Technical Note

A micro lab-scale compounder; a new rubber-mixing tool for EPDM/Carbon black composites

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Context

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 timeintensive (at least two personnel) to prepare a sample of one batch; cleaning is a difficult task (two Banbury for black and non-black samples), long turnaround time, etc. 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 of material; expensive additives are an issue. Besides, open mills and internal mixers occupy 6 m^2, mostly separate laboratory space because of carbon black (CB) 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.

In this work, we demonstrated the possibility of using the lab-scale Xplore MC15 HT high torque twin-screw microcompounder as a new rubber compound development tool. A lab-scale high torque twin-screw micro-compounder was used to prepare the EPDM-based compounds. The premixed rubber and carbon black, 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 sulfur and accelerator were added to the compound and mixed for 1 min. The rubber stripes were then taken from the compounder's die by changing the discharge valve's position (see Figure 1). The curing of EPDM composites was carried out in a hot press at 160°C.

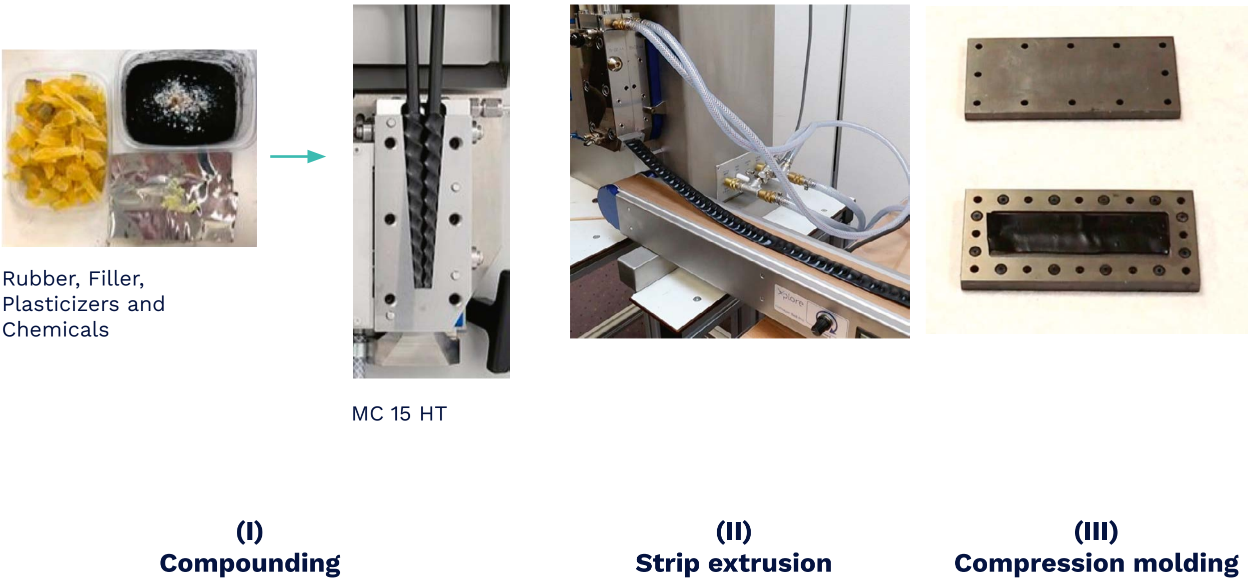


Figure 1. Flow chart for MC15 HT compounding of EPDM/CB

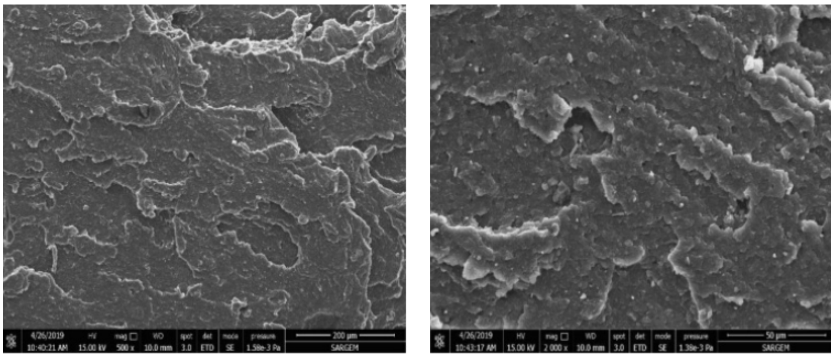
