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Properties of Post-used High Density Polyethylene Crates and Its Modification by Ethylene Vinyl Acetate Blending

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Abstract

In an attempt to recycle plastic waste of high density polyethylene (HDPE) crates used for bottle transportation in such a hot and humid environment, the characteristics and the mechanical properties of HDPE crates, as well as the processability of the polymer, were studied systematically along the period of utilization time. The results indicate that increasing the period of utilization time remarkably decreases the elongation at break, impact resistance, and flexural strength at yield of HDPE crates. The mechanical properties of post-used HDPE crates after 8-years of utilization time are generally worse than those of new HDPE crates, particularly the elongation at break and the impact resistance, possibly due to a photo-oxidative degradation process. The post-used HDPE crates were then blended with ethylene vinyl acetate copolymer (EVA), in order to improve the poor properties of post-used crates for recycling purposes. It was found that 5% EVA added in the post-used crates had noticeable effects on improving the elongation at break by more than 300%, raising the elongation at break of the blends almost up to that of the virgin material. This might suggest a compatibility in the blends of post-used HDPE crates and EVA.

Keywords : HDPE crate; EVA; Mechanical properties

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Introduction

Plastic recycling has been a topic of great interest for the last few decades due to the widespread concern about the world environment. Many studies have been made in an attempt to explore alternative routes to reduce plastic waste by recycling that is exclusive for each type of polymer and product (Bisio, *et al.* 1994; and Ehrig, 1992). High density polyethylene (HDPE), one of the most widely used commodity plastics, is used to produce a wide range of plastic containers. This is due to the advantageous properties of HDPE in the areas of high impact strength, toughness, good chemical resistance, excellent moisture and water resistance and recycleability. Plastic crates used for bottle transportation is one of the major products manufactured from HDPE. Because of the popularity in soft-drink consumption in such a hot and humid country as Thailand, the average demand in plastic crates in the soft-drink industry is around 200,000 – 300,000 containers per year (or 300 – 450 tons per year). Considering the whole world, hundred thousands of HDPE crates must be used. The average lifetime of plastic crates is about eight years. As a result, a huge amount of waste from plastic crates has been generated that should be handled properly in order not to cause any environmental pollution.

Previous studies on the mechanical recycling of post-used, yellow-pigmented HDPE crates using a restabilization technique by Kartalis, *et al.* (2000) has reported that Recyclossorb 550 (R550) significantly improves the processability during a repeated remelting cycle as well as an elongation at break of post-used crates due to a limitation in the free volume between the branched polymer chains of nonrestabilized post-used HDPE crates. Later on, Kartalis, *et al.* (2000) have reported the effect of artificial weathering on the properties of post-used, yellow-pigmented HDPE crates, using R550. The impact strength of the nonrestabilized grade dramatically decreases because of severe photo-oxidative degradation while the addition of the R550 improves the light stability of the recycled HDPE crates, resulting in excellent retention of impact strength for at least up to 8000 hours in artificial exposure.

A study on other post-used HDPE containers such as containers for liquid by Loulteheva, *et al.* (1997) reported that the rheological and mechanical properties of recycled HDPE containers were strongly dependent on the reprocessing conditions, i.e. temperature, residence time and applied stress. A report on the properties of recycled HDPE milk bottles by Pattanakul, *et al.* (1991) indicated that the elongation at break was affected the most by the composition of recycled HDPE. At an appropriate concentration of recycled HDPE in virgin HDPE, useful properties are not largely different from those of virgin materials.

Boldizar, *et al.* (2000) studied the recycling of post-used, uncolored HDPE bottles. The recycled materials were examined by a testing procedure consisting of reextrusion and subsequently accelerated by thermo-oxidative aging. It was found that the HDPE material did not degrade to any significant degree during 10 cycles of stimulated recycling.

This work, therefore, aims to study the possibility in recycling plastic wastes of HDPE crates used for soft drink bottle transportation. The properties of plastic crates, particularly used in hot and humid weather, including the mechanical and physical properties, the processability and the degradation of polymers were studied systematically. The raw materials are the red HDPE crates, produced under the same condition, which have been used for bottle transportation for 8, 3 and 0 years, respectively. In addition, the post-used HDPE crates were blended with ethylene vinyl acetate copolymer (EVA), a highly elastic polymer, in an attempt to improve poor properties of post-used crates for recycling purposes.

Experimental

Materials

The high density polyethylene used in this study is from red plastic crates for 10-ounce soft drink bottle transportation supplied by Srithai Superware Public Co., Ltd. and Thai Pure Drinks Co., Ltd. All materials can be classified according to different utilization times as shown in Table 1, including virgin HDPE granule. It should be noted that the utilization times are approximate from the production year, assuming that the crates were regularly used in the soft-drink transportation industry. Ethylene vinyl acetate copolymer (EVA) with 18% vinyl acetate content obtained from the Thai Petrochemical Industry Public Co., Ltd., was used to blend with post-used HDPE crates.

Table 1 Classification of HDPE samples

Code	Materials
VIRGIN	Virgin HDPE
HDPE#0	New HDPE crates
HDPE#3	3-years used HDPE crates
HDPE#8	8-years used HDPE crates
EVA5%	Blend of HDPE#8 : EVA at 95:5 (wt:wt)
EVA15%	Blend of HDPE#8 : EVA at 85:15 (wt:wt)

Sample preparation

All HDPE crates were washed in water, dried and then shredded into granules with a size of about 5mm. For the blends of post-used HDPE crates and EVA, two different compositions as shown in Table 1 were firstly prepared by dry blending in "BOSCO" Pot for 15 min and then melt blending by "COLLIN co-rotating twin screw Kneader ZK-25". The extrudate was cooled in a water bath at 25°C and fed to a "PLANETROL 075D2" pelletizer to cut into pellet form. The samples were collected for testing on processability and mechanical properties.

The specimens for viscosity measurement and mechanical property testing were prepared by a compression molding technique. The granules of HDPE and HDPE blends was heated up to 190°C for 7 min, compressed at a pressure of 160 kg/cm^2 for 5 min, and then cooled down with water to 25°C for 10 min. The thickness of the mold is 3 mm for mechanical testing and 1mm for viscosity measurement. Finally, the HDPE compressed sheet was cut into a standard size for each test.

Testing

The molecular weight of HDPE samples was determined using a "Water HT-GPC150C" instrument at 135°C using 1,2,4-trichlorobenzene as a solvent. The melting temperature and the degree of crystallinity of the HDPE samples was a determined by differential scanning calorimeter "NETZSCH DSC 2000".

The melt flow index was measured by using a "KAYNESS GALAXY I" extrusion plastometer with a piston load weight of 2.16 kg at 190°C. The test conditions were set according to ASTM D1238.

The viscosity of samples was measured by using a "HAAKE RHEOMETER RS 75". The tests were performed in dynamic mode using a parallel-plate geometric head under a frequency range between 100 to 0.01 Hz at 190°C. All measurements were carried out in the linear viscoelastic zone, which was at an applied stress of 1000 Pa.

