Studying the Pulverization Mechanism of Rubber with a Modified Design of the Solid-State Shear Extrusion Process

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ABSTRACT: Waste rubber cannot be reprocessed by melting because of its three-dimensionally crosslinked molecular structure. Mechanical size reduction is the most common method for recycling and reusing waste rubber. The modified solid-state shear extrusion (SSSE) process developed at the Illinois Institute of Technology was used for mechanical size reduction of rubber in a continuous process. In this work, an extended screw was used in conjunction with the original design of the SSSE apparatus to achieve greater rubber pulverization with a higher throughput rate and lower power consumption. The operating conditions of the modified SSSE apparatus were optimized to produce finer rubber particles that could be further used in a wide variety of applications. Controlling the residence time of rubber particles inside the pulverization section was crucial for producing a fine powder without agglomeration. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 119: 1075–1083, 2011

Key words: extrusion; particle size distribution; recycling; rubber

INTRODUCTION

Waste rubber from a variety of sources, including vehicle tires and byproducts of the rubber industry, is a growing environmental concern. Scrap rubber cannot be reprocessed by melting because of its three-dimensionally crosslinked molecular structure. However, rubber can be recycled efficiently via the breaking of the crosslinks created in the vulcanization process. Chemical, microwave, and ultrasonic treatments have been used to devulcanize waste rubber. In devulcanization, carbon and sulfur bonds break, and this yields raw rubber that can be blended with virgin rubber for a variety of applications.[1] However, in the aforementioned treatments, carbon–carbon bond breakage occurs along with carbon–sulfur bond breakage. The carbon–carbon bond breakage causes degradation of the rubber along with its devulcanization, and this results in inferior mechanical properties for products manufactured from the recycled rubber. Mechanical size reduction is an increasingly common method for recycling and reusing waste rubber because it preserves the carbon–carbon bonds.[1–3] Mechanical size reduction of waste rubber can be performed by a number of processes. In 1935, Bridgman[4] used the effects of compression and shear force to pulverize metal in an instrument known as the Bridgman anvil. The Bridgman anvil was initially used at the Center of Excellence in Polymer Science and Engineering (Illinois Institute of Technology) to study the pulverization of polymer particles.[2,3,5,6] Pulverization with the Bridgman anvil is achieved by the application of hydrostatic pressure to a particle placed between a fixed anvil and a rotating anvil.[4] Mechanical size reduction of rubber particles is generally achieved by cryogenic extrusion with liquid nitrogen, which yields a relatively uniform particle size distribution (PSD) but at higher cost.[7–9]

In 1998, Arastoopour and coworkers[10–13] developed a solid-state shear extrusion (SSSE) process for recycling rubber in a cheaper, noncryogenic, continuous way as an alternative to the cryogenic grinding process. The SSSE process has been shown to be effective in pulverizing rubber particles with high shear stress and compression forces. However, in this work, the throughput and efficiency of such a system were significantly improved with a modified SSSE apparatus using a separate screw at the end of the extruder and in a separate assembly.[5] Shahidi et al.[5] investigated the effect of the pulverization of rubber particles with a modified SSSE design with a 28–76-mm cylindrical housing. On the basis of their study, we investigated the use of the pulverization of rubber with a 14–28-mm cylindrical housing to increase the throughput of rubber particles and to reduce the erosion of the extended screw. The

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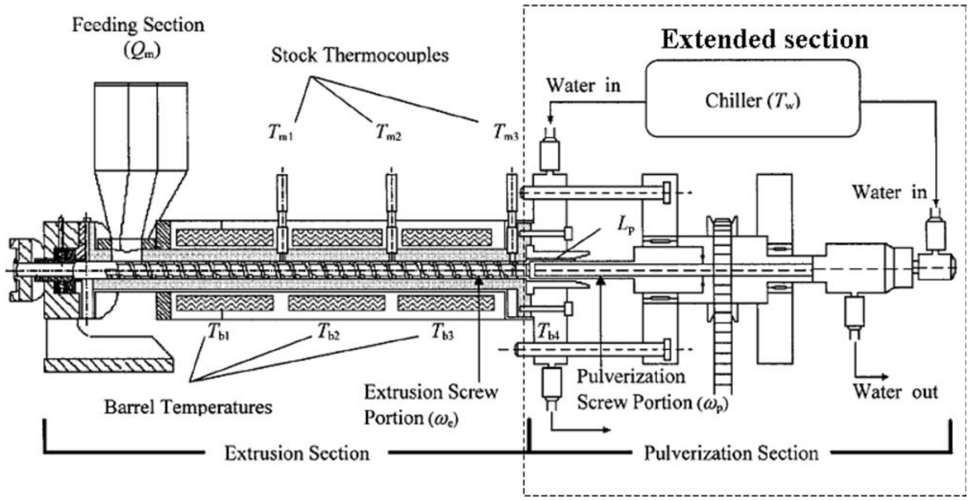

Figure 1 Schematic diagram of the extended design of the SSSE process.

impact of additional parameters such as the rotation rate of the original-design screw (ω_e) and the third-zone barrel wall temperature was also investigated.

