# Preparation and Characterization of Low-Density Polyethylene/Banana Starch Films Containing Compatibilizer and Photosensitizer

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**ABSTRACT:** Composite films containing various percentages of banana starch and low-density polyethylene (LDPE) were prepared. The effects of the compatibilizer, banana starch content, and photosensitizer content on the thermal and tensile properties of these films were investigated. The banana starch content was varied from 5 to 20 wt % of LDPE, whereas benzophenone was added as a photosensitizer in three different amounts (0.25, 0.5, and 1 wt %) based on LDPE. In these films PE-*graft*-maleic anhydride (PE-*g*-MA) was used as a compatibilizer at 10 wt % banana starch. It was found that the thermal stability of the composite films remained unchanged with respect to the amount of banana starch and benzophenone content. The addition of banana starch had no effect on the melting temperature and degree of crystallinity of the films. Similarly, PE-*g*-MA had no effect on the melting temperature but decreased the degree of crystallinity of the LDPE phase. Benzophenone caused an increase in the melting temperature but decreased the degree of crystallinity of LDPE in the films. Increasing the amount of banana starch decreased the tensile properties of the composite films. The addition of PE-*g*-MA as a compatibilizer increased the tensile properties compared with the uncompatibilized films. However, benzophenone had no effect on the tensile properties of the blend films. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 100: 2717–2724, 2006

**Key words:** polyethylene; biodegradable; thermal properties; starch; photosensitizer

## INTRODUCTION

The production and investigation of environmentally friendly polymeric materials has recently gained a lot of attention from the scientists around the world. This is because of the fact that the rapid rise of polymer consumption, especially in terms of packaging materials, became a real danger for the environment. Low-density polyethylene (LDPE), the most widely used packaging plastic,[1] is the worst offender. LDPE is highly resistant to degradation. One way by which the degradation of LDPE can be obtained is the incorporation of additives that increase its susceptibility to environmental degradation. Starch is an inexpensive natural biopolymer, which is totally biodegradable in a wide variety of environments. It can be used as a filler in traditional plastics.[2] Several studies have been carried out on the degradability of starch-filled plastics in various aspects.[3–9] Starches such as corn,[10] wheat,[11] rice, and potato[12] have been successfully added into LDPE.

Banana is a large herbaceous plant and it fourth on the list of the developing world's most important food crops, after rice, wheat, and maize.[13] In Thailand it is grown in all regions of the country. Banana is a highly perishable fruit. It can grow all year long and surplus fruits are often available year round. Because of high starch content (14–23% on a fresh weight basis or 61–76% on a dry weight basis[14]), banana starch is of interest as a possible resource for fillers in biodegradable materials.

Increasing the amount of starch causes a decrease in both the tensile strength and elongation at break, and the resulting materials have poor film forming properties. This deterioration arises from the different polar characterization of starch from most synthetic polymers, which leads to poor interfacial adhesion. In order to increase the adhesion, and therefore to improve the mechanical properties, a compatibilizer must be used. In previous studies[14] ethylene–vinyl acetate copolymer was used as a compatibilizer to prepare LDPE/banana starch biodegradable film.

Before an investigation of the application potential of these degradable films, their complete characterization and elucidation of the degradation process must be undertaken. Because their processing and possible end use will occur at elevated temperature, the determination of their thermal properties is of great importance. The thermal stability and melting behavior of the films to be processed can be obtained from ther-

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mogravimetric analysis (TGA) and differential scanning calorimetry (DSC), respectively.

Sunlight is another source of degradative energy. The radiation by UV or sunlight can reduce the polymer chain size of PE. In the present work, we focused on the preparation and characterization of both biodegradable and photodegradable LDPE/banana starch films containing compatibilizer and photosensitizer. PE/maleic anhydride graft copolymer (PE-*g*-MA) was used as a compatibilizer, and benzophenone was used as a photosensitizer to enhance the photodegradability of LDPE. The effects of the compatibilizer, banana starch content, and photosensitizer content on the thermal and tensile properties of these films were investigated. TGA and DSC were used to study the thermal properties of the composite films.

## EXPERIMENTAL

### Materials

Extrusion film grade LDPE (LD1902F, Cementhai Commercial Co., Ltd.) was used in this research. Its melt flow index and density were 2.0 g/10 min and 0.919 g/cm$^3$, respectively. Benzophenone was purchased from A.C.S. Xenon Limited Partnership. PE-*g*-MA with a 0.5–1 wt % graft level was kindly supplied by Dupont Co., Ltd. under trade name Fusabond. Banana starch was used as a biodegradable filler. The extraction of banana starch was reported earlier.[14] The granular size ranged from 5 to 45 $\mu$m, with an average size of 21–24 $\mu$m.

