

# Effect of Starch on Thermal, Mechanical, and Barrier Properties of Low Density Polyethylene Film

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**ABSTRACT:** Low-density polyethylene (LDPE) with different quantities of starch was compounded using a twin screw extruder and blown into films by a Konar K, blow-film machine. Mechanical properties, namely percent elongation, tensile, bursting, and tear strength, as well as barrier properties, such as water vapor and oxygen transmission rate, of the filled LDPE film were studied. Thermal properties of the films were studied using DSC and DMA. Master curves at reference temperature of 30°C were obtained using software linked to DMA. Incorporation of 1% starch in LDPE has marginally affected the thermal, barrier, and mechanical properties; however, that of 5% starch filled LDPE has affected the properties to a great extent. The mechanical

properties, such as percent elongation, tensile, tear, bursting, and seal strength, decreased by 19.2, 33.6, 3.60, 10.8, and 22.12%, respectively. Similarly, water vapor and oxygen transmission rate increased to 32.5 and 18.3%, respectively. Other physical properties, namely migration and thermal properties, were also affected in 5% starch filled LDPE; however, the film can still be used as packaging material. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 99: 3355–3364, 2006

**Key words:** barrier; DSC; mechanical properties; thermal properties; transition

## INTRODUCTION

Biodegradable plastics have aroused enormous interest in flexible packaging materials.<sup>1,2</sup> Studies also indicate that encapsulation of starch into polyethylene films renders it biodegradable.<sup>3</sup> However, the starch incorporation in plastics is a difficult task as it may affect barrier and mechanical properties.<sup>4,5</sup> Starch may lose its crystallinity on incorporation in synthetic polymers.<sup>6</sup> It has been reported that starch blended with polyethylene produces a transparent film, which disintegrates; the starch component biodegrades, leaving behind small particles of polyethylene.<sup>3</sup> Among packaging materials, low density polyethylene (LDPE) is the most extensively used because of its easy availability, processability, flexibility, improved optical properties, and seal strength. In addition, no other ingredient is required during processing of low density polyethylene (LDPE).<sup>7,8</sup>

The challenge is to convert this film into biodegradable ones by suitable fillers marginally affecting the properties.<sup>9</sup> Several investigators have studied the effect of different fillers, such as mica, silica, calcium carbonate, titanium dioxide, alumina, clay, potassium permanganate, and so forth, on the performance of

polyolefin's packaging material.<sup>10–12</sup> To optimize the polymer materials for suitable packaging application, an in-depth knowledge of the structural property relationship is required.<sup>13</sup> This has paved the way for investigation on optimum filling of starch into low density polyethylene (LDPE) without affecting the packaging properties.<sup>14</sup> However, analysis of the addition of starch on thermal properties by differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA) in LDPE packaging film has not so far been the focus of much research.

In this investigation, starch at 1 and 5% levels was incorporated into LDPE using a twin-screw extruder. Some of the physicochemical properties of starch filled LDPE were studied, along with DSC and DMA measurements to establish the structure-property relation. Master curves were also studied using software linked to DMA.

## METHODS

### Materials

LDPE (specifications: density 0.92 g/cm<sup>3</sup>, melt flow index 29/10 min; melting point 115–125°C) was obtained from IPCL, Baroda, India. Starch was obtained from Ranbaxy Laboratories, New Delhi, India.

### Compounding and film blowing

LDPE was compounded with the starch using a Haake Twin-Screw Extruder (CTW 100, Bersdorf, Germany)

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with L/D ratio 33 : 1. A temperature profile of 156–180°C was maintained during the process. The compounded LDPE was then blown into 75-micron thick films using a Konark single screw extruder with L/D ratio 22 : 1. Film blowing was done at a temperature profile of 170–190°C.

### Mechanical properties<sup>15</sup>

The tensile strength and elongation at break of the film were determined using ASTM methods.<sup>14</sup> The specimen in the form of strips of size 14 × 160mm were cut from cured free film. The specimens were conditioned for 24 h at 60% RH and were tested in a universal testing machine (Model 4302, Instron, UK).

### Bursting strength<sup>16</sup>

The strength of plastic films can be evaluated by means of a simple bursting device. The burst strength of films is the resistance it offers to a steadily increasing pressure, applied at right angles to its surface, under certain defined conditions. The burst strength is taken to be the pressure at the moment of failure, and is essentially a measure of the capacity of the film to absorb energy.

Burst strength was measured using a Goodbrand and Colt (UK) burst tester. The specimen is placed between the two angular rings of the machine. The inlet valve is opened to maintain an air pressure of 110 lbs/sq inch. The sample was exposed to air pressure at a controlled rate of increase until it failed. The pressure at failure in lb/in<sup>2</sup> is taken as the burst strength of the material.

### Tear strength<sup>17</sup>

Tear strength was measured using an Elmendorf tear tester (Goodbrand and Colt, U.K.). The sample is mounted in the jaws of the tear testing machine. The cut is initiated using a sharp blade provided in the machine. When the pendulum is released, it swings down under the force of gravity and the specimen is torn from the already begun slit/notch; because of the energy required to tear the film, the pendulum has less energy than if it had fallen freely. The difference in energy is indicated by a pointer on a calibrated scale. The pendulum is released, and the specimen is allowed to tear completely. The reading is noted down from the calibrated scale. Tear strength of packaging films is expressed in grams.