The modified design is depicted in Figure 1. The length of the original SSSE screw has been increased as a result of the separate screw at the end. The elongated cylindrical housing surrounds the extended screw and is used in place of the barrel wall. The rubber particles are compressed between the extended screw and the cylindrical housing and are delivered at the end of the cylindrical housing. The extended design has a separate motor to control the rotation rate of the additional screw, which is independent of ω_e. A separate water-cooling circulation unit is attached to the extended screw to remove the heat of pulverization of the rubber particles. It is well known that the pulverization of rubber particles by the SSSE process is more efficient with a 5 : 1 compression ratio (CR) of the screws versus lower CRs of 3 : 1 and 1.5 : 1.[2,3,14] However, the use of a 5 : 1-CR screw in extrusion entails higher power requirements and higher maintenance costs because of screw erosion under stress. We used a 1.5 : 1-CR screw for most of the experimental work. The shift of the pulverization phase of the recycling process to the extended section of the extruder provides higher cooling efficiency, reduced maintenance costs, and lower power requirements.[5]

EXPERIMENTAL

Materials

Rectangular slabs of rubber sheets obtained from an outside vendor company were cut into small square pieces with a shear cutter. These small rubber pieces were then fed into a Cumberland (Cumberland Engineering Corporation located in New Berlin, WI) granulator to obtain rubber granules. These granules were separated via sieving into fractions of three different sizes: 4.00–6.3, 2.00–4.00, and less than 2 mm. The 2.00–4.00-mm rubber granules were used as the raw feed for the SSSE process. Table I presents the composition of the rubber used in this work.

Apparatus

In this work (similar to that of Shahidi et al.[5]), the modified SSSE design with an extended pulverization section was used, as illustrated in Figure 1. The extrusion section depicted in Figure 1 is part of the original design of the SSSE process manufactured by C. W. Brabender (South Hackensack, NJ) with an attached intelligent interface (model PL2000). Bilgili et al.[3,14] and Eskandari and Arastoopour[2] discussed the original design in detail. In the original design, the extrusion section consists of a feeding section, an extrusion screw, and three separate zones that are temperature-controlled independently by heating/cooling elements surrounding the barrel wall. Air is generally used as the heating medium, and water is used as the cooling medium in the original design. An attached central interface controls ω_e and the barrel wall temperatures of the first, second, and third zones of the extruder (T_{b1}, T_{b2}, and T_{b3}, respectively). The original-design screw achieves a maximum rotation rate of 100 rpm and can withstand a maximum torque of 100 Nm within the extruder barrel. The diameter of the original-design screw increases along the length of the screw such that the flights on the screw push the material forward.[2] The first two zones of the extruder heat the rubber granules, and the screw flights push the granules forward along the extruder barrel. The clearance between the screw and the barrel wall continues to decrease and is lowest at a higher CR, so higher compressive and shear

TABLE I
Composition of the Rubber Used in the SSSE Pulverization Experiments

Component	wt %
Natural rubber CV60	55.34
Zinc oxide	2.77
Stearic acid	3.32
N-351 carbon black	22.69
2,2,4-Trimethyl-1,2-dihydroquinoline	0.55
Okerin 7666 (wax)	1.11
N-(1,3-Dimethylbutyl)-*N*′-phenyl-*p*-phenylenediamine	1.11
AC PE (A-C$^{(R)}$ Polyethylene)	1.11
Aromatic oil	9.96
Insoluble sulfur	1.38
N-Oxydiethylene benzothiazole sulfenamide (Akrochem)	0.55
Tetramethylthiuram disulfide	0.11

forces can be applied in the third zone to pulverize the rubber granules. Higher CRs of the extruder screw yield finer particles in the pulverization process, albeit at a higher shear force and a higher torque.

