

Effect of Corn Starch Content in Thermoplastic Starch/Low-Density Polyethylene Blends on Their Mechanical and Flow Properties

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ABSTRACT: The aim of this work is to prepare low-density polyethylene (LDPE)/thermoplastic corn starch (TPCS) compounds containing the highest amount of starch for biodegradable applications. It is intended to increase concentration of corn starch into LDPE without affecting basic required mechanical properties of LDPE plastics for disposable products. LDPE/TPCS blends containing different contents of TPCS (0–40 wt %) and a constant amount of LDPE grafted maleic anhydride [PE-g-MA (3 wt %)] are prepared using a single-screw extruder. The prepared blends are evaluated for their mechanical, flow, and water absorption properties. Scanning electron micrographs of the samples show improvement in dispersion of the starch particles in LDPE matrix in the presence of PE-g-MA as a compatibilizer. The Young's modulus and impact strength properties

reduce by increasing corn starch content in TPCS. The ultimate tensile strength and elongation at break of the samples show that the blends with 25 wt % TPCS have the required mechanical properties to produce plastic packaging products, as evidenced by ASTM D 4976-04. The results of rheological tests indicate that as shear rate increases, the apparent viscosity of the blends decreases (shear thinning). The addition of starch to LDPE also decreases the melt flow index values of the samples. The water absorption of the samples increases with increasing starch concentrations at a constant time of water immersion. © 2012 Wiley Periodicals, Inc. *J Appl Polym Sci* 000: 000–000, 2012

Key words: corn starch; concentration; mechanical properties; flow behavior; water absorption

INTRODUCTION

Polyolefins have significantly obtained a main position in packaging industry because of their low cost, light weight, required properties, and low-energy consumption during their processing.^{1,2} Among these polymers, LDPE, which is hard to degrade in environment, is one of the fastest growing commercial thermoplastic materials. However, the continuous use of polyethylene (PE) plastics in different applications leads to the growing problem of environmental pollution. To overcome this problem, production of degradable and biodegradable polyolefins is necessary.^{3,4}

Corn Starch is an inexpensive, renewable material which satisfies the requirement of adequate thermal stability, minimum interference with flow properties, and minimum disturbance of product quality.⁵ Griffin used granular starch as filler in PE matrix to increase the biodegradability of its blends.⁶ Starch-filled PE blends due to incompatibility showed poor mechanical properties. To improve their compatibil-

ity and enhancing interfacial adhesion between phases, various attempts have been made to modify either starch^{7–10} or PE.^{11,12} Also, utilizing a plasticizer to improve dispersion of starch in PE matrix and promoting interfacial adhesion between PE and starch has been investigated.^{13,14} Some materials, such as glycerol, sorbitol, glycol, urea, ethanolamine, ethylenebisformamide, and water are usually used to plasticize starch.^{15–20} Starch granules swell after absorbing plasticizers, like glycerol, through hydrogen bonding with their free hydroxyl groups, but they still retain their order and crystallinity.²¹ When these swollen granules are heated, the semicrystalline structure of starch changes into a homogeneous one and the hydrogen bonds are formed between plasticizer and starch.²²

The melt blending of thermoplastic starch with PE has been studied^{23–25} and it was reported that LDPE-plasticized starch blends performed better than dry granular starch blends.²⁶ It was also found that PE-plasticized starch blends due to good homogeneous distribution of starch in the PE matrix had more biodegradability rate than those with native starch.²⁷

The mechanical, water absorption properties, and biodegradation of PE-thermoplastic starch blends have already been studied.^{28–32} But, investigation of

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melt rheology of the LDPE-thermoplastic starch blends compatibilized with LDPE-grafted maleic anhydride (PE-g-MA) is scarce. Yin et al.³³ characterized rheological behavior of these blends containing ethylene acrylic acid as a compatibilizer and concluded that the blends showed shear-thinning behavior. They also indicated that viscosity of the blends has an Arrhenius dependence on temperature and a power law dependence on shear rate.