### Seal strength<sup>18</sup>

To measure the strength of a seal, the free ends of a standard size sealed film specimen are gripped by the jaws of a tensile tester and the movable jaw is driven

at a constant rate, peeling the sealed area apart. The strength of the peel is defined as the maximum force indicated in the test per inch of seal width. Heat seal strength is the measure of the force required to pull apart the pieces of film that have been sealed together. The test was carried out on an Instron tensile tester (UTM-4302). The films are sealed using a sealing machine to get a standard seal width of 4mm; the sealed films are cut into test samples of dimensions 5 × 1 in. The free ends of the sample are mounted on two grips of the tensile testing machine, and the movable jaw is driven at a constant rate of 500mm per minute, peeling the sealed area apart. The load required to fail the seal of the sample is noted. The seal strength is calculated by dividing the load at fail by the seal width.

$$\text{Seal strength} = \frac{\text{Load at fail}}{\text{Width of seal}} \text{ kg/10mm}$$

### Barrier properties

#### Water vapor transmission rate<sup>19</sup> (WVTR)

WVTR was determined using free film as per method ASTM D-96. Free films were cut to obtain a circular test specimen of 12cm diameter. The test specimen was fastened to a payne cup containing 8 mL of distilled water using a C-type clamp. The diameter of the film for water vapor to permeate was 10 cm. The cups were kept in a desiccator over fused CaCl<sub>2</sub>. The cups were weighed every 24 h until a constant water loss was obtained. WVTR was calculated as per the ASTM method. Inside the cup, the RH will be 100%; but outside the cup, that is, in the desiccator, the RH will be zero.

#### Oxygen transmission rate<sup>20</sup>

The oxygen transmission rate is the quantity of oxygen gas passing through a unit area of the parallel surfaces of the film per unit time under the conditions of the test. The test specimen used is of circular shape of size 10cm diameter. Unclamp the diffusion cell and open it. Apply a thin layer of sealing grease around the raised rim of the lower half of the diffusion cell. Remove the test specimen from the desiccator and place it upon the greased surface, taking care to avoid wrinkles or creases. Lower the upper half of the diffusion cell into place and clamp both halves lightly together. Before starting, the experiment zero voltage level (E<sub>o</sub>) is set. After establishing the E<sub>o</sub> value, the flow switch is put on and oxygen purged. Sensor output current, as indicated by the strip chart recorder, should increase gradually, ultimately stabilizing at a constant value. The constant value of the voltage on the strip chart recorder shall be recorded and labeled E<sub>e</sub>.

OTR is calculated using the formula given below:

$$\text{OTR} = \frac{(E_e - E_o) \times Q}{A \times R_1}$$

where  $E_e$  = steady state voltage level,  $E_o$  = zero voltage level,  $A$  = specimen area,  $Q$  = calibration constant (given),  $R_1$  = value of load resistance (given), and OTR is expressed in ml/m<sup>2</sup>/24 h.

### Migration properties<sup>21</sup>

#### Total global migration

Migration is considered to be an undesirable package/product interaction as a result of mass transfer from the package into the contained product. Migration is a complex process depending in part on the migration species diffusivity. Diffusivity, or the diffusion coefficient ( $D$ ), is the tendency of a substance to diffuse through the polymer bulk phase. Migration, therefore, can also be a mass transport process under defined test conditions (i.e., time, temperature, and the nature and volume of the contacting phase). During migration, residual monomers and other unconverted processing aids tend to migrate into the system. It is difficult to analyze for these residues; hence, representatives of various foods, called "food stimulant liquids," are used to contact with the packaging material, and leaching is computed as per the standard specifications.

The volumes of the food stimulant system were prepared as per the surface contact area at 1 mL/cm<sup>2</sup>. The sample is allowed to immerse into the solvent system and, after an interval of 24 h, a known quantity of this stimulant was pipetted out into a pre weighed empty petri dish and kept for evaporation. After evaporation and equilibrium to the ambient conditions in a desiccator, the petri dish is weighed again for the migrants. The difference in weight will give the total migration value for that polymer in the particular stimulant system. Then the total migration of the given sample is kept in mg/L.

### DSC experiments

The  $T_g$  values of the starch filled LDPE films were obtained using a DSC 2010 (TA Instruments, USA)

equipped with an intra cooler. The measurements were made using a sealed empty pan as the reference material and N<sub>2</sub> as a flushing agent over the head. The instrument was calibrated with indium. Samples of 5 to 10 mg were weighed into standard aluminum pans. The pans were sealed and each sample was heated from -50 to 100°C at the rate of 5°C/min. The measurements were made in triplicate. Results were analyzed using TA universal thermal analyzer software.