### Film preparation

Prior to mixing with LDPE in a twin-screw extruder (Thermoprism DSR-28), the banana starch was dried in an air oven at 80°C for 24 h. The operating screw speed was 25 rpm; and the temperatures of the five heating zones of the extruder were set at 100, 135, 140, 145, and 150°C. Banana starch content was varied from 5 to 20 wt % of LDPE. Three different amounts of benzophenone (0.25, 0.5, and 1.0 wt %) based on the LDPE content were added. In these composites PE-*g*-MA was used as a compatibilizer at 10 wt % based upon the banana starch content. After mixing into pellet form, starch-based LDPE films were prepared using a Collin chill roll cast film (model ECS-T10). The barrel temperature was 100–160°C and the screw speed was 25 rpm.

### Characterization of composite films

#### Chemical structure of films

Fourier transform IR spectroscopy was used to study the structure and functional groups of the blend films. For each spectrum, 32 consecutive scans with 4 cm$^{-1}$ resolution were used. The samples were scanned at a frequency range of 4000–400 cm$^{-1}$.

#### Morphological studies

Scanning electron microscopy (SEM; JEOL JSM 5410LV; Tokyo, Japan) was used to examine the fractured surface of the films. For the fractured surface investigation, the films were fractured immediately after freezing in liquid nitrogen and were sputter coated with gold to prevent any electrical discharge during examination.

#### Thermal properties

The thermal properties of the LDPE/banana starch films were evaluated by using a differential scanning calorimeter (PerkinElmer Dimond DSC) and a thermogravimetric analyzer (PerkinElmer TGA7).

TGA was carried out under a nitrogen atmosphere at a heating rate of 10°C/min from 20 to 650°C. Prior to conducting the experiments, approximately 10-mg samples were dried in a vacuum oven at 60°C for 24 h. The onset of the degradation temperature ($T_d$) was recorded for each sample.

For DSC analysis, a 4-mg average weight sample encapsulated in a hermitically sealed aluminum pan was prepared for each sample. The same temperature history was applied to all samples: heating from 20 to 200°C, quenching to 20°C to remove any previous thermal history, and heating again to 200°C at a heating and cooling rate of 10°C/min. Each run was also performed under a nitrogen atmosphere. The melting temperature ($T_m$) and heat of fusion ($\Delta H_f^*$) of the samples were obtained from the maximum peak and the area under the peak, respectively. The percent crystallinity of the LDPE phase was calculated using the following equation:

$$\% \text{ crystallinity} = \frac{\Delta H_f^*}{\Delta H_f^0} \times 100 \qquad (1)$$

where $\Delta H_f^0$ is the heat of fusion for 100% crystalline LDPE and $\Delta H_f^*$ is the heat of fusion for the semicrystalline LDPE phase.

#### Tensile properties

Tensile tests of rectangular film specimens (1.5 cm × 20 cm × 100 $\mu$m, width × length × thickness) were conducted using a universal testing machine (LLOYD LR 100k). Measurements were performed using a crosshead speed of 50 mm/min and a gauge length of 10 cm, according to ASTM D 882. At least five specimens of each film were tested, and the results were averaged to obtain a mean value.

