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# HDPE/ROSIN blends: I-Morphology, mechanical and rheological properties

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## SUMMARY

Melting mixtures of high density polyethylene (HDPE) and glycerol ester of hydrogenated rosin (ester gum) were prepared under select conditions using up to 50% of rosin. All blends showed phase separation. The size of rosin domains varied with the content of low molecular weight constituent. The 50/50 blend showed the highest dispersion and the presence of micro and macro rosin domains. Up to 20% rosin a slight decrease of Young's modulus was noticed while above this rosin content a slight increase occurred. The stress and elongation dropped markedly above 10% of rosin. The hardness of blends showed the same behavior as the elastic modulus. The melt flow index increased exponentially with the rosin content while processability has improved.

## INTRODUCTION

The effect of commercial low molecular weight resins, from mineral or vegetable source, on blends is scarcely explored in the literature. Only the isotactic polypropylene (iPP) / oligo(cyclopentadiene) (HOCP) system has been investigated in some detail; this mixture is a commercial product with good permeability and sealing characteristics (1-5). Recently, the morphology, mechanical and thermal behavior of high density polyethylene (HDPE) / HOCP blends was studied (6-7). This system was partially miscible and an increase of elastic modulus was detected. Also, the effect of HOCP on the morphology of iPP / HDPE binary blends was investigated (8). The purpose of this paper is to show the morphology, mechanical and rheological properties of HDPE / ROSIN (ester) blends prepared by melt mixing. All blends showed phase separation and the 90/10 blend displayed better compatibility than the others.

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## EXPERIMENTAL

### *Materials*

Commercial High Density Polyethylene (HDPE) produced by Ipiranga Petroquímica S.A., Brazil, $M_w = 22 \times 10^4$, $M_n = 1.6 \times 10^4$ (by GPC), density = 0.95 $g/cm^3$ (by liquid immersion), degree of crystallinity = 40% (by DSC), degree of branching = 5 methyl groups/1000 carbon atoms, hardness (9) = 50 (Shore D), melt flow index (MFI) (10) = 0,16 g/10min.

Commercial glycerol ester of partially hydrogenated rosin (ester gum) was by Hercules Inc., Brazil; $M_w = 1190$, $M_n = 920$ (by GPC), density = 1.1 $g/cm^3$ (by liquid immersion), color WG, acid number (11) = 8.7, softening point (12) = 101.5$^{o}$C.

### *Blend preparation*

The blends were prepared by melt mixing in a Haake Rheocord 9000 at 210$^{o}$C, 32 rpm for 10 minutes. The following HDPE/ROSIN w/w ratios were blended: 100/0, 90/10, 80/20, 70/30, 60/40 and 50/50.

### *Specimen Preparation*

Sheets of 9x7x0.1 cm were compression moulded. The sample was left for 5 minutes at 220$^{o}$C without pressure to melt completely. After this, 2.5 MPa of pressure was applied for 5 minutes and then cooled in air until to 110$^{o}$C. Finally, the mold was put in another press with water circulation to reach room temperature.

### *SEM experiment*

Scanning electron microscopy (SEM) was performed by a JEOL scanning microscope JSM 5300. The samples were fractured using liquid nitrogen. The broken area was covered with a thin layer of gold/palladium alloy and submitted for microscopic observation.

### *Mechanical test*

Tensile parameters were obtained using an Instron machine (model 4204), according to ASTM D 1708-93 (13). The cross head speed and gage length were 10 mm/min and 22.25 mm, respectively. The mechanical parameters were calculated using the average of 10 specimens.

## RESULTS AND DISCUSSION

Figure 1 shows a representative top view of fractured 100/0 HDPE/ROSIN blend. Fracture was effective due to the absence of plastic deformation; microspherulites and boundaries among these structures were observed. The photomicrograph of fractured 90/10 HDPE/ROSIN blend (Figure 2) revealed phase separation only at high magnification (x5000). The rosin domains were small and homogeneously dispersed in the HDPE matrix. When the rosin content was raised, evidence for phase separation was enhanced by SEM microfractographs of 80/20 and 50/50 HDPE/ROSIN blends (Figure 3). The photomicrograph of fractured 80/20 HDPE/ROSIN blend showed rosin domains as regular and well dispersed microspheres. The photomicrograph of fractured 50/50 HDPE/ROSIN presented rosin domains with at least two different sizes. The presence of micro and macrodomains was clearly seen. The microdomains were similar to those observed in a 80/20 HDPE/ROSIN blend. The macrodomains did not show defined shapes.

