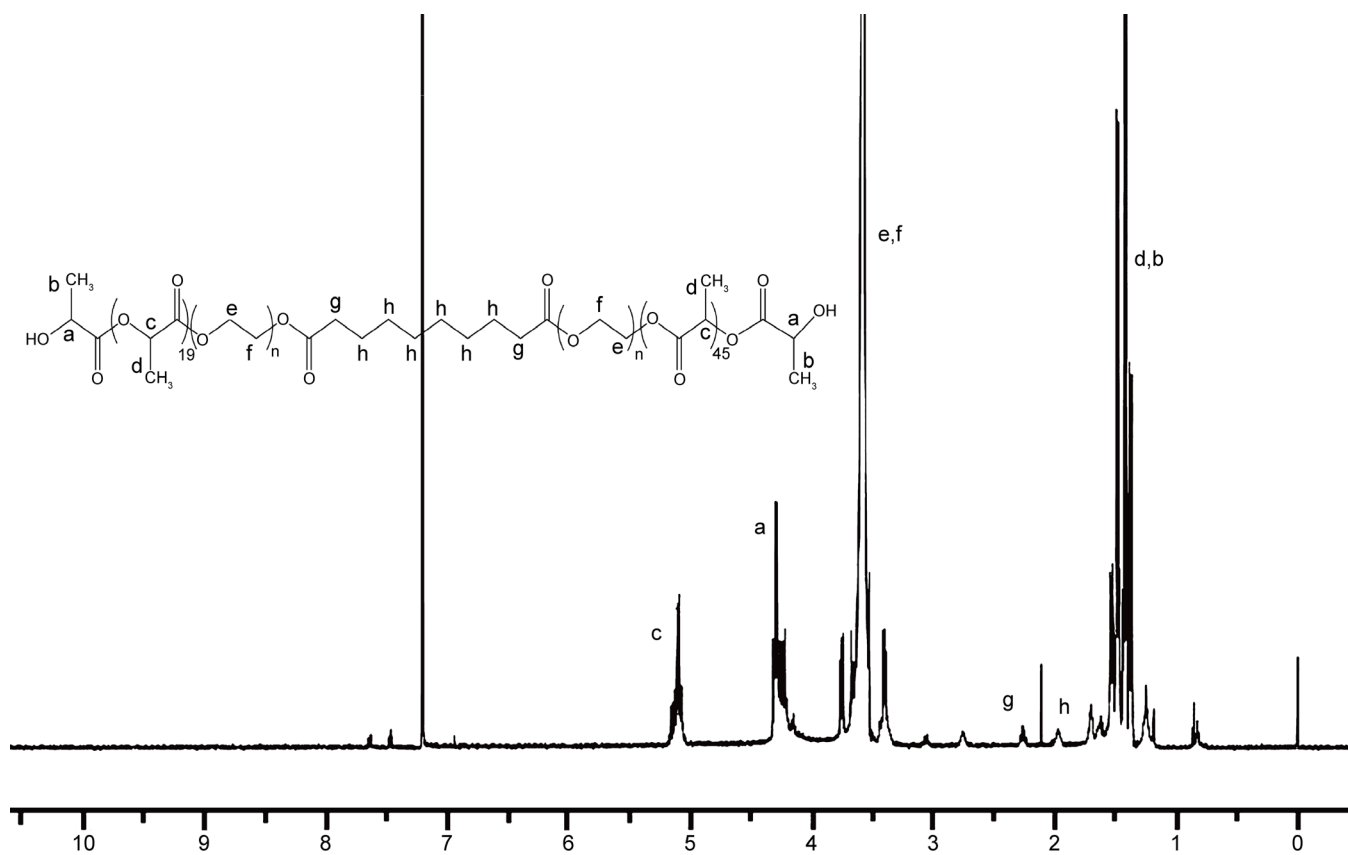
groups indicating that the formation of the PLA-PEG-SC-PEG-PLA block copolymers has taken place. By increasing the PLA chain length, the band associated with hydroxyl group would be less intense, increasing the band's intensity of carboxylic ester (C=O) and (C-O) groups was due to the increasing of molecular weight of the block copolymer with increasing the ratio of PLA in the copolymer, which caused less stretching for the hydroxyl group with increasing stretching of carboxyl one, and that confirmed that the reaction take place [14].