Compounding EPDM with carbon black was carried out in a 2 L Banbury mixer (Met-Gur, Turkish Model) at 25 rpm of rotor speed and an average temperature of 60°C. First, EPDM was masticated for 2 min. Carbon black and oil were added to the compound and mixed for 5 min, and then activator and antioxidant were incorporated and were mixed for a further 2 min. Finally, the accelerator and curing agent was incorporated into the EPDM compounds for 1 min. The complete compounding cycle was approximately 10 min. Rubber compounds were homogenised and shaped by using a laboratory-type high torque two-roll mill having a diameter of 15 cm and length of 30 cm. Milling speed was kept constant at 10 rpm, and the friction ratio was 1:1.1. The temperature was 60°C, and the distance between the cylinders was varied from 0.5 to 3 mm during mixing. The curing of EPDM composites was carried out in a hot press at 160°C.

The carbon black dispersion level in EPDM composites prepared in Banbury and MC15 HT was evaluated by scanning electron microscopy (SEM) analyses. Figure 2 shows the SEM micrographs of tensile fractured surfaces of EPDM/CB composites prepared in the Banbury and the MC15 HT with different screw speeds. As can be seen in Figure 2 that, carbon black was dispersed homogeneously in the EPDM matrix independent of the sample preparation method.

Sample BANBURY

x500 **x2000**

EPDM/CB



Sample MC 15 HT

x500 **x2000**

EPDM/CB -25 rpm

EPDM/CB -100 rpm

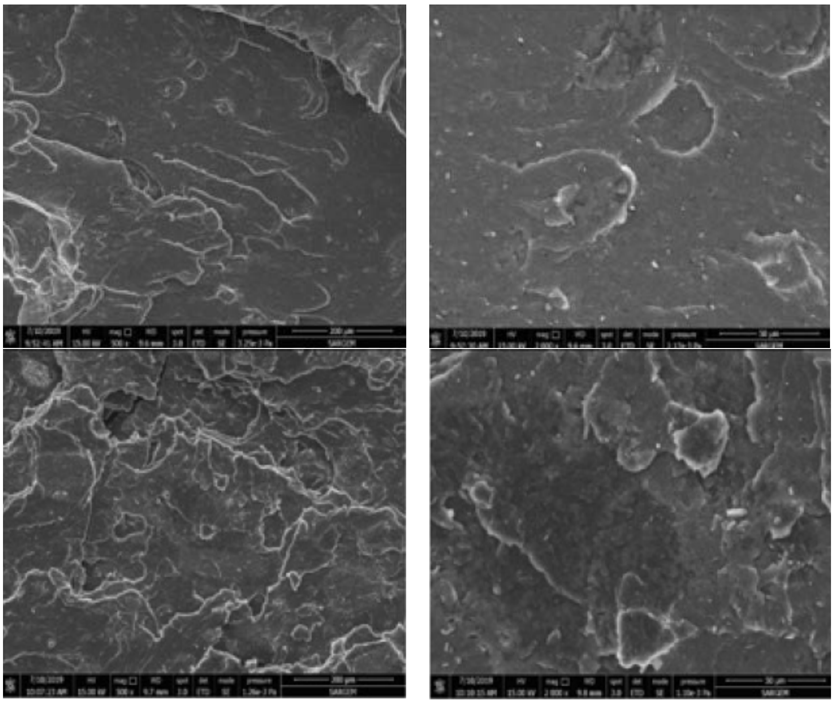


Figure 2.
Tensile fractured surface morphologies of the EPDM/CB composites prepared in a Banbury and in an MC15 HT (magnification x 500 and x 2000).

