Devulcanization of Waste Rubber Using Thermomechanical Method Combined with Supercritical CO₂

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Abstract: Rubber waste disposal is an environmental problem. Particularly, many researches are centered in the management of discarded tires. In spite of all different ways of handling used tires, the most common is to deposit them in a landfill, creating a stock of tires. These stocks can cause fire danger and provide ambient for rodents, mosquitoes and other pests, causing health hazards and environmental problems. Because of the three-dimensional structure of the rubbers and their specific composition that include several additives, their recycling is a current technological challenge. The technique which can break down the crosslink bonds in the rubber is called devulcanization. Strictly, devulcanization can be defined as a process where poly-, di-, and mono-sulfidic bonds, formed during vulcanization, are totally or partially broken. In the recent years, super critical carbon dioxide (scCO₂) was proposed as a green devulcanization atmosphere. This is because it is chemically inactive, nontoxic, nonflammable and inexpensive. Its critical point can be easily reached (31.1 °C and 7.38 MPa), and residual scCO₂ in the devulcanized rubber can be easily and rapidly removed by releasing pressure. In this study thermomechanical devulcanization of ground tire rubber (GTR) was performed in a twin screw extruder under diverse operation conditions. Supercritical CO₂ was added in different quantities to promote the devulcanization. Temperature, screw speed and quantity of CO₂ were the parameters that were varied during the process. The devulcanized rubber was characterized by its devulcanization percent and crosslink density by swelling in toluene. Infrared spectroscopy (FTIR) and Gel permeation chromatography (GPC) were also done, and the results were related with the Mooney viscosity. The results showed that the crosslink density decreases as the extruder temperature and speed increases, and, as expected, the soluble fraction increase with both parameters. The Mooney viscosity of the devulcanized rubber decreases as the extruder temperature increases. The reached values were in good correlation (R= 0.96) with de the soluble fraction. In order to analyze if the devulcanization was caused by main chains or crosslink scission, the Horikx's theory was used. Results showed that all tests fall in the curve that corresponds to the sulfur bond scission, which indicates that the devulcanization has successfully happened without degradation of the rubber. In the spectra obtained by FTIR, it was observed that none of the characteristic peaks of the GTR were modified by the different devulcanization conditions. This was expected, because due to the low sulfur content (\sim 1.4 phr) and the multiphasic composition of the GTR, it is very difficult to evaluate the devulcanization by this technique. The lowest crosslink density was reached with 1 cm³/min of CO₂, and the power consumed in that process was also near to the minimum. These results encourage us to do further analyses to better understand the effect of the different conditions on the devulcanization process. The analysis is currently extended to monophasic rubbers as ethylene propylene diene monomer rubber (EPDM) and natural rubber (NR).

Keywords : devulcanization, recycling, rubber, waste

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