#### Isothermal crystallization

A TA instruments DSC 2010 with an intra cooler was also used for isothermal measurements. All experiments were carried out with a sealed empty pan as the reference material and N<sub>2</sub> as a flushing agent over the head. Samples (5–10mg) were weighed into standard aluminum pans. Each sample was held at an initial temperature of -30°C to ensure no crystallization for 3 min. The sample was then ramped up at a maximum rate (200°C/min) to the final isothermal holding temperature of 250°C, where it was held for 20 min during which time crystallization was observed. Triplicate measurements were made at each chosen temperature.

### DMA experiments

#### Glass transition temperature ( $T_g$ )

DMA measurement was carried out using an Eplexor (Gabo instruments, Germany) in tensile mode. The sample was prepared according to ASTM D-882. A stress in tensile mode was applied to the sample at 5Hz, and the stress was measured at a strain of 40%. The samples were heated from 30 to 80°C at a rate of 3°C/min. The storage modulus ( $E'$ ), loss modulus ( $E''$ ), and loss tangent ( $\tan \delta = E''/E'$ ) accompanying the heating of the samples were measured.

#### Time-Temperature superposition

The software "mastering" provided in the instrument Eplexor (Gabo instruments, Germany) was used for generating "master curves." Mastering was carried out with the data of temperature-frequency sweep.

TABLE I  
Mechanical Properties of Pure LDPE and Starch Filled LDPE Films

Sample	Percentage of elongation	Tensile strength (MPa)	Tear strength (g)	Bursting strength lb/in <sup>2</sup>	Seal strength kg/10 mm
LDPE	327 ± 7.2	18.8 ± 0.5	412 ± 6.5	12.9 ± 0.5	1.13 ± 0.01
1% starch + LDPE	284 ± 6.1	16.5 ± 0.4	396 ± 5.8	11.5 ± 0.5	1.01 ± 0.01
5% starch + LDPE	264 ± 5.6	12.5 ± 0.3	364 ± 5.4	10.8 ± 0.5	0.88 ± 0.01

±Standard deviation of three replicates.

**TABLE II**  
Barrier and Migration Properties of Pure LDPE and Starch Filled LDPE (75 Micron) Films

Sample	Total migrants (g/m <sup>2</sup> )				Water vapor transmission rate g/m <sup>2</sup> /24 h at 38°C + 90% rh	Oxygen transmit rate (ml/m <sup>2</sup> /24 h)
	Water	3% acetic acid	50% alcohol	n-heptane		
LDPE	0.005	0.065	0.108	0.128	7.9 ± 0.09	298 ± 5.1
1% starch + LDPE	0.126	0.205	0.150	0.250	27.2 ± 0.08	314 ± 6.0
5% starch + LDPE	0.360	0.516	0.350	0.450	36.85 ± 0.08	352 ± 6.0

±Standard deviation of three replicates.

The shift factors were also generated through the mastering software. The specimen was analyzed at frequencies of 0.01, 0.02, 0.05, 0.1, 0.2, 0.5, and 1. It is always better to use a minimum of five frequencies over two decades while generating a master curve. The specimens were also heated from 0 to 60°C.

#### Statistical analysis

The standard deviation (SD) was calculated using Stastica Software 99, and the values were expressed as mean ± SD.

## RESULTS AND DISCUSSION

### Mechanical properties:

The different mechanical properties of the starch filled LDPE are given in Table I. It can be observed that with incorporation of 1% starch, there was a marginal decrease in the mechanical properties of LDPE films. However, in the case of 5% starch filled LDPE film, mechanical properties such as percent elongation, tensile, tear, bursting, and seal strengths decreased by 19.23, 33.65, 3.88, 10.8, and 22.12%, respectively. Even though the decrease in tensile and seal strengths was large for the 5% starch filled film, it could be used as flexible packaging material.<sup>14</sup>

### Permeability properties

The water vapor transmission rate (WVTR) and oxygen transmission rate (OTR) of the starch filled LDPE have been given in Table II. It may be observed that WVTR and OTR values increased with the incorporation of starch, which may be attributed to the weakening of intermolecular forces in the polymer chain.<sup>22</sup> In the case of the 5% starch filled film, the increase in WVTR and OTR was by 32.5 and 18.3%, respectively. The increase in WVTR in starch filled LDPE may be attributed to the water-soluble starch portion in the film.

### Migration properties

Any packaging material coming in contact with food products must be evaluated for compatibility through the overall migration of plastic additions into food simulants, such as distilled water (for aqueous food products), 3% acetic acid (for acidic products), 50% ethanol (for alcoholic beverages), and *n*-heptane (for fatty foods) under simulating conditions. Migration values for all the LDPE starch blend film (Table II) revealed that there was a significant increase in migration value for the films containing starch. The migration values were very high in starch filled LDPE, and these films may not be suitable as primary packages as well as for packaging of liquid food.