<sup>1</sup>H NMR technique was used to confirm the structure of the new prepared triblock copolymers (AL<sub>10</sub>, AL<sub>25</sub>, AL<sub>50</sub>, BL<sub>10</sub>, BL<sub>25</sub>, BL<sub>50</sub>, CL<sub>10</sub>, CL<sub>25</sub>, and CL<sub>50</sub>). <sup>1</sup>H NMR of (A, B, and C) polymers and triblock copolymers of PLA-PEG-SC-PEG-PLA were recorded on a Bruker 400 MHz spectrometer, using CDCl<sub>3</sub> (Sigma-Aldrich, 99.8% atom D). **Figure 3** shows the NMR spectrum of poly (ethylene glycol)-sebacate-poly (ethylene glycol) having (PEG 2000), polymer A; the methylene protons in (CH<sub>2</sub>) group of PEG were around 3.65 ppm (a and b), the peaks at 2.2 ppm represented the end methylene protons (CH<sub>2</sub>) of sebcoyl chloride bonded to PEG segments, and at 2 ppm represented the methylene (CH<sub>2</sub>) protons non bonded. The same position peaks were appeared for polymer B and C.

**Figure 4** exhibits the NMR spectrum of triblock poly (L-lactide)<sub>50</sub>-poly (ethylene glycol)-sebacate-poly (ethylene glycol)-poly (L-lactide)<sub>50</sub> (CL<sub>50</sub>), indicate the presence of methine (CH) and Methyl (CH<sub>3</sub>) protons in PLA was observed around 4.3 ppm (c) for (CH) of end group and around 5.2 to the (CH) bonded inside the polymer chain, and 1.3 - 1.5 ppm (d, b), for the (CH<sub>3</sub>) end group and inside group respectively. The methylene protons in (CH<sub>2</sub>) group of PEG were around 3.65 ppm (e and f), the peaks at 2



**Figure 3.** NMR spectrum of poly(ethylene glycol)-sebacate-poly(ethylene glycol) having (PEG 2000), polymer A.



**Figure 4.** NMR spectrum of triblock poly(L-lactide)<sub>50</sub>-poly(ethylene glycol)-sebacate-poly(ethylene glycol)-poly(L-lactide)<sub>50</sub> (CL<sub>50</sub>).

ppm (g) represented the end methylene protons ( $\text{CH}_2$ ) in the sebcoyl chloride bonded to PEG segments, the peak at 2.2 ppm (h) represented methylene protons ( $\text{CH}_2$ ) of sebcoyl chloride non bonded, the figure is very similar to the previously reported spectrum [15] [16], and confirm the synthesis of the PLA-PEG-SC-PEG-PLA block copolymers with three different units (10,25, and 50 units) on the end of the bis-poly (ethylene glycol) sebacate.

The molecular weight distributions (Mw/Mn) were calculated for all prepared triblock copolymers and **Table 3** shows the GPC results. It appears that the PDI is ranging between 1.02 - 1.10 indicating that the copolymers molecular weight distribution is nearly monodispers or from narrow distribution type. All these results are confirmed the expected structure and composition of the prepared copolymers and promoted this matter with great accuracy the results of GPC as shown significant closely with theoretical calculations of molecular weight and result the awesome polydispersity index (PDI) is close to one.