In this work, the modified SSSE apparatus was used; it includes an additional screw encased in a cylindrical housing to increase the efficiency of the extrusion and pulverization processes. The clearance between the cylindrical housing and the screw is kept very low to achieve higher compression and shear forces, and this results in improved pulverization of the rubber granules. The CRs of the original screw and extended-design screw were kept at 1.5 : 1 and 5 : 1, respectively, for most of the experiments. The original-design screw and the extended-design screw can rotate independently of each other by separate motors. The rotation rate of the extended-design screw (ω_p) varies between 1 and 150 rpm. The extended section of the SSSE process employs a circulating water-cooling system to remove heat efficiently from the pulverized particles to avoid agglomeration of the pulverized particles. The cooling system consists of an inner cooling tube in the extended screw portion along with a cooling jacket surrounding the cylindrical housing. A thermocouple, attached to the outer surface of the cylindrical housing, measures the temperature of the pulverization section (T_p). Figure 2 illustrates a cross-sectional view of the extended-design screw attached to the original design of the single-screw extruder with the help of a cylindrical housing. The rubber particles first pass between the barrel wall and the original-design screw and then between the cylindrical housing and the extended-design screw. The cylindrical housing is not a straight, hollow cylinder but is conical at the end to allow for ease of delivery of the rubber particles. The dimensions of the cylindrical housing and extended-design screw are given in Table II. The effective length of the cylindrical housing participating in the pulverization (L_e) is the length of the straight cylinder. The conical part does not participate in the pulverization of rubber particles. The screws used in the extended design either have flights or do not have flights, and the screw in the original design has flights.

Experimental procedure and measurements

The pulverization of rubber granules was conducted under several different operating conditions of the SSSE process. To understand the effects of external parameters on the pulverization of rubber, certain independent and dependent variables were considered. The independent variables for the process were L_e, T_{b1}, T_{b2}, T_{b3}, CR of the original-design screw, the presence or absence of flights on the extended screw, ω_e, and ω_p. The dependent variables for the process were the material temperatures in the first, second, and third zones of the original design (T_{m1}, T_{m2}, and T_{m3}, respectively), T_p, the torque on the screw in the extrusion section (τ), the

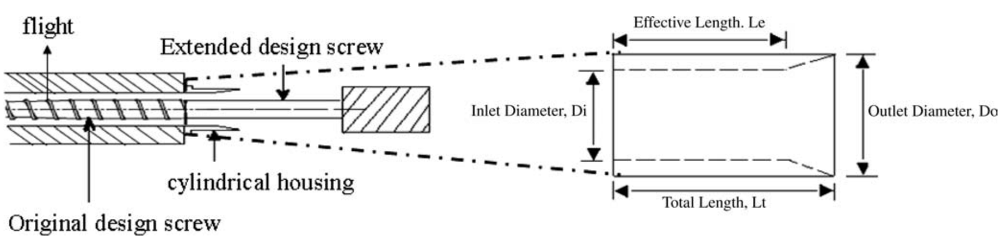

Figure 2 Cross-sectional view of the extended-design screw attached to the original design of the SSSE process.

TABLE II
Dimensions of the Cylindrical Housing and Extended-Design Screw

Parameter	Long cylindrical housing	Short cylindrical housing
Inlet diameter (mm)	19.1	19.1
Outlet diameter (mm)	21.5	21.5
Total length (mm)	46	28
Effective length (mm)	28	14
Screw diameter (mm)	17.32	17.32

material mass flow rate (Q_m), and the PSD of the final pulverized rubber. Table III lists the different processing conditions that were used to pulverize the rubber. The independent variables were varied systematically to optimize the pulverization process for rubber particles and to assess the effects of independent variables on the dependent variables. The effects of changes in the CR of the original-design screw (tests T101–T103), the presence or absence of flights on the extended screw (tests T104 and T105), the length of the cylindrical housing (tests T106–T108), the rotation rates of the extrusion section screw (tests T108, T111, and T112) and pulverization section screw (tests T108–T110), and T_{b3} (tests T108 and T113–T115) were analyzed with respect to the PSD of rubber. Each experiment (tests T101–T115) was repeated three times to evaluate the reproducibility of the SSSE process.