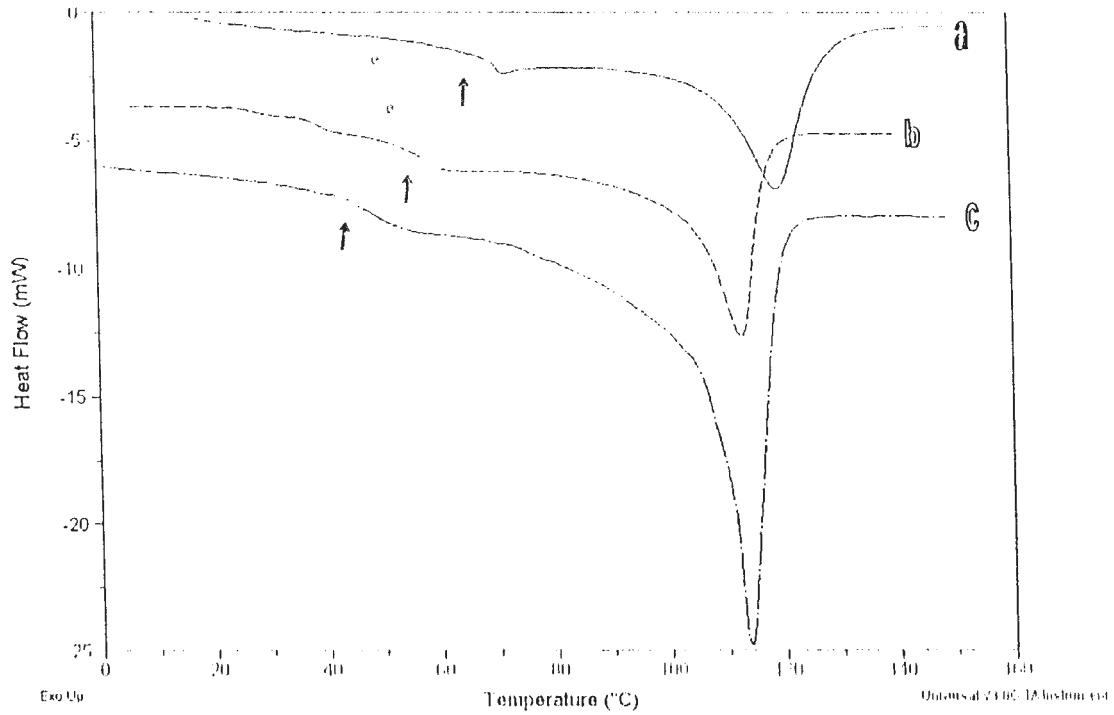
### Glass transition temperature (T<sub>g</sub>)

Glass transition temperature was measured using DSC as well as DMTA, and the values have been given in Table III and depicted in Figures 1 and 2. It can be observed that both techniques provided significantly different values of T<sub>g</sub>. This was expected as the T<sub>g</sub> is not an absolute value and depends on conditions and method of determination.<sup>23</sup> The definition of T<sub>g</sub> in the DSC is the onset of change in the heat capacity/slope, whereas in DMA it is change in modulus (E') or of tan δ. The T<sub>g</sub> determined by DMA is associated with molecular relaxation. The primary relaxation/change in heat capacity is labeled as α and is due to the glass-rubber relaxation/transition in the amorphous state. The data in Table III show the difference between the

**TABLE III**  
Thermal Properties of Pure LDPE and Starch Filled LDPE Films

Sample	T <sub>g</sub> from DSC	T <sub>g</sub> from DMA	ΔH from DSC mw
LDPE	67.7 ± 1.5	58.3 ± 0.9	40.5 ± 1.5
1% starch + LDPE	56.8 ± 1.2	47.5 ± 0.8	60.8 ± 1.5
5% starch + LDPE	48.5 ± 1.1	41.6 ± 0.8	472.5 ± 1.5

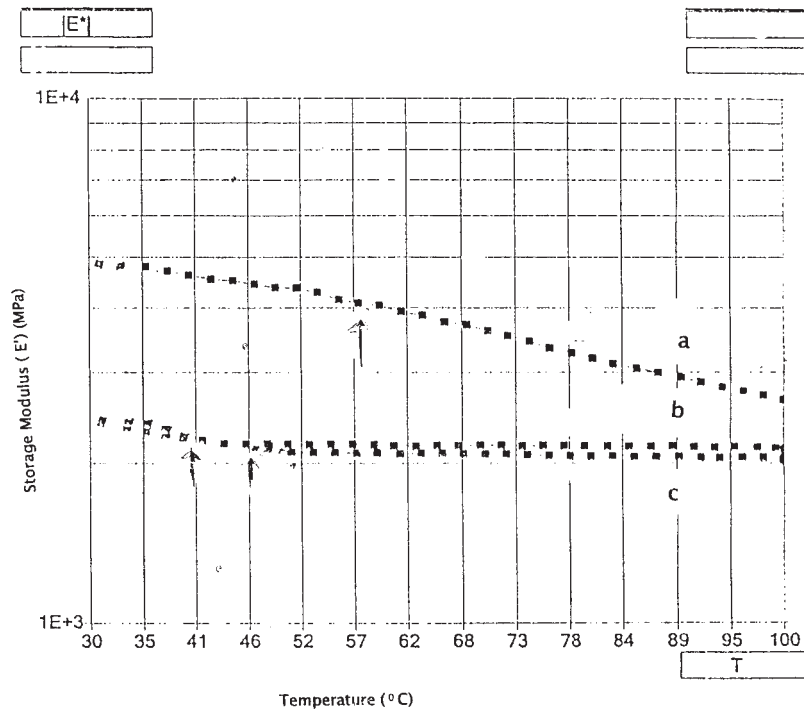
±Standard deviation of three replicates.