Tensile testing was measured using a "HOUNSFIELD" universal testing machine following ASTM D638. The dumbbell shaped samples (type II) were used.

Izod impact testing was carried out according to ASTM D256 on a "CEAST" pendulum impact tester.

Flexural testing was measured by using a "HOUNSFIELD" universal testing machine. The test conditions follow ASTM D790.

The Rockwell hardness of samples was determined by using the "MATSUZAWA" following ASTM D785.

Results and Discussion

The properties of post-used HDPE crates at various utilization times

Characterization of HDPE

Table 2 presents the results on weight average molecular weight (M_w), number average molecular weight (M_n), molecular weight distribution (MWD), melting temperature and the degree of crystallinity of virgin HDPE and HDPE crates at various utilization times. The M_w of new HDPE crates is higher than that of a virgin HDPE granule by 2.3% while the M_n of new HDPE crates is lower than that of a virgin HDPE granule by 14.2%. It can be seen that a slight increase in M_w and a decrease in M_n, resulted in a broadening in the MWD. The result corresponds to the work on HDPE from milk bottles compared to virgin HDPE by Pattanakul, *et al.* (1991). It was reported that after the molding process, a slight decrease in the M_n and an increase in the M_w had occurred, resulting in a broadening in the molecular weight distribution. They have suggested that a chain scission and crosslinking mechanism probably has occurred during the processing. Further study by Epacher, *et al.* (1999) on the effects of chemical reactions during the processing of stabilized polyethylene found that chemical reactions of the polyethylene can proceed in two main directions namely chain scission and crosslinking. The ratio of the two reactions depends on the amount of oxygen and amount of stabilizer added. In the absence of excessive oxygen, polyethylene has a strong tendency to crosslink.

Table 2 Characteristics of virgin HDPE and HDPE crates at various periods of utilization time

Code	M_w	M_n	MWD	Melting Temperature (^oC)	Degree of Crystallinity (%)
VIRGIN	204,504	48,684	4.2	134	77
HDPE#0	209,245	41,744	5.0	132	67
HDPE#3	208,445	35,996	5.8	135	61
HDPE#8	208,451	37,152	5.6	134	63

Considering at various utilization times, the M_w of HDPE crates at various utilization times, more or less, remains constant, but the M_n of HDPE crates at the utilization time of 3 and 8 years is lower than that of new crates by 13.8% and 11%, respectively. Therefore, the MWD of HDPE crates at the utilization time of 3 and 8 years is broader than that of new HDPE crates by 16% and 12%, respectively. The broadening in the MWD can be noticed in the molecular weight distribution curve shown in Figure 1. This indicates that the utilization time of HDPE crates has an effect on the molecular weight of HDPE crates, in particular M_n. There is a change that occurs in polymeric molecules during the use of the crates for bottle transportation in a hot and humid weathering exposure, leading to changes in molecular weight. A change in HDPE molecules could be due to the degradation process of HDPE which is a result of competing reactions of crosslinking and chain scission that lead to molecular weight changes (Hinsken, *et al.* 1991). Other reports related to the molecular weight and degradation process are the work of Jabarin and Lofgren (1994) who found that weathering exposure time has an extreme effect on the decrease in M_n and M_w of HDPE film as a result of photo-oxidative degradation. Valadez-Gonzalez, *et al.* (1999) reported that, during the first 300 hours of HDPE exposure in an accelerated weathering chamber, the viscosity average molecular weight (M_v) decreases slightly and after that it drops abruptly in the irradiation time interval between 300 and 800 hours. It is evident from the results that there is chain scission reaction during the degradation of the material.

From the DCS results on the melting temperature and the degree of crystallinity reported in Table 2, the melting temperatures of the HDPE crates varies in a very narrow range of 132–135°C indicating no change in the melting temperature of HDPE crates during the utilization time. The degree of crystallinity of virgin HDPE granules is higher than that of HDPE crates. The degree of crystallinity of HDPE crates at the utilization time of 3 and 8 years is lower than that of new crates by 9% and 6%, respectively. A decrease in the degree of crystallinity implies that the crystallized region or the ordered structure in the HDPE chains has decreased.

Processability

The melt flow index of virgin HDPE and HDPE crates at various utilization times shown in Figure 2 indicates that the melt flow index of new HDPE crates is higher than that of virgin HDPE granules by 6.4%. This could be the result from several additives added during the injection process. It can also be seen that the melt flow index of HDPE crates is decreased after a period of utilization time. HDPE crates after 3 and 8 years of utilization show a decrease in melt flow index by 15.7% and 14.1%, respectively, in comparison with that of new HDPE crates. This behavior could possibly be related to changes in the polymer chain structure since HDPE crates were exposed to a very hostile environment such as sunlight, heat, physical stress and impact during use for bottle transportation. A drop in melt flow index could be the due to photo-oxidative degradation that might occur during the service life.