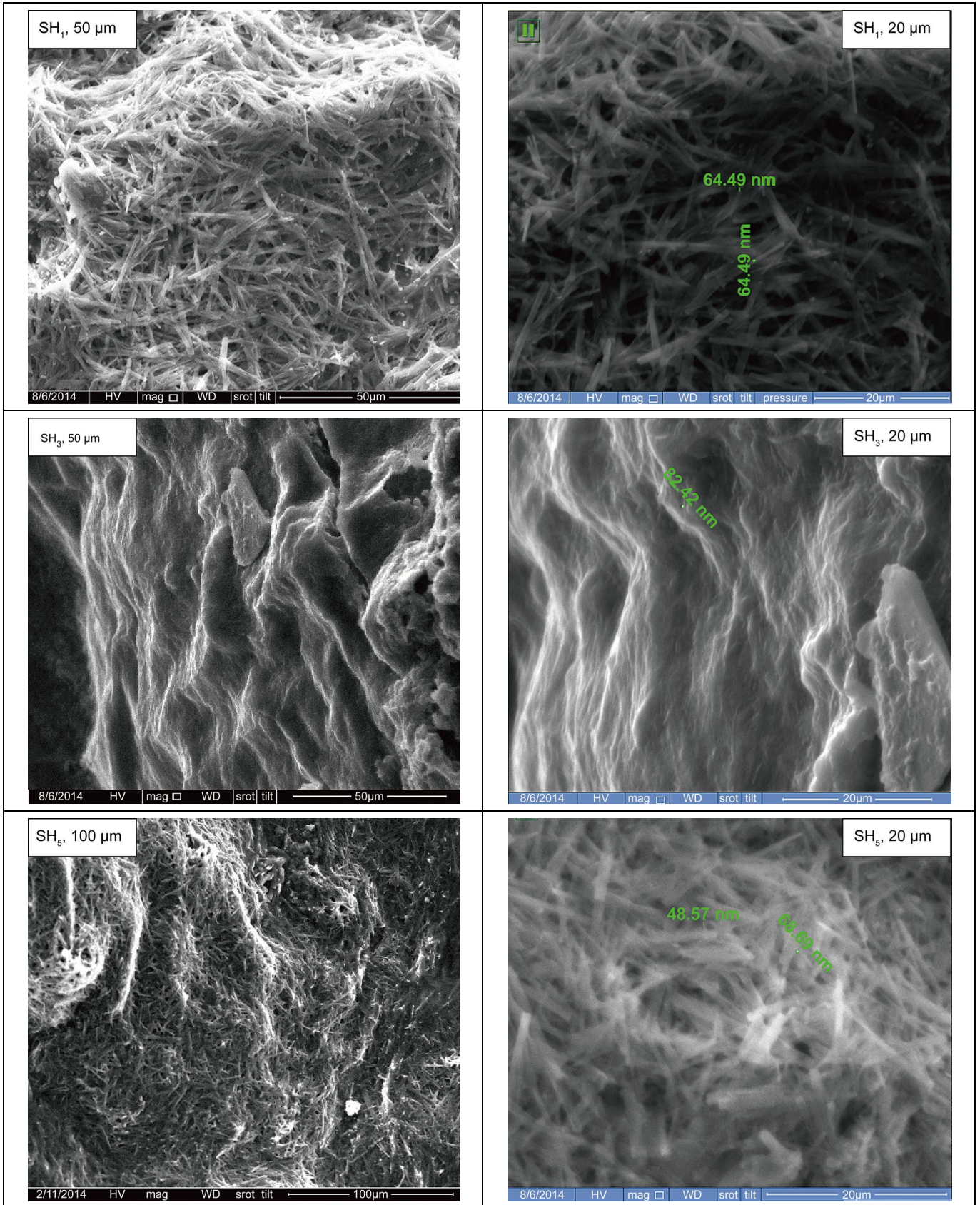
The examination of the morphology and fiber geometry of the IPN's was performed by using SEM. **Figure 5** shows the SEM images of uncoated xerogel IPN's which demonstrated the fibers of the prepared IPN's. The size of the fibers was found to be in the range of (48 - 97) nm and the images of SEM measurements showed the clarity in the fibrous characterization of the IPN's with increasing the PLA chain length. Prior to be used as tissue engineering scaffolds, alginate-based materials need to be crosslinked to preserve their structure in an aqueous environment. Ionic crosslinking with divalent ions (e.g.  $\text{Ca}^{2+}$ ) avoids potentially toxic materials used in chemical crosslinking reactions.