## RESULTS AND DISCUSSION

### Characterization of composite films

Chemical structure of films

Figure 1 shows the IR spectra of pure LDPE, LDPE/ 20% banana starch, LDPE/20% banana starch/10% PE-*g*-MA, and LDPE/20% banana starch/10% PE-*g*-MA/1% benzophenone films. The IR spectra of starch-filled LDPE film displayed different bands from the pure LDPE spectra. Obviously, because of the presence of starch in the LDPE film, three significant IR bands occurring at 3000–3650, 1640, and 960–1190 $cm^{-1}$ are designated as O—H stretching, O—H bending, and C—O stretching, respectively. For the compatibilized blend with 10% PE-*g*-MA, it can be seen that, even after using the PE-*g*-MA as a compatibilizer, there was no significant difference between the two spectra. It was anticipated that PE-*g*-MA, which contains an anhydride group, could develop hydrogen bonds with the hydroxyl groups of starch and form ester groups at 1735 $cm^{-1}$. However, it must be realized that the concentration of anhydride groups in the blend is very low. For example, for a blend containing 20 wt % starch and 10 wt % PE-*g*-MA (based on starch), the total concentration of PE-*g*-MA in the blend film is 2 wt %. Taking into account that PE-*g*-MA contains 0.5–1 wt % anhydride groups, the total concentration of the latter in this blend will not exceed 0.01–0.02 wt %. With such a low concentration, the degree of hydrogen bonding is very limited. Nevertheless, it can still affect the compatibilization of the blend films, as will be discussed later. For LDPE/ starch blends with photosensitizer (1% benzophenone), the spectrum of the LDPE/starch blend with 1% benzophenone was almost the same as that of the uncompatibilized blend, except for a tiny peak occurring at 1710 $cm^{-1}$. A small difference in the blend with photosensitizer is assigned to the carbonyl group (C=O) from benzophenone. The expected broad band
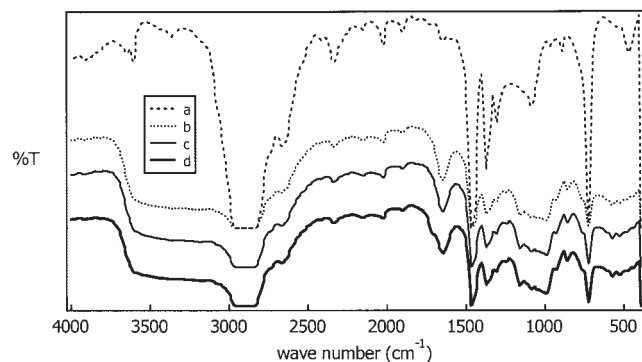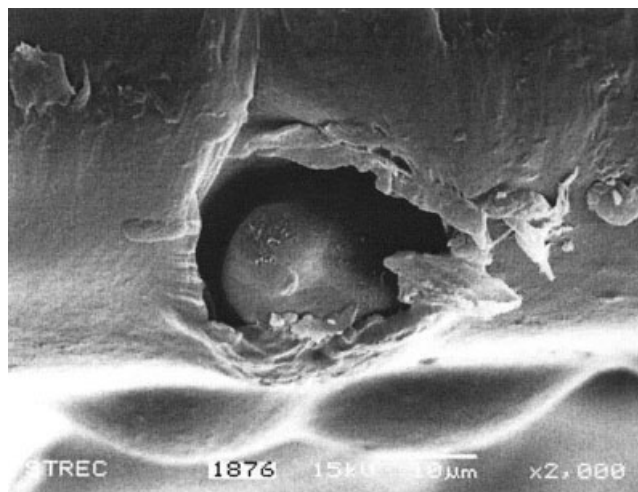


**Figure 1** IR spectra of pure LDPE (spectrum a), LDPE/20% starch (spectrum b), LDPE/20% starch/10% PE-*g*-MA (spectrum c), and LDPE/20% starch/10% PE-*g*-MA/1% benzophenone (spectrum d).



(a)



(b)

**Figure 2** SEM micrographs of LDPE/banana starch of (a) an uncompatibilized blend and (b) a compatibilized blend with 10% PE-*g*-MA.

at 3000 $cm^{-1}$ attributable to the aromatic groups (=C—H stretching) of benzophenone occurred coincidently with the broad and intense O—H stretching band of the starch molecule.

Morphological studies

The SEM micrographs of LDPE/banana starch film with and without 10% PE-*g*-MA as a compatibilizer are presented in Figure 2. The morphology of the fractured surface of the uncompatibilized film is different from the film containing PE-*g*-MA in that the uncompatibilized film exhibited a gap between the starch granule and LDPE matrix, whereas the film
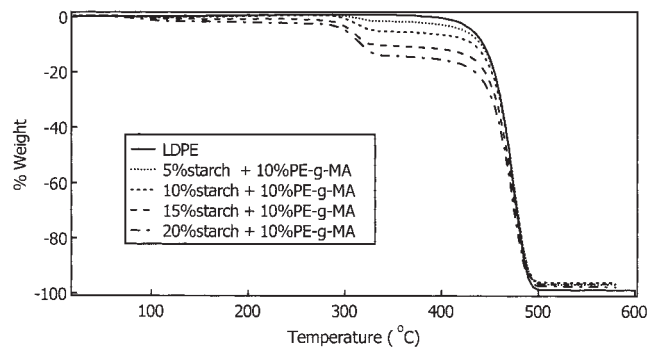
**Figure 3** TGA curves of LDPE blend films containing 10% PE-*g*-MA and varying banana starch contents.