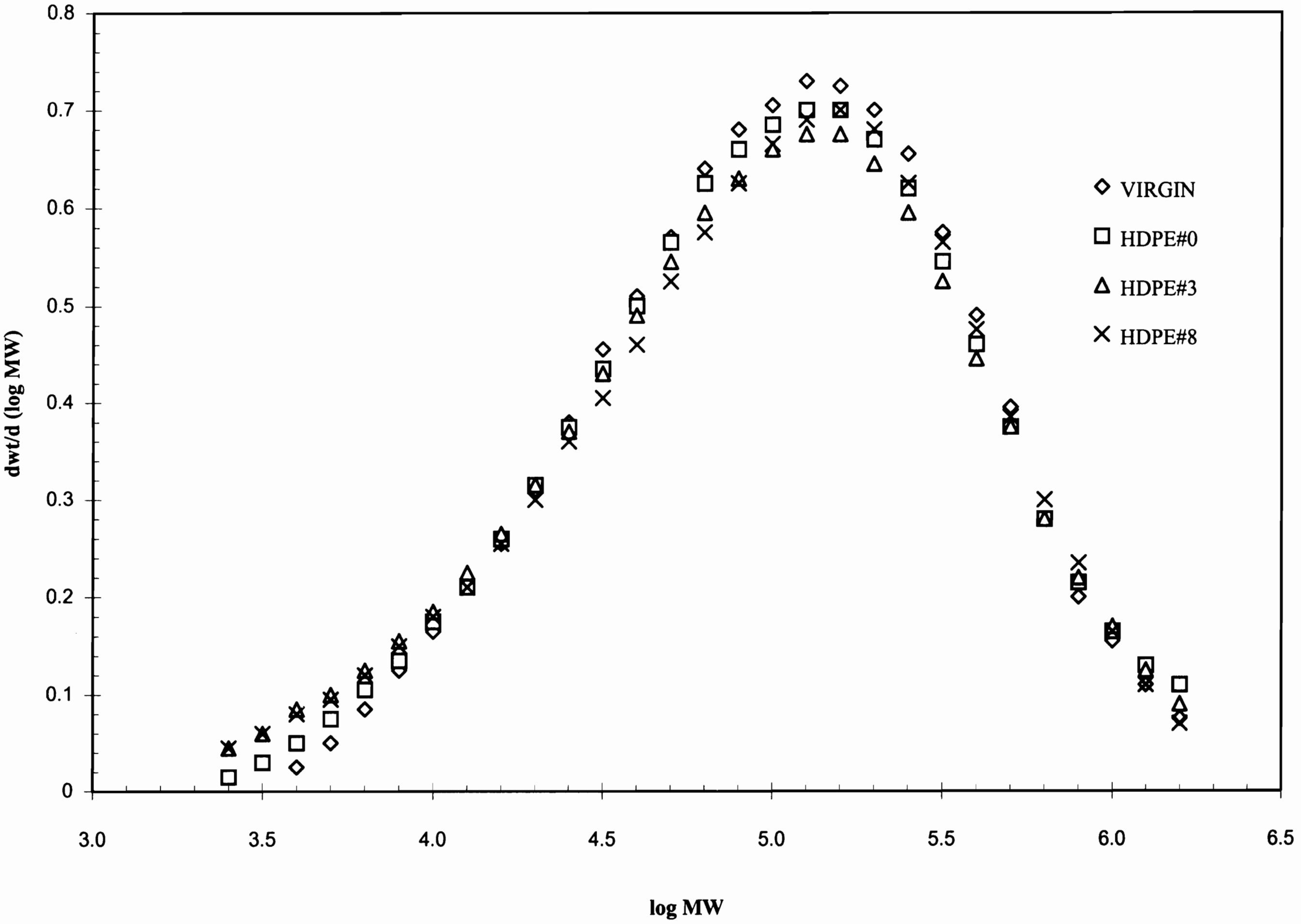

Figure 1 Molecular weight distribution curves of virgin HDPE and HDPE crates at various utilization times.

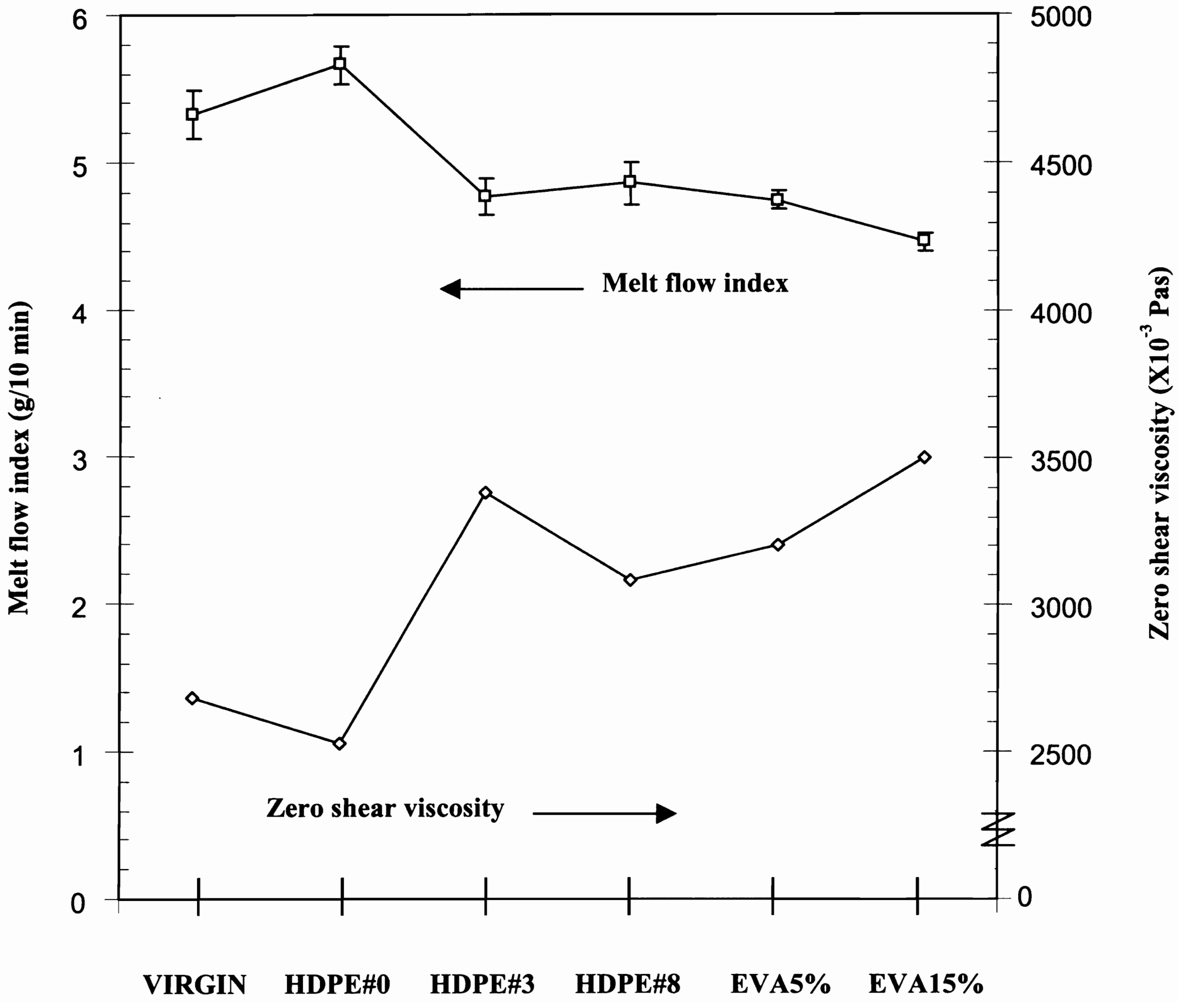

Figure 2 Melt flow index and zero shear viscosity of blended post-used HDPE crates with EVA compared to virgin HDPE and HDPE crates at various utilization times.

The results on the zero shear viscosity of the polymers which represents the flow ability in the melted state in Figure 2 show that the zero shear viscosity of new HDPE crates is lower than that of virgin granules by 5.6% and the zero shear viscosity of used HDPE crates increases compared to that of new crates. The zero shear viscosity of HDPE crates at the utilization time of 3 and 8 years increases by 33.6% and 21.7% from that of the new HDPE crates, respectively. Compared with the results on melt flow index, an increase in the viscosity of used HDPE crates is in agreement with a decrease in the melt flow index of used HDPE crates. This results from changes in polymer chain structure from photo-oxidative degradation as discussed earlier. Chain scission, chain branching and crosslinking would occur during the degradation process. Theoretically, an increase in M_w results in an increase in zero shear viscosity. Unfortunately, our results on viscosity did not agree with the theory possibly due to changes in chain structure.