**Figure 1** Glass transition temperature of: (a) LDPE, (b) 1% starch filled LDPE, and (c) 5% starch filled LDPE film from DSC thermogram.

$T_g$  values determined using DSC and DMA was about  $8^\circ\text{C}$ . The  $T_g$  values determined for starch filled films of LDPE were almost the same. It was also observed that

$\Delta H$  value determined by DSC increased with an increase in starch content in the LDPE film. This is probably due to the fact that the starch component



**Figure 2** Glass transition temperature of: (a) LDPE, (b) 1% starch filled LDPE, and (c) 5% starch filled LDPE film from DMA thermogram.



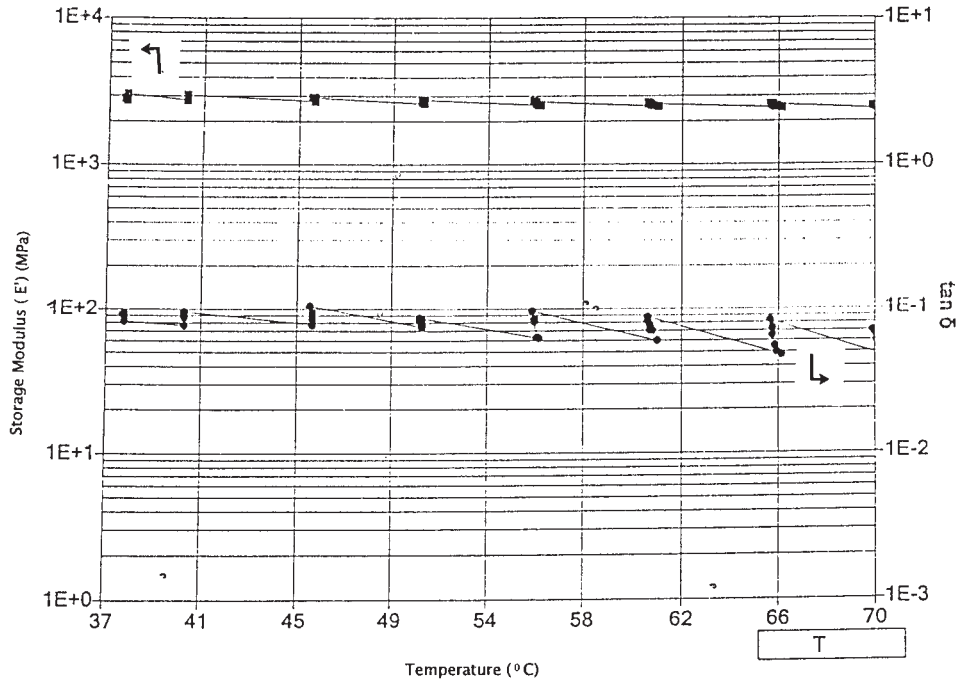


Figure 3 Typical temperature-frequency sweep of pure LDPE film for application of time-temperature superposition principle.

**WLF Equation**

$$\text{Log } a_t = \frac{C1 (Tg - T_0)}{C2 + (Tg - T_0)}$$

$T_0 = 30^{\circ}\text{C}$

Temp. Shift.  
C 1 : 31.54

C 2 : 89.40

60° C	-1.342
55° C	-1.137
50° C	-0.938
45° C	-0.709
40° C	-0.478
35° C	0.231
30° C	0.000
25° C	0.214
20° C	0.445
15° C	0.659
10° C	0.824
5° C	1.005
0° C	1.236