To determine the quality of the EPDM compounds and carbon black dispersion in EPDM, DisperGrader analyses were carried out. Figure 3 shows the images, average particle size, and percent dispersion obtained from the DisperGrader analysis indicating the dispersion of carbonblack in EPDM. Generally, the dispersion of carbon black in a polymer matrix is related to reducing the size of carbon black in the form of aggregates. It was obtained for EPDM/CB composites that the dispersion of CB in EPDM was much better in the composites prepared with an MC15 HT compared to the Banbury. The lab-scale high torque twin-screw micro-compounder MC15 HT yielded better dispersion levels for carbon black in comparison to that obtained in Banbury.

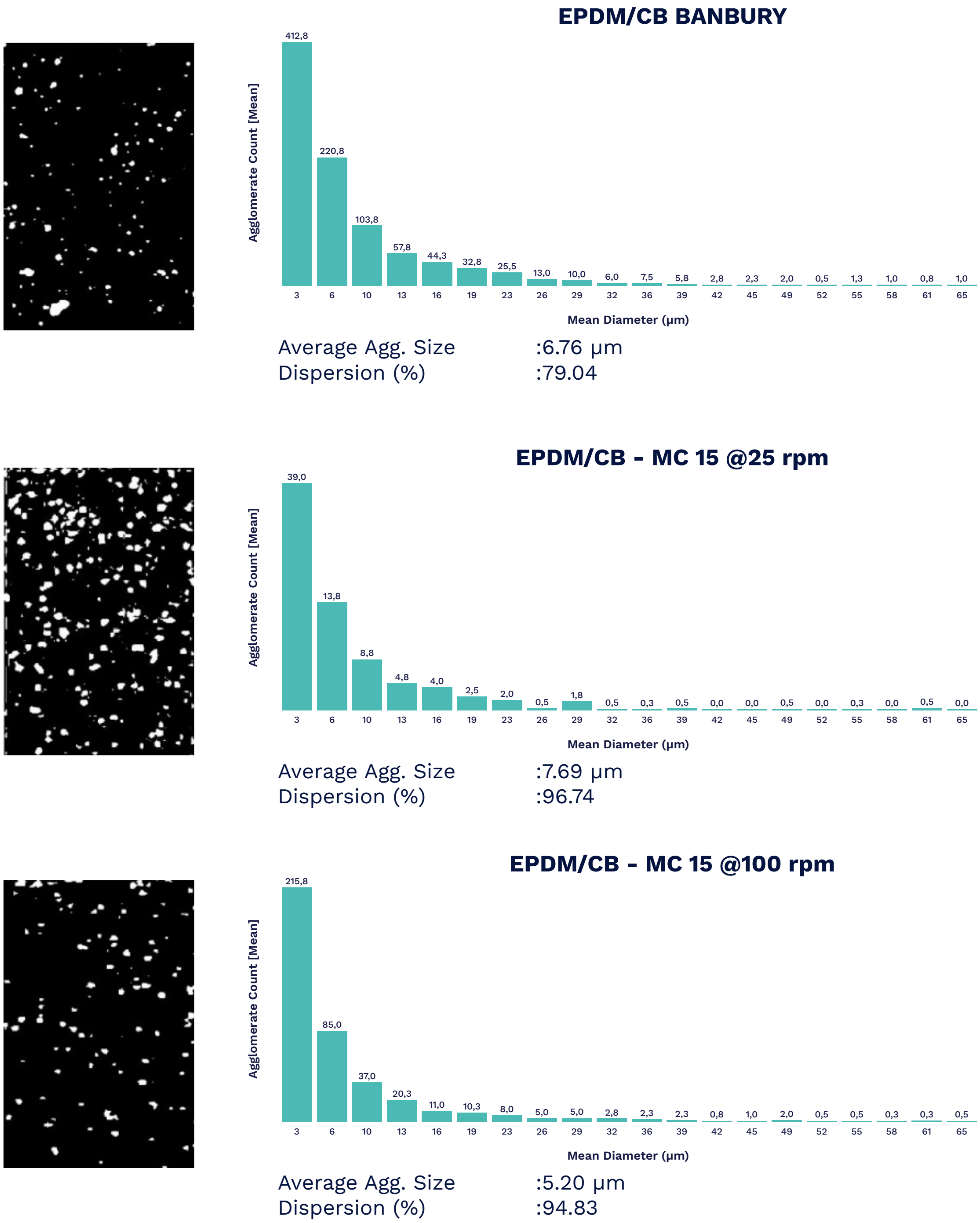


Figure 3.
Carbon black dispersion in EPDM composites prepared in a Banbury and an MC15 HT.