Mechanical properties

Figure 3 shows tensile strength at yield of virgin HDPE and HDPE crates at various utilization times. The results show that the tensile strength at yield of virgin HDPE is approximately the same as those of new and used HDPE crates. In Figure 4, it can be noticed for HDPE crates that there is a decrease in elongation at break after a period of utilization time. The elongation at break of HDPE crates at the utilization time of 3 and 8 years is decreased by 49.7% and 36.7% from that of new HDPE crates, respectively. These results clearly indicate that the elongation at break is significantly decreased after use, which could be caused by outdoor exposure. The result is in agreement with the work of Jabarin and Lofgren (1994) who have studied the photo-oxidative degradation effects on the properties and structure of HDPE and found that weathering exposure time has an extreme effect on a decrease in the elongation at break of HDPE. Their results indicated that HDPE embrittlement was influenced by chain scission.

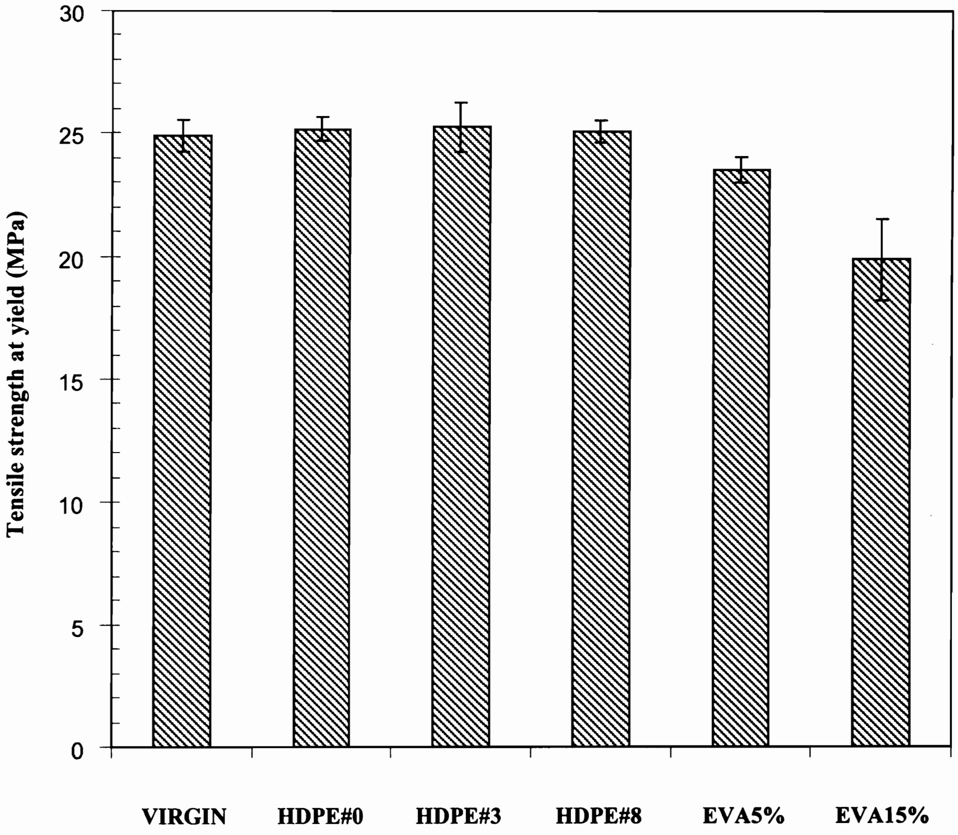

Figure 3 Tensile strength at yield of blended post-used HDPE crates with EVA compared to virgin HDPE and HDPE crates at various utilization times.

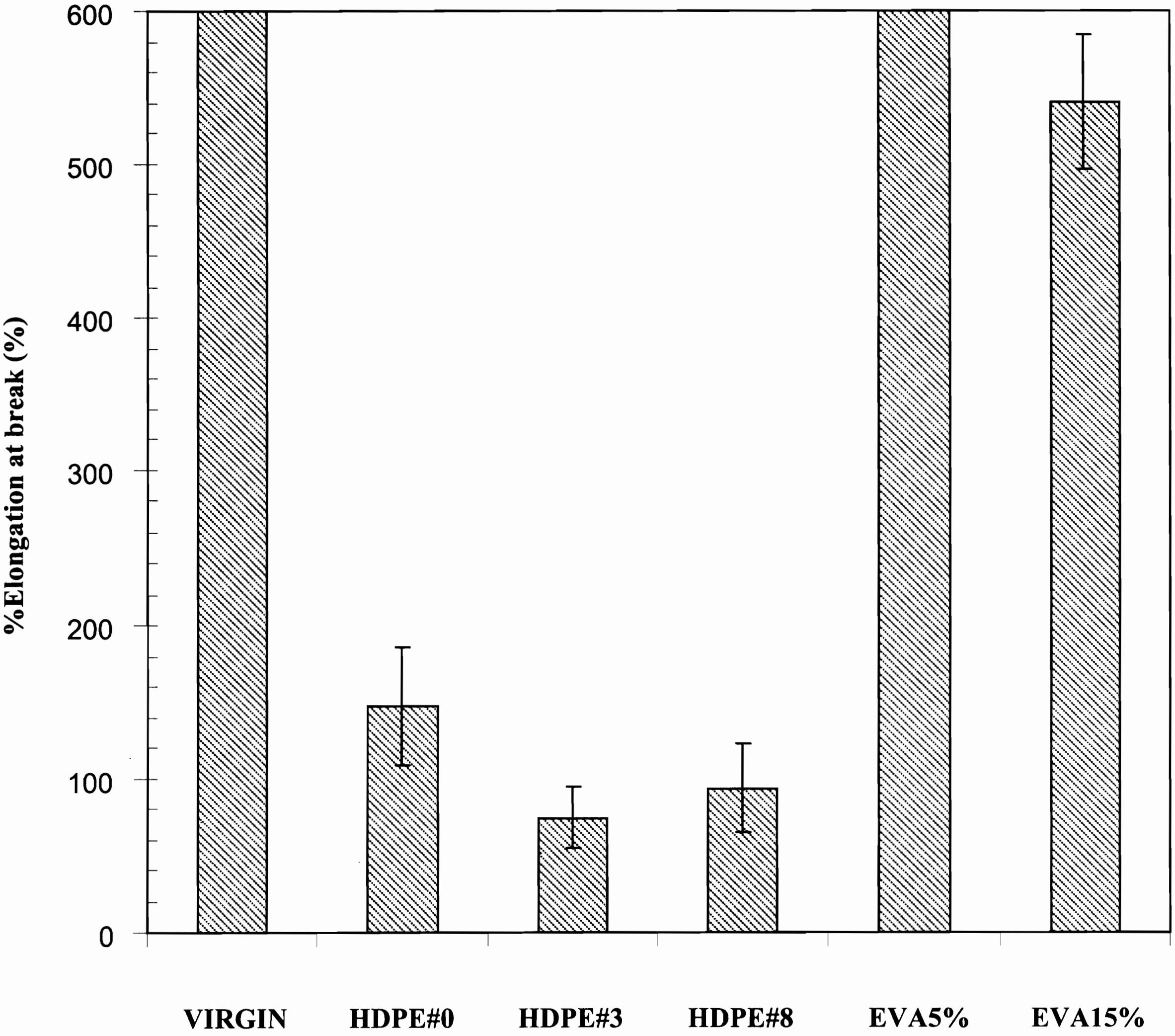

Figure 4 Elongation at break of blended post-used HDPE crates with EVA compared to virgin HDPE and HDPE crates at various utilization times.

