Development of a continuous process for EPDM devulcanization in an extruder
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Document Version
Publisher's PDF, also known as Version of record

Publication date:
2006

Link to publication in University of Groningen/UMCG research database

Citation for published version (APA):

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CHAPTER 7

TECHNOLOGICAL ASSESSMENT: APPLICATION OF THE EXTRUDER MODELS FOR OTHER DEVULCANIZATION PROCESSES

1. Methodology

A methodology for designing a continuous setup for a devulcanization process has been developed within this project. The results discussed in Chapter 4 demonstrate that the devulcanization process studied in the batch system (Chapter 3) can be successfully applied in a continuous extrusion operation.

1.1. Designing procedure

The screw designing procedure for a continuous devulcanization process is depicted schematically in Figure 7.1. When the devulcanization kinetics (showing the influences of the rate determining parameters on the conversion) is provided, the screw configuration can be designed by performing simulations. The chosen screw configuration should provide enough residence time and shear to give the required conversion at certain operating conditions. Kinetics of the devulcanization can be developed in a batch setup by first conducting screening experiments (Chapter 2) and subsequently extensive kinetics experiments (Chapter 3). The kinetics study should include all measurable factors that influence the devulcanization rate.

On the other hand, when there are not enough kinetic data to build a physical model of the extrusion, finding the optimum devulcanization conditions in a batch setup and trying to replicate the same conditions in the continuous process (the route on the right side of Figure 7.1) proves an alternative route. Since the residence time is then, consequently, estimated by not taking into account the changes in the degree of fill in transporting and pumping zone due to the lack of physical and kinetic data, some deviation of the results might occur. In this case, an experimental design method for the flow rate and rotation speed variation might be effective in studying the process response in the operating condition ranges studied (as has been discussed in Chapter 5). Fine-tuning of the operating conditions can then be done using the derived model.
1.2. Estimation of the Key Parameters

The screw configuration of the extruder is designed according to the residence time and shear rate required to achieve the desired conversion. The shear rate calculation is quite straightforward as compared to that of the residence time.

The required shear rate can be obtained by adjusting the flow rate and the screw rotation speed according to equations proposed by van der Wal et al. [1,2], or Meijer and Elemans [3]. For practical purposes, the shear rate in the kneading zone is the most important one. It should meet the shear rate in the Brabender since its value in the kneading zone is the highest and hence the conversion occurs mainly here.

Figure 7.1. Flow diagram to design the screw configuration for devulcanization purpose
On the other hand, the residence time calculation is not straightforward, as depicted in Figure 7.2. The residence time must be calculated per screw section, depending on the maximum drag capacity of the screw, the feed rate, the phase state (solid/liquid) and the backflow. The maximum drag capacity is influenced by the rotation speed and the screw geometry. The backflow is a function of the maximum drag capacity, the backpressure, and the throughput.

![Figure 7.2. Factors influencing the residence time (+: positive effect, -: negative effect)](image)

2. **Influence of the Processing Steps on the Product Performance**

The mechanical properties of the reclaimed rubber are to some extent dependent on the processes involved, which can be grouped into two parts: the devulcanization part and the revulcanization one. The process conditions during the devulcanization step will influence the characteristics (namely viscosity) of the devulcanizate produced. In the revulcanization step, the curing system and the reclaim/virgin ratio will affect the product performance. In order to obtain the best results in mechanical properties, these parameters should be studied further.

Considering the broadness of the revulcanization techniques and the product possibilities, a product technology approach is required to find the best product that can be made out of the devulcanized EPDM rubber. Product technology here involves recursive impacts between the chemistry, the processing, and the product properties and market price (Figure 7.3). Product properties are the results of the chemistry and processing parts of the technology. On the other hand, the processing techniques and chemistry are very much dependent on the product value, which is in turn related to the product properties. Chemistry and processing are to be considered simultaneously in optimizing the product’s properties since they give feedback to each other. In this
research, the effect of the processing on the product’s properties has been studied, as has been discussed in chapter 6.

The importance of this product technology approach can also be seen in the economic evaluation of the recycling process. Economic feasibility of the whole process is, in the end, the most important factor to be considered. Figure 7.4 shows the balance that should be made between the operational cost and the profit of a recycling process for the feasibility study. A devulcanization process is cost intensive due to the high operating temperature required to break a quite stable crosslinked structure in EPDM’s. Taking a look at the factors considered in Figure 7.4, almost all can be assumed reasonably fixed for different devulcanization process, except the material cost for re-vulcanization and the product price. Therefore, the balance is now really dependent on these two factors, which are in turn dependent on the product optimization as depicted in Figure 7.3.
3. Applications of the Reclaim Rubber

Three revulcanization possibilities have been studied within this project so far. The first one is introducing the devulcanized EPDM profile into the virgin compound (in the 50/50 and 25/75 of devulcanized/virgin ratio), which results in mechanical properties that are comparable to the virgin one. The second one is the revulcanization of the roofing sheet material (100% from the devulcanized roofing sheet), which results in slightly inferior mechanical properties compared to the virgin one (Table 7.1). The third one is the mixing of EPDM profile material into the virgin roofing sheet compound that has been conducted by Rubber Group of University of Twente. The compound was cured according to the roofing sheet recipe and resulted in good mechanical properties (Table 7.1), all higher than the UEAtc standard.

Table 7.1. Mechanical properties of the roofing sheet material produced from devulcanizates

<table>
<thead>
<tr>
<th>Material</th>
<th>Tensile strength (MPa)</th>
<th>Elongation at break (%)</th>
<th>Tear strength (N/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Roofing sheet reclaim</td>
<td>7-10</td>
<td>400-500</td>
<td>15</td>
</tr>
<tr>
<td>Profile reclaim</td>
<td>9-13</td>
<td>620-800</td>
<td>20-35</td>
</tr>
<tr>
<td>Virgin roofing sheet</td>
<td>13</td>
<td>650</td>
<td>20</td>
</tr>
</tbody>
</table>

*The UEAtc standard is 8 MPa for tensile strength, 400% strain at break, and 20 N/mm tear strength [4]*

The first two represent the attempt to reuse the rubber for its original application, while the last one is an example of reusing the rubber in a different application. Considering the possibilities in using the reclaim rubber, an extensive study on the application is required as a continuation of this research.

4. Conclusions

The work presented here is of industrial importance, as has been discussed by Fukumori et al. [5], having the final aim to minimize the production waste.

A method to develop a continuous devulcanisation system in an extruder based on a known kinetics has been presented here. When the kinetics is not available, experimental design can be used in studying the response of the process in a certain range. Anyhow, preliminary study in a batch setup is required for both cases.

Developing a feasible devulcanization process is a challenge. The solution to it requires an extensive study on finding the best-recycled product with good market value at lowest possible re-vulcanization cost.
References

4. UEAtc (union européenne pour l'agrément technique dans la construction) (1988)