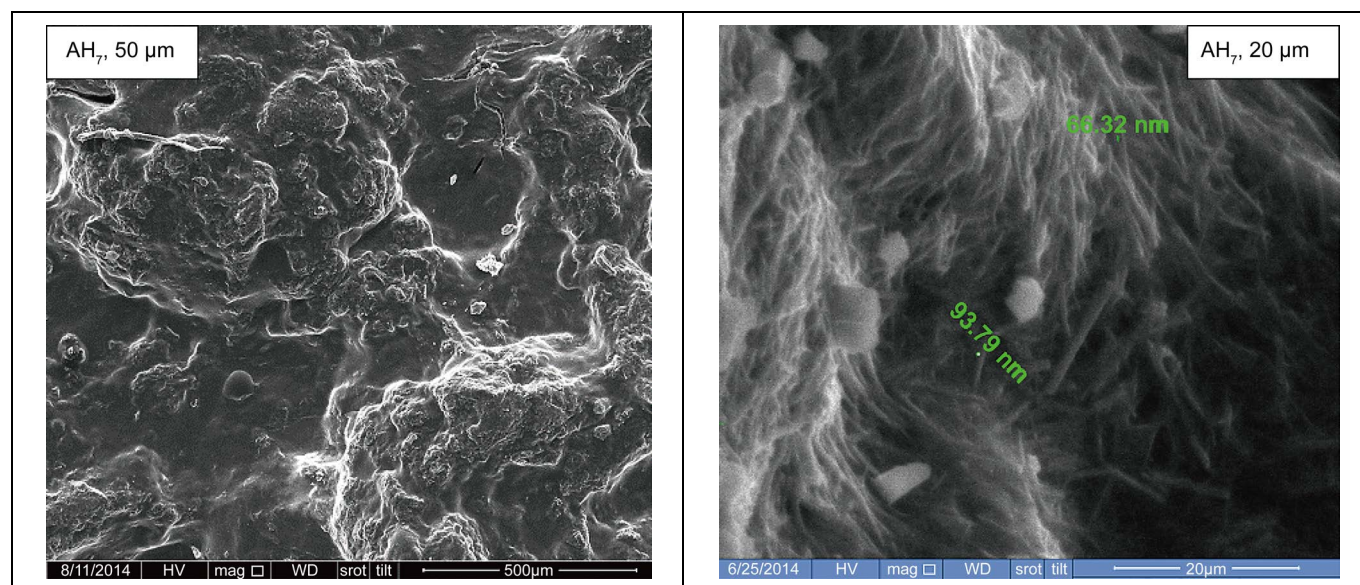
From the figure, it is easy to observe the presence of spherical structures due to crosslinking of sodium alginate by divalent calcium ion. Also, the SEM images reveal that it is possible for the nanofibers to cover the surfaces of particles and the gaps between them [17]. The miscibility of ethylene oxide in the pegelated lactide and alginate may explain the smoothness of the surface exhibited in the SEM micrograph. It is known that a single polymer cannot satisfy all the requirements of an ideal biomaterial,

**Table 3.** Molecular weights and polydispersity index obtained from GPC measurements.

Triblock copolymer	Calculated M. Wt	Mn (GPC)	Mw (GPC)	PDI
AL <sub>10</sub>	5642	5685	5861	1.03
AL <sub>25</sub>	7802	8084	8603	1.06
AL <sub>50</sub>	11,402	11,845	12,070	1.02
BL <sub>10</sub>	9642	9772	10,037	1.03
BL <sub>25</sub>	11,802	11,782	12,611	1.07
BL <sub>50</sub>	15,402	15,250	15,590	1.02
CL <sub>10</sub>	21,642	21,758	23,446	1.07
CL <sub>25</sub>	23,802	24,014	25,500	1.06
CL <sub>50</sub>	27,402	27,453	30,323	1.10







**Figure 5.** SEM images of fiber SH<sub>1</sub>, SH<sub>3</sub>, SH<sub>5</sub> and SH<sub>7</sub> at different amplifications.

such as poly (ethylene glycol) [18]. Therefore, it may need other combinations of polymers, like PEO and Na-alg., to increase the density of fibers on the mesh produced, which is the case here. The SEM micrographs of prepared fiber IPN's revealed that dense materials were obtained.

