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C. Albano<sup>a</sup>; G. Sanchez<sup>a</sup>; A. Ismayel<sup>a</sup>; P. Hernández<sup>a</sup>

<sup>a</sup> Universidad Central de Venezuela, Facultad de Ingeniería, Escuela de Ingeniería, Los Chaguaramos, Caracas, Venezuela

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# Recovery of Plastic Low-Density Polyethylene/High-Density Polyethylene (LDPE/HDPE) Wastes

C. ALBANO\*, G. SÁNCHEZ, A. ISMAYEL and P. HERNÁNDEZ

*Universidad Central de Venezuela, Facultad de Ingeniería, Escuela de Ingeniería Química, P.O. Box 48146, Los Chaguaramos, Caracas 1041 A, Venezuela*

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In order to show the importance of plastic recycling, a study of the mechanical, thermal and thermodegradative properties of low-density polyethylene (LDPE) blends with virgin and recycled high-density polyethylene (HDPE) was made to assess the possibility of using recycled polymers, and consequently, reducing environmental pollution brought about by those materials. The formation of an additional phase was determined through thermal analysis of LDPE blends with virgin and recycled HDPE. In the study of mechanical behavior of these blends, this intermediate phase was observed to increase tensile strength and flexibility of samples, thus acting as an interfacial agent. Regarding activation energy obtained in thermogravimetric studies, it was concluded that a phenomenon occurs which delays decomposition process at 30% and 40% recycled HDPE, because at higher compositions, properties completely deteriorate. It is possible to use recycled HDPE in blends with LDPE, since raw materials cost is reduced and properties of the finished product are enhanced.

**Keywords:** Polyblends; Mechanical behavior; Recycled HDPE; HDPE/LDPE; Thermal behavior; Thermodegradation

## INTRODUCTION

Plastics represent a serious environmental problem when they become waste. In the last decades, total consumption of plastics, as well as the number of applications have increased dramatically. Plastics are used in packaging, construction, buildings, transport, vehicle manufacture,

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\* Corresponding author.

electronic products, agriculture, and for many household goods. Because of this, it is impossible to avoid the generation of great amount of plastic waste, with the resulting severe environmental, ecological and economic problems.<sup>[1]</sup>

The most commonly used plastics, specifically in packing and wrapping, are polyolefins (polypropylene (PP), HDPE, LDPE, etc.). This is shown in Table I, which reflects the total consumption expressed as a percentage of different plastics used in the European Union and Venezuela. These polymers represent about 70% of the plastics used. This high consumption is reflected in wastes levels which consequently cause environmental and ecological problems.<sup>[1-3]</sup> Reinink<sup>[4]</sup> projected that by the year 2000, the volume of plastic waste in Western Europe will grow to 20 million tons, as compared to 13 million tons produced in 1989.

Recycling polymers is an essential process, however, it is important to determine the mechanical, thermal and thermodegradative behavior of these materials in order to optimize the virgin polymer/recycled polymer relation and to obtain suitable properties that do not result in deterioration of the finished product.

One of the goals pursued in the current study is to develop new materials from recycled polyolefins, especially those produced and used in Venezuela. Therefore, the main objective of the present paper is to study the technical feasibility of polyolefin recycling through the evaluation of the mechanical, thermal, thermodegradative, and morphological properties of LDPE with virgin and recycled HDPE.

Studies on mechanical properties are important because they help to detect if such properties deteriorate when recycled material is used, and, therefore, to determine the proper amount of recycled polymer

TABLE I Total consumption of plastics (%) for packaging in Venezuela and the European Union<sup>[1-3]</sup>

<i>Plastics</i>	<i>EU</i>	<i>Venezuela</i>
LDPE	37.9	28.9
HDPE	17.5	20.8
PP	11.2	14.5
PS + ABS	11.7	12.4
LLDPE	6.6	—
PET	3.5	—
Other	1.2	3.2
PVC	10.4	20.2

to be used so that values of tensile and impact properties are not affected. Likewise, analysis of thermal properties are necessary because they show fusion and crystallization temperatures of mixtures, values which are important to determine processing conditions, as well as their crystallinity, a very important characteristic influencing some tensile properties.

Thermodegradative studies help to detect conditions under which material is degraded, especially if one of the components is a recycled one. Thus, by means of a comparative study of blends with virgin material and recycled polymer, it can be determined if it is possible to decrease raw material cost. Also proper conditions can be found so that composition is not accelerated, since it strongly influences final properties of the manufactured produced, especially if one of the components is a recycled material.