Gupta et al prepared and characterized biodegradable LDPE-glycerol plasticized potato starch composites and films using equal amounts of LDPE and LDPE-g-MA with various starch contents.^{32,34} LDPE-thermoplastic starch blends have also been prepared by Wang et al.¹⁴ They studied tensile properties of the blends and reported the homogenous distribution of starch in LDPE matrix and improvement in the interfacial adhesion between starch and LDPE by using glycerol as a plasticizer and PE-g-MA as a compatibilizer.

In this work, we used glycerol as a plasticizing agent to improve the dispersion of starch in LDPE and to reduce agglomeration of the starch particles. The interfacial adhesion between LDPE and starch was improved with the addition PE-g-MA as a compatibilizer.^{35,36} The aim of this work is to prepare a LDPE/thermoplastic corn starch (TPCS) blend containing the highest amount of starch without affecting the required mechanical properties of LDPE for biodegradable applications. To investigate processing behavior of the blends by considering flow property and its sensitivity to water absorption, these properties were also measured.

EXPERIMENTAL

Materials

Low-density polyethylene [LDPE, film grade, with density 0.924 g/cm³ and a melt flow index (MFI) of 4.7 g/10 min (at 190°C, 2.16 kg load)] which is appropriate for packaging usage was obtained from Aria Sasol Polymer Petrochemical, Iran. The corn starch used was unmodified commercial grade (30% amylose and 70% amylopectin, wt/wt) which was supplied from Glucosan, Iran. PE-g-MA containing 2 wt % maleic anhydride was prepared using the method reported previously.³⁷ The amount of grafted maleic anhydride on LDPE was measured using the method reported in the literatures.³⁸ Glycerol was reagent grade from Hansa, Germany.

Sample preparation

Corn starch was dried in a vacuum oven for 24 h at 80°C. TPCS was prepared by pre-mixing corn starch in powder form with 35 wt % liquid glycerol at

room temperature. The mixtures were stored under airtight conditions for a day until starch granules were swelled. Then, the homogeneous compound was melt-blended using a Haake internal mixer, model SYS 90 (with a volumetric chamber capacity of 300 cm³) at 140°C with rotor speed of 60 rpm for 8 min. The mixture was taken out from the chamber and cooled to room temperature.

TPCS was melt-blended with LDPE in a laboratory scale Brabender single-screw extruder [screw diameter (d) = 19 mm, screw ratio (L/D) = 25 : 1]. The screw speed was 60 rpm and the temperature profile along the extruder barrel was 180, 190, 185, and 190°C (from feed zone to die). TPCS loadings were varied from 0, 10, 20, 25, 30, and 40 wt %. PE-g-MA was used at a constant amount of 3 wt % on the basis of the blend in all cases throughout the study. The obtained blend was extruded again to improve the homogeneity of the mixture.

LDPE-TPCS blends were compression molded using a hydraulic press machine. A pre-heating at 150°C for 6 min was carried out, followed by compression with a pressure of 10 MPa for 3 min. All the compression molded sheets (180 × 180 × 2 mm³) were cold pressed at a rate of 25°C/min to room temperature.

Mechanical properties of the blends

Measurements of the tensile properties [ultimate tensile strength (UTS), elongation at break, and Young's modulus] were carried out according to the ASTM D 638-03, using a tensometer, model Zwick 1446-60 equipped with a 10 KN cell and a data acquisition system. Dumbbell-shaped specimens, (2-mm thick) of the LDPE/TPCS blends cut from the compression molded sheets. The samples were placed in the grips of the testing machine and stretched with strain rate of 5 mm/min at room temperature. Five replicates were tested for each sample, and the average values of the measured properties were reported. The modulus was determined from the slope of the linear portion of the stress-strain curves.