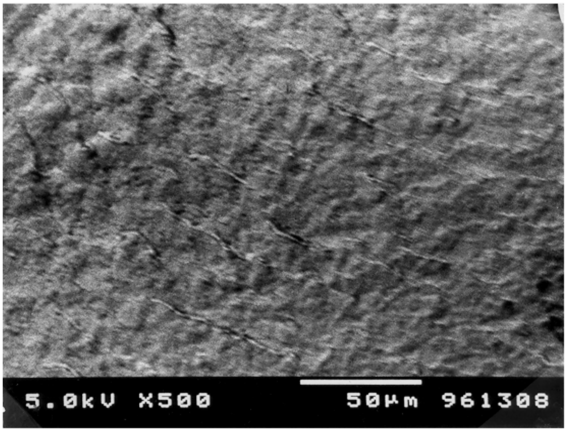



Figure 1
SEM photomicrograph of fractured 100/0 blend

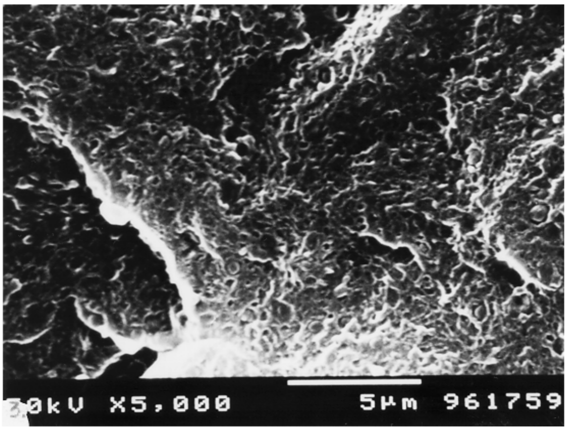
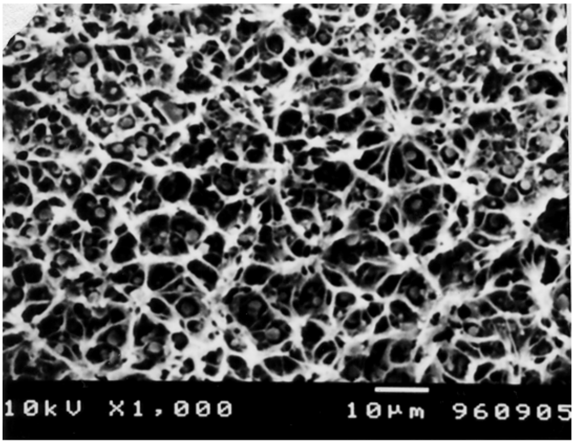
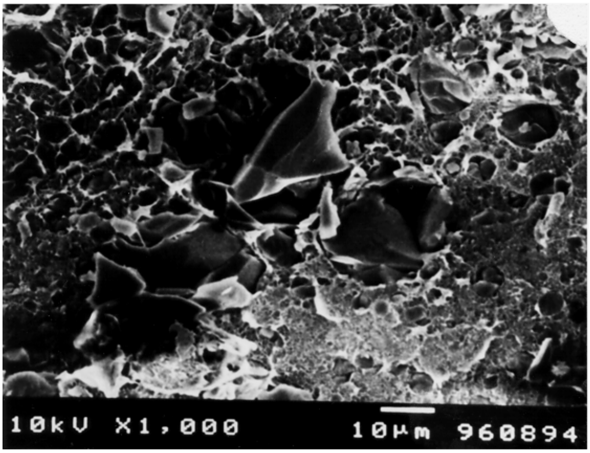



