# Obtaining of Biodegradable Composite Films Based on Polyethylene and Pectin

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#### ABSTRACT

In this work, biodegradable polymer films based on a modified pectin low-density polyethylene (LDPE) composition were obtained, where a graft copolymer of LDPE with maleic anhydride (LDPE-g-MA) was used as a compatibilizer. Chemical modification of pectin was carried out by grafting acrylamide using cerium ammonium (IV) nitrate as an initiator. The characteristics of the graft copolymer and the resulting composition were studied by FTIR spectroscopy, X-ray diffraction, and thermal gravimetric analysis (TGA). The degree of grafting was determined to be 71% and the yield of the final product was 14.2% of copolymerization. It was shown that the polymer composition is susceptible to biodegradation, and a weight loss of 23% during 45 days was revealed.

Keywords: pectin, modification, graft copolymerization, LDPE, biodegradable film.

#### **INTRODUCTION**

Plastic is one of the most essential materials in our daily life. However, environmental pollution from consumable plastics in the form of packaging materials, disposable containers, and films is becoming a serious problem [1]. In connection with the emerging problem of the rapidly growing use of synthetic thermoplastics, research and synthesis of biodegradable polymers is a very urgent task [2]. There is an increased interest in the creation and use of fully biodegradable polymers and the final purpose of it is replacing synthetic plastics [3]. The development of biodegradable films based on polyolefins has attracted great interest due to their low price, special and operating properties. It is generally accepted that the complete degradation of films based on polyolefins includes two stages: oxidative degradation (oxodegradation) and biodegradation [4-6]. In this connection, the addition of natural fillers to synthetic polymers is one of the interesting approaches to impart biodegradable properties to them [7]. Among the various types of natural fillers such as starch [8], cellulose and its derivatives [9], chitosan [10], lignin [11], pectin has undoubtedly scientific and practical interest, the main chain of which consists of 1,4-linked residues of α-D-galacturonic acidcontaining partially esterified carboxyl groups [12]. It has been used successfully for many years in the food industry as a thickener, gelling agent and a colloidal stabilizer [13]. However, pectin has poor stability when exposed to shear forces during processing, because, its melting point is higher than the degradation temperature, it cannot be processed by traditional technologies used for thermoplastic polymers [14,15]. In this connection, it is advisable to modify it to improve processing properties and compatibility with polyolefins by chemical modification. Considerable interest has been focused on the modification of natural polysaccharides such as cellulose and starch by grafting with a synthetic polymer [16-19]. It was reported about the use of a cerium ion to initiate the graft copolymerization of pectin with vinyl monomers [20], such as methyl acrylate, ethyl acrylate, and ethyl methacrylate [21], acrylamide (AAm) [22] and 4-vinylpyridine [23]. In general, it has been observed that graft copolymers having long macromolecules with less branching are stable during processing. A large number of graft copolymers were obtained by grafting acrylamide onto polysaccharides such as starch and carboxymethyl cellulose [24,25].

In addition, thermoplastic polymers such as polyethylene, polypropylene, polyvinyl chloride are not compatible with polysaccharides, including pectin, due to their differences in nature, which affects their performance properties. Compositions based on them with improved properties can be obtained by using a third component, a compatibilizer [26]. To obtain a homogeneous mixture of LDPE, a copolymer of ethylene and vinyl acetate (EVA) [27], PP-g-MA [28], and LDPE-g-MA [29] are used as compatibilizers.

In the present research, an attempt was made to obtain biodegradable films based on PE and pectin modified by grafting with acrylamide and studying their properties.

#### **Methods and Material**

We used apple pectin with an average molecular weight of 147 kDa (degree of esterification 55%) manufactured in Germany (Herbstreith & Fox), acrylamide (98% degree of purity) manufactured in Shandong, China (MX CHEM), ammonium cerium (IV) nitrate chemically pure, made in France (Biochem), maleinized polyethylene compatibilizer (grafting degree 2%, density =  $0.86 \text{ g/cm}^3$ ) made in the USA (DOW Chemical), linear low-density polyethylene (LLDPE) grade F-0320 (density 0.918-0.922 g/cm<sup>3</sup>) production Uzbekistan (UNG Shurtan GKM).