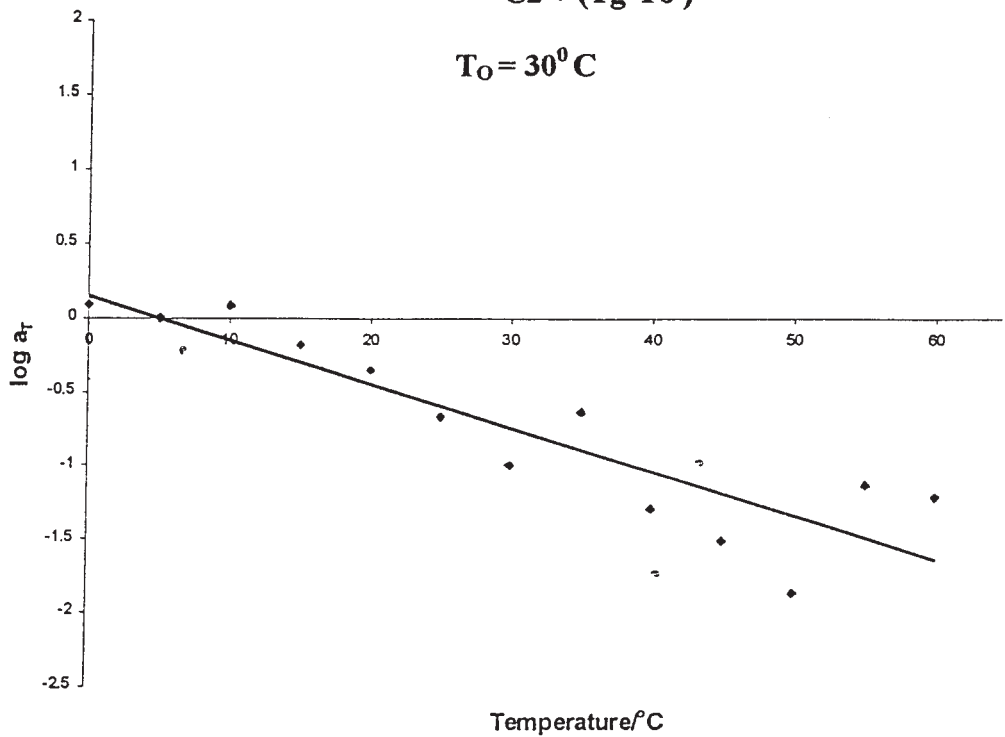


Figure 4 WLF equation (shift factor fit).

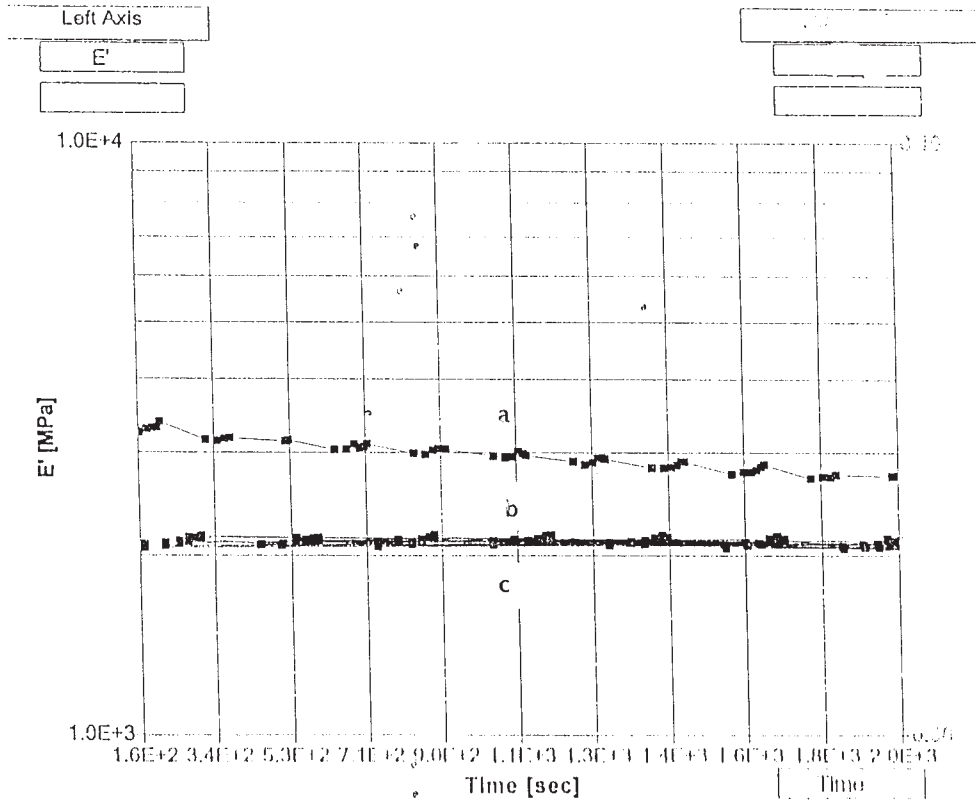


Figure 5 Master curve for: (a) LDPE, (b) LDPE + 1% starch, and (c) LDPE + 5% starch.