Izod impact strength was also performed on a Zwick Impact instrument, (model 5102), according to ASTM D 256-04. Each notched sample was placed between the grips of the testing machine and the pendulum-type hammer was released from a specific height to hit the specimen and break it. The impact strength was determined from the energy absorbed by the sample. For each formulation, six measurement values were averaged and reported. The relative impact strength (RIS) of the samples was calculated by dividing the impact strength value of each sample to the value of the control (i.e., LDPE/PE-g-MA blend).

Flow properties measurements

Melt flow index and viscosity measurement

The rheological behavior of the LDPE–TPCS blends was studied by a MFI (2.16 kg/190°C) apparatus and a parallel plate rheometer.

MFI of the blends is determined using an apparatus (Zwick 4100, Germany), with a capillary die of 8-mm length, 2-mm diameter, according to ASTM D 1238-04 (procedure B). Seven grams of each sample was loaded into the barrel of the apparatus, which has been heated to the specified temperature of 190°C. A driving weight of 2.16 kg for the material was applied to the plunger and the molten sample was forced through a die. The timed extrudate was collected and weighed. MFI values were calculated in g/10 min.

The apparent viscosity measurement of the LDPE/TPCS blends was carried out using a parallel plate rheometer (model Paar Anton MCR300, Austria) with a gap of 1-mm space at 190°C which covered the processing temperature range. One of the plates rotates and other is fixed. Each sample was placed on the stationary plate and the rotational plate placed into it.

Surface morphology

The surface morphology of the blend samples was studied using a scanning electron microscopy (SEM), model AIS-2100, Korea, which operated at an accelerating voltage of 14 KV. Before the test, the sample surfaces were coated with a thin layer of gold to prevent electrostatic charging and poor resolution during examination. SEM micrographs observed at magnification of 2000× to identify changes on the surface of the samples. Dispersion of the minor phase in the LDPE matrix of the blends was compared.

Water absorption measurements

Water absorption of the blend samples was measured using 76.2 × 25.4 mm² strips of 2-mm thick-

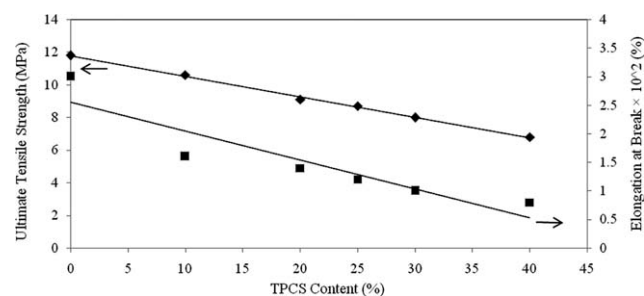


Figure 1 Effect of corn starch concentration on ultimate tensile strength and elongation at break of the LDPE–TPCS blends.

TABLE I
Tensile Modulus of the LDPE–TPCS Blends

TPCS content (wt %)	Young's modulus (MPa)
0	250
10	231
20	214
25	200
30	160
40	140

ness, according to the ASTM D570-98 method. Before the water absorption measurements, the samples were dried at 80°C for 24 h in a vacuum oven. Then the samples were weighed immediately after being taken out from the oven. Water absorption measurements were performed by soaking the samples in distilled water. The weighed samples were placed inside a container and enough water was added for their complete immersion. At regular time intervals, each sample was removed from the container, dried, and subsequently weighed to determine its water absorption. The samples were placed back in the water after each measurement again. The amount of water absorbed by the samples was determined by weighing them until they were saturated and a constant weight was obtained. The percentage of water absorption at any time (wt %) was calculated according to: w_t (%) = $[(w_2 - w_1)/w_1] \times 100$, where w_1 and w_2 were the weight of dried sample and the sample after immersion at time t , respectively. The percentage of equilibrium water absorption was calculated after water saturation in the samples (wt %).