Figure 5 shows stress-strain curves of virgin HDPE and HDPE crates at various utilization times. After the yield point, the high tensile strength at yield of HDPE crates sharply decreases because some parts of the samples broke but some still extended. As a result, the tensile strength at break is low. It may be because the irregular changes of properties in different parts of the crates occurred during use and/or scrap HDPE crates were not thoroughly mixed during the processing. In addition, the area under the stress-strain curve which is proportional to the energy required to break for HDPE crates decreases for 3- and 8-years old HDPE crates compared to the new HDPE crates. This implies that the new HDPE crates are tougher than the used HDPE crates.

Figure 6 shows the impact resistance of virgin HDPE and HDPE crates at various utilization times. The impact resistance of virgin HDPE is higher than that of new HDPE crates by 8.3% while the impact resistance of HDPE crates at a utilization time of 3 and 8 years is lower than that of new HDPE crates by 17% and 25%, respectively. The utilization time of HDPE crates has an effect of lowering the impact resistance of HDPE. This is in agreement with the results on the degree of crystallinity that the loss of crystallized regions of post-used HDPE crates were responsible for the decrease of the impact strength. Our results are similar to the previous work by Pages, *et al.* (1996) on weathering aging of HDPE. The article has demonstrated that a long exposure time in the Canadian winter has caused specific chemical transformations in the polymer chain. These reactive phenomena decrease the linear character of the polymer chains caused by the formation of bulky groups, which leads to an increase of the amorphous content and a decrease in the impact resistance.

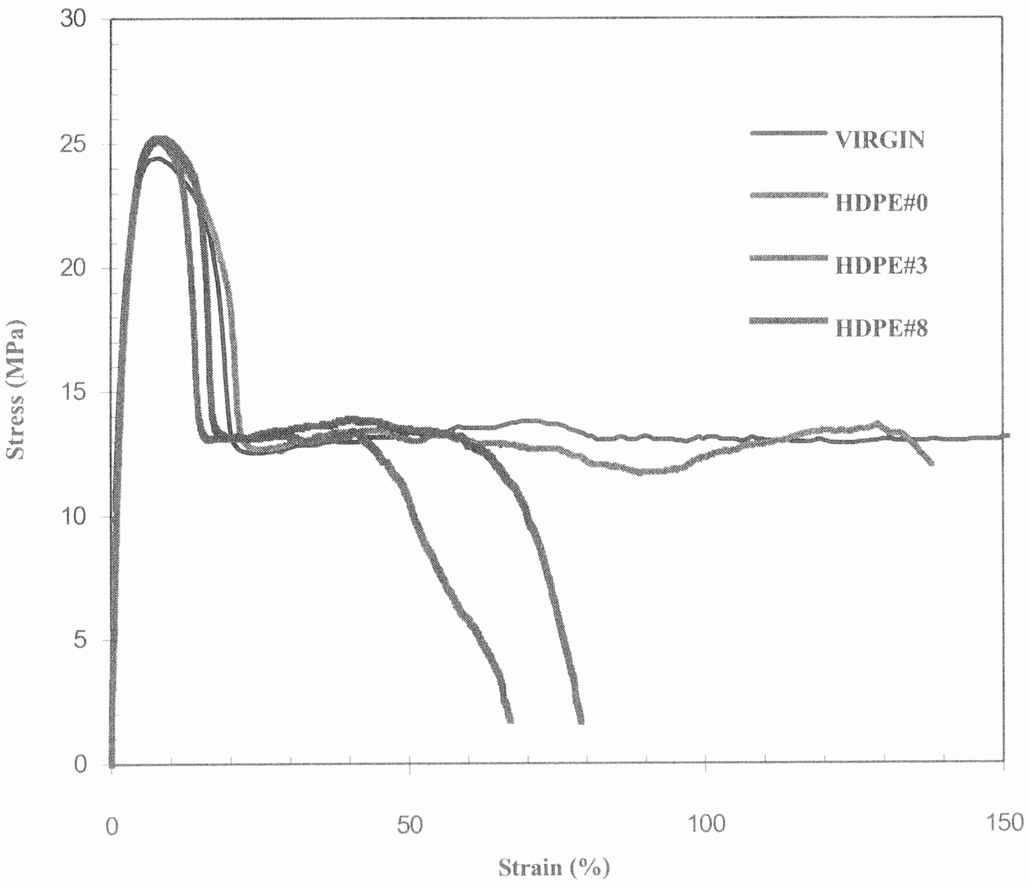

Figure 5 Stress-stain curves of virgin HDPE and HDPE crates at various utilization times.

