



## RECOVERY OF VULCANIZED ELASTOMERS, USING A MICROWAVE GENERATION UNITY AT IPEN

Liliane Landini<sup>1\*</sup>, Sumair G. Araújo<sup>1</sup>, Ademar B. Lugão<sup>1</sup>, Hélio Wiebeck<sup>2</sup>

<sup>1\*</sup>Centro de Química e Meio Ambiente, IPEN-CNEN/SP - Av. Prof. Lineu Prestes, 2242 - Cid. Universitária, CEP:05508-900, São Paulo/SP, Brazil, - landini@usp.br; <sup>1</sup>IPEN-CNEN/SP - sgaraujo@ipen.br; <sup>1</sup>IPEN-CNEN/SP - ablugao@ipen.br; <sup>2</sup>Poli/USP - hwiebeck@usp.br

One of the concerns with polymeric materials is the environment protection after its discard. The microwave can break the three-dimensional network of reticulation of some materials without depolymerization, allowing a new vulcanization with similar properties to the original compound. The aim of this work is to use this radiation to recover vulcanized rubber. These samples (vinyl ethylene-acetate and butyl rubber) are being prepared and irradiated in a microwave generation unity, developed and assembled at IPEN, using variable energy doses at a specific frequency. The properties of these materials are being analyzed to verify the trustworthiness of the method.

### Introduction

It is known that polymeric materials (plastics and rubbers) do not decompose easily (it takes long for natural degradation due to their crosslinked structure), so the disposal of waste polymers (worn-out products and scrap of some artifact production) is a serious environmental problem. In the case of rubbers, large amounts are used as tires inner tubes, o-rings, rubber hoses, soles, carpets, flask caps, wire and cable coating etc. So one of the most important problems we have to face in the 21st century is waste disposal management, because production is continuously growing for all kinds of materials.

Recycling of these wastes is a good approach to try to solve this problem. This way can help the preservation of raw materials that exist in nature and reduce the trash volume, air and water pollution.

Researches have been made all over the world to identify qualitatively and quantitatively and define the mechanism of microwave-material interaction, in rubber devulcanization. In Brazil the application of this process is a very recent field and has been studied as a new tool in materials processing, which use high temperatures.

Thus, the objective of this work was to make the preliminary assays of rubber recovery, by using EVA and butyl rubbers (vulcanized) from scraps rubber of sole production and caps for pharmaceutical industry, respectively, to try to devulcanize them through microwave technology. This understanding of microwaves and devulcanization process is valuable for appreciating the advantages of this technology over other possible solutions [1].

### Experimental

#### Materials

Vinyl ethylene-acetate (EVA) rubber compounds are used in shoe manufacturing in several parts (sole, insole, leather). It has a lot of advantages such as lightness, flexibility, bright colors and it is comfortable as well. In general it has good process ability, thermal stability during processing, resistance to impact, weariness, resilience, tenacity and flexibility.

Butyl rubber (IIR) is the copolymer of isobutylene and a small amount of isoprene. The primary attributes of this kind of rubber are excellent impermeability/air retention and good flex properties, resulting from low levels of insaturation between long polyisobutylene segments.

These materials have many applications and there is a significant market for them today [2, 3]. So national market has great interest in recovering these products. Production leftovers of EVA shoe soles and butyl rubber caps were the samples used in the irradiations with microwaves.

#### Methods

##### - Microwave Process

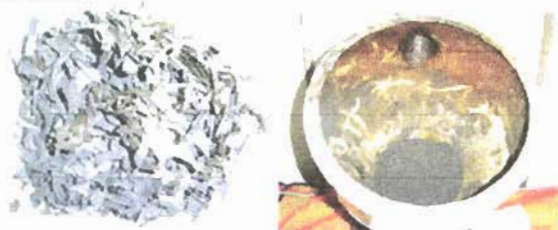
Microwaves (MW) are electromagnetic waves in the frequency band from 300 MHz to 300 GHz.

When an electric field, such as generated by microwave energy, interacts with some kinds of materials, such as some insulators, electrons do not flow freely, but re-orientation or distortions of induced,

11204

or permanent dipoles, can give rise to heating. Microwaves penetrate these materials and release their energy in the form of heat as the polar molecules vibrate at high frequency to align themselves with the frequency of the microwave field. Others substantial benefits are also speed, temperature homogeneity and absorption of almost all MW energy in the heated material. For rubbers, microwave energy can generate the necessary heat to try devulcanize them and at specified frequency with an amount sufficient can break some bonds. Thus in this process elastomer waste can be reclaimed without depolymerization to a material capable of being recompounded and revulcanized having physical properties essentially equivalent to the original vulcanizate. Moreover this method is very useful because it provides an economical, ecologically sound method of reusing elastomeric waste to return it to the same process [1].

In this work, a heat generating equipment was developed and installed (by IPEN-CNEN/SP) with high frequency (2450MHz) and RF valves (maximum 3000W). It consists of a wave generator, wave guides, a cavity to be filled with the material to be irradiated (Figure 1), which has a residual gas outlet. Also, a digital control panel was projected, manufactured and installed; that permits automatic monitoring and handling of irradiation time and power applied to the material.



**Figure 1** IIR sample (after irradiation) and internal view of the cavity of the heat generating equipment (MW) filled with EVA.

#### *- Irradiations*

Samples of EVA were irradiated in the MW reactor, at powers of 1000W, 2000W and 3000W. For butyl rubber samples, at 1000W. The times were: 1min up to 5min (EVA); 10min and 20min (IIR). All irradiations were monitored through a thermocouple.

#### *- Processing*

After being irradiated, the EVA samples were processed in an open mixer (cylinder), to homogenize the material and take the rubber straps. Some of these samples were used in rheometric analysis and others were pressed to make masterbatches and test bodies (150°C during 15min).

#### *- Rheometric Analysis*

The EVA samples were analyzed in a rheometer, model Rheometer 100 (Rheotec), for 12min at 160°C.

### **Results and Discussion**

The preliminary tests were made to evaluate the equipment and technique performance in recovering the EVA. At the end of the irradiations, the maximum temperature value of the EVA samples was 55°C. It was not possible to obtain rubber plates, according to the specifications from ASTM, because the produced laminated (mass) were not homogeneous. It had the appearance of orange-peel and were hard to the touch (Figure 2), confirmed by rheometric analysis which curves were not representative of rubbers with good characteristics to vulcanize again. In the case of butyl rubber, the samples were well heated and tests are still in progress.



**Figure 2** EVA samples after irradiation showing appearance of orange-peel (after processing in the cylinder).

### **Conclusion**

In this study with EVA rubber, the microwave technique did not show potential application, with these relations between irradiation time and power supply. So the samples are still not good enough in quality to be widely used. It is necessary to perform other experiments, to try to break the crosslinked structure of this type of rubber. Certainly, other properties should also be examined.

### **Acknowledgements**

The authors wish to thank: FAPESP, for the financial support; Anvil industry, for allowing the use of laboratories and some equipments; Grendene and Anvil industries, for supplying the samples.

### **References**

1. B.Adhikari; D.De; S.Maiti *Prog. Polym. Sci* 2000, 25, 909.
2. Polímeros e Elastômeros. [www.vulcanizar.com.br](http://www.vulcanizar.com.br). Access: October/2004.
3. *Materiais Solados e Palmilhas de Montagem. Série: Tecnologia Básica em Calçados, CTCCA (Centro Tecnológico do Couro, Calçados e Afins), Novo Hamburgo, May/2002.*