#### Modification of apple pectin with acrylamide

Pectin (1 g) was dissolved in 80 ml of distilled water with continuous stirring at room temperature. The resulting solution was purged with nitrogen for 30 minutes to remove dissolved oxygen. Then, acrylamide (5 g) and 4 ml of a freshly prepared aqueous solution of ammonium-cerium (IV) nitrate (0.04 g/ml) was added to the reaction mixture with constant stirring. During the reaction, the mixture was kept in a nitrogen atmosphere to avoid interaction with oxygen for 5 h with continuous stirring. After completion of the reaction, the graft copolymer was precipitated in an excess of ethyl alcohol. The product was purified by extracting unreacted components, including polyacrylamide homopolymer (which can be obtained during polymerization) by washing with an ethyl alcohol-water mixture (30:70). The procedure was repeated 5 times. Thus obtained pure modified pectin was finally washed with ethyl alcohol and dried in a drying oven for 12 h at a temperature of 60°C.

The degree of grafting G (%) and the yield of the final product Y (%) were determined by the gravimetric method and calculated as follows [30].

$$G (\%) = \frac{(W_2 - W_1)}{W_1} \times 100 \%$$
$$Y (\%) = \frac{(W_2 - W_1)}{W_3} \times 100 \%$$

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where  $W_1$ ,  $W_2$ , and  $W_3$  are the mass of pectin, copolymer after drying, and the mass of monomer, respectively.

# Composite obtaining

Compositions were prepared by mixing LLDPE in the presence of a compatibilizer with modified pectin, under the action of high-temperature shear deformations on a rotary laboratory mixer of the Brabender type with a rotational speed of 60 rpm at a temperature of 160°C, varying the content of modified pectin from 10 to 30% in the composition. Film samples were obtained by pressing compositions on a hand-held hydraulic press of a circular shape with a diameter of 6 cm and a thickness of 60  $\mu$ m.

# FTIR spectroscopy

FTIR spectra were measured on an INVENIO S spectrometer (Bruker, Germany). The spectra were recorded in transmitted light at  $T = 23 \pm 2^{\circ}C$  in the wavelength range  $4600 \le v \le 450 \text{ cm}^{-1}$ .

# TGA

Thermogravimetric analysis of the samples was carried out on a TGA DSC STA PT 1600 derivatograph (Linseis, Germany). The mass of the samples was 5-10 mg, the heating rate was 10°C/min, the analyzed temperature range was 50-600°C.

# X-ray diffraction

X-ray studies were carried out on a DRON-3M diffractometer with monochromatic Cu K $\alpha$  radiation at a voltage of 20 kV and an amperage of 10 mA.

# Water absorption

Film samples of the material were placed in test tubes with distilled water. The tubes were placed in a thermostat with a temperature set in it of 30°C. At certain time intervals, the tubes were removed from the thermostat, the samples were removed from the tubes, blotted with filter paper, weighed, and placed back. The experiment was carried out for 45 days, till reaching equilibrium water absorption by all materials. When the samples are immersed in water, two oppositely directed processes occur simultaneously: water absorption and leaching of the filler. Unfortunately, it is not possible to take into account the factor of washing out of the filler from the sample at each weighing, but it is possible at the last weighing. Thus, after weighing the samples on the 45th day of exposure in water, they were brought to constant weight in an oven at 100°C. The percentage of water absorption is calculated as follows:

Water absorption = 
$$\frac{(W_2 - W_1)}{W_1} \times 100 \%$$

where  $W_2$  and  $W_1$  are the final weight and dried weight of the sample, respectively. The difference in the mass of the original and the dried sample after the experiment on water absorption is directly indicative of the washout effect.

### Biodegradability

The biodegradability of the test sample was studied using the weight loss of the composite in some time in the soil. A round film sample with a diameter of 6 cm and a thickness of 60  $\mu$ m was weighed on an analytical balance and then buried in boxes with holes containing soil. The soil moisture was maintained at about 30% of the soil mass, and the sample was buried to a depth of 15 cm. The sample was removed from the soil every 15 days, washed in a water-alcohol solution, dried, and weighed. The percentage of weight loss was calculated using the following equation.