## THEORETICAL BACKGROUND

In kinetic studies of decomposition, the concentrations of the reactants and products are major factors. Reactions are considered irreversible, and kinetics depends on the activation energy and order of the reaction. The parameters can be calculated from a dynamic experimental curve. When we combine the reaction velocity and the Arrhenius equation, we have the following basic expression:

$$\frac{d\alpha}{dt} = f(\alpha)A \exp\left(-\frac{E_a}{RT}\right), \quad (1)$$

where  $T$  is the temperature (K),  $d\alpha/dt$  is the velocity of reaction ( $t^{-1}$ ),  $f(\alpha)$  is a function of  $\alpha$  depending on the reaction mechanism,  $\alpha$  is the reacted fraction at time  $t$ ,  $E_a$  is the activation energy (kcal/mol),  $A$  is the pre-exponential factor, and  $R$  is the gas constant. With Equation (1), we can develop different methods to evaluate the activation energy.

With integral method of Coats and Redfern,<sup>[5]</sup> the expression is as follows when  $n \neq 1$  ( $n$  is reaction order):

$$\log\left\{\frac{1 - (1 - \alpha)^{1-n}}{T^2(1 - n)}\right\} = \log\frac{AR}{BE_a} \left[1 - \frac{2RT}{E_a}\right] - \frac{E_a}{2.3RT}, \quad (2)$$

where  $B$  is the rate of heating (degree/time). When  $n = 1$ , the expression becomes

$$\log\left(-\frac{\log(1-\alpha)}{T^2}\right) = \log\frac{AR}{BE_a}\left(1 - \frac{2RT}{E_a}\right) - \frac{E_a}{2.3RT}. \quad (3)$$

Van Krevelen *et al.*<sup>[6,7]</sup> using the same methodology as Coats and Redfern,<sup>[5]</sup> obtained, for  $n \neq 1$ :

$$\ln\left(\frac{(1-\alpha)^{1-n} - 1}{1-n}\right) = \ln\left(\frac{A}{B}\left(\frac{0.368}{T_n}\right)^{E_a/RT_n}\left(\frac{1}{E_a/RT_n + 1}\right)\right) + \left(\frac{E_a}{RT_n} + 1\right) \ln T, \quad (4)$$

where  $T_n$  is the temperature at the maximum rate (K). When  $n = 1$ , the expression becomes

$$\ln(\ln(1-\alpha)) = \ln\left(\frac{A}{q}\left(\frac{0.368}{T_n}\right)^{E_a/RT_n}\left(\frac{1}{E_a/RT_n + 1}\right)\right) + \left(\frac{E_a}{RT_n} + 1\right) \ln T. \quad (5)$$

Horowitz and Metzger<sup>[8]</sup> is a simplification of that of Coats and Redfern.<sup>[5]</sup> Based on Equations (2) and (3), the characteristic temperature is defined  $\theta = T - T_s$ , where  $T$  is the temperature at any time, and  $T_s$  the TGA differential curve peak temperature. For  $n \neq 1$ , the following expression is obtained:

$$\ln(1 - (1-\alpha)^{1-n}) = -\frac{E_a\theta}{RT_s^2} + \ln(1-n). \quad (6)$$

When  $n = 1$ , Equation (7) becomes

$$\ln(-\ln(1-\alpha)) = \frac{E_a\theta}{RT_s^2}. \quad (7)$$

## EXPERIMENTAL

Since the objective is to recycle polymers already used, therefore, degraded, the material has to be prepared to carry out experiments required for its evaluation. To obtain degraded HDPE with characteristics of a recycled material and unpolluted with other polymers or additives, virgin HDPE was degraded up to a decomposition percentage between 15 and 20. To this end degradation conditions were selected based on work by Arribas *et al.*,<sup>[9]</sup> and the polymer was subjected, at different exposure times, to a temperature of 280°C. Decomposition percentage was measured by determining viscosity variation of the polymer as a function of exposure time.

During the first part of the experiment, LDPE (Lagotene, density: 0.925 g/cm<sup>3</sup>, MFI: 17 g/10 min) and virgin (Polímeros del Lago, density: 0.958 g/cm<sup>3</sup>, MFI: 6.0 g/10 min) and recycled HDPE (MFI: 5.6 g/10 min) were mixed in an extruding machine, at different compositions.

Mechanical properties, after compression molding, were measured using an Instron Tensile tester with a crosshead rate of 25 mm/min. Impact strength measurements were made on notched samples using a Zwick impact testing machine. Differential scanning calorimetry (Perkin Elmer, model DSC7 and DuPont, model 990) was used to observe melting and solidification behavior. To erase thermal history, the first cooling and the second heating thermograms were detected with a scanning rate of 5°C/min in the first cooling and 10°C/min in the second heating. The morphology of each sample was observed through a transmission electron microscope (Hitachi, model H-500).

Thermograms were obtained with TGA (DuPont, model 951) in which samples were heated to a temperature of 773 K at a rate of 10°/min, in a nitrogen atmosphere. Subsequently, the blend was examined kinetically using the different integral methods to determine the global energy of activation.

## RESULTS AND DISCUSSION

As shown in Table II, Young's modulus for both blends increases with increasing percentages of virgin or recycled HDPE in the composition.

