xplore

Technical Note

Processing and Characterization of EPDM Rubber/Silica Compounds using an MC15HT

N. Yazici, M. Kodal and G. Ozkoc

Introduction

The global rubber market is expected to grow from USD 27 billion in 2017 to USD 34 billion by 2022, at a compound annual growth rate (CAGR) of 4.6% from 2017 to 2022 [1]. Moreover, academic studies related to rubbers, including patents, proceedings, etc., have significantly increased in the last decade. Therefore, it is believed that rubber science and technology will be one of the essential areas in the coming decade. The research and development (R&D) in rubber formulation development requires reproducible, repeatable, fast, accurate, and efficient sample preparation. Rubber compounding is a conventional process that combines rubbers and various chemicals such as vulcanizing agents, stabilizers, and fillers to produce rubber-based products of the desired properties. Open mills or internal mixers (Banbury) are used to prepare rubber compounds.

After that, the resulting compound is shaped on the mills, extruders, or calendars prior to the vulcanization process. One of the most important criteria and complications in rubber compounding is the increasing dispersion of fillers such as silica, and the chemicals such as sulphur, antioxidants, accelerator, etc., in the matrix. To improve the dispersion of additives and fillers in rubber compounds, robust mixers facilitate enough shear and residence time and need to be reproducible, user-friendly, and fast. The lab-scale formulation development is conventionally carried out using small-scale internal mixers and two-roll mills.

These conventional compounding methods contain some critical disadvantages, such as being labor and time-intensive (at least two personnel) to prepare a sample of one batch, cleaning is a difficult task and long turnaround time. In addition, from a safety point of view, two roll mills are dangerous since they have an open mixing area. It needs at least 300–400 cc material; expensive additives are therefore an issue. Besides, both open mills and internal mixers occupy large lab area, mostly separate laboratory space because of carbon black or fumed silica dusting.

However, high torque laboratory twin-screw micro-compounder, which have been serving the plastic industry for more than 30 years, can be used to formulate new rubber compounds for fast and accurate sample preparation that on top can contribute to the economics of R&D. Moreover, micro-compounders in rubber compounding at laboratory scale have some other critical features such as, one person can operate, easy, less labor-intensive and the compound is ready in 5 min. It needs only small amounts of materials to prepare compounds with expensive materials. More accurate formulations can be prepared. It occupies small lab space as compared to conventional ones. Besides, it is easy to clean and safe to operate.

In this technical note, we reported the possibility of using a labscale 15 mL high torque twin-screw micro-compounder as a tool for new rubber compound development. For this purpose, weformulated ethylene propylene diene monomer (EPDM) /silica recipes through conventional way using a Banbury mixer followed by a two-roll mill, and through a new way by using a lab-scale 15 mL high torque twin-screw micro-compounder. The conventional way of sample preparation was compared with a possible way of sample preparation based on materials parameters and ease of operation as well.