Veight loss% = 
$$\left[\frac{(M_o - M_d)}{M_o}\right] \times 100$$

where  $M_o$  is the initial mass of the sample, and  $M_d$  is the mass of the sample during the decomposition period for every 15 days.

#### **RESULTS AND DISCUSSIONS**

Pectin was modified by grafting with acrylamide. The suggested vaccination mechanism is shown in the diagrams below. The reaction begins with the formation of a complex of cerium IV with hydroxyl groups –OH at  $C_2$  and  $C_3$  of the elementary unit of the pectin macromolecule (**Scheme 1**).



Scheme 1. Formation of cerium IV complex

Then, oxidation occurs with rupture of the pyranose ring between  $C_2$  and  $C_3$ , and the formation of a macroradical initiating the grafted polymerization of the monomer (**Scheme 2**).



**Scheme 2. Oxidation** 

Chain growth is terminated by the interaction of the growing chain macroradical with  $Ce^{4+}$  (Scheme 3).



Scheme 3. Chain termination

The influence of conditions on the yield of the final product and the degree of grafting of copolymerization was investigated.

**Table 1.** Change in the degree of grafting of the copolymer and the yield of the final product from the reaction conditions and temperature

The ratio of the components in mass parts	Condition	T (°C)	G (%)	Y (%)
1:0,3	atmospheric	60	2,8	8,4
1:2,0	N <sub>2</sub> atm	25	22	11
1:3,0	atmospheric	80	30,5	10,17
1:5,0	$N_2$ atm	25	71	14,2

G (%) - percentage grafting

Y (%) - yield of graft copolymerization

**Table 1** shows the degree of grafting and the yield of the final product data dependence of the acrylamide amount. The increase in the amount of acrylamide in the reaction mixture leads to the increase in the degree of grafting and the yield of the final product. The optimal conditions for the modification of pectin were found: the ratio of pectin to acrylamide 1:5, temperature 25°C, time 5 hours.

The obtained copolymer was studied by FTIR spectroscopy. **Figure 1** shows the FTIR spectra of pectin, acrylamide, and copolymer based on them. In the spectrum (**Figure 1** (1)) between 3600 and 2800 cm<sup>-1</sup>, absorption bands characteristic of pectin can be observed: v O– H (peak 3424.10 cm<sup>-1</sup>) and v C–H (peak 2929.97 cm<sup>-1</sup>). And also between 1800 and 1500 cm<sup>-1</sup> of the spectrum, two bands can be observed at 1754 cm<sup>-1</sup> and 1632 cm<sup>-1</sup>, which are associated with v C=O in COOCH<sub>3</sub>, COOH and with v C=O in COO<sup>-</sup>, respectively. The

FTIR spectrum of acrylamide (**Figure 1** (2)) contains the (v NH<sub>2</sub>) vibration of amide-I linked by hydrogen bonds at 3353.54 cm<sup>-1</sup> and 3190.35 cm<sup>-1</sup>, absorption bands at 1675.04 cm<sup>-1</sup> (v C=O) and at 1613.76 cm<sup>-1</sup> ( $\delta$  NH<sub>2</sub>) corresponding to amide-I and amide-II. Wide vibration band –NH 700-600 cm<sup>-1</sup>. The copolymer spectrum combines bands associated with pectin and acrylamide (**Figure 1** (3)). The peak at 3424.10 cm<sup>-1</sup> refers to both –OH and –NH groups. The main changes occur in the 1400-1800 cm<sup>-1</sup> region, the peak at 1744 cm<sup>-1</sup>, which is characteristic of carbonyl groups in pectin.



**Figure 1.** FT-IR spectrum of pectin (1), acrylamide (2), and modified pectin (3) The characteristic peak at 1648  $\text{cm}^{-1}$  was due to the band of amide-I of the amide group of polyacrylamide. These data confirm the grafting of acrylamide respectively to pure pectin.

Figure 2 presents the TGA of pectin and graft copolymer.

The figure indicated that pure pectin showed the presence of a small endothermic peak at 96°C, corresponding to the glass transition temperature (Tg) of pure pectin. There is another endothermic peak, observed at about 184°C, and it can be attributed to the melting temperature (Tm) of pure pectin.