TABLE II Mechanical properties of virgin LDPE with virgin and recycled HDPE blends (E: Young's modulus;  $\sigma_B$ : breaking strength;  $\varepsilon_B$ : elongation to break; IS: Impact strength;  $\sigma_f$ : yield strength;  $\varepsilon_f$ : yield elongation, v: virgin, r: recycled)

HDPE (%)	E (kg/mm <sup>2</sup> )	$\sigma_B$ (kg/mm <sup>2</sup> )	$\varepsilon_B$ (%)	$\sigma_f$ (kg/mm <sup>2</sup> )	$\varepsilon_f$ (%)	IS (J/m)
0r	15 ± 2	0.90 ± 0.02	83 ± 4	1.02 ± 0.1	18.4 ± 0.8	> 200
10r	21 ± 3	0.94 ± 0.01	252 ± 10	1.18 ± 0.09	14.8 ± 0.6	117 ± 10
20r	26 ± 2	0.97 ± 0.03	325 ± 20	1.23 ± 0.11	14.9 ± 0.9	62 ± 8
30r	35 ± 4	1.09 ± 0.04	487 ± 20	1.40 ± 0.06	14.3 ± 0.8	32 ± 6
40r	41 ± 3	1.13 ± 0.02	515 ± 50	1.58 ± 0.08	13.5 ± 1.0	23 ± 6
50r	35 ± 2	0.49 ± 0.01	9 ± 2	1.24 ± 0.11	8.2 ± 1.1	22 ± 4
100r	55 ± 3	1.57 ± 0.03	560 ± 60	2.33 ± 0.12	10.9 ± 0.6	38 ± 5
10v	21 ± 2	0.93 ± 0.02	130 ± 10	1.06 ± 0.06	16.7 ± 0.8	85 ± 8
20v	27 ± 2	1.01 ± 0.01	280 ± 20	1.26 ± 0.04	15.0 ± 0.9	45 ± 5
30v	21 ± 3	0.95 ± 0.04	308 ± 40	1.34 ± 0.12	14.5 ± 0.9	25 ± 4
40v	29 ± 4	1.08 ± 0.08	394 ± 60	1.52 ± 0.08	13.7 ± 0.8	23 ± 4
50v	34 ± 4	1.14 ± 0.09	630 ± 45	1.67 ± 0.14	13.0 ± 0.8	20 ± 3
100v	53 ± 6	2.23 ± 0.12	960 ± 100	2.73 ± 0.16	10.4 ± 0.9	28 ± 2

With recycled HDPE, the modulus values are slightly higher and show synergism when 40% recycled HDPE is used. From these results, it can be concluded that the blends become more rigid due to the presence of HDPE, but not as rigid as HDPE itself. Synergism refers to the situation in which the values of some properties for the blend are higher than for either pure HDPE or LDPE. Clearly, such a peak in the plot of property versus composition is not predicted by any additivity relationship.<sup>[10]</sup>

The modulus of crystalline polymers greatly depends on spherulitic morphology, the size of spherulites, and the lamellar thickness.<sup>[11]</sup> These virgin and recycled polymers present a high nucleation density and the size of the small spherulites have a granular appearance (Figure 1). For this reason, the increase in modulus can be explained by the change in the size of the lamella, depending on HDPE content. Thus, it can be deduced that the HDPE and LDPE lamellas have different thickness, as shown in Figure 2. There it is possible to detect qualitatively (because both micrographs have the same magnification) that LDPE lamellas are thinner and shorter than those of HDPE, either virgin or recycled. Pereira *et al.*<sup>[12]</sup> recently found by means of X-ray diffraction studies (WAXS) that LDPE shows a smaller crystal thickness than HDPE.

As shown in Table II, elongation to break of LDPE with virgin HDPE blends increases with increasing HDPE content. In the case of

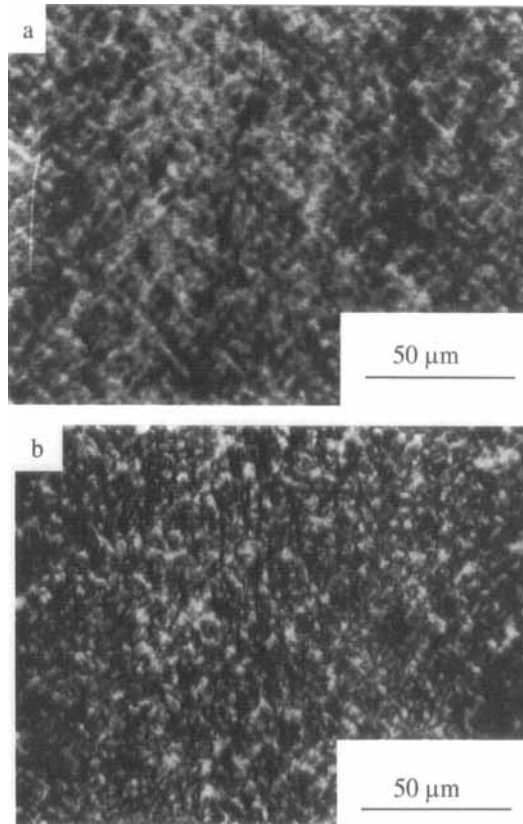


FIGURE 1 Optical micrographs of (a) virgin HDPE and (b) recycled HDPE.