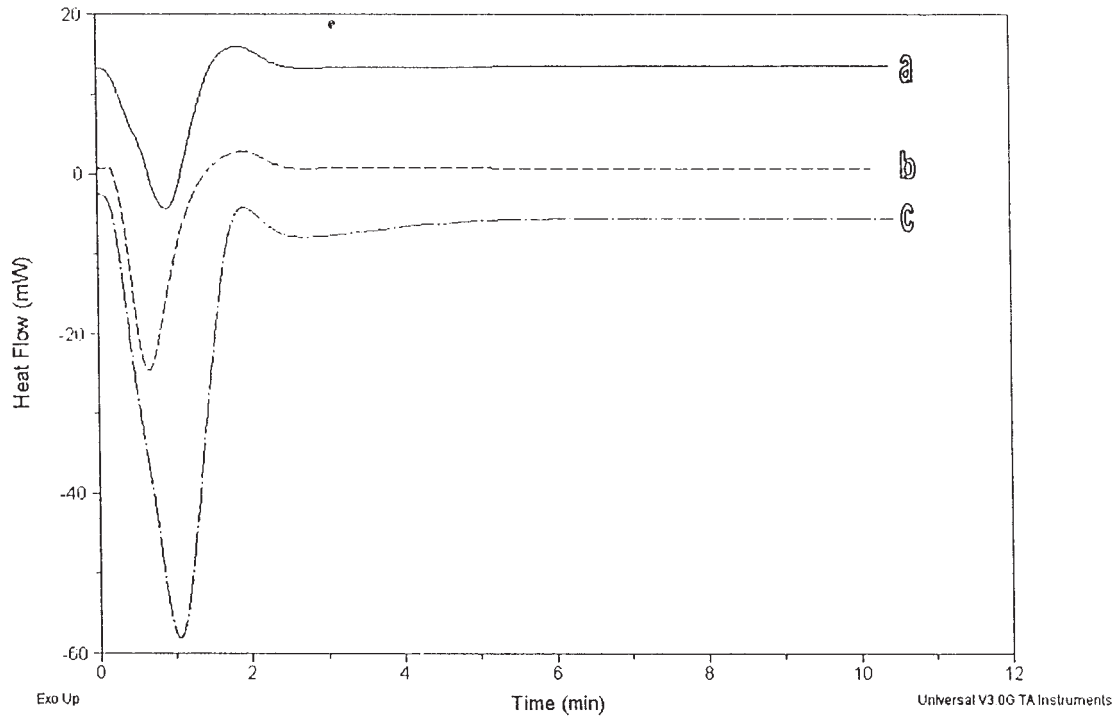


Figure 6 Isothermal crystallization plot for (a) LDPE, (b) LDPE + 1% starch, and (c) LDPE + 5% starch.

**TABLE IV**  
Crystallization Kinetics Parameter from Avrami Equation for pure LDPE and Starch Filled LDPE Films

Sample	$\Delta H$ (J/g)	Avrami index (n)	Log k
LDPE	$97.05 \pm 2.8$	$2.9 \pm 0.05$	$-7.63 \pm 0.05$
1% starch + LDPE	$83.05 \pm 2.5$	$2.6 \pm 0.05$	$-11.63 \pm 0.05$
5% starch + LDPE	$61.05 \pm 1.8$	$2.1 \pm 0.05$	$13.73 \pm 0.05$

$\pm$ Standard deviation of three replicates.

absorbs more heat for melting. Melting temperature ( $T_m$ ) of LDPE films decreased with the incorporation of starch (Fig. 1). It has been reported that plasticizers, which weaken the polymer chain, decrease the  $T_g$ .<sup>22</sup> It can be observed from Table III that the  $T_g$  of LDPE films decreased with an increase in starch content. Therefore, it can be inferred that starch acted as a plasticizer in the LDPE films. Plasticizers are known to act as flexibilizers and, hence, increase the percent elongation, which is evident from Table I. It can also be observed in Figure 2 that the storage modulus decreased with the incorporation of starch, which is in agreement with the decrease in tensile strength (Table I).

#### Time-Temperature superposition (TTS) principle

William, Landel, and Ferry<sup>24</sup> proposed an empirical equation that described the temperature dependence of relaxation times in the glass transition region and is given by  $\text{Log } a_t = C_1 (T_g - T_0) / C_2 + (T_g - T_0)$ , in which  $T_0$  is an arbitrary reference temperature while  $C_1$  and  $C_2$  are universal constants and  $a_t$  is the shift factor. The WLF equation is very useful in applying the principle of time-temperature superposition (TTS). To apply the TTS principle, the temperature-frequency sweep is necessary, and then the shift factor can be calculated by using the WLF equation. Figure 3 shows typical temperature-frequency sweep curves for pure LDPE. The curve created by the superposition is called a "master curve" and represents the time (frequency) response of the material at the reference temperature of 30°C.<sup>25,26</sup>

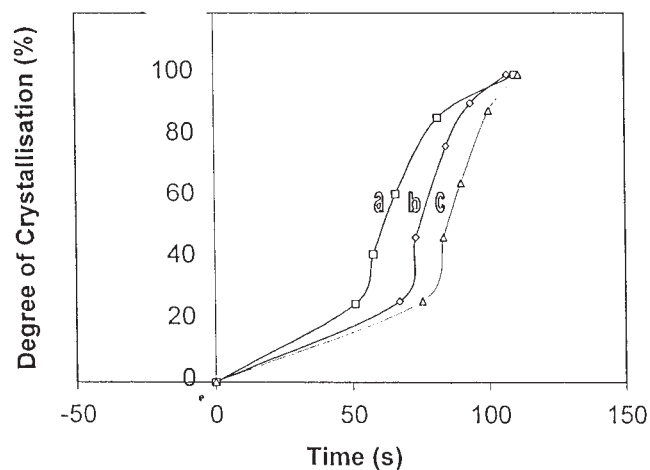
The underlying basis for time/temperature superpositioning is the demonstrated equivalence between time (or frequency) and temperature. It has been proven in WLF theory that viscoelastic data collected at one temperature can be superimposed upon data obtained at a different temperature simply by shifting (shift factor) one of the curves along the time (or frequency) axis.<sup>27</sup> The shift factor obtained by the software kit associated with DMA instruments has been graphically shown as a function of temperature in Figure 4. Once the shift factor is obtained by the software kit, then the master curve can be derived at

any desired temperature. The master curves obtained from these data were reduced by the WLF shift procedure, while the excellence of superposition showed the shape of the mechanical distribution of relaxation.<sup>24</sup>