The rheometer results of the EPDM compounds are shown in Figure 4. It is seen that for EPDM/CB compounds, the Banbury and MC15 HT@25 rpm yielded nearly similar results; however, MC15 HT@100 rpm exhibited higher MH values in comparison to the samples prepared in Banbury. This can be due to the better dispersion of the CB in EPDM at higher rpm. Moreover, the highest torque difference (MH–ML), which can be related to the crosslink density of the composites, was obtained for EPDM/CB samples prepared in MC15 HT@100 rpm. This increment in crosslink density can be attributed to the increased interfacial interaction between carbon black and EPDM.

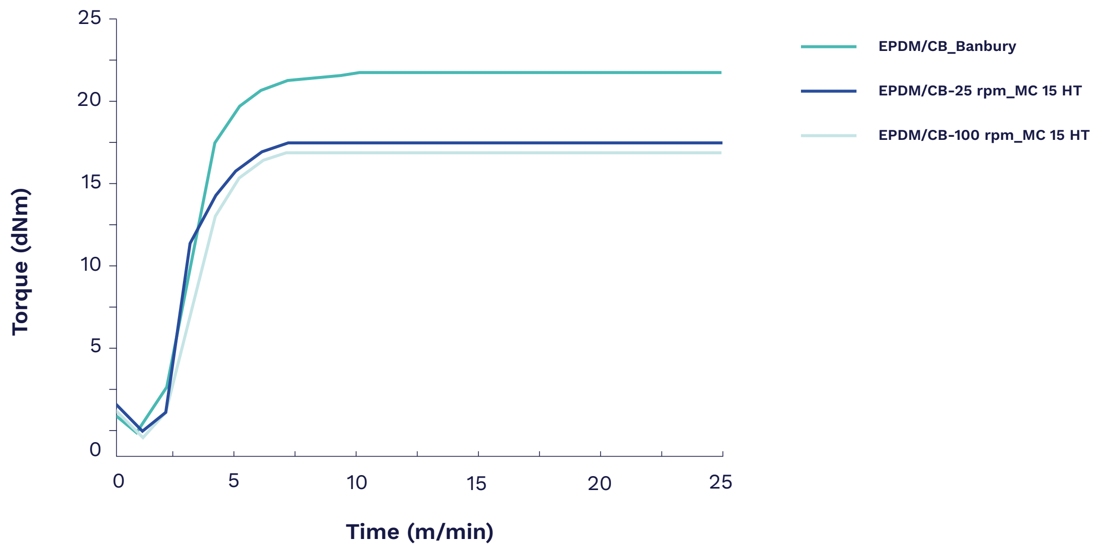


Figure 4.
Rheometer curves of the composites EPDM/CB prepared in a Banbury and an MC15 HT.

In summary, this work demonstrates the versatility of an Xplore MC15 HT.
An MC15 HT is a must have for every R&D institution and quality control lab working with plastics, resins, compounds, elastomers, or performing reactive extrusion activities.

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