LDPE with recycled HDPE, there is an increase of elongation to break, up to 40% composition of recycled polymer. Subsequently, there is a steep decline of the property in the case of 50% composition. In the cases where HDPE is recycled, the values are slightly higher. The behavior of these blends, with the exception of that having 50% recycled HDPE, shows a certain degree of interaction in the amorphous phase or compatibility. The reduction detected at a 50% composition may be due to previous degradation of HDPE, which weakens the tying molecules. Therefore, the material becomes weaker and the rupture occurs earlier than expected.



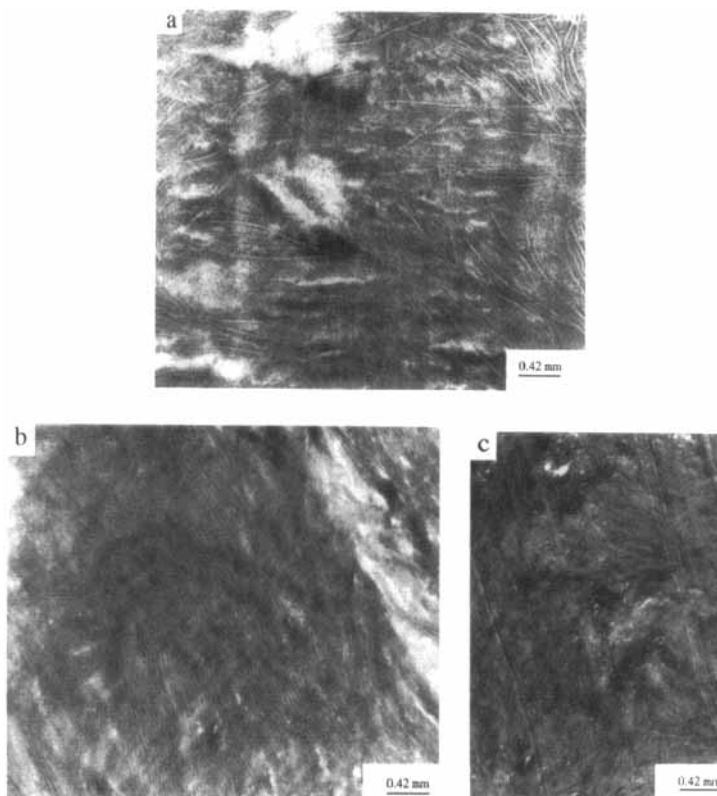


FIGURE 2 TEM micrographs of (a) virgin HDPE, (b) recycled HDPE and (c) LDPE.

Breaking strength increases slightly in both blends and, in the case of a 50% composition of recycled HDPE, it suddenly decreases. The increase in breaking strength can be attributed to the fact HDPE presents more tying molecules and intercrystal bonds. These types of links play an important role in the transmission of force between lamellas. However, in blends of 50% recycled HDPE, the tying molecules weaken or break, and the material has less resistance due to previous degradation reactions.

Morphology obtained by TEM (Figures 3 and 4) shows that there is no apparent dispersed phase of the HDPE observable in the matrix of LDPE. This shows that there is some compatibility or partial miscibility that improves the mechanical properties with an increase in the

