Devulcanization of EPDM (Ethylene-Propylene-Diene Rubber) Wastes

Rubber Recycling of Washing Machine Gaskets

EPDM wastes occur as crosslinked elastomers in production. They are highly elastic and chemically resistant, but cannot be remelted. The crosslinks must therefore be broken before the polymer is reused. For this purpose, the Fraunhofer ICT has analyzed the potential of ultrasound-assisted extrusion. The recycled elastomers are used as feedstock for the production of new gaskets in the automotive sector and washing machines.

Wastes generated during the production of washing machine gaskets can be recycled.

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In Germany, rubber is recycled in various ways. The most common methods are mechanical and chemical recycling, which includes pyrolysis. In mechanical recycling, the rubber wastes are first shredded and subsequently ground. The rubber pieces are cut into small chunks or rubber pellets until they can then be used as pellets for manufacturing new rubber products (**Fig. 1**). During chemical recycling, in contrast, the resulting rubber waste is broken down into its original components. These components can then be used as raw materials for manufacturing new products. In pyrolysis, the rubber wastes are heated in a closed system at high temperatures to break them down into oil, gas and carbon. The oil and gas obtained can be used as a fuel, while carbon can be used as a raw material for new rubber products.

In Germany, most rubber wastes are mechanically recycled, since this is the simplest and most cost-effective method. However, the elastomers that are recovered usually only perform » the function of fillers, which limits the maximum amount that can be integrated into virgin material because of the increasingly worsening properties. It would be desirable to have devulcanized rubber recyclates that can be recrosslinked again after appropriate compounding and added to virgin material in large quantities without loss of properties.

Fig. 1. Strings of production wastes (left) and shredded sprues of washing machine gaskets are fed back into the production of virgin material as fillers. © Fraunhofer ICT

Re-use: Turning Crosslinked Rubber Wastes into New Gaskets

In the Re-use project, a twin-screw extrusion process for continuous devulcanization of the secondary materials was developed for compounding the vulcanized EPDM wastes. In the trials, an extruder with a screw diameter of 27 mm and a process length of 52 D was used. The EPDM production machines used were derived from the extrusion of gasket profiles for the automotive industry and injection-molded sprues of gaskets for washing machines.

The main aim of the project was the targeted devulcanization of the crosslinked rubber wastes and investigations into their reuse as feedstock or revulcanizable rubber recyclates in virgin material. For this purpose, the EPDM wastes were first shredded into pellets to ensure robust dosing and subsequently subjected to extensive metering tests. In the next step, the pellets were metered into the extruder via gravimetric metering equipment. To convey the pellets within the extruder to the individual process zones, an optimized screw for depolymerization was developed, as can be seen in **Figure 2**. Besides the feed zone (1) for EPDM pellets and additives, the process zone of the extruder consists of the following four further process zones: shredding zone (2), additive homogenization/dispersion zone (3), ultrasound zone (4) and degassing and discharge zone (5).

In the shredding zone, the fed pellets and additives, such as devulcanization aids and recrosslinking inhibitors, are shredded. The surface area is thus enlarged for subsequent treatment and finally optimally distributed in the homogenization and distribution zone. The heart of the process is the ultrasound zone, in which treatment with ultrasound takes place. In the ultrasound zone, the material is subject to intensive shearing and ultrasonic energy. In this zone, the crosslinked structure is cleaved, which leads to a significantly reduced material viscosity. Depending on the process conditions used, as regards temperature, throughput, pressure and input ultrasonic energy, various degrees of devulcanization and therefore material properties could be realized. Furthermore, the process resulted in a (partially) devulcanized product, in which reactive areas, such as double bonds or free radicals, could be identified by breaking the bonds. The additives introduced into the process prevent undesirable recrosslinking in the extruder. Finally, there are the degassing and discharge zones, in which the volatile components are removed and the devulcanized EPDM material is discharged.