The relative swelling was determined gravimetrically after immersing uncoated discs in water, Simulated Intestinal Fluid (SIF), and Simulated Gastro Fluid (SGF). The degree of swelling was calculated according to the following equation:

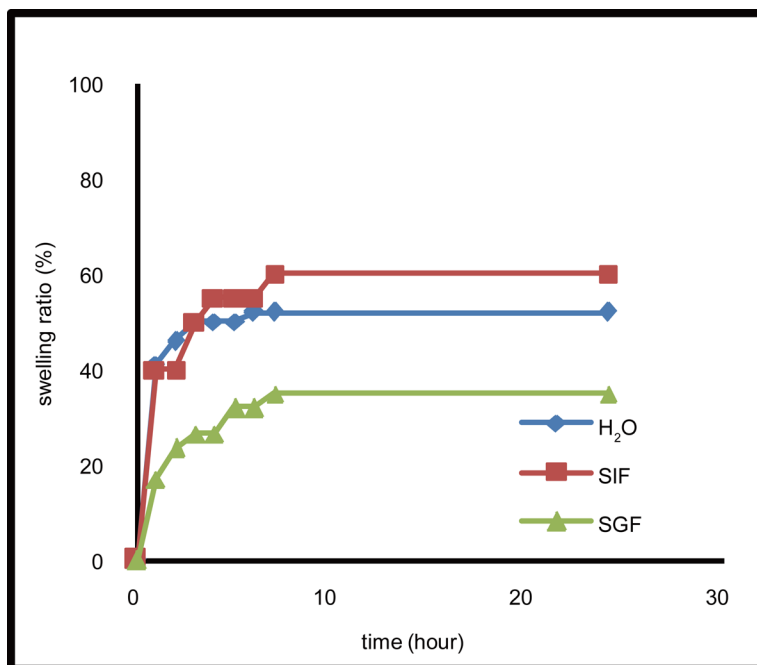
$$S_w (\%) = (W_s - W_d) / W_d \times 100$$

where,  $W_s$  and  $W_d$  represent the weight of swollen and dry samples, respectively.

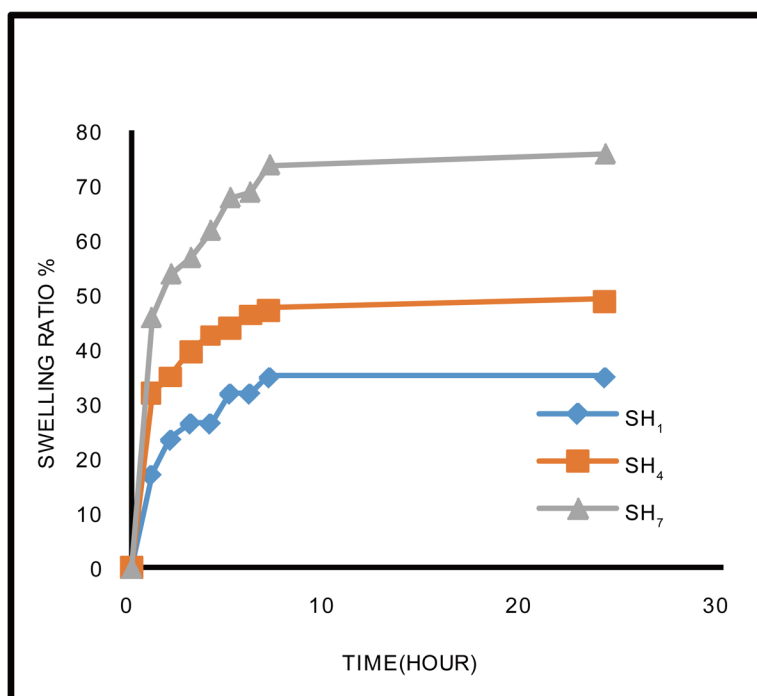
The study of swelling of uncoated fiber IPN's affected by a number of varieties, including changes in the pH of immersing solution, different molecular weight of PEG in the triblock copolymers in the IPN's, and changing of PLA chain length. As expected, the swelling ratio of the triblock copolymers increases with an increasing proportion of the hydrophilic block [19] [20].