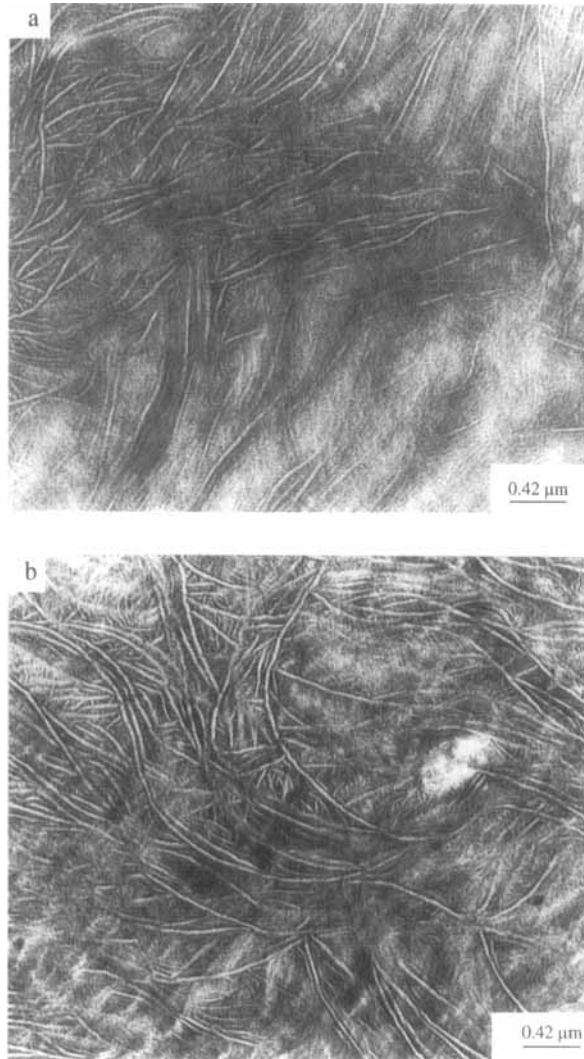


FIGURE 3 TEM micrographs of LDPE with (a) 30% virgin HDPE and (b) 50% virgin HDPE.

concentration of HDPE. At 50% composition of recycled HDPE, this polymer does not increase the thickness of lamellas (Figure 4b). This factor influences negatively on ductile properties. This fact could explain the behavior observed at this particular composition of

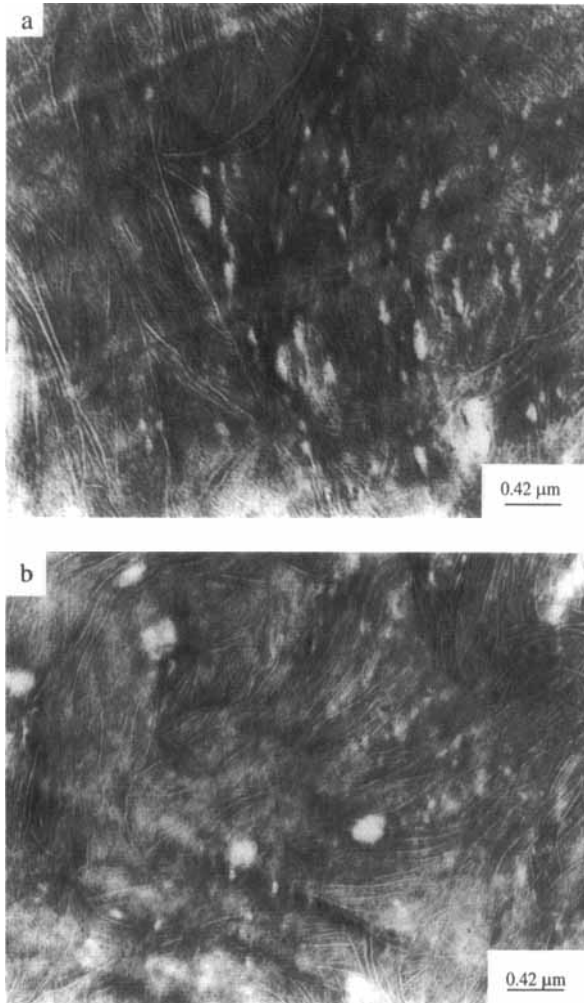


FIGURE 4 TEM micrographs of LDPE with (a) 30% recycled HDPE and (b) 50% recycled HDPE.

this recycled material. In Table II, resistance to impact is extremely high in the case of LDPE, and could not be measured with available test equipment, which is caused by the higher tenacity of LDPE in relationship to HDPE.

As the concentration of virgin and recycled HDPE increases in the LDPE blends, the impact strength decreases. As a result, the material loses tenacity, since its behavior becomes less ductile. This leads to the conclusion that HDPE does not provide the mechanism of energy dissipation. However, the compositions behave in a similar way to a polymer with rigid filler. Thus, in both blends, the impact strength shows an incompatible behavior, because this property exhibits a minimum when plotted versus blend proportion.<sup>[10]</sup>

The results of the thermal behavior are shown in Table III. The fusion temperatures of the virgin and recycled HDPE are 133°C and 134°C, respectively. Similarity in these values is the result of the fact that although HDPE decomposition percentage is between 15 and 20, fusion temperature of the recycled material is not affected.

When HDPE is degraded there are chain rupture reactions which decrease molecular weight, as well as intercrossing reactions which affect crystal regularity.<sup>[13]</sup> Moreover, intercrossing acts as a defect in molecular structure.<sup>[14]</sup> For these reasons, fusion temperature of recycled HDPE should decrease. This does not occur in the case under study, because decomposition percentage is too low for fusion temperature to decrease. Another reason for this behavior is that MFI of recycle HDPE is almost equal to that of virgin LDPE.