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TG Pectin — TG Pectin-AAm – – – DTA Pectin – – – DTA Pectin-AAm

Figure 2. TGA of pectin and graft copolymer

The weight loss curve of the grafted pectin indicated that there are small endothermic peaks. The first peak at temperature 71°C is due to the segmental motion of the molecules within the grafted polymer chain and it can be assigned to the glass transition temperature (Tg) of the grafted pectin. The second peak at temperature 111°C indicates the melting temperature (Tm) of the latter. The decrease in Tg and Tm may be explained by the disruption of the crystalline structure during the grafting of pectin with polyacrylamide, which indicates the formation of the graft copolymer.

The composition was obtained by mixing LLDPE and the compatibilizer (LDPE-g-MA) with modified pectin, varying the content of the latter from 10 to 30%.

Sample code	LDPE (%)	Pure pectin (%)	Modified pectin (%)	Compatibilizer (%)	Temperature (C°)
PPCPE 1	85	10	_	5	180
PPCPE 2	75	20	_	5	170
PPCPE 3	65	30	-	5	160
PPCPE 4	80	10	_	10	150
PPCPE 5	70	20	-	10	140
PPCPE 6	60	30	_	10	130
MPCPE 1	85	_	10	5	180
MPCPE 2	75	—	20	5	170

Table 2. Parts of composition

MPCPE 3	65	_	30	5	160
MPCPE 4	80	_	10	10	150
MPCPE 5	70	_	20	10	140
MPCPE 6	60	_	30	10	130

PPCPE - Pure pectin/ Compatibilizer /LDPE

MPCPE - Modified pectin / Compatibilizer /LDPE

In the process of mixing the temperature regime of the extruder was change depending on the composition (**Table 2**). Samples containing pure pectin were dark in color due to the charring of the pectin. Such samples do not meet technical requirements. The mixing temperature is also influenced by the compatibilizer, which acts as a performing agent to improve the mechanical and technological characteristics. The principle of the compatibilizer operation is explained by the peculiarity of its structure. It contains both hydrophilic and hydrophobic moieties, allowing them to bind the polar filler to the non-polar polymer. By varying the content of the latter from 5 to 10% the optimal formulation of composition and the mixing temperature was found.

Peaks are observed in the FTIR spectra of the composition (**Figure 3**) at 2922.68 cm<sup> $^-1$ </sup>, 2850.3 cm<sup> $^-1$ </sup>, 1465.73 cm<sup> $^-1$ </sup>, 727.86 cm<sup> $^-1$ </sup> interpretive LDPE and at 3439.77 cm<sup> $^-1$ </sup>, 1672.12 cm<sup> $^-1$ </sup>, 1130.06 cm<sup> $^-1$ </sup>, 1026.57 cm<sup> $^-1$ </sup> modified pectin.



**Figure 3.** FTIR spectrum of LDPE (1), modified pectin (2), and compositions based on them (3)

In addition, a new peak appears at 1079.95  $\text{cm}^{-1}$ , indicating the association of the amide group of the modified pectin and the carbonyl group of LDPE-g-MA.

Figure 4 shows a comparative curve of TG, LDPE, its composition with modified pectin. Kinetic parameters can reveal changes that occur in molecular chains as a result of the

degradation process. The onset temperature of the LDPE indicates the only degradation zone that occurs at 339°C for the LDPE. The thermogram for the composition displays two stages of degradation.  $T_{onset}$  appears at 225°C and  $T_{peak}$  appears at 485°C.



#### Figure 4. TGA of LDPE and composition

Kinetic parameters such as decomposition temperature (DT), integral decomposition temperature (IDT), and activation energy ( $E_a$ ) are calculated (**Table 3**).  $E_a$  is calculated by the Broydo equation [31].

It can be seen from **Table 3**, that the  $E_a$  value decreases in the order LDPE> Modified pectin / Compatibilizer /LDPE > Pure pectin/ Compatibilizer /LDPE and is 8.02, 7.95, and 7.54, respectively.

