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CURE KINETICS OF BUTYL RUBBER CURED BY PHENOL FORMALDEHYDE RESIN

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Abstract–– **The characterization of rubber compounds is a necessary step for simulating rubber products manufacturing processes. We study experimentally and model computationally compounds of Butyl rubber cured by Phenol Formaldehyde Resin, a rubber used for making curing bladders, a key component in the tire industry.**

Several compounds, differing in the proportions of resin, Zinc Oxide, Polychloroprene (CR) and Stearic Acid, are experimentally tested during the curing process using a moving die rheometer. The experimental results are used to find the parameters of two well established kinetic models, namely the Nth order and the Kamal-Sourour models. Both models include an ordinary differential equation for the degree of cure and an algebraic relation between torque and temperature. The fitting of the model parameters is rearranged as an optimization problem, and solved using the Levenberg-Marquadt algorithm.

The results show that both models are able to represent the behavior of these compounds, although the more involved Kamal-Sourour model performs better for early times, as expected. The parameter values found are useful for numerical simulations of curing bladder manufacturing processes, and as a reference for further studies of similar compounds.

Keywords–– **Butyl rubber, Vulcanization, Kinetic model, Levenberg-Marquadt.**

I. INTRODUCTION

Butyl rubber is a copolymer of Isobutyle and 2% Isoprene which, depending on the final product application, can be cured using different cure systems (Rodgers *et al.*, 2008). This work is focused on resin cure systems, more specifically on Phenol formaldehyde resin.

The use of this resin results in slow cure kinetics but produces crosslinks stable enough to give excellent heat aging resistance and good flexibility and impermeability performance (Tawney *et al.*, 1960). These properties make them suitable to be used in the tire industry to make tire curing bladders which are to be exposed to high temperatures, high pressure steam, and hot water.

The techniques to characterize cure kinetics include rheometry, differential scanning calorimetry, chemical analysis, and equilibrium swelling, among others. The standard device used in the industry, both for compound development and for quality control, is the rheometer.

In this device the sample is placed over an oscillating plate inside a heated cavity. The crosslink density is correlated with the resistance of the sample to be deformed. Two cavity types are available; the oscillating disk rheometer (ODR), which has an oscillating biconical rotor inside the cavity (ASTM-D2084), and the moving die rheometer (MDR), which has an oscillating lower die (ASTM-D5289). The rheometer output is a torque evolving curve.

Two types of mathematical models have been developed to study cure kinetics, namely, phenomenological and mechanistic. Mechanistic models aim to represent the actual chemical reactions; therefore, a detailed knowledge of the reaction path is required. Consequently, the model developed will only be useful for a given type of compound. The first attempt of a mechanistic model is probably that of Coran (1964), who developed a model to represent the scorch delay kinetics. Several authors later adapted this model to take into account more reactions or other compounds (Ding *et al.*, 1996; Liang Fan *et al.*, 2002; Jeong *et al.*, 2002). Later on, several authors developed models for different compounds using the Population Balance Method (Sato *et al.*, 2002; Ghosh *et al.*, 2003; Likozar and Krajnc, 2007; Anandhan *et al.*, 2012).

On the other hand, phenomenological models aim to represent the behavior (macrokinetics) observed in curing tests, i.e. *Induction*, *Curing*, and *Post-curing*. The parameters of these models do not have correlation with the actual chemical reactions; therefore, the same model can be used for several rubber compounds. The model parameters have to be fitted to the experimental data. The mathematical representation of phenomenological models include one or more algebraic or differential equations (Isayev and Deng, 1988; Kamal and Sourour, 1973; Han *et al.*, 2000; Milani and Milani, 2010). Phenomenological models are widely used to analyze the effect of rubber formulation on the curing kinetics (He *et al.*, 2013; Marzocca and Goyanes, 2004; Sun and Isayev, 2009; Han *et al.*, 1999) or to numerically simulate the rubber behavior in the mold during the filling and/or curing stages (Sadr-Bazaz *et al.*, 1984; Khouider *et al.*, 1986; Armand *et al.*, 1986; Chan *et al.*, 1993). Among the authors who have used phenomenological models it is important to highlight the work of He *et al.* (2013), who used them to study the Chloride Butyl Rubber vulcanization.