RESULTS AND DISCUSSION

Mechanical properties measurements

Figure 1 shows variation of the UTS and elongation at break (ϵ_b %) of the blend samples versus TPCS concentrations. As it is seen, there is a continuous decrement in UTS and a reduction in ϵ_b % by increasing the starch concentrations.

Table I presents the effect of starch content on Young's modulus (E) of the samples. This modulus is obtained from the slope of linear portion of the stress–strain curves of the samples. It is seen that a decrease in the modulus with increase in the starch content is observed. These results could be explained as: (1) agglomeration of the starch particles in the interfaces of LDPE–starch acts as stress concentrator which can be intensified by increase in the starch content, (2) lower UTS, modulus and ϵ_b % of TPCS (1.44, 50 MPa, and 30%) compared with the LDPE matrix (12, 250 MPa, and 280%), (3) plasticization effect of glycerol in the blends which is expected to decrease UTS and increase ϵ_b %. However,

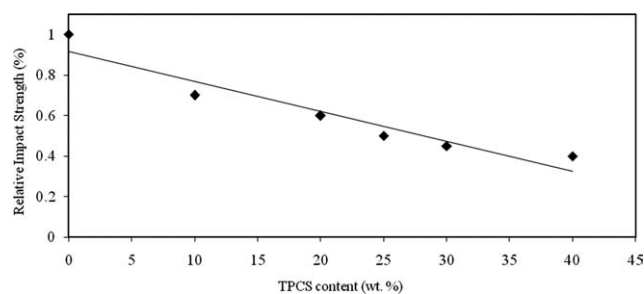


Figure 2 Variation in relative impact strength of the LDPE-TPCS blends with increasing in starch concentration.

predominant effect in decreasing ε_b % is the Case 1 as is explained above, (4) probable degradation of the polymers (LDPE, starch, PE-g-MA) during melt processing.

Reduction in the tensile properties of the LDPE-TPCS blends in this work is in agreement with the PE-thermoplastic starch blends properties that have been reported by other workers.^{14,29,35} Investigation of the blends such as LDPE-thermoplastic rice starch,¹⁴ HDPE-tapioca starch²⁹ and LDPE-thermoplastic sago starch showed reduction in their tensile properties (i.e., UTS, ε_b %, E).

Figure 2 shows changes in the RIS of the blend samples versus TPCS concentrations. It can be seen that as the starch content increases, the RIS values of the samples decrease. A linear relation between the RIS values and the starch contents is obtained.

Glycerol in the blends despite its hydrogen bonding with the starch is expected to increase molecular mobility of the polymers in them which is in favor of impact resistance of the samples. The interaction between maleic anhydride groups in PE-g-MA and hydroxyl groups in both starch and glycerol together with compatibilizing action of PE-g-MA in the blends would lead to improve the impact strength of the samples. However, as it is explained in the Case 1 for the tensile properties microaggregation of the starch particles in the LDPE-starch interphase act as stress concentrator leading to a significant decline in the impact strength of the samples. Increase in the starch content (i.e., larger particles size) intensifies the stress concentrators at the interphase of the blends which causes a more reduction in the impact strength of the samples.

The most effective way to enhance the compatibility and subsequently mechanical properties of the PE-starch blends is to improve the dispersion and interfacial properties between two phases with addition of a compatibilizer. Also, a good distribution of a dispersed phase in the matrix and high interfacial adhesion between phases are essential to improve the mechanical properties of these blends. By decreasing particle size of the dispersed phase and

achievement of homogenous distribution of it into the LDPE matrix, better mechanical properties of the blends are obtained.¹⁴

In this work, addition of PE-g-MA (3 wt %) improves dispersion of the starch particles and interfacial adhesion between the two phases. In other words, branched and crosslinked macromolecules that are produced by the chemical reaction between TPCS and PE-g-MA presumably decrease interfacial tension between the phases, improve interfacial adhesion, and facilitate stress transfer from the LDPE matrix to the dispersed phase (i.e., TPCS).³⁵