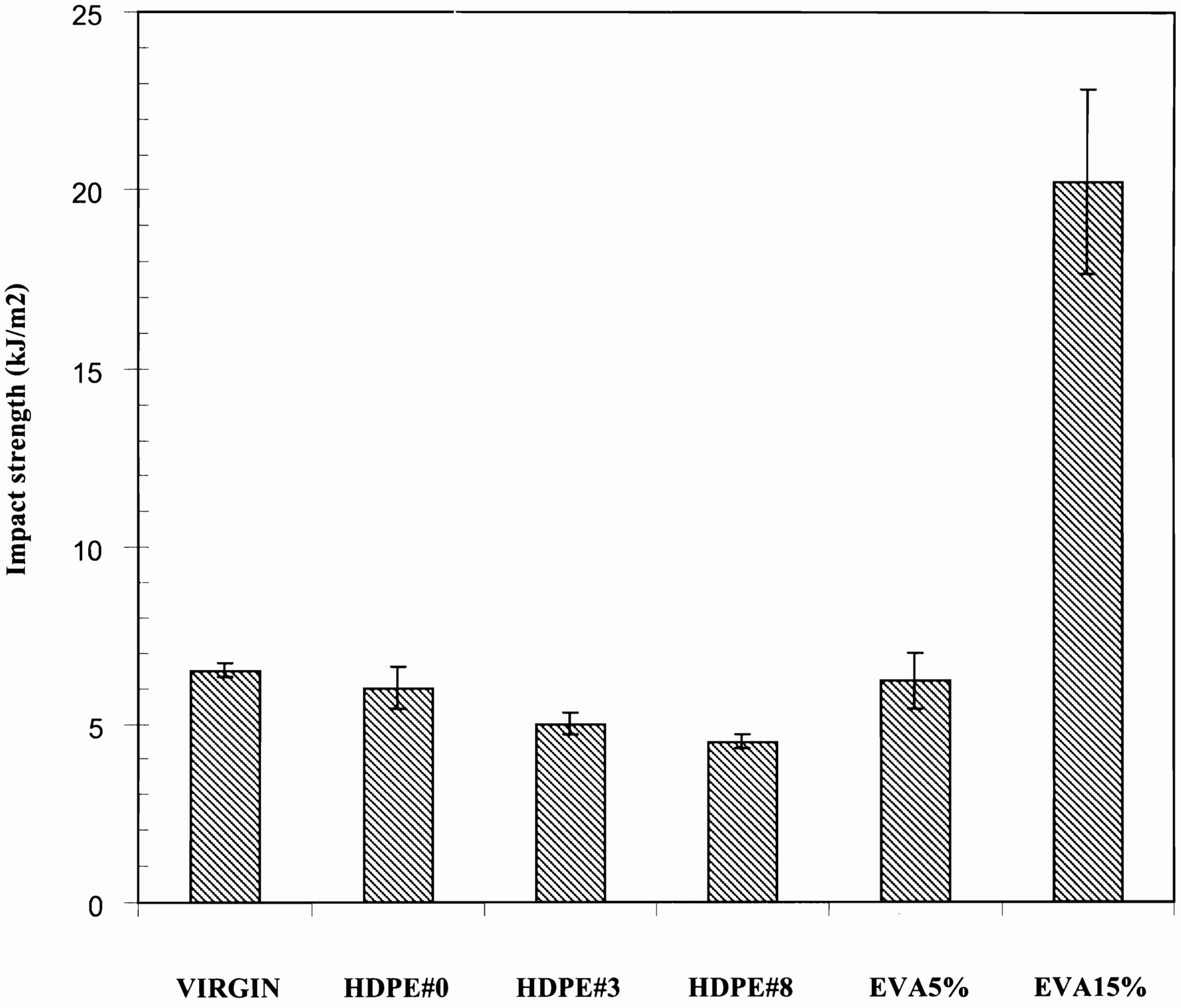

Figure 6 Impact strength of blended post-used HDPE crates with EVA compared to virgin HDPE and HDPE crates at various utilization times.

In Figure 7, the flexural strength at yield of HDPE shows a similar trend as observed in the impact strength. Figure 8 shows the Rockwell hardness of virgin HDPE and HDPE crates at various utilization times. The Rockwell hardness of new HDPE crates is higher than that of virgin HDPE by 3.9%, this could be due to many additives added during the processing. The Rockwell hardness of HDPE crates does not significantly vary along the utilization time.

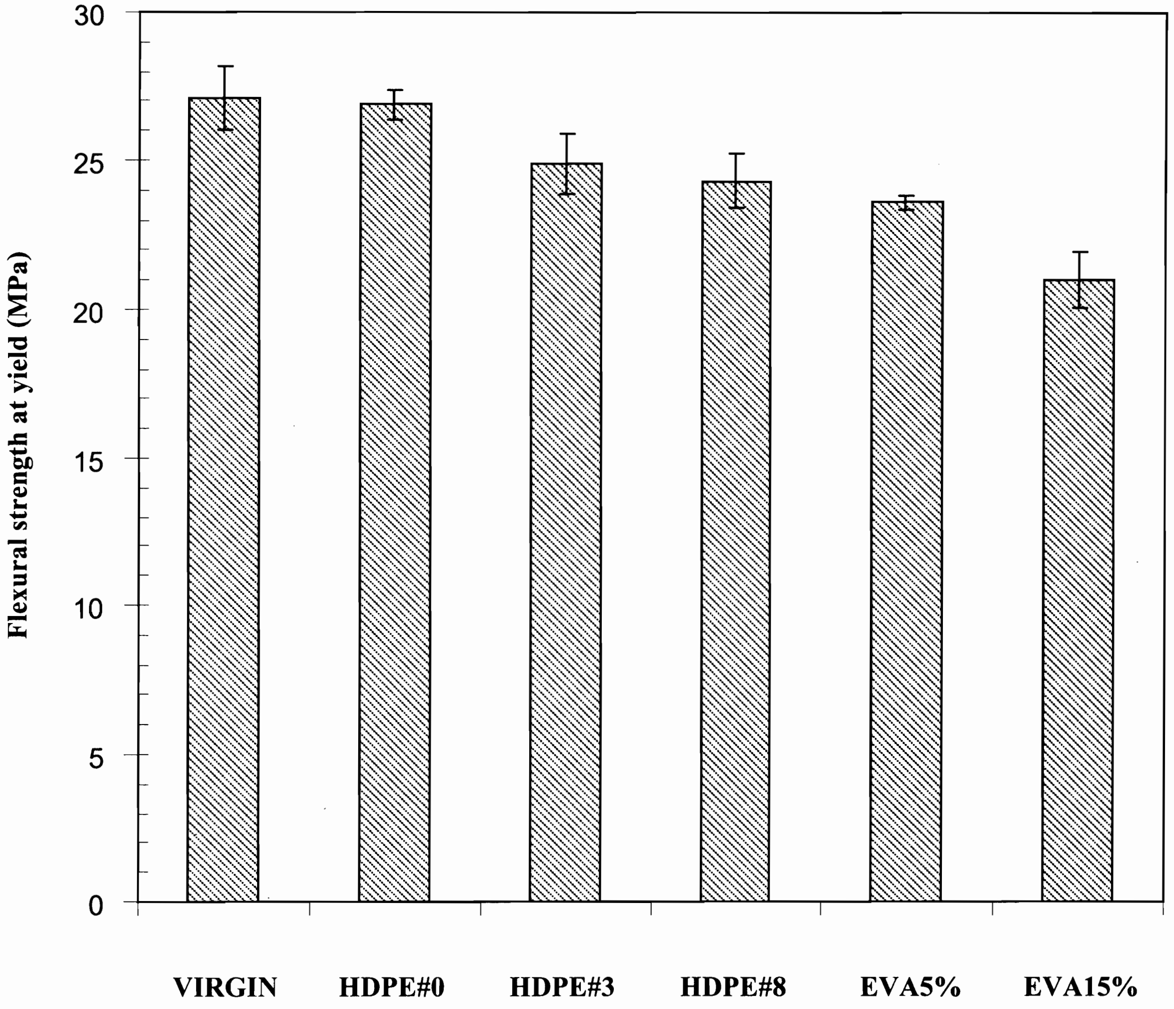

Figure 7 Flexural strength at yield of blended post-used HDPE crates with EVA compared to virgin HDPE and HDPE crates at various utilization times.