The endothermic curves of the blends present two peaks corresponding to the fusion temperatures of homopolymers (Figures 5 and 6),

TABLE III Values obtained from DSC studies the virgin LDPE with virgin and recycled HDPE blends. ( $T_f$ , fusion temperature;  $\Delta H_f$ , fusion heat;  $t$ , theoretical;  $e$ , experimental;  $X_c$ , crystallinity;  $v$ , virgin;  $r$ , recycled)

HDPE (%)	$T_{f1}$ (°C) ± 1	$T_{f2}$ (°C) ± 1	$T_{f3}$ (°C) ± 1	$\Delta H_{fe}$ (J/g) ± 5	$\Delta H_{ft}$ (J/g) ± 5	$X_c$ (%) ± 5	$X_t$ (%) ± 5
0	111	—	—	105	—	35	—
10r	108	117	124	126	113	44	40
20r	107	120	124	126	121	45	43
30r	107	122	125	138	130	48	46
40r	108	—	126	146	138	51	49
50r	110	—	125	130	146	46	52
100r	—	—	133	189	—	73	—
10v	108	119	123	100	117	35	41
20v	109	121	125	117	126	41	45
30v	110	122	125	130	138	46	49
40v	108	—	126	142	151	50	53
50v	107	—	125	159	159	56	56
100v	—	—	134	213	—	82	—

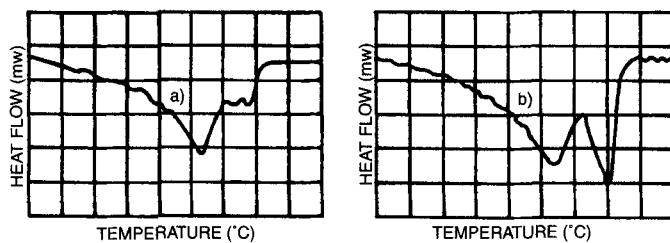


FIGURE 5 DSC curves of LDPE with virgin HDPE blends heating  $10^{\circ}\text{C}/\text{min}$ . (a) 90/10, (b) 80/20.

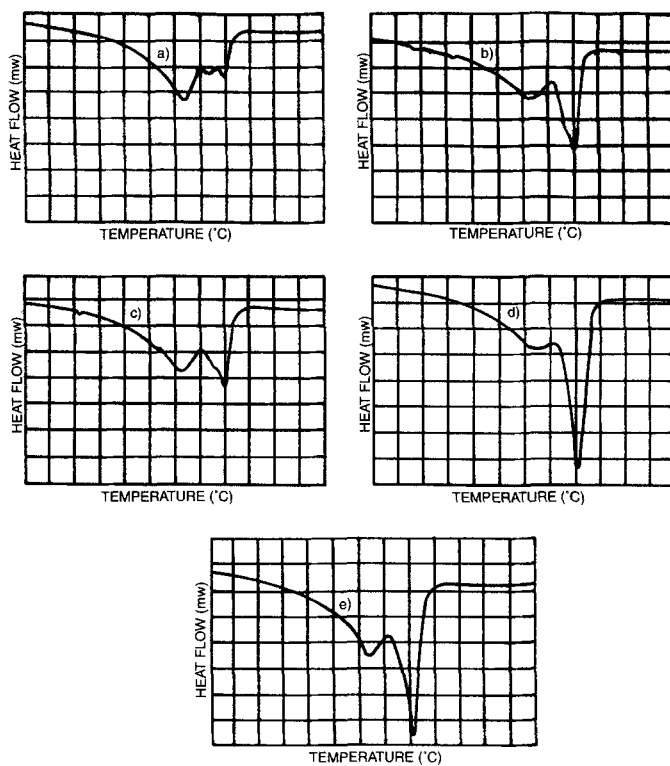


FIGURE 6 DSC curves of LDPE with recycled HDPE blends heating  $10^{\circ}\text{C}/\text{min}$ . (a) 90/10, (b) 80/20, (c) 70/30, (d) 60/40, (e) 50/50.

signifying immiscibility. Fusion temperatures of both components (virgin and recycled HDPE and LDPE) of the blends decrease with increasing HDPE content. This behavior is related to an interplasticizer effect, caused by some molecules of one of the components acting as a diluent in the crystalline zone of the other component.

In compositions formed with 90–70% LDPE, a third endothermic peak is observed (Figures 5 and 6). This is probably due to a new crystalline phase being formed in the polyblend, as a result of an interaction of both components. This assumption agrees with some works carried out with Raman spectrometry with HDPE/LDPE blends. In these studies, researchers recognize three regions: crystalline, amorphous and interfacial, this latter being product of interactions at the molecular level.<sup>[15]</sup>

Bonotto *et al.*<sup>[16]</sup> and Siegmann *et al.*<sup>[17]</sup> found similar results in LLDPE/LDPE polyblend: they observed two endotherms, and in one of them two peaks were detected, which implies the existence of an additional phase which can be formed by cocrystallization of both polyethylenes.

It can be observed in Figures 5 and 6 that for the mixtures studied, this third peak appears at higher temperatures as HDPE concentration is increased. As a consequence, the fusion temperature of the new phase is superposed on that of HDPE. This behavior is similar in both blends virgin and recycled HDPE. Similar results have been obtained with crystallization exotherms (Figures 7 and 8).