If the measured tensile properties of the blends are compared with those required for PE packaging products, according to ASTM D 4976-04, the sample containing 25 wt % TPCS in the blend will fulfill the required properties (i.e., 8.5 MPa UTS, 100% ε_b %).³⁹

Flow properties

Melt flow index and viscosity measurement

Figure 3 shows the changes in the MFI values of the blends versus TPCS concentrations. As it is seen, the MFI values decrease with increasing corn starch content in the TPCS. There is a linear relation between the amount of corn starch in the blends and such decrease in the MFI. It is believed that starch particles decrease MFI giving melt stability to the LDPE. This effect is enhanced by increasing starch concentration in the TPCS. The starch melt stabilizing effect may be explained by the macroalkyl radical trapping action of starch particle surfaces (or some additives in the native corn starch).^{40,41} These macroalkyl radicals result from mechanoscission of the polymer chains due to the high shearing forces operating on the polymer during the extrusion process when the viscosity of the polymer blend is high. The macroalkyl and macroalkoxy radicals in the extruder (in a deficiency of oxygen) cause crosslinking of the LDPE⁴² which decreases the MFI.

Using PE-g-MA as a compatibilizer in the blends also causes interactions and probably chemical

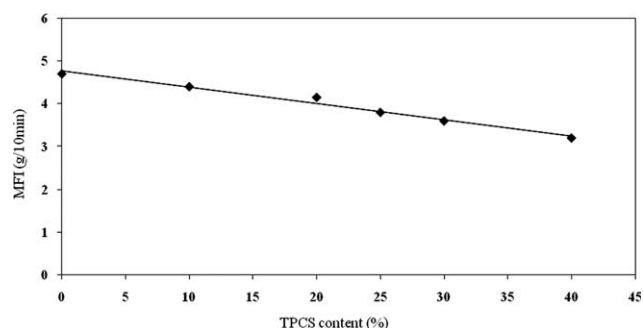


Figure 3 Melt flow index of the LDPE-TPCS blends versus TPCS concentrations (3 wt % LDPE-g-MA is present in each blend).

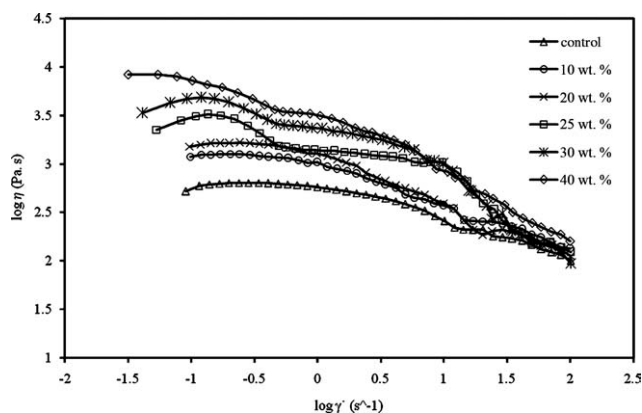


Figure 4 Variation of apparent viscosity versus shear rate of the LDPE-TPCS blends, effect of various concentration (wt %) of corn starch in TPCS.

reaction between hydroxyl groups in the starch and anhydride in PE-g-MA which decreases the MFI values too. On the other hand, the presence of glycerol in the blends, as a plasticizer, which facilitates flowing of the amylopectin part of the starch and subsequently melt flow in the extruder, reduces the rate of decrease in the MFI values.⁴³

The apparent viscosity (η) versus shear rate ($\dot{\gamma}$) of the LDPE-TPCS blends was plotted using a double logarithmic scale in Figure 4. It can be seen that as

shear rate increases from 0.1 to 100 s^{-1} , the apparent viscosity of all the samples decreases, suggesting that the blend melts exhibited a shear-thinning behavior. An increase in the apparent viscosity at a constant shear rate is observed by increase in the starch concentration. Figure 4 indicates that the presence of starch in the samples increases the apparent viscosity at constant shear rate as compared with the control (i.e., LDPE/PE-g-MA blend).