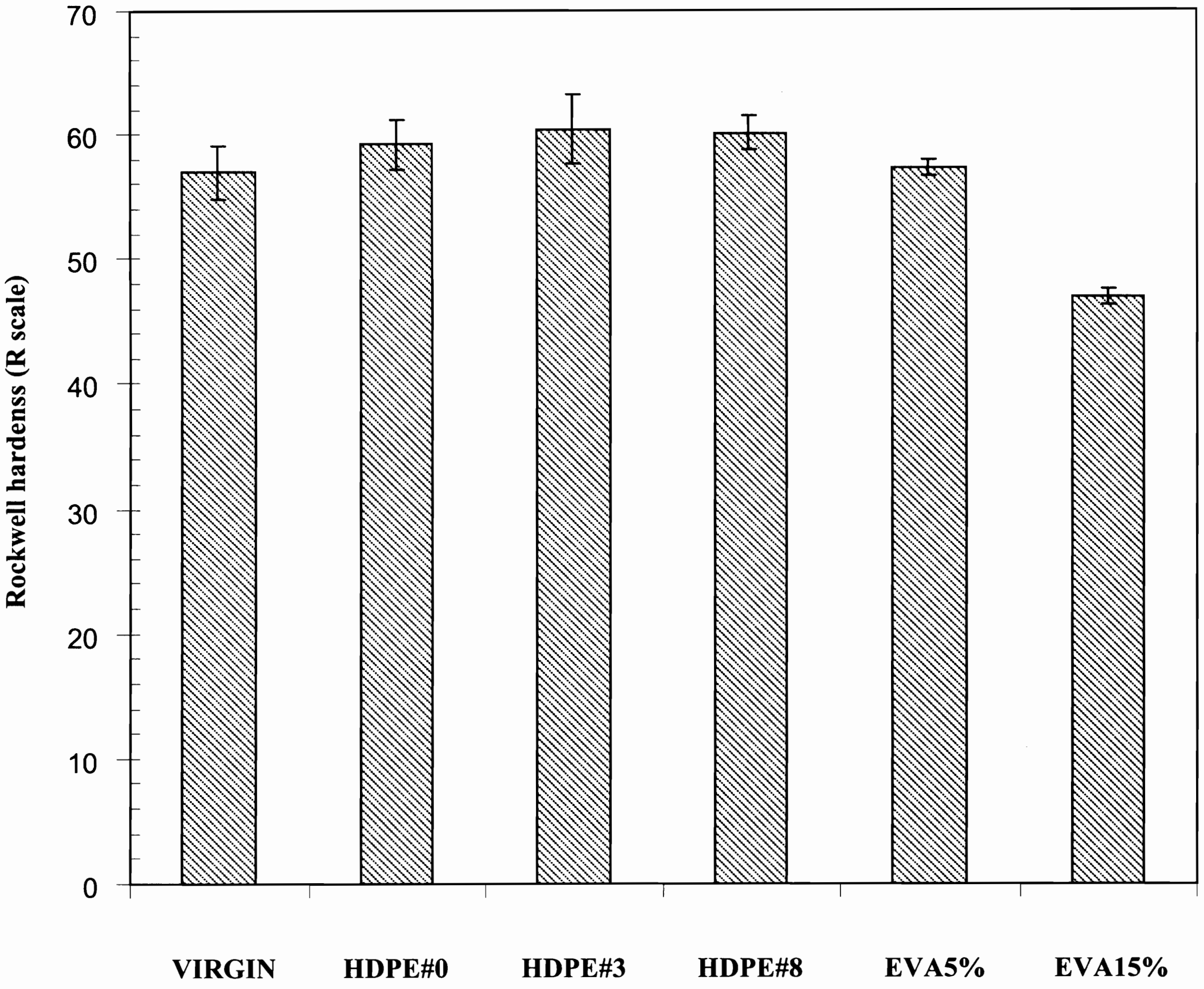

Figure 8 Rockwell hardness of blended post-used HDPE crates with EVA compared to virgin HDPE and HDPE crates at various utilization times.

The properties of blended post-used HDPE with EVA

Processability

For blends of post-used HDPE crates with EVA, it can be noticed from Figure 2 that post-used HDPE crates after blending with 5% and 15% EVA have lower melt flow indexes than that of post-used HDPE crates by 2.3% and 8% respectively. EVA, by a significant amount, makes the melt flow index remarkably decrease due to the low melt flow index of EVA (2.3 g/10 min). Comparing the zero shear viscosity of the blends of post-used HDPE crates and EVA with the unblended sample, it is shown that the zero shear viscosity of post-used HDPE crates is increased by increasing the concentration of EVA. The viscosity of the blends of post-used HDPE crates and EVA at an EVA concentration of 5% and 15% is higher than that of post-used HDPE crates by 4% and 13.6%, respectively. The results indicate that EVA has a remarkable effect on the processability of the post-used HDPE crates. The results on viscosity of the blends of post-used HDPE crates and EVA are in agreement with the melt flow index results.

Mechanical Properties

In Figure 3, it can be seen that the tensile strength at yield of post-used HDPE crates after blending with EVA at the EVA concentration of 5% and 15% is lower than that of post-used HDPE crates by 6.2% and 20.7%, respectively. The results indicate that the tensile strength at yield of post-used HDPE crates is slightly decreased at an EVA concentration of 5% and continues to be rapidly decreased when increasing the amount of EVA content in the blend. This behavior could be due to the softness of EVA compared to HDPE and the good dispersion of EVA in the HDPE phase.

Comparing the blends of HDPE#8 and EVA with the unblended sample in Figure 4, the blend of HDPE#8/EVA at the weight ratio of 95/5 (EVA5%) can be elongated more than 600%. The ability on the elongation at break of EVA5% exceeds the detection limit of the testing machine at the crosshead speed of 50 mm/min, similar to that found in the case of virgin HDPE granule. This point will be further clarified. For the blend of HDPE#8/EVA at 85/15 (EVA15%), the elongation at break of EVA15% is higher than that of HDPE#8 by 480%. It could be suggested that the addition of EVA remarkably improves the elasticity of HDPE.

Since the EVA5% and virgin HDPE granules can be elongated more than 600%, at this point, the tensile properties of both samples are rechecked at a higher crosshead speed of 100 mm/min (Previous test was done at the crosshead speed of 50 mm/min). The results on tensile properties of virgin HDPE and EVA5% at a crosshead speed of 50 mm/min and 100 mm/min are shown in Table 3. Increasing strain rate from 50 mm/min to 100 mm/min, Young's modulus increases with a corresponding decrease in elongation at break. At the speed of 100 mm/min, it is clearly noticed that the elongation at break of EVA5% is lower than that of virgin by HDPE 26%.