The maximum throughput rate achievable with the configuration used for devulcanization is 5kg/h. An increase of the throughput rate is not possible, since an increase of the throughput rate significantly reduces the residence time and the treatment time is therefore not sufficient for devulcanization. The degree of crosslinking (crosslink density = CLD value) was measured by the swelling method and the Flory-Rehner equation. The suitability of the devulcanized material samples for use as inactive/active components was again investigated and evaluated in the case studies of washing machine gaskets and extruded gaskets for the automotive industry as admixture in virgin material for different EPDM recyclate components.

Results Obtained and Outlook

The results of the Re-use project impressively show that sulfur-crosslinked EPDM materials can be successfully devulcanized in the co-rotating twin-screw extruder. In some cases, the use of ultrasound does not show a significant improvement in the devulcanization as measured by the CLD values. For the components treated with ultrasound as well as those treated exclusively mechanically, a CLD value reduction of over 90 % could be determined. However, the case studies show a positive influence with the use of ultrasound, since, in both applications, a higher proportion of the ultrasound-treated devulcanized material can be integrated into production mixtures, and thereby a higher proportion of recyclate can be obtained. The devulcanized material could be used either as an inert filler or in revulcanized form, with the proportion being higher

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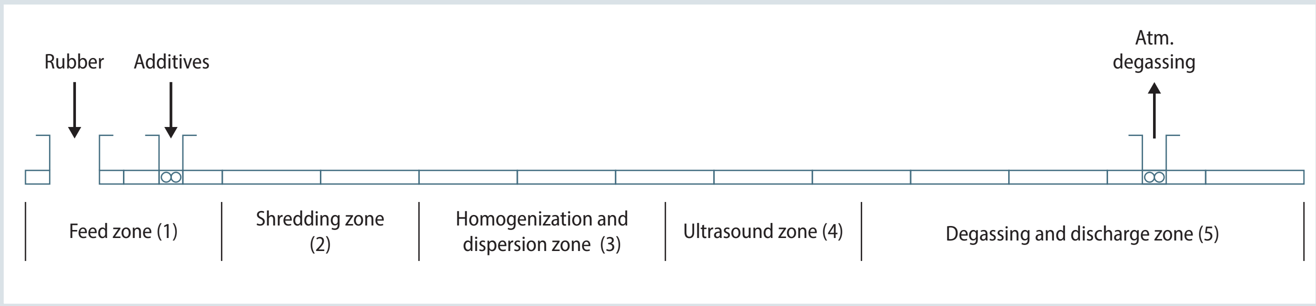

Fig. 2. Extruders with process zones. Source: Fraunhofer ICT, graphic: © Hanser

in the case of revulcanization because of the better incorporation. However, both variants significantly extend the possibility of integrating recyclates into raw mixtures, resulting in a saving potential of raw rubber and fillers.

Proportions of Recyclates: Up to 25 % in Washing Machines

The first case studies with the devulcanized materials were carried out under real production conditions at an industrial partner for manufacturing car gasket systems and an industrial partner for manufacturing washing machine gaskets. In the manufacture of the gasket systems for the automotive industry, the case studies showed that the devulcanized filler could be mixed under real production conditions into a rubber compound in a proportion of 5 % and subsequently extruded to form a profile. This profile met all mechanical requirements, such as hardness, density and tensile strength. The analytical studies did not show any significant changes in the fingerprint of the EPDM mixture, which allowed it to be mixed into existing material formulations. Only the characteristics for fogging could not be achieved and can be reached by adapting the material formulations. In the manufacture of the washing machine gasket, it was found that 15 to 25 % of the rubber mixtures can be replaced by the devulcanized material. In this case study, all the mechanical values and aging tests at the production level could also be met. Only the results of the lifetime test showed slightly reduced characteristic values.

To sum up, it can be reported that EPDM production wastes, which currently are usually incinerated, can be successfully returned to production. Furthermore, the project results clearly show the savings potential compared to virgin material by the use of recyclates in rubber processing with the example of EPDM. The amounts of waste generated during production can thereby be significantly reduced and so make a positive contribution to environmental protection. ■