The changes in the apparent viscosity at a constant shear rate due to the effect of TPCS (Fig. 4) are explained: (1) by the stiffening effect of the swollen starch particles dispersed in the LDPE matrix on the flow. The chemical (branched and linear) and physical (crystalline) structures of the corn starch as the expanded swollen solid particles in the LDPE matrix during the melt blending could be responsible for such effect.⁴⁴ (2) Glycerol as a plasticizer increase macromolecular mobility causing reduction in the apparent viscosity. (3) The interactions and chemical reaction between anhydride groups in PE-g-MA and hydroxyl groups of TPCS molecules increase apparent viscosity of the blends. (4) Effect of other factors, such as starch stabilization on LDPE degradation, variation in the mobility of PE molecules, in the boundary layers and starch-glycerol-PE interactions.²¹

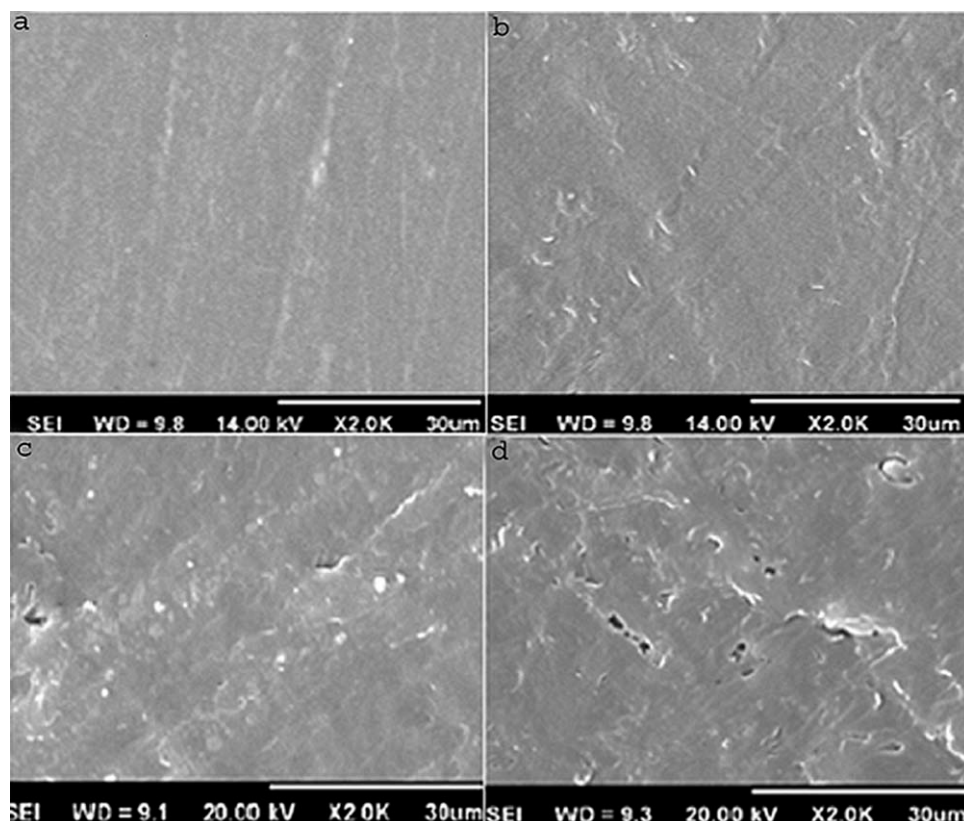


Figure 5 SEM micrographs of the LDPE-TPCS blends: (a) control (LDPE/PE-g-MA), (b) LDPE-TPCS (90/10), (c) LDPE-TPCS (75/25), (d) LDPE-TPCS (60/40), magnification 2000 \times .