Figure 1. Forced feeding area of the intermeshing screws

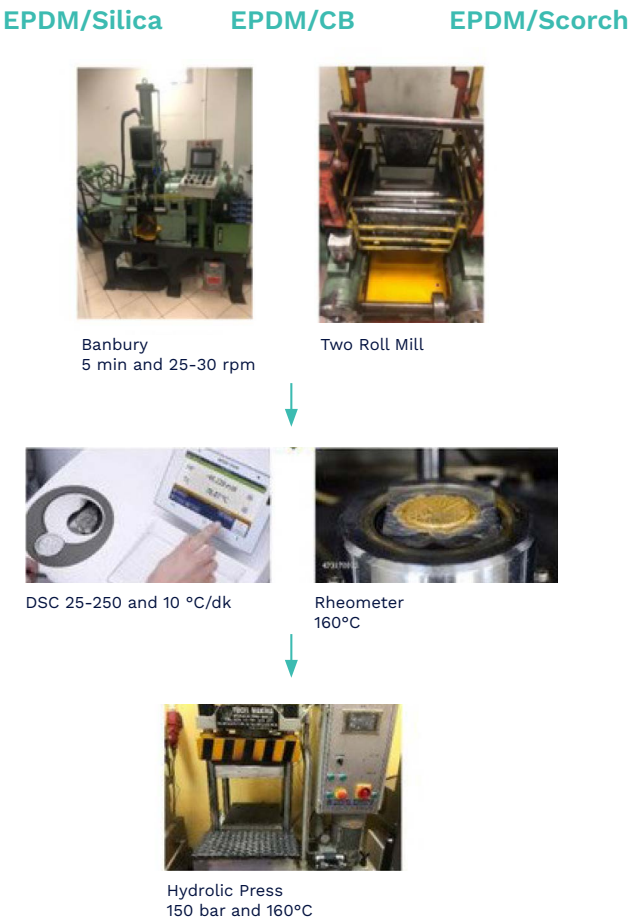


Figure 2. Flow chart for Banbury compounding of EPDM/Silica

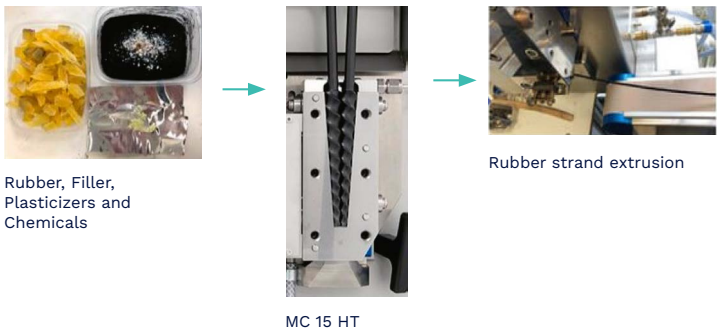


Figure 3. Flow chart for MC15 HT compounding of EPDM/Silica

Experimental

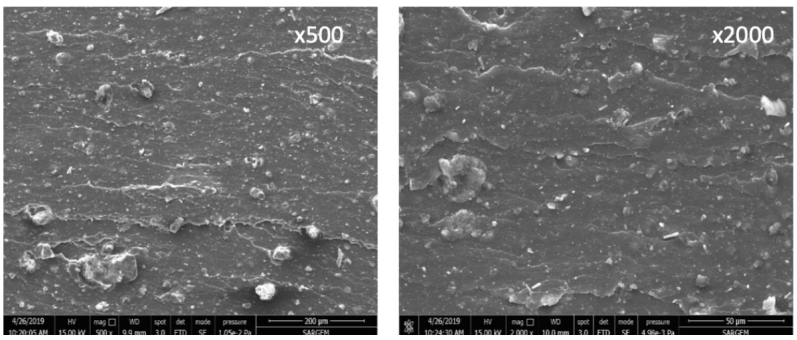
EPDM (Keltan 9650Q) was purchased from Lanxess. Silica, and other chemicals such as zinc oxide, stearic acid, 2,2,4-Trimethyl-1,2-Dihydroquinoline (TMQ), Tetramethylthiuram disulfide (TMTD), 2-Mercaptobenzothiazole (MBT) were provided by RubberChem. Paraffinic oil was obtained from Petro Yag, Turkey. The silica ad sulphur contents were kept at 25 and 2.5 phr, respectively.

Compounding EPDM with silica was carried out in a 2 L Banbury mixer at a 25 rpm of rotor speed and an average temperature of 60 °C. First, EPDM was masticated for 2 min. Silica and oil were added to the compound and mixed for 5 min, and then activator and antioxidant were incorporated and were mixed for further 2 min. Finally, accelerator and curing agent were incorporated into the EPDM compounds for 1 min. The complete compounding cycle was approximately 10 min and shown in Figure 2. After two-roll milling, the compounds were crosslinked using a hot-press at predefined crosslinking time and temperature that were obtained from DSC and rheometer studies, respectively. A lab-scale high torque twin-screw micro-compounder (Xplore Instruments MC 15 HT, The Netherlands) was used to prepare the EPDM-based compounds. The premixed rubber and ingredients, except sulphur and accelerator, were fed to the compounder. The barrel temperature was controlled using water-cooling jackets to keep it at 60 °C. At the end of 2 min, the sulphur and accelerator were added to the compound and mixed for 1 min. The rubber strands were then taken from the die of the compounder by changing the position of the discharge valve (Figure 3). After two-roll milling, the compounds were crosslinked using a hot-press at predefined crosslinking time and temperature similar to that of conventional Banbury compounds.

