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# Influence of Devulcanization and Revulcanization of Ground Tire Rubber in Dynamic Mechanical Properties of Blends Ground Tire Rubber/High Density Polyethylene

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## INTRODUCTION

In no other phase of human development was produced such amount of waste as currently. Its composition and quantity are directly related to the way the population lives, socioeconomic condition and the ease of access to consumer goods. The irregular disposition of such waste may cause harmful environmental impacts. One of the most dangerous solid wastes is the vulcanized rubber, which besides having high natural degradation time, has many chemical additives on its formulation and the possibility to store rain water when disposed in landfills, and may become breeding of vectors. So, recycling comes against this problem and devulcanization a way of recycling that restores the fluidity of the rubber. One of the applications of this devulcanized rubber is in the production of polymeric blends, like dynamically revulcanized ones.

## MATERIALS AND METHODS

### Materials

Ground waste truck tire (GTR) separated from non-elastomeric components was kindly supplied by Pirelli Pneus Ltda. As vulcanization additives, were used rubber accelerator TBBS (N-tert-Butyl-2-benzothiazole sulfenamide) and sulphur, both kindly donated by Pirelli Pneus Ltda. The TBBS is accelerant agent and sulphur the vulcanization agent. The high density polyethylene (HDPE) used was a commercial product of Braskem with the trade name IA-59, used for injection molding applications.

### Devulcanization of GTR

GTR was devulcanized in a system comprised of a conventional microwave oven adapted with a motorized stirring system with a speed control. The whole devulcanization process was done by using the maximum power of the oven, i.e., 820 W. About 65 g of the GTR sample was put into a 250 mL glass beaker with a stirring speed of 100 rpm. The time at which the material was exposed to microwaves was 5.5 min.

After the devulcanization process, all the samples were homogenized in a laboratory two roll mill and were added 1 phr of TBBS and 1 phr of sulphur, respectively. Shortly after the process, the resulting tacky rubber sheets were cut manually (dimensions of approximately 3 mm x 3 mm) for use in the production of blends in twin screw extruder.

### Production of the blends in twin screw extruder

Blends were prepared in a twin-screw extruder of type Process 11 Thermo Scientific with 2 feeders. The temperatures of the barrel zones were fixed at 180°C, except the third zone for the second feeding mode whose temperature was fixed at 120°C to facilitate the introduction of the rubber phase. The screw speed was 250 rpm.

The nomenclature adopted is type X GTR Y+ad/Z HDPE, where Y represents the time of exposure of the GTR to the microwaves, '+ad' the presence of vulcanization additives in the sample, X and Z the amount of GTR and HDPE in the sample (in mass), respectively.

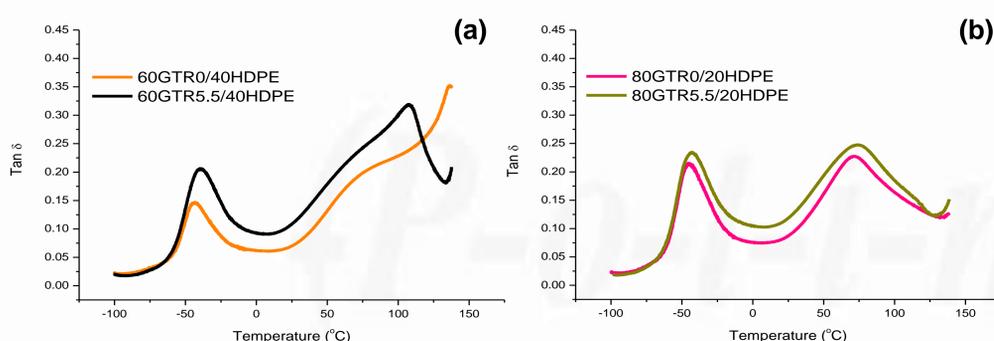
### DMA

The dynamic-mechanical properties of polymer blends were analyzed by using a dynamic-mechanical analyzer DMA Q800 of TA Instruments. The analyses were carried out in Single Cantilever mode, 1 Hz of frequency, in heating ramp from -100 to 140°C and heating rate of 3°C/min. Glass transition temperatures of the rubbers were extracted from the maximum value of the tan  $\delta$  peak. The samples were previously pressed in a hydraulic press in a rectangular mould with the dimensions of 17,5x12,5x2 mm to 180°C for 5 minutes.

## RESULTS AND DISCUSSION

### Effect of devulcanization of elastomeric phase: Nonrevulcanized blends

Tan  $\delta$  curves as function on the temperature of the blends containing GTR0 and GTR5.5 for verification of effect of GTR devulcanization on dynamic-mechanical properties of the nonrevulcanized blends are presented in Figure 1.