Particle size analysis

Pulverized powder obtained from the SSSE process was sieved with a W.S. Tyler (Mentor, Ohio) model RXS86 sieve shaker. The system used a set of sieves with different pore diameters (1400, 850, 600, 425, 250, 150, and 90 μm). The sieves were arranged from top to bottom with decreasing pore size. An electric motor attached to the sieve shaker created circular and hammering motions to break rubber agglomerates. Each sample was sieved for 45 min, and the particles obtained from each sieve were weighed and converted into weight percentages for each size range. The PSD curve was obtained via the plotting of weight percentages on the y axis against the average particle diameters on the x axis. The average particle diameter was calculated from the average pore diameter of the sieves above and below the position at which the particles were collected. The cumulative weight percentage of the particles in each sieve was determined by the addition of the average weight percentages of particles in all sieves below it. The cumulative PSD was plotted to study the effect of the agglomeration of rubber particles on the pulverization process.

RESULTS AND DISCUSSION

Effect of the CR of the original design

The effect of the CR of the original-design SSSE process on the PSD was studied extensively by Bilgili et al.[3] This section describes the effect of the CR of the original-design screw on the PSD in the presence of the extended design. Figure 3(a,b) shows the PSD of the pulverized rubber resulting from tests T101–T103 conducted with 5 : 1, 3 : 1, and 1.5 : 1 CRs of the original-design screw. The extent of pulverization was highest with a 5 : 1-CR screw in the original design, with a shift in the PSD toward the left indicating the generation of finer particles. The extent of pulverization increased with CR because of higher compression and shear forces caused by reduced clearance between the extrusion screw and the barrel wall. On the other hand, the PSD shifted toward coarser particles when a lower compression

TABLE III
Operating Conditions of the SSSE Process in the Rubber Pulverization Tests

Test	SSSE design	L_e (mm)	T_{b1} (°C)	T_{b2} (°C)	T_{b3} (°C)	CR	Flight	ω_e (rpm)	ω_p (rpm)
T101	Extended	14	120	80	33	5 : 1	No	75	150
T102	Extended	14	120	80	33	3 : 1	No	75	150
T103	Extended	14	120	80	33	1.5 : 1	No	75	150
T104	Extended	14	120	80	33	1.5 : 1	No	75	110
T105	Extended	14	120	80	33	1.5 : 1	Yes	75	110
T106	Original	0	120	80	33	1.5 : 1	—	75	—
T107	Extended	14	120	80	33	1.5 : 1	No	75	75
T108	Extended	28	120	80	33	1.5 : 1	No	75	75
T109	Extended	28	120	80	33	1.5 : 1	No	75	110
T110	Extended	28	120	80	33	1.5 : 1	No	75	150
T111	Extended	28	120	80	33	1.5 : 1	No	25	75
T112	Extended	28	120	80	33	1.5 : 1	No	50	75
T113	Extended	28	120	80	25	1.5 : 1	No	75	75
T114	Extended	28	120	80	45	1.5 : 1	No	75	75
T115	Extended	28	120	80	65	1.5 : 1	No	75	75

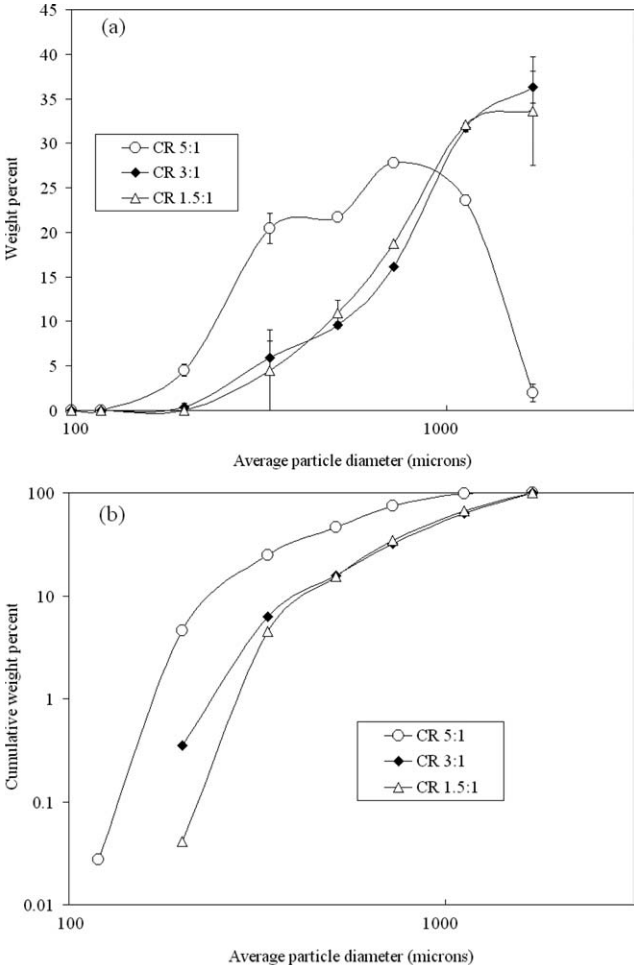

Figure 3 Effect of the CR of the original-design screw on (a) the PSD and (b) the cumulative PSD (tests T101–T103).