From the crystallinity obtained in the experiments, we noticed that the crystallinity values of blends, where HDPE is recycled, were higher than those for virgin HDPE with LDPE. Moreover, the experimental crystallinity is higher than obtained from the additivity rule for the blends with recycled HDPE.<sup>[18]</sup> Similar results are observed with heat of fusion (enthalpy values). When blending LDPE with recycled HDPE, a new crystalline phase could have been formed, as in the blends where both polymers are virgin (LDPE/HDPE). From these results, an increase of the modulus and breaking strength is expected, as well as an increase of the elongation at break, because this phase acts as a link in the interface and improves mechanical properties in general. In addition, in the blends of LDPE with recycled HDPE, this phase generates increased crystallinity that also improves Young's modulus.

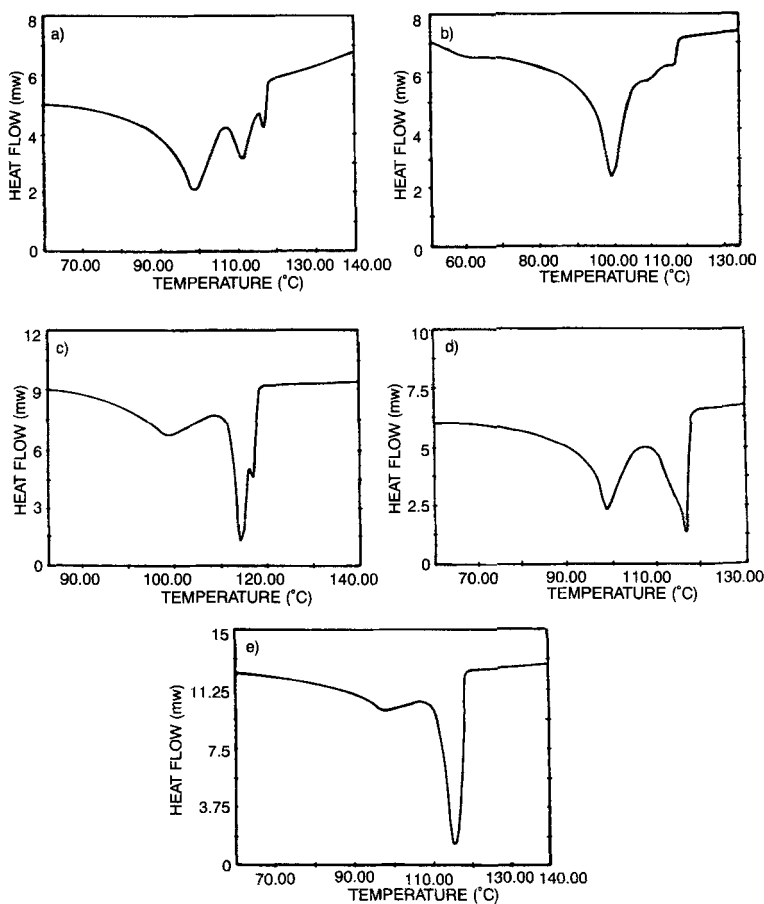


FIGURE 7 DSC curves of LDPE with virgin HDPE blends cooling  $5^{\circ}\text{C}/\text{min}$ . (a) 90/10, (b) 80/20, (c) 70/30, (d) 60/40, (e) 50/50.

In Table IV, the kinetic parameters of the pure components and the blends are shown. Those parameters are obtained through different methods. The methods applied to determine the activation energy were Van Krevelen (VK), Coats–Redfern (C–R), and Horowitz–Metzger (H–M). The activation energy of the decomposition process of LDPE is lower than the corresponding values of the pure and recycled HDPE. This fact agrees with the results obtained by Allen and Chirinos,<sup>[19]</sup> the decomposition kinetics is inversely proportional to the crystalline phase content.