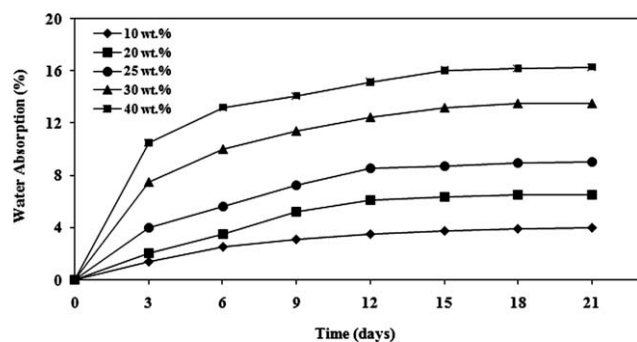


Figure 6 Variation in water absorption of the LDPE/TPCS with time of immersion. Effect of various concentrations (wt %) of corn starch in the TPCS (a day = 24 h).

Surface morphology

SEM is used to examine the effect of starch concentration on the surface texture of the LDPE/TPCS blends, compared with the control (i.e., LDPE/PE-g-MA blend). Figure 5(a–d) depicts the micrographs of the blends samples containing different amounts of corn starch.

The image of the sample containing 10 wt % TPCS [Fig 5(b)] shows a good dispersion of starch particles on the sample surface. As the starch content in the blends increases, uniformity of particles (finer distribution) is decreased [Fig 5(c,d)]. However, a good uniformity of particles is observed with starch content of 25 wt % [Fig 5(c)].

The rheological and mechanical properties of the blends depend on the microstructure, which is primarily governed by the degree of the starch dispersion.⁴⁵

It is known that melt blending of the starch with the LDPE due to the hydrophilic nature of the starch has compatibility problem. To make corn starch compatible with the LDPE and to ensure homogeneity in the blend at microscopic level, it is necessary to reduce the interfacial tension between the two phases. This can be achieved by the chemical modification of starch or LDPE and use of the compatibilizers containing groups capable of hydrogen bonding with starch hydroxyls.^{46,47} In this work, we used PE-g-MA and glycerol to solve the compatibility problems of the blends.

Water absorption

Figure 6 shows the variation in water absorption versus water immersion time of the blend samples containing various amounts of starch. It can be observed that water absorption increases with both time of immersion and corn starch concentration. The higher starch content, the greater water absorption. A rapid water absorption is observed for all the

samples during the first 5–10 days of the immersion time followed by a slow increase. After 21 days, the saturation of the samples occurs and a steady state value of water absorption is achieved.

The increase in water absorption is mainly due to hydrophilic nature of the starch particles. It may also be due to the presence of branched or cross-linked macromolecules that are produced by the reaction of the PE-g-MA with the hydroxyl groups in TPCS.²⁹

The increasing in water absorption with increase in both starch content and immersion time is in agreement with the previous studies on PE-starch blends by other workers.^{29,48,49}

CONCLUSIONS

LDPE-TPCS blends containing different amounts of TPCS (0–40 wt %) and a constant amount of LDPE-g-MA (3 wt %), were prepared using a single-screw extruder. The tensile properties and RIS of the samples decreased with increasing the TPCS concentration. However, addition 25 wt % did not affect mechanical properties of pure LDPE (as evidenced by ASTM D 4976-04). This blend fulfilled the required mechanical properties for PE packaging products application. A linear relation between the MFI reduction and starch content of the blends was observed. Also the apparent viscosity of the blends by increasing both starch concentration and shear rate increased and decreased, respectively.

The starch particles are mainly responsible for the water absorption with increasing to a maximum and by the immersion time (21 days) and increasing the starch concentration. It is believed that if the blend sample containing 25 wt % TPCS is processed into fabricated products (e.g., disposal packaging) it will be bioassimilated by combined biodegradation and humidity absorption in the environment after being used.

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