**Figure 4** TGA curves of LDPE blend film with 10% banana starch, 10% PE-*g*-MA, and different amounts of benzophenone.

containing compatibilizer showed a bond between the starch granule and the LDPE matrix. These results indicate that the addition of PE-*g*-MA improved the interfacial bond strength of the LDPE/starch film. Good compatibility and enhanced adhesion between the two phases were evidenced by the increase in tensile properties of the film compared to the uncompatibilized film, as will be discussed in the next section. The SEM micrographs ensure the role of a compatibilizer in reducing the interfacial energy and thus promoting the interfacial adhesion between the banana starch phase and the LDPE phase.

## Thermal properties

### TGA

The effect of the banana starch content on the thermal degradation behavior of LDPE/starch films is shown in Figure 3. The LDPE films with different amounts of banana starch showed three decomposition stages. The first stage at around 100°C was attributed to the loss of moisture content of banana starch because starch is hydrophilic in nature and it is well known that starch can strongly absorb moisture. The second degradation zone located around 290–300°C was associated with the thermal degradation of banana starch because of it was similar to that of pure banana starch.[14] The third stage appearing at around 450°C was associated with the greatest weight loss, and it
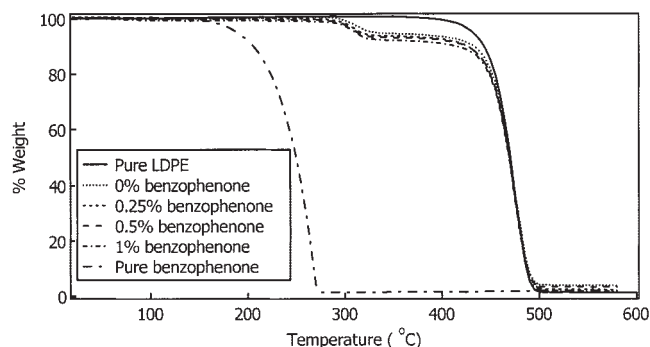
was termed the main stage. This stage was due to the complete thermodegradation of the LDPE backbone.

Table I summarizes the onset of the degradation temperature [$T_d$(onset)] for the second and third stages, the weight loss, and the residue at specific temperatures for all films. As expected, the second $T_d$(onset) showed no significant difference when the amount of starch increased. In other words, the starch content does not affect the $T_d$(onset). However, as listed in Table I, the banana starch content affected the amount of weight loss of the films at this stage. For example, at 350°C, the weight loss of the films increased as a function of the banana starch content. These results confirm that the second weight loss must be due to the decomposition of banana starch. Nevertheless, there was no effect from the banana starch content at the third stage of thermal decomposition. These results imply that no matter how much banana starch was added, the LDPE composite films absolutely decompose at approximately the same temperature of 500°C. The residue percentages at 550°C of the LDPE/banana starch films were considerably greater than that of the pure LDPE. This implied that the origin of the residue left at high temperature may be the banana starch.

The effect of the benzophenone content on the thermal degradation behavior of the composite films is presented in Figure 4. As shown, pure benzophenone exhibited a degradation temperature at around 227°C.

**TABLE I**
**Effect of Banana Starch Content on Thermal Stability of LDPE/Banana Starch/10% PE-*g*-MA Films**

| Sample | $T_d$ (°C) (onset) | | Weight loss (%) | | | Residue at 550°C (%) |
|---|---|---|---|---|---|---|
| | Second | Third | 120°C | 350°C | 450°C | |
| LDPE | — | 450.3 | — | — | 15.06 | 1.38 |
| LDPE + 5% starch | 295.0 | 451.1 | 0.02 | 1.65 | 15.87 | 3.31 |
| LDPE + 10% starch | 295.2 | 451.6 | 0.22 | 5.30 | 18.35 | 3.96 |
| LDPE + 15% starch | 294.5 | 450.3 | 0.66 | 10.61 | 23.75 | 3.50 |
| LDPE + 20% starch | 293.6 | 452.9 | 1.57 | 14.14 | 27.87 | 2.57 |

| Sample | $T_d$ (°C) (onset) | | Weight loss (%) | | Residue at 550°C (%) |
|---|---|---|---|---|---|
| | First | Second | 350°C | 450°C | |
| LDPE | — | 450.3 | — | 15.06 | 1.38 |
| 0% benzophenone | 295.2 | 451.6 | 5.30 | 18.35 | 3.96 |
| 0.25% benzophenone | 293.9 | 450.0 | 6.30 | 20.33 | 2.49 |
| 0.5% benzophenone | 292.4 | 448.8 | 6.80 | 20.53 | 3.40 |
| 1% benzophenone | 292.0 | 448.2 | 7.81 | 21.48 | 1.85 |