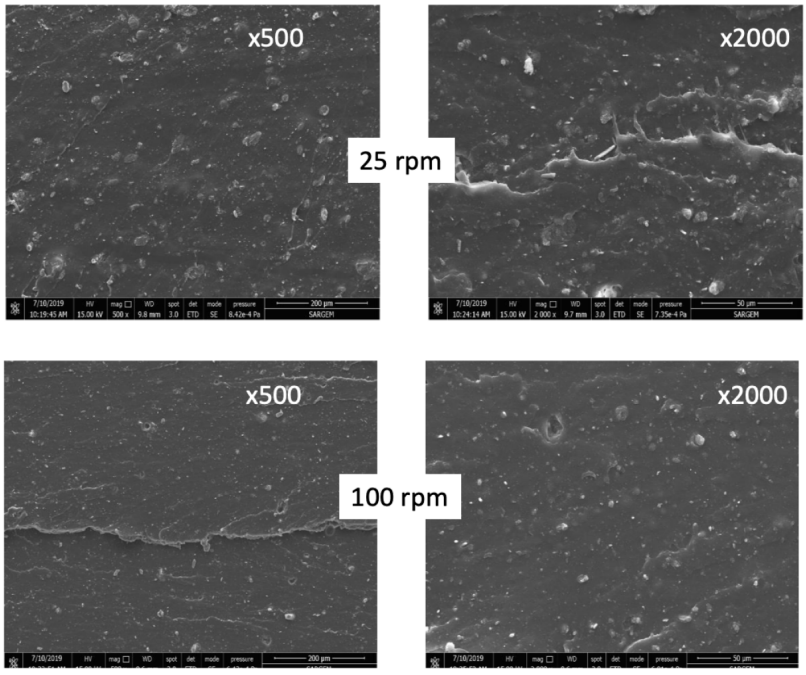
The performance of the rubber compounds were characterized using moving die rheometer (MDR), tensile tests, scanning electron microscopy (SEM) and dispergrader analysis.

Results and discussion

The level of dispersion of silica in EPDM composites prepared in Banbury and MC 15 HT was evaluated by SEM analyses. Figure 4 shows the SEM micrographs of tensile fractured surfaces of EPDM/silica composites prepared in both Banbury and MC 15 HT with different screw speeds.



(a) EPDM/Silica Compounds prepared in Banbury



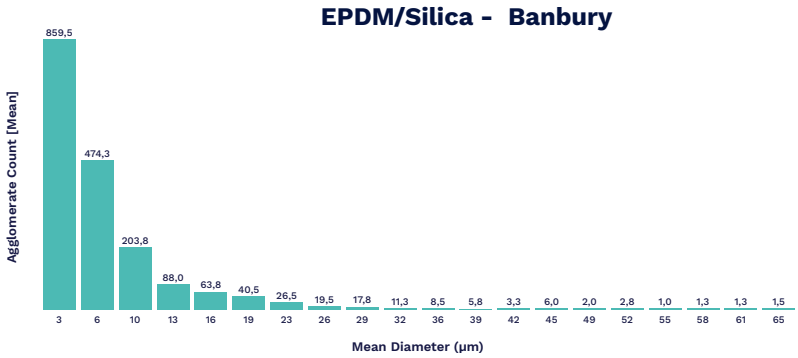
(b) EPDM/Silica Compounds prepared in MC15 HT

Figure 4. Tensile fractured surface morphologies of the EPDM/Silica composites (magnification ×500 and ×2000).