Results in **Figure 6** shows the effect of pH (8.2, 7.2, and 1.2) onto the swelling ratio of the IPN's (SH<sub>1</sub>). Studies of swelling ratio shows that the pH has a significant effect because it was found that the swelling ratio increased with increasing pH. This can be related to their ability to form ionizable groups ( $\text{COO}^-$ ) in Sodium alginate which leads to increase the bonding characteristics of the fiber IPN's with water.

The swelling ratio increased with increasing poly (ethylene glycol) molecular weight due to increase the hydrophilic polymer chain length of PEG. The swelling ratio for fiber IPN's containing PEG 2000 (SH<sub>1</sub>), 4000 (SH<sub>4</sub>) and 10000 (SH<sub>7</sub>) are found to be equal to 60%, 79% and 90% respectively, and the same effect is found for all other fiber IPN's. **Figure 7** and **Figure 8** are shown examples for this effect.



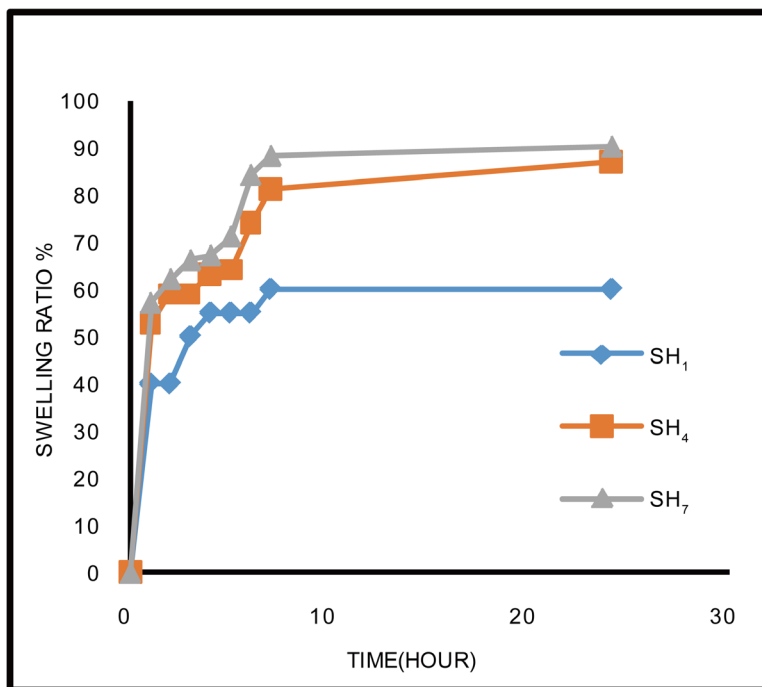
**Figure 6.** Effect of pH on swelling ratio of SH<sub>1</sub> in (SGF, SIF, and H<sub>2</sub>O).