screw was used in the original design. With 3 : 1- and 1.5 : 1-CR screws, there was negligible pulverization in the original design as well as a small amount of pulverization in the extended design. Although the extended design was equipped with a 5 : 1-CR screw, L_e was small and did not contribute to reducing the particle size significantly.

The variation of dependent variables such as T_{m2}, T_{m3}, T_p, τ, and Q_m with the CR of the original-design screw is shown in Figure 4. T_{m2} and τ of the original-design screw increased with CR of the screw increasing. The clearance between the barrel wall and screw decreased at higher CRs, and this resulted in an accumulation of particles, with higher frictional and compression forces increasing the material temperature and power consumption (τ). At low CRs of 3 : 1 and 1.5 : 1, the clearance between the screw and barrel wall was high, and this resulted in an easy flow of material with reduced material temperatures and τ values and coarser particles. Q_m of the rubber particles decreased with higher CRs of the screw because of higher resistance to the flow of rubber. T_{m3} and T_p did not change significantly with changes in the CR of the screw because of efficient water cooling in the third zone and in the pulverization section.

Finer particles were obtained with higher CR screws in the original design. However, the power consumption and τ values of the screw under such conditions were significantly higher. The screw in the original design eroded heavily at high CRs, and this resulted in higher maintenance and replacement costs. The original-design screw, driven by a motor attached by a safety pin, has a τ limit of 100 Nm and breaks the pin at higher τ values to avoid motor damage. Operation of the extruder with a 5 : 1-CR screw resulted in very frequent pin breakage because of τ overshoots. Tests T103–T115 were conducted with a 1.5 : 1-CR screw in the original design (used as the extrusion section), whereas the extended-design screw was used as the pulverization section. Therefore, in the modified SSSE process, the pulverization was shifted to the extended design even with a lower CR screw in the original design. This process also reduced the maintenance cost of the original-design screw while improving rubber pulverization.

Effect of the screw design

Figure 5 compares the PSD of rubber obtained with screws with and without flights in the extended design. Table IV lists the dimensions of the screws with and without flights. L_e was kept at 14 mm, and the screws with and without flights were used in tests T104 and T105. Approximately 40% of the rubber particles had size fractions between 1400 and 2000 μm. L_e was not sufficient to pulverize the rubber particles into a fine powder. Figure 5 shows no significant difference in the PSD of pulverized rubber because of a lack of sufficient pulverization in

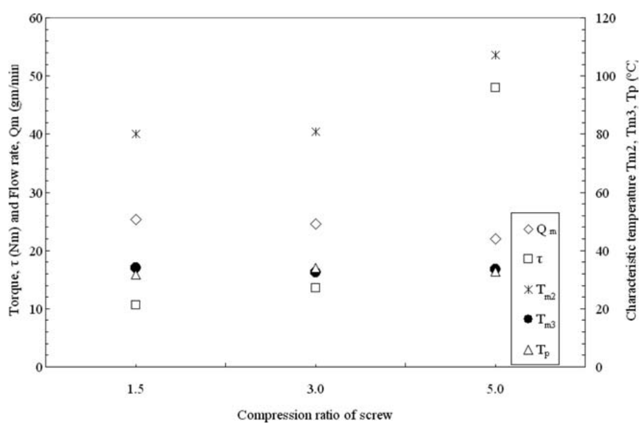

Figure 4 Effect of the CR of the original-design screw on the material temperatures in the extrusion and pulverization sections, Q_m of rubber particles, and τ of the original-design screw (tests T101–T103).