However, the composite films showed only two stages of thermal decomposition. The first stage was attributed to banana starch decomposition, whereas the second $T_d$(onset) was from the LDPE decomposition. No evidence of benzophenone decomposition was found in the composite films. This was probably due to the fact that the amount of benzophenone was quite small compared to other compositions. The highest content was only 1 wt %. Therefore, because the amounts of starch and PE-*g*-MA were kept constant, all thermograms for all films were completely superimposed. Similar to Figure 3, all the composite films further degraded up to about 500°C, where the composites almost completely decomposed. Table II shows the onset of the degradation temperature and percent weight loss for LDPE composite films with various amounts of benzophenone.

## DSC

The second heating and cooling cycles of the DSC thermograms for pure LDPE and LDPE composite films are displayed in Figures 5 and 6, respectively. The $T_m$ of the films was taken as the maximum of the endothermic peak from the second heating whereas the crystallization temperature ($T_c$) was taken as the maximum of the exothermic peak from the cooling

cycle. Compared with the DSC thermogram of pure LDPE film, we found that the $T_m$ and $T_c$ of the composite films did not change significantly. As listed in Table III, a small increase was observed in the LDPE blend with 20% banana starch/10% PE-*g*-MA/1% benzophenone. Although no other endothermic peak occurred for the LDPE/20% banana starch and LDPE/20% banana starch/10% PE-*g*-MA films, this does not mean that the films are compatible. The endothermic transition that occurred is attributed solely to the LDPE phase because the banana starch, like any other starch, has no melting temperature, instead having gelatinization and degradation temperatures.

Table III provides the $T_m$, $T_c$, and degree of crystallinity for LDPE and LDPE/starch films. A value of $\Delta H_f^0$ (276 J/g according to Thakore et al.[8]) for purely crystalline PE and the $\Delta H_f^*$ obtained from the total area of the melting were used to calculate the degree of crystallinity. Comparing the difference between pure LDPE and LDPE/20% starch films, there was an apparent decrease in the heat of fusion as the amount of starch increased. However, when the heat of fusion was corrected by taking into account only the LDPE content in the film, it is evident that there was no change in the degree of crystallinity of the LDPE phase. Thus, it can be suggested that LDPE is hardly miscible with the banana starch. For the compatibi-
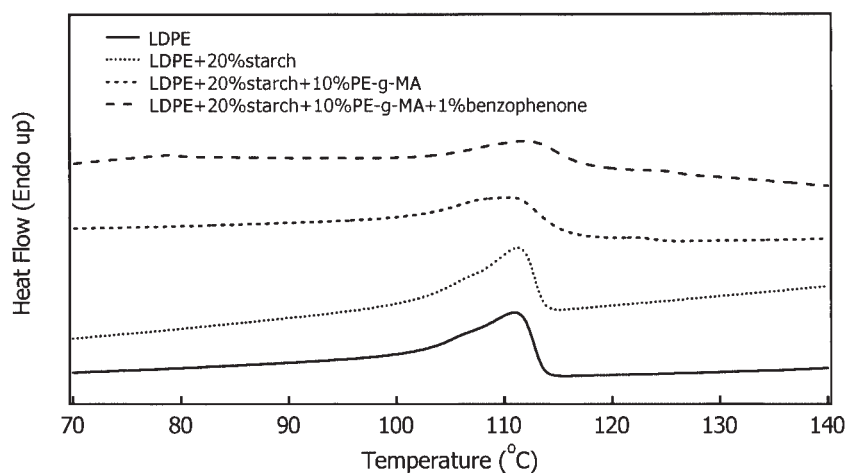