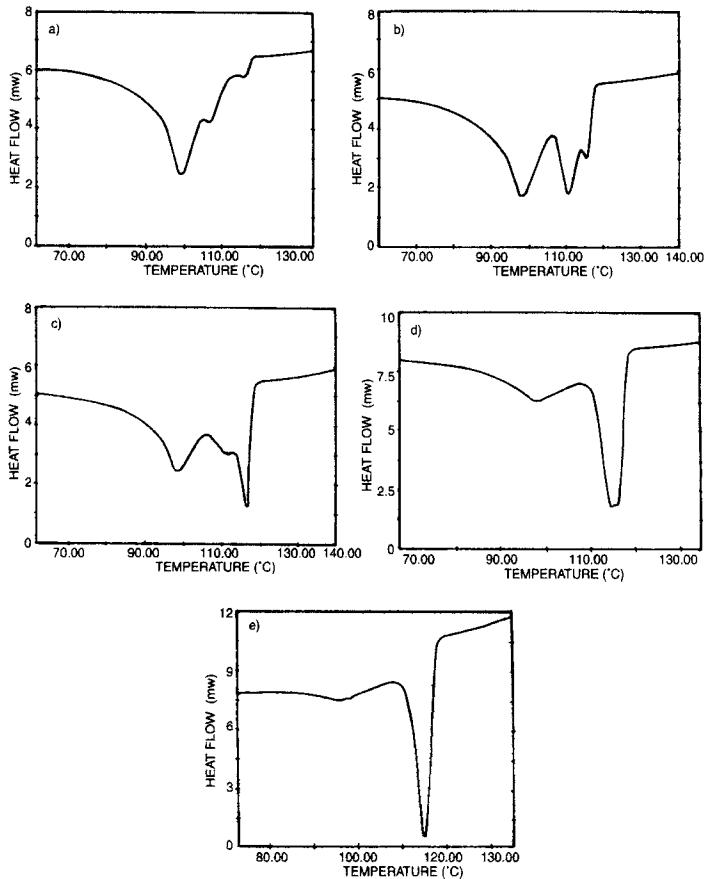


FIGURE 8 DSC curves of LDPE with recycled HDPE blends cooling  $5^{\circ}\text{C}/\text{min}$ . (a) 90/10, (b) 80/20, (c) 70/30, (d) 60/40, (e) 50/50.

Table IV shows that the activation energy for the blends with virgin material is lower than  $E_a$  obtained for pure LDPE, except for 50% composition. One can infer that one polymer accelerates the decomposition process of the other polymer. Besides, it is shown that the activation energy increases slightly with the concentration of the recycled and virgin HDPE. The effect is considerable when there is previous recycled material in the blends.

At compositions of 70–60% LDPE with 30–40% recycled HDPE, there is a slight synergistic effect, since the activation energy obtained



TABLE IV Global activation energy ( $E_a$ ) for the degradation of LDPE, recycled HDPE (r) and virgin HDPE (v) and their blends (Values in parenthesis are theoretical,  $E_{ab}$ )

HDPE (%)	$H-M$ (kcal/mol) $\pm 5$	$V-K$ (kcal/mol) $\pm 5$	$C-R$ (kcal/mol) $\pm 5$
0	45	44	42
10v	30 (48)	28 (47)	26 (45)
20v	35 (51)	34 (50)	33 (47)
30v	35 (53)	32 (52)	31 (50)
40v	35 (57)	34 (55)	33 (53)
50v	48 (60)	46 (58)	45 (56)
100v	74	72	69
10r	37 (48)	36 (47)	35 (45)
20r	46 (51)	44 (50)	42 (47)
30r	59 (54)	56 (53)	52 (50)
40r	68 (57)	64 (57)	61 (53)
50r	52 (61)	52 (59)	49 (56)
100r	76	73	69

is higher than  $E_a$  of the pure LDPE. This would indicate that there is some phenomenon that impedes the acceleration of the degradation process, due to the addition of this recycled material.

Polikorpov *et al.*<sup>[20]</sup> showed that the activation energy is related to the intermolecular interaction energy in the amorphous phase and this phase is related to the mobility of chains. One can deduce that at these compositions, the recycled HDPE influences positively the thermal stability of the LDPE. This is possibly due to the new phase formed in the interphase, which could give rise to a reduction in the mobility of the chains of both polymers.

Analysis of the parameter relating to  $E_a$  (theoretical  $E_a$ ) additivity obtained from the activation energy of corresponding homopolymers in the following equation is useful.<sup>[18]</sup>  $E_{ab} = \sum E_{ai} X_i$  where  $E_{ai}$  and  $E_{ab}$  are the activation energies corresponding to the homopolymers and the blend, and  $X_i$  the weight fraction of the homopolymers.

When  $E_a$  of a blend is higher than the additive behavior (theoretical  $E_{ab}$ ) of  $E_a$  corresponding to the homopolymers (Table IV), a delay has occurred in the decomposition process, that is, thermal stability of materials has increased. In the studied blends,  $E_a$  values are lower than those obtained by additivity and than  $E_a$  of the pure components. This confirms that one of the polymers speeds the decomposition process of the other. This accelerating action could even have a catalytic

character. In the case of 30% and 40% recycled HDPE, one can deduce that at these compositions, there is a stabilization of the blends, because activation energy is higher than that obtained by additivity.

## CONCLUSIONS

Thermal studies of LDPE blends with virgin and degraded HDPE allow us to establish the formation of an additional phase. Traction and impact essays show that this additional phase increases tensile strength and flexibility of mixtures.

Thermogravimetric analysis allow us to conclude that at 30% and 40% HDPE compositions, a delay occurs in the decomposition process.

In summary, the mechanical, thermal and thermodegradative behaviors of the blend with virgin and recycled material suggests that the additional phase behaves as an interfacial agent, which improves the adhesion at the interface. Therefore, it is possible to use the recycled HDPE in blends with LDPE (40/60% and 30/70%), since it appears to enhance the properties of the finished product.

## Acknowledgments

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