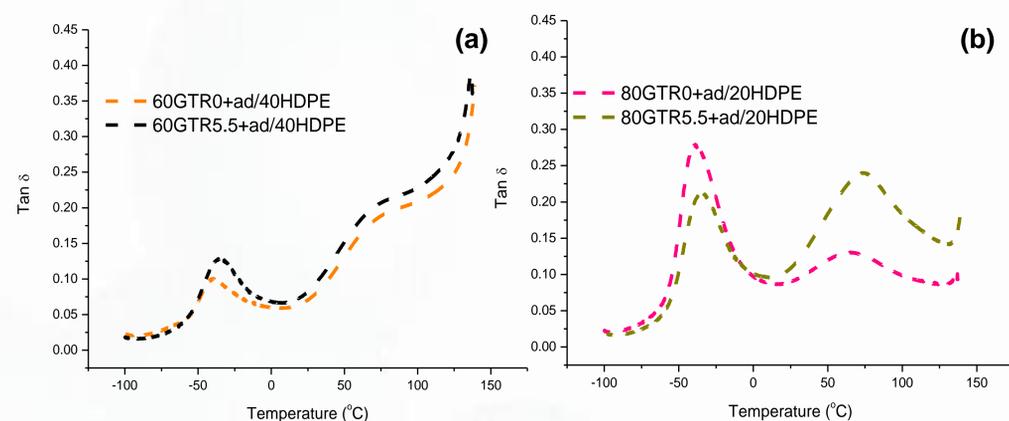


**FIGURE 1:** Tan  $\delta$  curves as function of the temperature of the blends: (a) 60GTR/40HDPE, (b) 80GTR/20HDPE, containing GTR0 and GTR5.5 for verification of the effect of devulcanization in dynamic-mechanical properties of the nonrevulcanized blends.

According to Figure 1, there are two transitions on the phases of the blends: around -40° C refers to the  $T_g$  of the GTR and the other refers to the  $\alpha$  transition of HDPE ( $T_{\alpha}$ ), which occurs around 100° C. The existence of two distinct transitions proves the character of immiscible blend. The temperature at which the transition  $\alpha$  occurs is related to the crystallinity degree and the average thickness of the crystallites. It is also related to the occurrence of rotations of small molecular sequences in the crystallites, which can be subdivided into smaller subdivisions known as pre-melting temperature. The transition refers to the relaxation of the limits of the grains, a subdivision of  $\alpha$  transition known as  $\alpha'$ .

### Effect of devulcanization and revulcanization of elastomeric phase: Revulcanized blends

Tan  $\delta$  curves as function on the temperature of the blends containing GTR0 and GTR5.5 for verification of effect of GTR devulcanization in dynamic-mechanical properties of the revulcanized blends are presented in Figure 2.



**FIGURE 2.** Tan  $\delta$  curves as function of the temperature of the blends: (a) 60GTR+ad/40HDPE, (b) 80GTR+ad/20HDPE, containing GTR0 and GTR5.5 for verification of effect of devulcanization and revulcanization in dynamic-mechanical properties of revulcanized blends.

According to the Figure 2, it was observed a reduction in the area under the  $T_g$  peak of GTR phase of blends containing GTR0 due to the restriction on the mobility of the chains by the increase of cross-linking density. Such behavior is due to the probable coexistence of two three-dimensional networks, the first from the vulcanization of the GTR and the second of dynamic revulcanization, what does not mean that it has higher cross-linking density. This is also due to the increase in the  $E'$  values of GTR0 phase, which probably hindered the breaking of the GTR0 particles during processing; resulting in larger rubber particles.

$T_g$  values of GTR phase of the analyzed blends were determined from the maximum points from the first peak of tan  $\delta$  curves as function of temperature, and they are presented in Table 1.

**TABLE 1.**  $T_g$  values of the elastomeric phase of revulcanized and non revulcanized blends GTR0/HDPE and GTR5.5/HDPE.

Sample	$T_g$ (°C)
60GTR0/40HDPE	-44.17
60GTR0+ad/40HDPE	-40.40
80GTR0/20HDPE	-45.46
80GTR0+ad/20HDPE	-39.63
60GTR5.5/40HDPE	-39.39
60GTR5.5+ad/40HDPE	-35.65
80GTR5.5/20HDPE	-43.19
80GTR5.5+ad/20HDPE	-34.83

## CONCLUSIONS

In general, the devulcanization of GTR phase changed the crystallinity of HDPE phase of some blends, possibly due to changes in sizes of particles of devulcanized GTR and its distribution in the matrix. Concerning the revulcanization of the elastomeric phase, generally, it was observed the shift of the  $T_g$  peak of the GTR to higher temperatures, and reduction in the area under the peak of tan  $\delta$  for the  $T_g$  of the same phase, as a result of the restriction on the mobility of the GTR by dynamic revulcanization.