Figure 5 represents the master curve of the storage modulus  $E'$  as a function of time at the reference temperature of 30°C. It can be observed that the starch filled LDPE films had lower modulus compared to that of the unfilled one, but the modulus remained almost constant with respect to time. In the case of unfilled LDPE film, the master curve indicated that initially the modulus was higher than that of the starch filled one; however, the modulus decreased drastically with respect to time. The main advantage of the master curve is to predict the properties of plastic with respect to time. If it is possible to evolve optimum (desired) mechanical properties by other means, then the master curve can predict the time in terms of days, months, or years required to deteriorate/attain that critical value. Master curves have been used for predicting the shelf life of composite materials.<sup>25</sup>

#### Isothermal crystallization

Figure 6 indicates the isothermal crystallization kinetics of starch filled LDPE films. The calculated heat of crystallization ( $\Delta H$ ) was obtained by integrating the peak with time and is given in Table IV. The  $\Delta H$  values were found to increase with an increase in starch content in the LDPE films. Taking the beginning of exotherm as time zero, that is,  $\theta(t) = 0$ , and the end when the trace has returned to base line, the crystallization was considered to be completed when  $\theta(t) = 1$ . The degree of crystallization  $\theta(t)$  was calculated by integrating the peaks with respect to time. The



**Figure 7** Plot of degree of crystallization versus time for: (a) LDPE, (b) LDPE + 1% starch, and (c) LDPE + 5% starch.



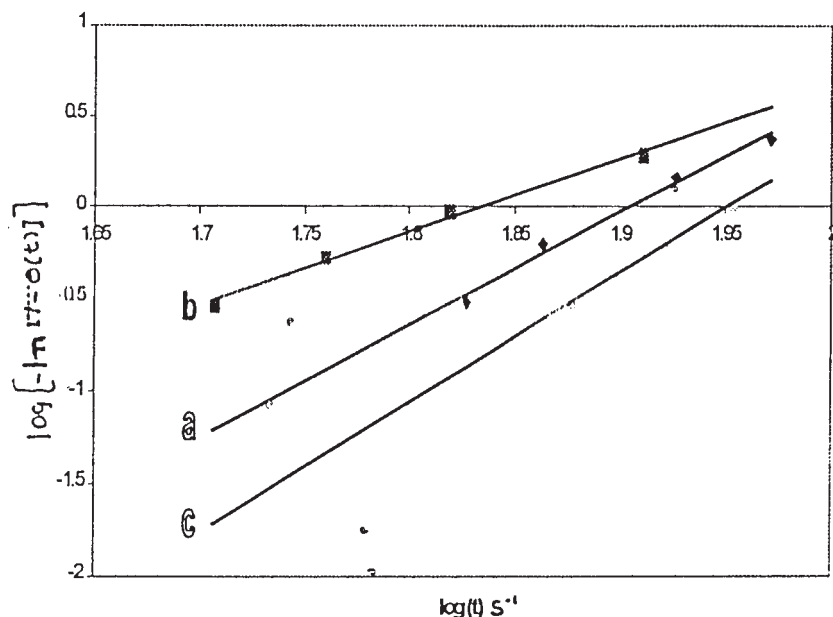


Figure 8 Avrami plot for: (a) LDPE, (b) LDPE + 1% starch, and (c) LDPE + 5% starch.

relative crystallinity  $\theta(t)$  was plotted against time for each peak (Fig. 7). It can be seen in Figure 7 that the incorporation of starch in LDPE retarded the degree of crystallization. The results were analyzed using the Avrami equation<sup>28,29</sup>:

$$\theta(t) = 1 - e^{-kt} \quad (1)$$

where  $k$  is the rate constant, depending on crystallization rate, and  $n$  is an exponent describing the growth order.  $\theta(t)$  is the degree of crystallization at time  $t$ . Rearrangement of eq. (1) gives

$$\log_{10}k + n \log_{10}t = \log_{10}(-\ln(1 - \theta(t)))$$

The plot of  $\log[-\ln(1 - \theta(t))]$  against  $\log t$  is a straight line and is shown in Figure 8. The slope gives Avrami exponent ( $n$ ), which varies from 3 to 4. This fact indicated that the crystallization of starch filled LDPE films was not an instantaneous nucleation but multi dimensional.<sup>26,27</sup> The intercept of the straight line with the Y-axis gave the rate constant and has been given in Table IV. Hence, it can be inferred from the data that starch interfered in the crystallization process of LDPE, and rate constant values indicated that the starch had a retarding effect on the kinetics of crystallization of LDPE. This is also evident from Figure 7.

## CONCLUSIONS

The master curve obtained by DMA indicated that the storage modulus of LDPE films decreased with respect to time; whereas that of starch filled LDPE films,

even though the modulus was lower, remained constant with respect to time. The  $T_g$  decreased with increase in the starch content of LDPE film, indicating the plasticizing or flexibilizing effect of starch. Mechanical properties marginally decreased with incorporation of starch in LDPE suggesting that the films could be used for solid food application but not for liquid ones because there was a drastic increase in WVTR and migration properties of starch filled LDPE. The isothermal crystallization kinetics indicated that the starch decreased the rate of crystallization in LDPE films.

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