Figure 2
SEM photomicrograph of fractured 90/10 blend




80/20 HDPE/Rosin blend




50/50 HDPE/Rosin blend

Figure 3- SEM photomicrographs of fractured 80/20 and 50/50 blends

The Figures 4, 5, 6, 7 and 8 show the variation of tensile parameter with rosin content. The elastic modulus (Figure 4) presented a slight decrease up to 20% of oligomer while over 30% this property showed some increase. Up to 20% the rosin plasticized the HDPE however, above this limit it became a hardener. The yield stress (Figure 5) decreased monotonically with rosin content. For the 90/10 blend the stress was close to that of HDPE. The elongation at yield (Figure 6) decreased and reached a constant value over 30% of oligomer. The mechanical properties at break were also affected by rosin content. The variation of stress is shown in Figure 7. For the 90/10 blend this parameter was similar to that of HDPE. Over 10% of rosin the stress diminished markedly. The elongation is shown in Figure 8. For the 90/10 blend the elongation was similar to that of HDPE however it dropped abruptly over 10% of rosin. These observations are related to the morphology. The blends with over 10% rosin showed pronounced phase separation by SEM. The morphology of these blends became coarse when the oligomer content was raised in the blends which could explain the loss of mechanical characteristics.

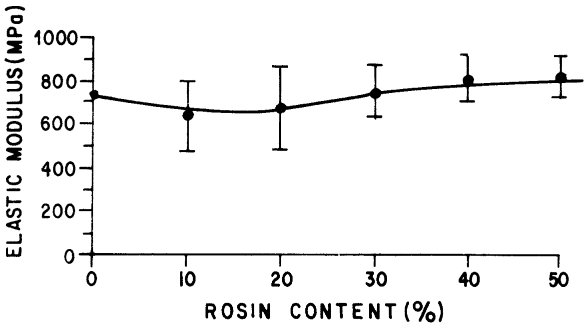



Figure 4
Elastic modulus *versus* rosin content of blends

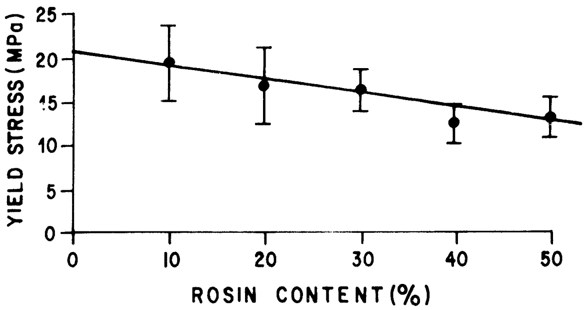



Figure 5
