

Butyl rubber reclamation by combined microwave radiation and chemical reagents

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ABSTRACT: Rubber recycling is growing worldwide because of increasing raw material costs. In addition, extensive use of rubber articles and their eventual disposal is a major concern for the environment. Butyl rubber (IIR) was devulcanized by microwave radiation with the aid of devulcanizing reagents and oil. The effect of several devulcanizing reagents, hexadecylamine (HAD), diphenyl disulfide (DPDS), *N*-cyclohexyl-2-benzothioyl sulfenamide (CBS), and tetramethylthiuram disulfide (TMTD) on devulcanization process, was studied. The investigation was carried out in various microwave radiation temperatures and different amounts of aromatic and paraffinic oils. The results showed that waste rubber powder with a median particle size of 279 μm was devulcanized efficiently. It was confirmed when devulcanization % of the devulcanizates were measured by swelling tests. Among of the devulcanizing reagents and based on Horix analysis, HDA with the sol fraction, crosslink density (CLD), and devulcanization percent of 14%, 25 mol/m³ and 64%, respectively, was the most suitable devulcanizing reagent. In addition, compound with 30 and 6 phr paraffinic oil and HDA in formulation, respectively, had maximum devulcanization % (83%). The devulcanizing temperature of this compound was 180°C. For the most of all compounds, during microwave radiation, a part of disulfides crosslinks were broken and released sulfurs giving new mono and polysulfides bridges. This resulted in the higher CLDs for some compounds when they compared with CLD of the initial waste rubber. In the morphological study of the devulcanized compounds, vulcanized rubbers clearly showed in the SEM micrographs surrounded by a matrix of devulcanized rubber. © 2016 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2016**, *133*, 43363.

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INTRODUCTION

Rubber recycling is growing in importance worldwide because of increasing raw material costs, diminishing resources, and the growing awareness of environmental issues and sustainability.¹ In rubber devulcanization, three-dimensional rubber networks which are the outcome of vulcanization reactions, are broken. In sulfur vulcanization, the formation of both C—S and S—S bonds takes place, and it is therefore expected that during devulcanization, only C—S and S—S bond cleavage should occur. In fact, in an ideal devulcanization process, crosslinks should be broken without main-chain scission.² Several techniques were used for rubber devulcanization.³ They are chemical, thermochemical, mechanical, radiation, and biological processes. Radiation process may include microwave radiation. The three-dimensional rubber network can be broken down by microwave.^{4–8} The energies required to break monosulfidic C—S, polysulfidic S—S, and peroxide C—C bonds are 270, 240, and 345 kJ/mol, respectively.⁹ Hence, enough caution should be considered during microwave radiation to break, only C—S and S—S bonds during devulcanization.

RECYCLING OF BUTYL RUBBER (IIR)

Butyl rubber (Scheme 1¹⁰) is the common name for the copolymer of isobutylene and 1 to 3% isoprene produced by cold (−100°C) cationic solution polymerization.¹⁰ It was developed in 1937, by researchers William J. Sparks and Robert M. Thomas, at Standard Oil of New Jersey's Linden, N.J., laboratory.¹¹ Isoprene provides unsaturation required for vulcanization. The most distinguished characteristics of butyl rubber refer to high level of its saturation, i.e., high resistance to the diffusion or solution of gas molecules.¹⁰ Butyl rubber, the same of other rubbers reaches to its end of the service and life and subsequently, its recycling is a must. Few research works were done on butyl rubber recycling and devulcanization.^{12–15}

Kuan *et al.*¹² used supercritical CO₂ along with devulcanizing reagent, diphenyl disulfide (DD) to completely devulcanize butyl rubber. They found reclaimed rubber could not be cured by sulfur due to numerous decreases in the active crosslinking sites and the remaining DD. In another attempt,¹³ microwave without any chemical devulcanizing reagent, was used to

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