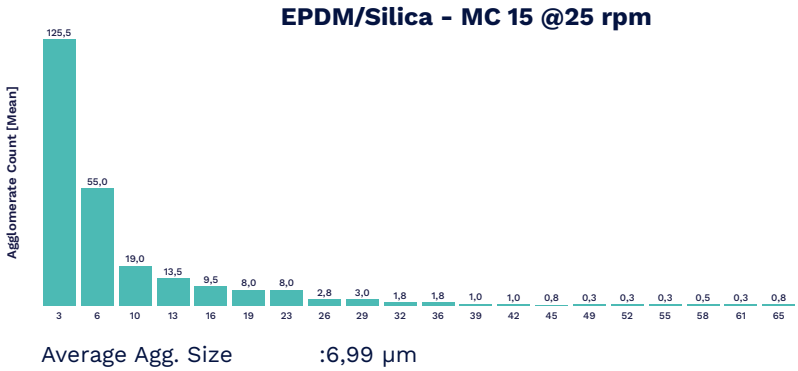
Silica dispersion was found coarser for EPDM/silica composites prepared in Banbury,. The particle size of silica in EPDM ranged from 10 to 110 µm. On the other hand, the preparation of EPDM/silica composites in MC HT 15@25 rpm led to a decrease in the diameter of silica agglomerates. The average diameter of silica was found 20 µm.

Moreover, a significant improvement in the dispersion of silica in EPDM composites prepared in MC 15 HT@100 rpm was obtained, as shown in Figure 3. To determine the quality of the EPDM compounds and silica dispersion in EPDM, DisperGrader analyses were carried out.

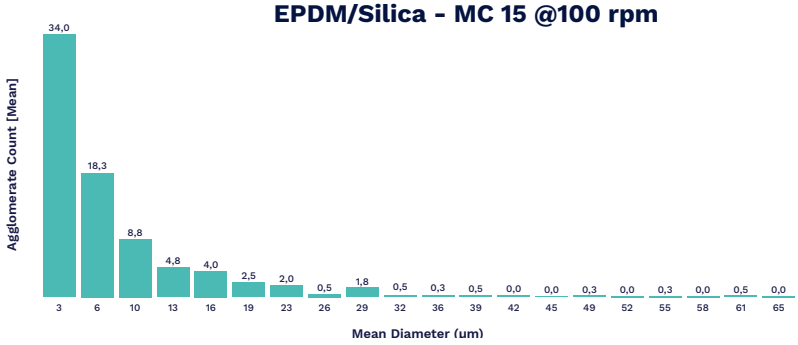
Figure 5. shows the images, average particle size, and percent dispersion obtained from the DisperGrader analysis indicating the dispersion of silica in EPDM. Generally, the dispersion of silica in a polymer matrix is related to reducing the size of silica in the form of aggregates. It was obtained for EPDM/silica composites that the dispersion of silica in EPDM was much better in the composites prepared with MC 15 HT compared to the Banbury. Moreover, the average particle size of silica decreased significantly from 9.2 µm to 7.0 µm and, the dispersion quality percentage of silica was much better in the composites prepared in MC 15 HT.



Average Agg. Size :9.20 µm
Dispersion (%) :67.04



Average Agg. Size :6,99 µm
Dispersion (%) :94.38



Average Agg. Size :6.95 µm
Dispersion (%) :98.08

Figure 5. The dispersion grade for EPDM/Silica compounds with respect to processing route