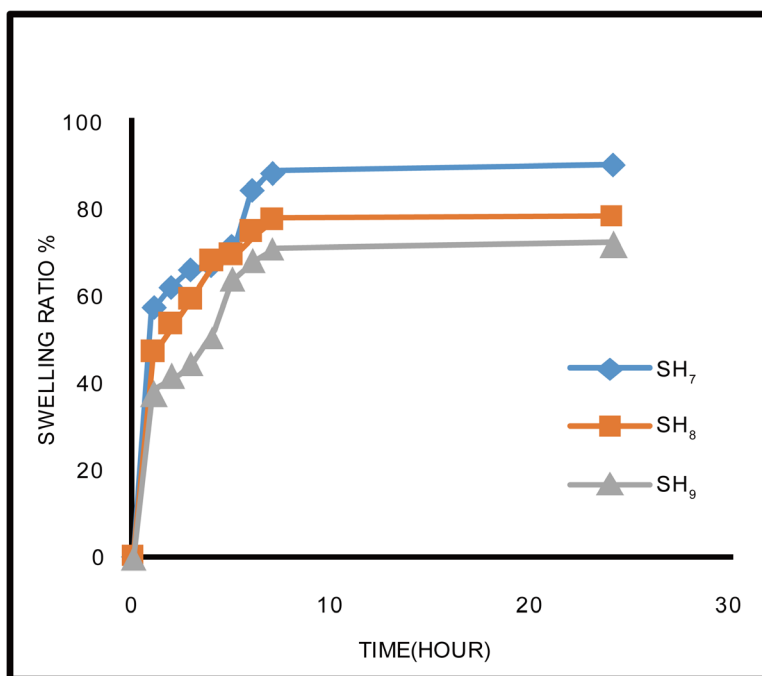
**Figure 7.** Effect of molecular weight of PEG on swelling ratio of SH<sub>1</sub>, SH<sub>4</sub>, and SH<sub>7</sub> in SGF.

In one series of block copolymer with the same molecular weight of PEG (AL<sub>10</sub>, AL<sub>25</sub> and AL<sub>50</sub>), the swelling ratio of fiber IPN's (SH<sub>1</sub>, SH<sub>2</sub>, and SH<sub>3</sub>) decreased with increasing of PLA chain length, because of the increasing of LA ratio will increase the hydrophobicity of the copolymers.

The swelling ratio decreased with increasing lactide chain length because the lactide groups represent the hydrophobic groups in the new triblock copolymer chains leading to decreasing of water uptake from the fiber IPN's. **Figure 9** and **Figure 10** are exhibited examples of this effect.



**Figure 8.** Effect of molecular weight of PEG on swelling ratio of SH<sub>1</sub>, SH<sub>4</sub>, and SH<sub>7</sub> in SIF.



**Figure 9.** Effect of lactide chain length on swelling ratio of SH<sub>7</sub>, SH<sub>8</sub> and SH<sub>9</sub> in SIF.