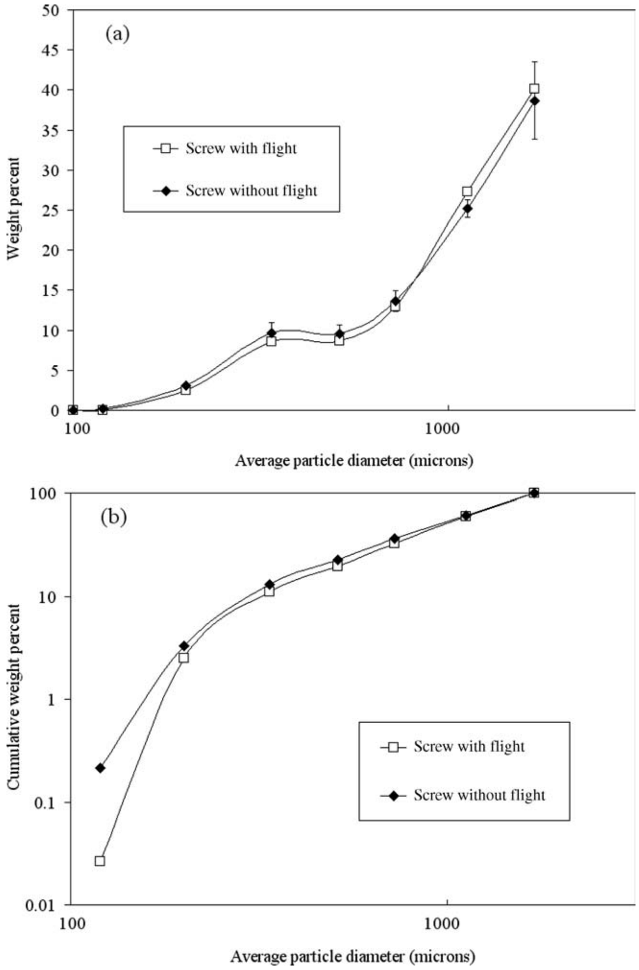

Figure 5 Effect of the presence and absence of flights in the extended-design screw on (a) the PSD and (b) the cumulative PSD (tests T104 and T105).

the extended design at lower L_e values. The pulverization was slightly efficient with a screw without a flight because the absence of flights caused the particles to accumulate and break further.

Effect of the length of the cylindrical housing in the extended design

To determine the effect of L_e in the extended design of the SSSE process, two cylindrical housings of various lengths were used (see tests T107 and T108). The size distribution of the pulverized particles thus obtained was compared to that obtained by the original-design process (see test T106) under similar process conditions (Fig. 6). The extent of pulverization was highest with a 28-mm-long cylindrical housing and lowest with the original design of the SSSE process. This comparison was reported by Shahidi et al.[5] with a longer cylindrical housing. The pulverization was improved with a longer cylindrical housing, but this could cause erosion of the screw and higher maintenance. In the original design of the SSSE process, the CR of the original-design screw was low (1.5 : 1) and thus yielded negligible pulverization. More than 80% of the produced rubber particles were 1000 μm in size or coarser. The increase in the length of the cylindrical housing increased the residence time of rubber particles in the pulverization section and produced fine particles. With a 28-mm-long cylindrical housing, a higher fraction of finer pulverized particles followed a bimodal distribution. However, very low pulverization was achieved with a 14-mm assembly because of the insufficient residence time of the

TABLE IV
Dimensions of the Extended-Design Screw

	Screw diameter (mm)	Flight diameter (mm)
Screw with flights	17.32	18.97
Screw without flights	17.32	—

