The Solid State Shear Extrusion process optimization for cross-linked polymers

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Reducing the size of the solid material specially polymeric material is an important process in industry. Using small particle size provides a homogeneous feed, near fluid-like handling characteristic, a desired temperature distribution among material during processing or molding, enhanced effective surface area and good mixing.

In order to obtain polymer powders basically there are three technologies:

- Suspension or emulsion polymerization,
- Precipitation of powder from dilute polymer solutions,
- Mechanical grinding of solid polymers [12].

. The first and the second technologies are used to obtain virgin powder polymers [12], although removing emulsifier and organic solvents from produced polymers are costly and usually cannot entirely be removed (such as emulsifier). Mechanical grinding can be used for both virgin and waste polymers although the produced particles may not have exactly the same quality. Beside the flexibility of the mechanical grinding for pulverization of different polymeric material, the other advantage of the mechanical grinding is its high capacity and large scale operation capability [12]. The fundamental idea in the mechanical size reduction technique is to subject the solid material to sufficient stresses such that material breaks into small pieces. In general, mechanical pulverization falls into these categories: crushing, impacting, cutting, and pulverizing at high compression and shear force.

The idea of using high compression and shear force at the same time has been proposed by Bridgman [4], who established an apparatus with two disks that could apply hydrostatic pressure on metal sample between them and pulverize material [4]. Enikolopian extended the idea of the high pressure and shear force to pulverize polymers by using extrusion process

[8]. This process is a non-cryogenic pulverization process and it works in room temperature. Further a Bridgman anvil was used in Center of Excellence in Polymer Science and Engineering (CEPSE) at Illinois Institute of Technology (IIT) [6] to study the pulverization mechanism of the polymers. The search for finding the mechanism of the pulverization in the extrusion process for both linear and cross-linked polymers in CEPSE has been pursued till present.

In this work, we applied Solid State Shear Extrusion (SSSE) process as a mechanical size reduction technique to waste low-cross-link-density natural rubber. The objective was to optimize the SSSE process and to determine the mechanism of the pulverization during the SSSE process, particularly for low-cross-link-density natural rubber. The Particle Size Distribution (PSD) of the produced particles, which was obtained from the process, was analyzed. The first goal of this analysis was to optimize the process condition to obtain a desirable output PSD (desired average particle size, or a narrow PSD); and the second goal was to find a relation between the process conditions and the output PSD. We showed that the produced particles in this pulverization process was reproducible and the variation coefficient of such powders was less than 3 percent [2]. In order to satisfy the second goal, the produced PSD and torque were measured at different combinations of the temperature in heating zone along the screw length and rotating speed. Our results showed a non-monotonic behavior of both PSD and required torque with rotation speed (rpm) at different operating temperatures. Based on these results and the previous works on the SSSE process using polyethylene and polystyrene, it can be concluded that the pulverization mechanism depends on the molecular structure of the material, the distribution of the dispersed phase in matrix (in case of the filled polymer or blend polymers) and the nature of the interactions between the dispersed phase and the matrix. Since rubber was the matrix material that was subjected to the SSSE process, we focused on the structure of rubber, and its filler, Carbon Black (CB). We used two rubber samples with the same recipe but one with CB and the other one without CB. The difference between the average particle size of the sample with CB and without CB (around 30 percent) is an evidence for the effect of the second component on the produced particle size by the SSSE process. There are evidences that adding second polymer to the first polymer in the SSSE process changes the produced particle size same as adding filler. The magnitude of the change in the produced particle average size depends on the mechanical properties of the filler, the interaction between filler and polymer and the size distribution of the filler in polymer matrix. Pulverization also causes change in cross-link density of the polymer [7].

Based on theses results it can be concluded that the smallest length scale,

which undergoes through the breakage in the SSSE process is smaller than the size of the average distributed filler in the polymer matrix and it may be in molecular length scale, but because of high shear and compression forces and poor temperature control, the broken polymer chains react with each other rather than creating new surface and further they agglomerate. In order to examine this hypothesis, we have designed a new extended extruder, which provides high temperature control and shear force. The produced particle average size of our new extended extruder design supports our hypothesis. In principal, the SSSE process is a multi-length-scale process and capable of producing very fine particles. In order to achieve this capability, it is required to improve the process and one of the ways is modelling this process. A material model as a function of the temperature and deformation rate may be used to practice the process condition (the screw geometry, temperature change due to heating the barrel, bond breakage, and energy dissipation [5, 8, 1], rotation rate, and particulate flow) and further to improve the process. Developing a material model based on the molecular approach can provide sufficient information for such a multi-length-scale model. Our parallel work has been establishing a Gaussian slip-link model for cross-linked polymers to satisfy this requirement.

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