Yield stress *versus* rosin content of blends

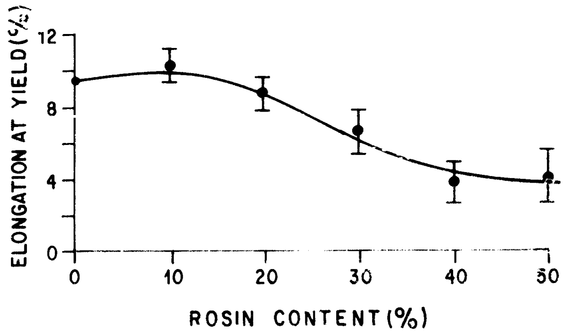



Figure 6
Elongation at yield *versus* rosin content of blends

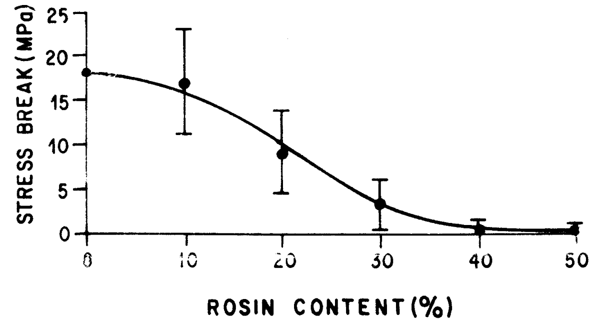



Figure 7
Stress at break *versus* rosin content of blends

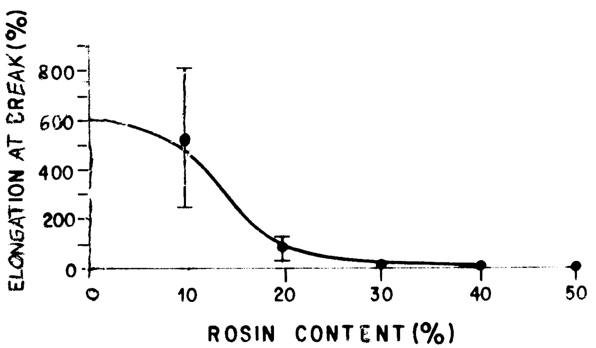



Figure 8
Elongation at break *versus* rosin content of blends

The hardness of blends (Figure 9) was similar to that of elastic modulus. Up to 20% of rosin a slight decrease is observed while over this content a increase occurred. The melt flow index of blends (Figure 10) changed exponentially with the rosin content. The variation of torque with rosin content (Figure 11) showed a decrease. The presence of rosin improved the processability of the polyolefin.

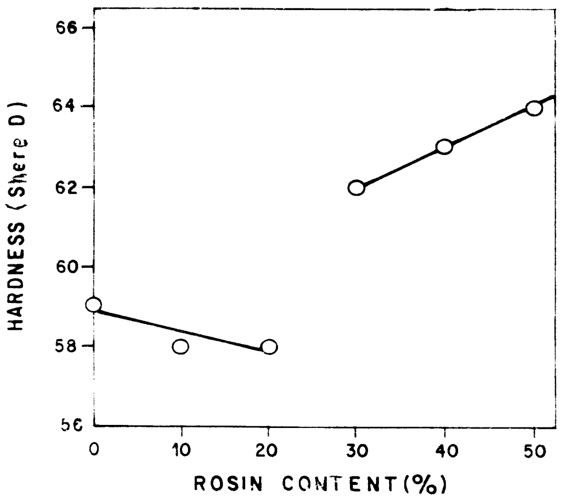



Figure 9
Hardness *versus* rosin content of blends

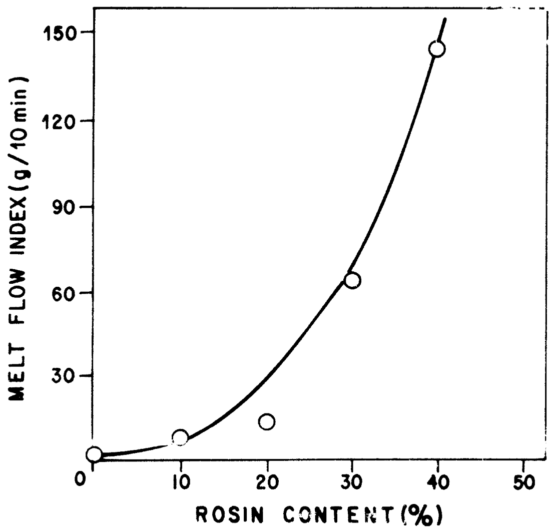



Figure 10
MFI *versus* rosin content of blends

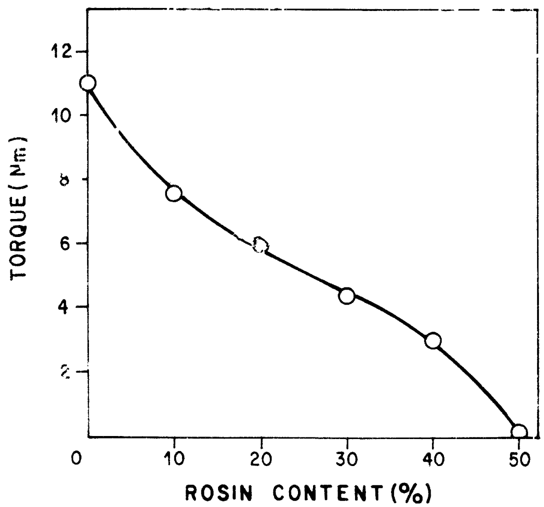



Figure 11
Torque *versus* rosin content of blends

**CONCLUSION**

The HDPE/ROSIN blends exhibit phase separation. The morphology of blends becomes coarse with the increase of rosin content. The blend with 10% of oligomer presented mechanical properties similar to that of HDPE. Above this rosin content the stress and elongation decreased. The blend hardness showed behavior similar to that of elastic modulus. The blend melt flow index changed exponentially with the rosin content and improved the processability of HDPE.

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