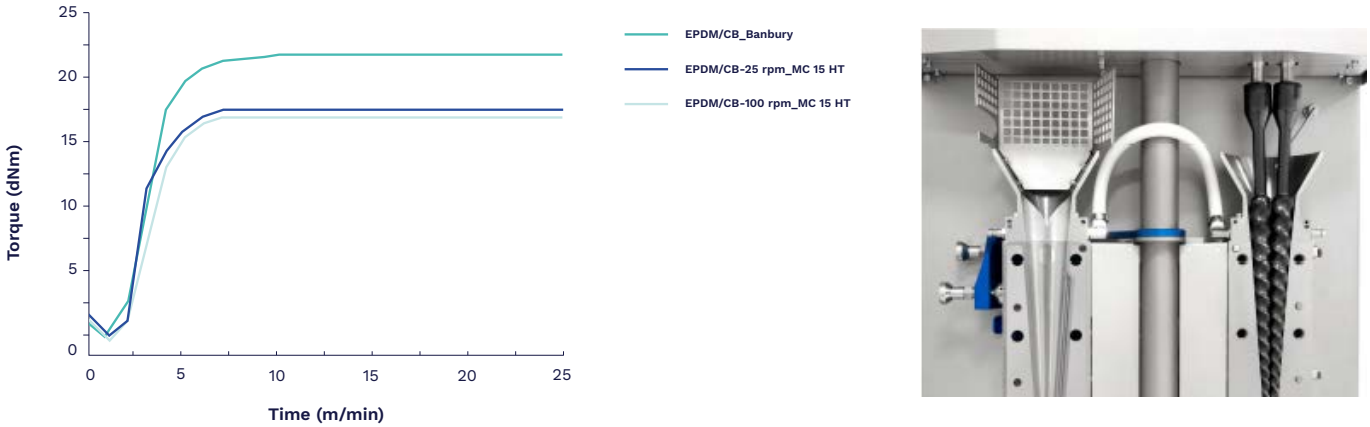


Figure 6. MDR curves of EPDM/Silica compounds

The rheometer results of the EPDM compounds in Figure 6. The samples prepared in Banbury and the samples prepared in MC 15 HT@25 rpm showed nearly similar results; however, the samples prepared in MC 15 HT@100 rpm yielded higher t90 with a marching behavior. This is due to the higher interfacial area between EPDM and silica, where silica retards the vulcanization of EPDM. Moreover, the acidic nature of silanol groups of finely dispersed silica particles in EPDM compounds prepared at MC 15 HT@100 rpm significantly affected the curing time and cure rate index (CRI). This situation increased the interaction of silica with the activator, which resulted in higher t90 and lower CRI [2,3].

On the other hand, finely dispersed silica particles in EPDM compounds prepared in MC 15 HT@100 rpm adversely affected the crosslink density of compounds. As stated in the literature, silica can react with a cure accelerator via a hydrogen bond.

This will accelerate the bond dissociation of N–S [4]. Therefore, the consumption of cure accelerator in the presence of welldispersed silica particles in EPDM compound led to a decrease in cure extent values of EPDM compound prepared in MC 15 HT@100 rpm.

Conclusion

Laboratory-scale mixing devices serve as cost and time-efficient research facilities and provide possibilities for processing a few grams of material.

They have the capability of continuous or batch processing with the same thermal environment as a conventional extruder. These micro-compounding devices can be used as fast screening tools in the field of polymer and rubber-based nanocomposite development. In this study, EPDM/silica composites were prepared in both a conventional rubber-mixing device and an MC 15 HT micro-compounder.

Moreover, different screw speeds were used to prepare EPDM compounds at MC 15 HT to evaluate the effect of rpm on the dispersion of silica in the EPDM matrix. It was shown that comparable dispersion and rheological behavior could be obtained from EPDM/silica compounds prepared in MC 15 HT at much shorter time. It can be concluded that compared to the traditional rubber mixing method, the lab-scale high torque twinscrew micro-compounders as a new rubber-mixing tool can be convenient and efficient to prepare rubber composites containing finely dispersed silica.

References

1- Baranwal, G.; Singh, M.; Vidyarthi, D.P. A framework for IoT service selection. J. Supercomput. 2020, 76, 2777-2814.

2- Mohd Zaini, N.A.; Ismail, H.; Rusli, A. Sepiolite hybridized commercial fillers, and their effects on curing process, mechanical properties, thermal stability, and flammability of ethylene propylene diene monomer rubber composites. Iran. Polym. J. 2018, 27, 663–675.

3- Hair, M.L.; Hertl, W. Acidity of surface hydroxyl groups. J. Phys. Chem. 1970, 74, 91–94.

4- Choi, S.-S.; Park, B.-H.; Song, H. Influence of filler type and content on properties of styrene-butadiene rubber(SBR) compound reinforced with carbon black or silica. Polym. Adv. Technol. 2004, 15, 122–127.

Xplore Instruments BV
Arendstraat 5
6135 KT, Sittard
The Netherlands

Tel: +31 46 208 97 70
Fax: +31 46 208 97 71

info@xplore-together.com
www.xplore-together.com
Trade Register: 60040114

Find out more at www.xplore-together.com