Table 3 Tensile properties of virgin HDPE and a blend of post-used HDPE crates with 5% EVA

Code	Young's Modulus (Mpa)	Tensile Strength at Yield (MPa)	% Elongation at Break (%)
Crosshead speed 50 mm/min			
VIRGIN	1474	24.91	>600
EVA5%	1389	23.52	>600
Crosshead speed 100 mm/min			
VIRGIN	2140	24.98	124
EVA5%	2298	23.16	91

The tensile stress–strain curves of recycled post-used HDPE crates with EVA are shown in Figure 9. It can be seen that the toughness of blended post-used HDPE crates with EVA is strongly improved. Adding EVA can dramatically reduce tensile strength at yield. This indicates that blends of post-used HDPE crates and EVA are softer and much tougher than post-used HDPE crates. The tensile strength at break of blended post-used HDPE crates with EVA is also higher than that of post-used HDPE crates. Especially, the blend of post-used HDPE crates with 5% of EVA generally gives the much improved tensile properties comparable to virgin HDPE granules. In addition, adding EVA might affect the crystal characteristics of the HDPE structure which could be another factor for changes in tensile properties. This can be further proved by DSC.

In Figure 6, it can be noticed that the impact resistance of post-used HDPE crates is rapidly increased with an increase in the concentration of EVA. The impact resistance of EVA5% and EVA15% is higher than that of HDPE#8 by 37.8% and 348.9% respectively. The impact resistance of post-used HDPE crates is effectively improved after blending with EVA at the concentration of 15% comparing with post-used HDPE crates. This could imply a good compatibility of the blends of EVA and HDPE thus giving a much better impact resistance.

In Figure 7, it can be noticed that the flexural strength at yield of post-used HDPE crates is decreased by 3% and 13% when adding EVA at a concentration of 5% and 15%, respectively. The same trend is also observed in the Rockwell hardness of blended post-used HDPE crates with

EVA in Figure 8. Generally, loss of crystallinity is a factor causing a decrease in hardness. As the degree of crystallinity of EVA is lower than that of HDPE, the compatibility in the blend could result in a decrease in the Rockwell hardness and the flexural strength.

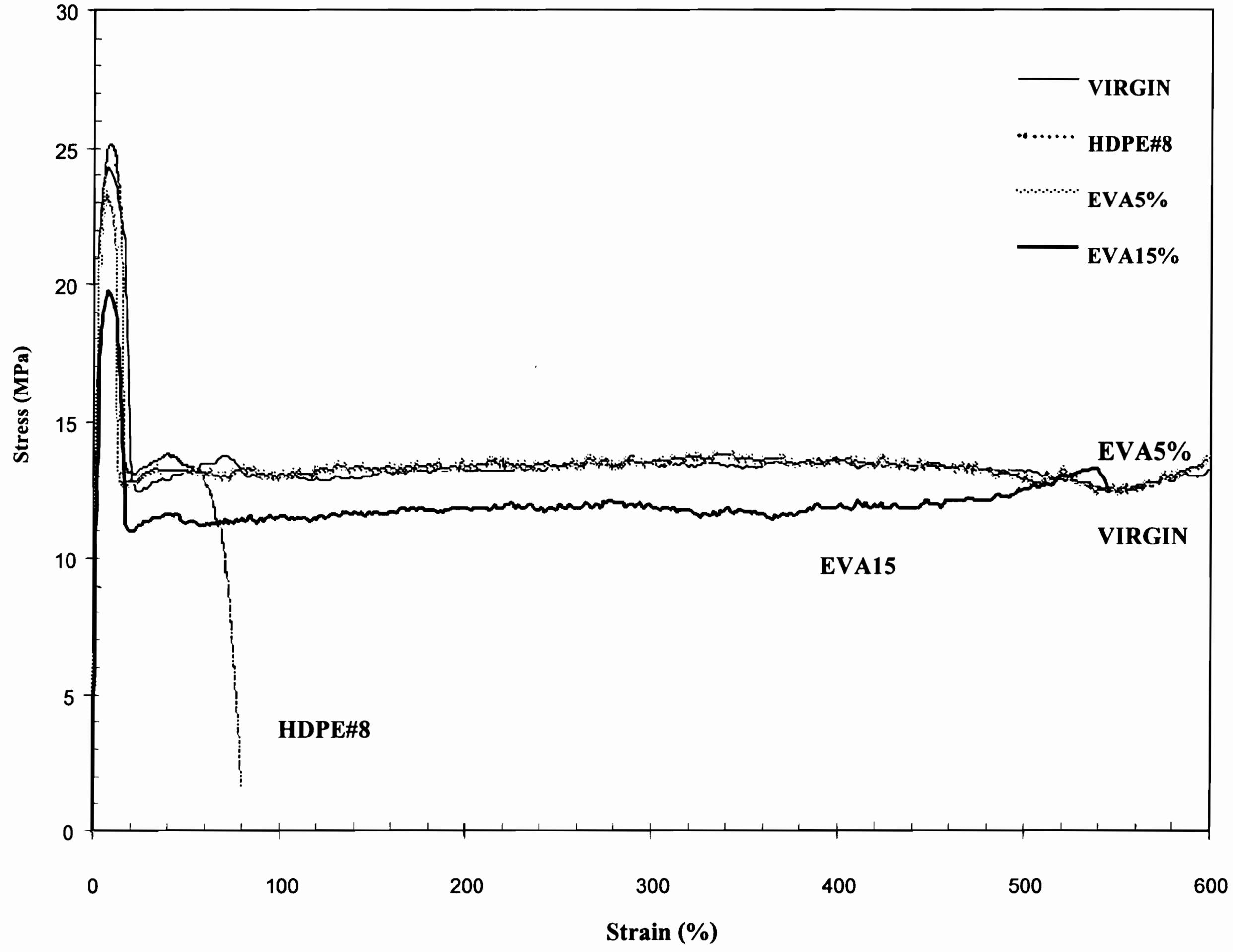

Figure 9 Stress-strain curves of blended post-used HDPE crates with EVA compared to virgin HDPE and post-used HDPE crates.

Conclusions

The characteristics and the properties of HDPE crates have been changed after a period of utilization time in such a hot and humid environment, possibly due to the photo-oxidative degradation that occurred. An increase in the period of utilization time tends to decrease the processability of HDPE crates and dramatically lowers the elongation at break, impact resistance, and flexural strength at yield of HDPE crates. The mechanical properties of post-used HDPE crates at a utilization time of 8 years are generally worse than those of new HDPE crates, particularly the elongation at break and the impact resistance. However, the variable factors that cannot be controlled in the experiment are the conditions of service of the HDPE crates. Some crates might be exposed to the sunlight, some were heavily pressed and some were crashed for different periods of time. For these reasons, the results of the experiment exhibit a wide variety.

The addition of EVA at a concentration of 5% and 15% in post-used HDPE crates has an effect on processability by increasing the zero shear viscosity and lowering the melt flow index of post-used HDPE crates. The elongation at break and the impact resistance of the blends of post-used HDPE crates and EVA are remarkably increased by more than 300%. Particularly, the blend of post-used HDPE crates with 5% EVA gives the most improved elongation properties comparable to the virgin HDPE. This could imply that EVA continually disperses in the HDPE phase giving a good compatibility with HDPE and can effectively improve the elasticity of HDPE. This can be further proven by Scanning Electron Microscopy.

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