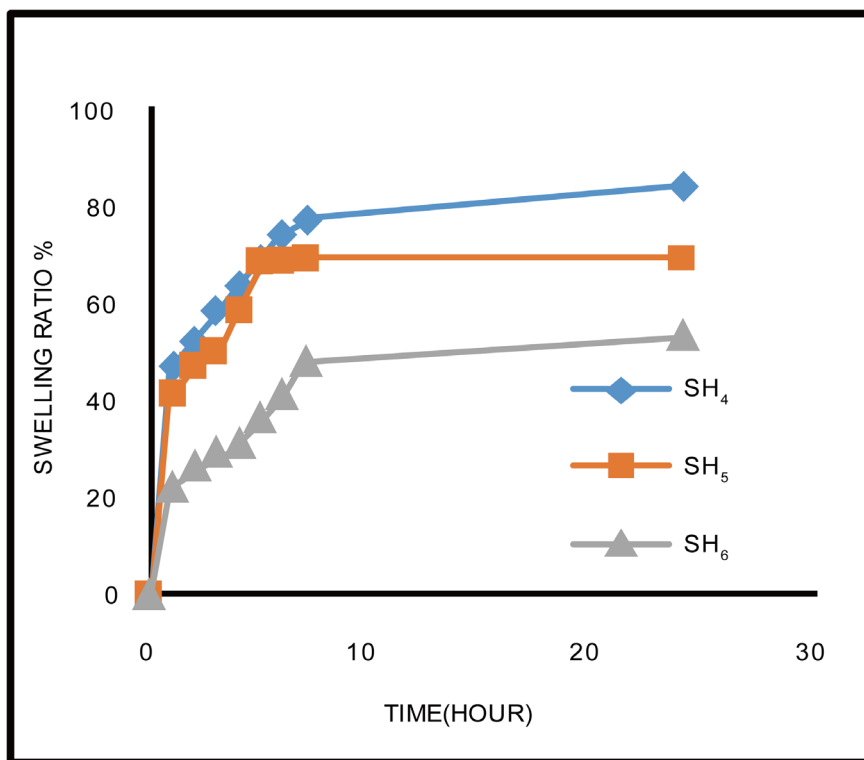


Figure 10. Effect of lactide chain length on swelling ratio of SH<sub>4</sub>, SH<sub>5</sub> and SH<sub>6</sub> in H<sub>2</sub>O.

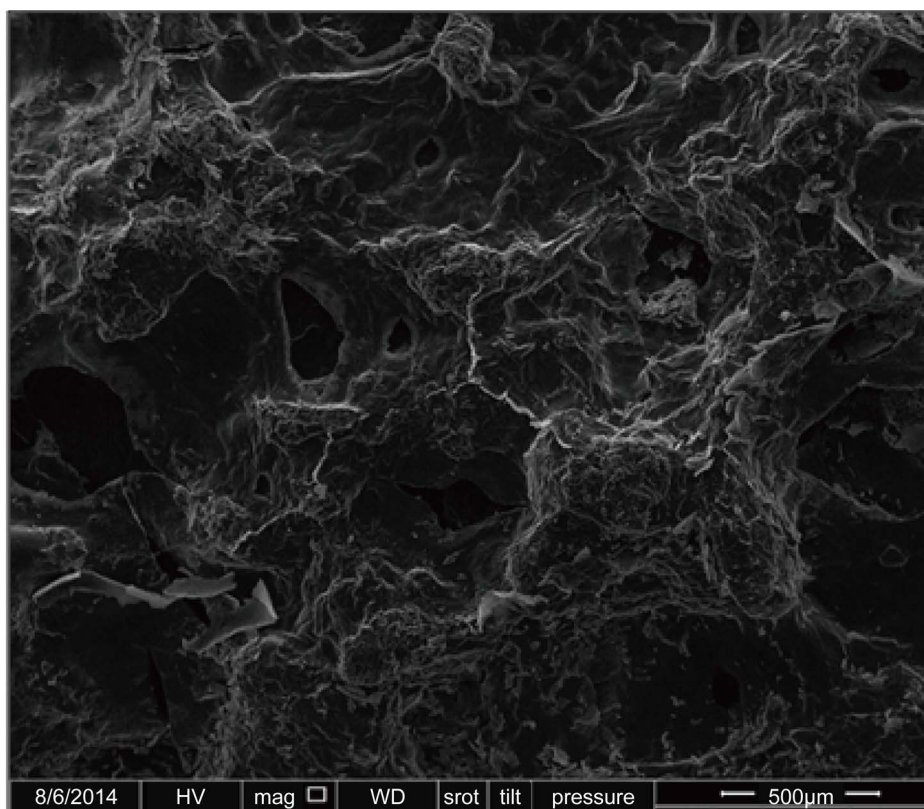


Figure 11. SEM image of swelling fiber SH<sub>5</sub> in water.

The surface of the fiber IPN's prepared was observed in the sinking area, due to the swelling characteristics of poly (ethylene glycol), the size of pores was found to be in the range of (3.406 - 15.346)  $\mu\text{m}$ , and **Figure 11** shows the SEM image of SH<sub>9</sub> IPN's in water.

#### 4. Conclusion

The spectroscopic characterization revealed that true triblock copolymers were prepared with narrow molecular weight distribution. The swelling results imply the possibility of using these polymeric IPN's in the field of polymer drug delivery especially for Insulin delivery.

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