**Figure 5** The DSC melting thermograms of pure LDPE and LDPE composite films.
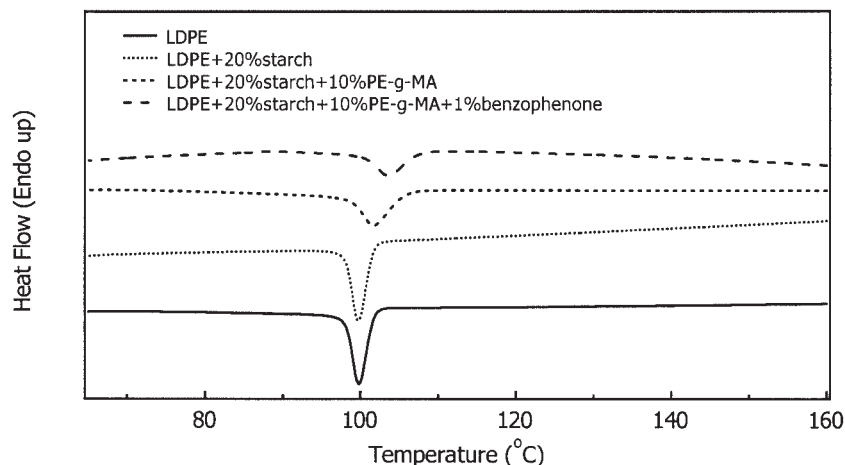
**Figure 6**  The DSC crystallization thermograms of pure LDPE and LDPE composite films.

lized film with PE-*g*-MA, the presence of PE-*g*-MA significantly decreased the degree of crystallinity of the LDPE phase in the blend. This decrease is due to the interaction between LDPE and starch, which hinders the close packing of LDPE chains.

For the LDPE/20% banana starch/10% PE-*g*-MA/1% benzophenone film, the addition of the benzophenone caused an increase in the $T_c$. This implies that benzophenone functions as a nucleating agent, allowing LDPE to form its crystals on the nucleating site very fast and at higher temperature compared to pure LDPE and the LDPE/banana starch films without benzophenone. These nucleating agents provide nuclei for heterogeneous crystallization, raising the crystallization rate and crystallization temperature. In addition, the degree of crystallinity of the LDPE phase was dramatically decreased. This is because more and smaller spherulites are consequently produced, resulting in the lowering of the degree of crystallinity.

## Tensile properties

Figures 7 and 8 illustrate the effect of the starch content on the tensile properties of pure LDPE and LDPE/starch composite films. Obviously, the tensile properties are highly dependent on the starch content. The tensile strength and elongation at break decreased

with increasing amounts of starch. This decrease can be associated with lack of compatibility between the two components because of the difference in the hydrophilic and hydrophobic characters of starch and LDPE, respectively, resulting in separation between the two phases. It is of interest that the tensile strength of the LDPE/starch film gradually decreased as the starch content was increased from 0 to 20% whereas the elongation at break showed a considerable decline at 5% starch content. Moreover, this reduction did not change much when the starch content increased to 20%. In other words, the reduction of the elongation at break appeared to plateau at higher starch concentrations. These results are however in good agreement with those previously reported by others.[15–17] Kang et al.[15] showed that the elongation at break for corn starch and high-density PE (HDPE) films decreased from 180% (100% HDPE) to about 60 and 40% when the starch content was increased to 5 and 20%, respectively. Similarly, the elongation at break for corn gluten meal (CGM)–LDPE films decreased approximately 750% upon increasing only 7% CGM content; thereafter, the elongation at break did not vary significantly as the CGM content was increased to 18%.[16] As also reported by Ratto and coworkers,[17] the elongation at break decreased approximately 200% upon increasing to 5% corn starch. In addition, they found that the

**TABLE III**
**Thermal Properties and Degree of Crystallinity for LDPE and LDPE/Starch Films**

| Sample | $T_m$ (°C) | $T_c$ (°C) | $\Delta H_f^*$ Total (J/g) | $\Delta H_f^*$ LDPE (J/g) | Crystallinity (%) Total | Crystallinity (%) LDPE |
|---|---|---|---|---|---|---|
| LDPE | 111.13 | 100.20 | 48.68 | 48.68 | 17.64 | 17.64 |
| LDPE + 20% starch | 111.23 | 100.08 | 42.60 | 47.33 | 15.43 | 17.15 |
| LDPE + 20% starch + 10% PE-*g*-MA | 110.76 | 101.78 | 33.55 | 41.02 | 12.16 | 14.86 |
| LDPE + 20% starch + 10% PE-*g*-MA + 1% benzophenone | 112.23 | 103.86 | 19.19 | 23.62 | 6.95 | 8.56 |