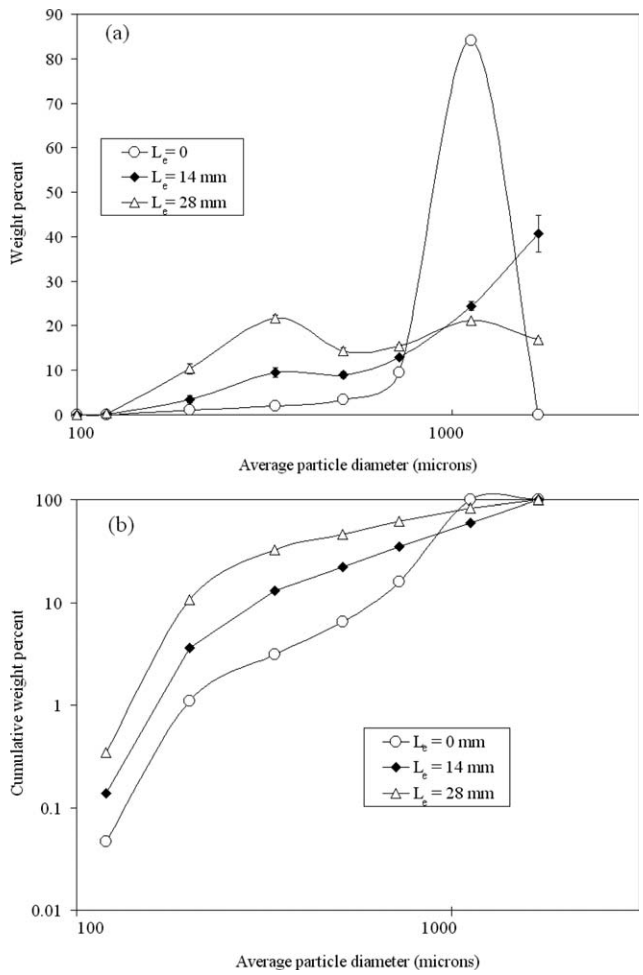

Figure 6 Effect of the length of the cylindrical housing on (a) the PSD and (b) the cumulative PSD (tests T106–T108).

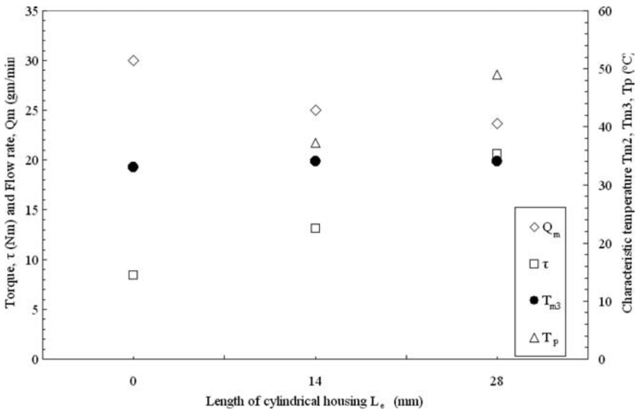

Figure 7 Effect of the length of the cylindrical housing on the material temperatures in the extrusion and pulverization sections, Q_m of rubber particles from the extruder, and τ for the original-design screw (tests T106–T108).

rubber particles in the pulverization section, as shown in Figure 6.

Figure 7 shows the effects of various lengths of the cylindrical housing on the dependent variables used in this work (tests T106–T108). τ for the original-design screw increased with an increase in the length of the cylindrical housing. For the original-design screw without the extended design (test T106), τ was low because of a lower CR of the screw and no additional resistance to the flow of rubber particles; this resulted in a higher Q_m value. In the extended design, the resistance to the flow of the rubber material was high because of an additional pulverization section with a low Q_m value and a higher τ value for the original-design screw. T_{m3} remained constant for different lengths of the cylindrical housing because of efficient water cooling. T_p was high with a 28-mm-long cylindrical housing because of the longer residence time of rubber particles and increased compression and shear forces.

Effect of ω_p

To investigate the effect of ω_p on the PSD of pulverized rubber and other dependent variables of the SSSE process, tests T108–T110 were conducted; the results are shown in Figures 8 and 9. ω_e was kept constant at 75 rpm. ω_p controlled the residence time of the rubber particles in the pulverization section and the extent of the compression and shear forces on the material. The residence time of the particles in the pulverization section was increased by the reduction of ω_p because the movement of particles along the length of the screw was reduced. However, at a very high residence time (a low rotation rate), the agglomeration of particles yielded coarser particles. At 150 rpm, the rubber particles flowed freely from the SSSE process with a very low residence time; this resulted in lower τ, lower T_p, and higher Q_m values. The extent of pulverization was higher at the screw rotation rate of 110 rpm in comparison with the rates of 150 and 75 rpm. Figure 8(a) shows that the average particle size of the rubber particles was reduced at 110 rpm versus 150 rpm because of the higher residence time and at 110 rpm versus 75 rpm because of less agglomeration of particles. The cumulative PSD indicated that a larger amount of the finest particles was produced at 75 rpm, as shown in Figure 8(b). The PSD at 110 rpm showed a fine powder between 300 and 700 μm. The cumulative size distribution of particles obtained at 75 rpm with the extended-design screw showed particle agglomeration. T_p was higher at 110 rpm because of increased pulverization and higher frictional and shear forces. τ for the original-design screw was higher at 75 rpm with the extended screw versus 110 and 150 rpm because of the accumulation of rubber particles at low ω_p values, as shown in Figure 9. There was no significant change in T_{m3} with various ω_p values.