N⁰	Simple code	The onset temperature of each stage (°C)		DT (°C)	IDT	Ea (kJ/mol)	
		Ι	II	III			
1.	LDPE	335	_	_	446	440.6	8.12
2.	PPCPE	209	435	_	428	433.2	7.53
3.	MPCPE	225	485	_	451	442.3	7.95

Table 3. Thermal kinetic parameter of PPL and GPAmL

Consequently, the activation energy of each composite system is showing a gradual decrease in trend as compared to pure LDPE. This may be due to the improved interfacial interaction between modified pectin and LDPE due to the presence of acrylamide, which increases their affinity, through interaction with LDPE-g-MA. The diffractogram (**Figure 5**) of LDPE (A) shows crystalline peaks at 21.43° and 23.82°  $2\theta$  typical of LDPE, while the diffractogram of composition (B) shows a peak at 19.50°  $2\theta$  attributable to modified pectin [32], and the percentage crystallinity of LDPE and the composition was 73.5% and 40%, respectively.



**Figure 5.** X-ray diffraction analysis of (A) LDPE and (B) Composition Diffraction patterns showed the decrease in the crystallinity of LDPE when it is filled with modified pectin. From the diffraction patterns, it can be concluded that the decrease in crystallinity in the composition may be associated with the inclusion of more bulky groups of modified pectin in the polymer matrix of LDPE, which, in its turn, reduces intermolecular hydrogen bonds, and this depends on the amount of acrylamide used in the modification of pectin.



Pure pectin as well as its graft copolymer with acrylamide are water-soluble.

Figure 6. Water absorption

According to **Figure 6**, there is a slow water absorption during all the experiment. Further, in a day, the increase in the water absorption of the samples is observed. Filled compositions

have higher water absorption rates than LDPE, which increases the availability of materials for microorganisms.

In this connection, the biodegradable properties of compositions with pure and modified pectin can be determined by three phases of the soil burial test. The first stage is a surface erosion process resulting from a microbial attack on the amorphous polymer-pectin chain when weight loss begins. At the second stage, deeper microbial contamination occurs, which, together with moisture, leads to extensive degradation of the material. In the last phase, the decrease in the pectin content leads to the decrease in the number of microorganisms. Most of the research has been done to characterize biodegradation on thin films. The analysis of the first stage included an assessment of the stepwise weight loss of each sample during 45 days in 10% compost in the minimum environment shown in **Table 4**. **Table 4**. Biodegradation of PPL and GPAmL

N⁰	Simple code	15-day weight (%) reduction	30-day weight (%) reduction	45-day weight (%) reduction
1	PPCPE	10	14	20
2	MPCPE	11	16	23

In most cases, weight loss increased with increasing pectin content and time. The moisture absorption of the samples is mainly due to pure and modified pectin. Compositions with higher moisture absorption are generally more susceptible to attack by microorganisms. This likely allows microorganisms such as bacteria and fungi to access the bottom of the polymer using water as a medium; This suggests that microorganisms consume pure and modified pectin as a nutrient medium, which leads to the destruction of the surface and structure of the polymer. Compositions with modified and pure pectin have a polar nature; therefore, contact with water is higher, which leads to their greater degradation. As shown in **Table 4**, after 15 days, the maximum weight loss obtained with the modified pectin composition is 11%. When the biodegradation test was completed after 45 days, the modified pectin LDPE composition showed the maximum percentage of weight loss compared to the composition containing pure pectin.

# CONCLUSION

Chemical modification of apple pectin was carried out by graft copolymerization with acrylamide. By choosing the synthesis conditions, the degree of grafting was reached 71%. Compositions based on Pure pectin/ Compatibilizer /LDPE and Modified pectin / Compatibilizer /LDPE of various compositions were obtained, the structure of the sample was studied using IR, X-ray structural, and thermal analyzes. The miscibility between pectin or modified pectin and LDPE was improved by LDPE-g-MA as a compatibilizer. A decrease in the thermal properties and kinetic parameters of the composite is observed due to the addition of a natural polymer to LDPE. According to the observation results, the thermal stability of the modified pectin was close to that of LDPE. The biodegradability of the composition was investigated by determining water absorption and changes in morphology and mass in the soil. The results show the decrease in weight during time, and the highest rate of weight loss was observed after 45 days, at 23%.

The results of the present work allow us to conclude that the graft copolymer of pectin and acrylamide can be used as a filler for LDPE with a content of 30% to obtain a biodegradable composition.

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