experimental results for the elongation at break are consistent with the theoretical values predicted by Nielsen[18] for filled systems with poor adhesion between the filler and matrix. Therefore, these elongation data suggest that there is a poor adhesion between the hydrophilic starch granules and the hydrophobic polymer matrix, as confirmed by SEM analysis [see Fig. 2(a)]. The addition of the immiscible component to a ductile matrix generally decreases the elongation properties considerably at break point. The elongation will therefore depend on the state of the interface in such cases. For the tensile strength, because the adhesion between the starch particles and LDPE matrix was poor, poor stress transfer occurred at the starch particle–polymer interface. The stress concentration occurred in the continuous phase around the starch particles because of a lack of stress transfer across the LDPE–filler interface, causing the
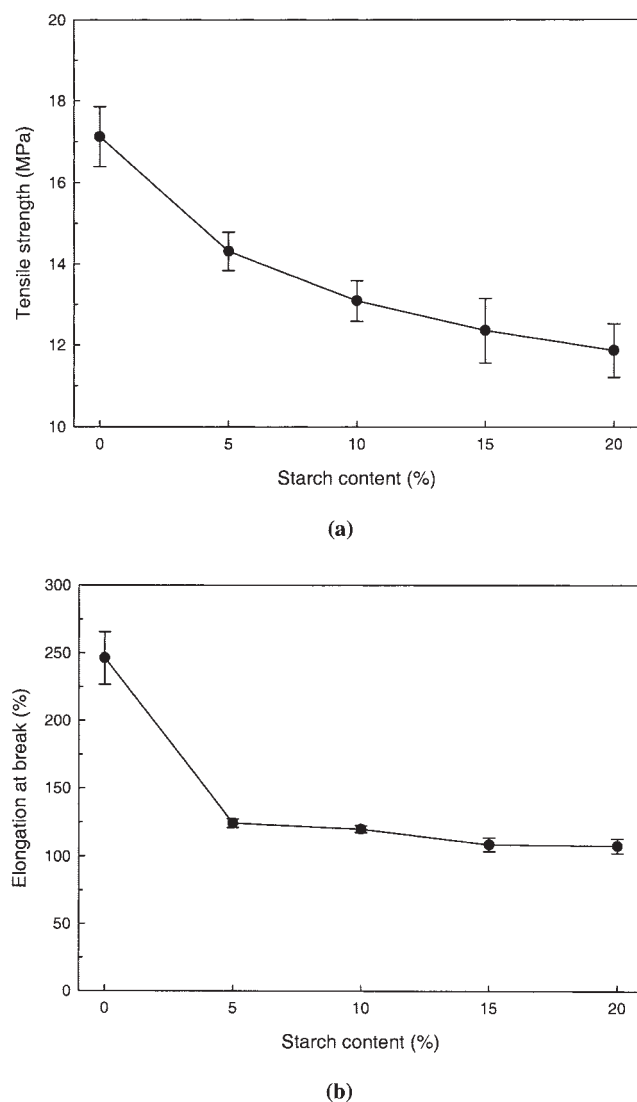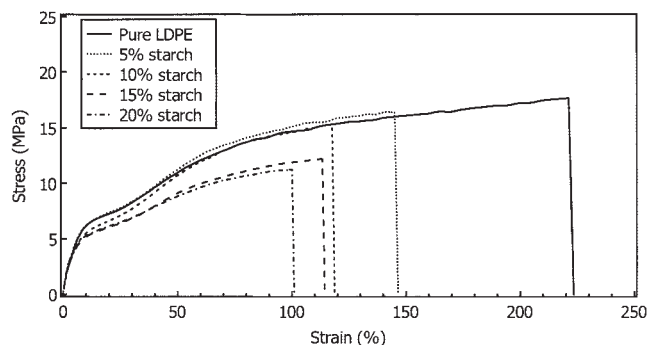


**Figure 8** Stress–strain curves of pure LDPE and LDPE/banana starch films with different contents of starch.

reduction in tensile strength of the films. In order to increase the adhesion, and therefore to improve the tensile properties, a compatibilizer must be used.

The tensile strength of the composite films with and without compatibilizer are shown in Figure 9(a). In both cases, a decrease in tensile strength was observed as the amount of starch increased. The decrease in tensile strength of the compatibilized films, however, was smaller compared to the corresponding decrease in the uncompatibilized films. The compatibilized films also exhibited a slightly greater elongation at break compared to the uncompatibilized ones, as seen in Figure 9(b). Although there was not much difference between the compatibilized and uncompatibilized films, the trend was similar to that of the tensile strength. It seems that the addition of PE-$g$-MA compatibilizer into the films increases the tensile strength and elongation at break compared to the uncompatibilized films. As previously described, the tensile strength properties in polymer films are partly affected by the interfacial adhesion between the polymers. LDPE and banana starch are immiscible because of their different polar character. In contrast to the hydrophobic LDPE, banana starch is a hydrophilic polymer because of the presence of hydroxyl groups in the starch molecules. As a result of this immiscibility, the tensile properties in the film decreased. The addition of PE-$g$-MA proved to be a very effective compatibilizer.

As for the effect of the benzophenone content, there was no significant effect on the tensile strength and elongation at break of the films. This might be attributable to the small amount of benzophenone in the films.

## CONCLUSION

The decomposition temperatures of the films were determined from TGA measurements. The composite films showed three decomposition stages. The first stage at around 100°C is due to the loss of moisture



**Figure 7** The (a) tensile strength and (b) elongation at break of LDPE/banana starch films.
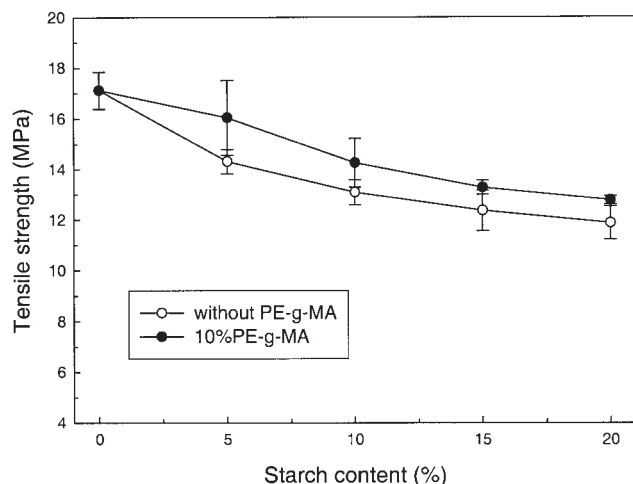
**(a)**
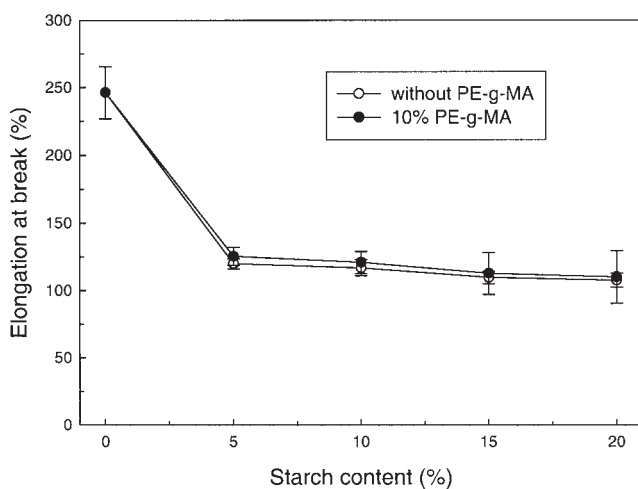


**(b)**

**Figure 9** The (a) tensile strength and (b) elongation at break of LDPE/banana starch films with and without 10% PE-*g*-MA.

content in banana starch, whereas the second stage around 290–300°C is due to the banana starch decomposition. The third stage at around 450°C is attributed to LDPE decomposition. The thermal stability of the films remained unchanged with respect to the amount of banana starch and benzophenone content.

In the DSC measurements banana starch had no influence on the $T_m$, $T_c$, and degree of crystallinity of LDPE/banana starch films. Similarly, PE-*g*-MA had no effect on the $T_m$ and $T_c$, but a small decrease in the

degree of crystallinity of the LDPE phase in the film was observed. However, the addition of benzophenone caused a slight increase in the $T_c$, resulting in a decrease in the degree of crystallinity of the LDPE phase. Overall, the changes in the $T_m$ and $T_c$ of the films were well within experimental error. Thus, the processibility of LDPE was not affected by the incorporation of additives.

The tensile strength and elongation at break of the composite films decreased with increasing amounts of banana starch. However, the addition of PE-*g*-MA as a compatibilizer in the film increased the tensile properties compared to the uncompatibilized film, whereas the benzophenone had no effect on the tensile properties of the films.

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