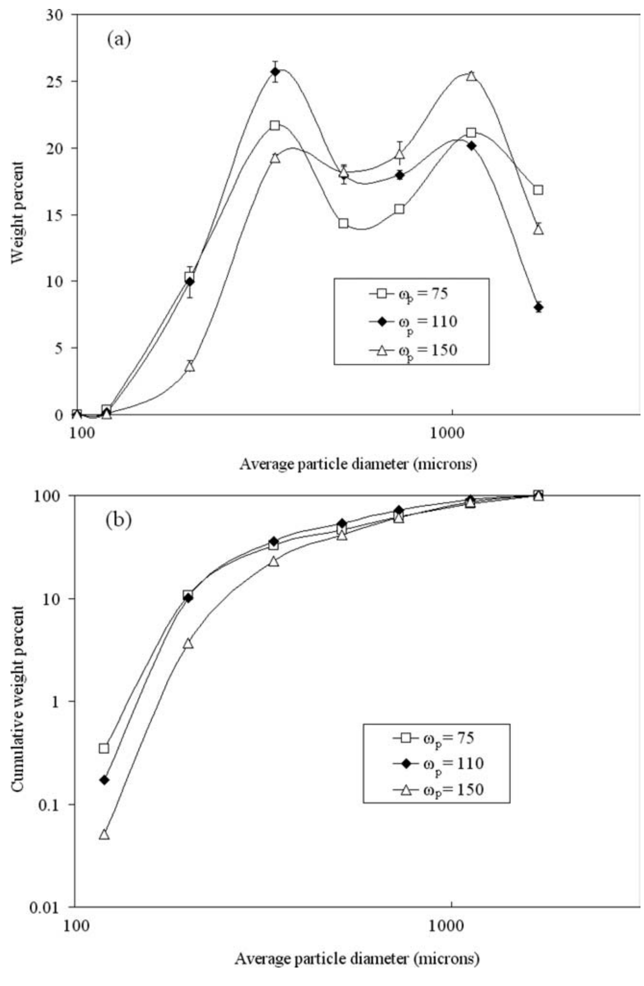

Figure 8 Effect of ω_p on (a) the PSD and (b) the cumulative PSD (tests T108–T110).

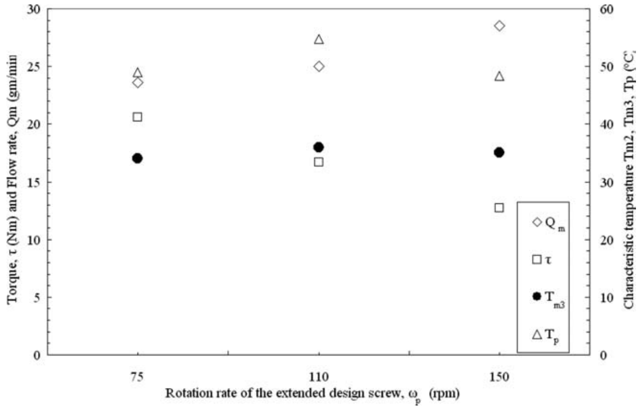

Figure 9 Effect of ω_p on the material temperatures in the extrusion and pulverization sections, Q_m of rubber particles from the extruder, and τ for the original-design screw (tests T108–T110).

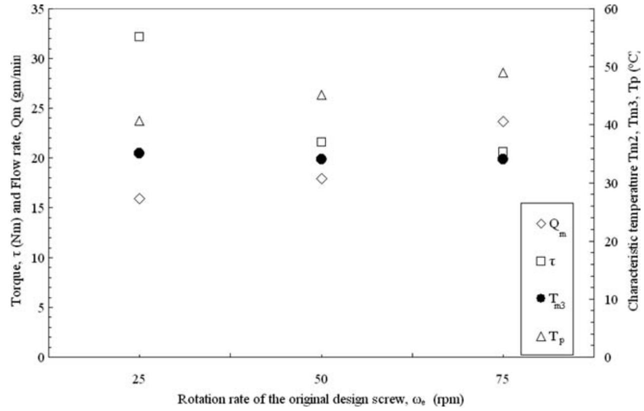

Figure 11 Effect of ω_e on the material temperatures in the extrusion and pulverization section, Q_m of rubber particles from the extruder, and τ for the original-design screw (tests T108, T111, and T112).

Effect of ω_e

Figures 10 and 11 show the effects of ω_e on the PSD of pulverized rubber and on other dependent variables of the SSSE process (see tests T108, T111, and T112). ω_p was kept constant at 75 rpm. ω_e controlled the residence time of rubber particles in the extrusion section. At 75 rpm, the rubber particles flowed freely from the extrusion section with a very low

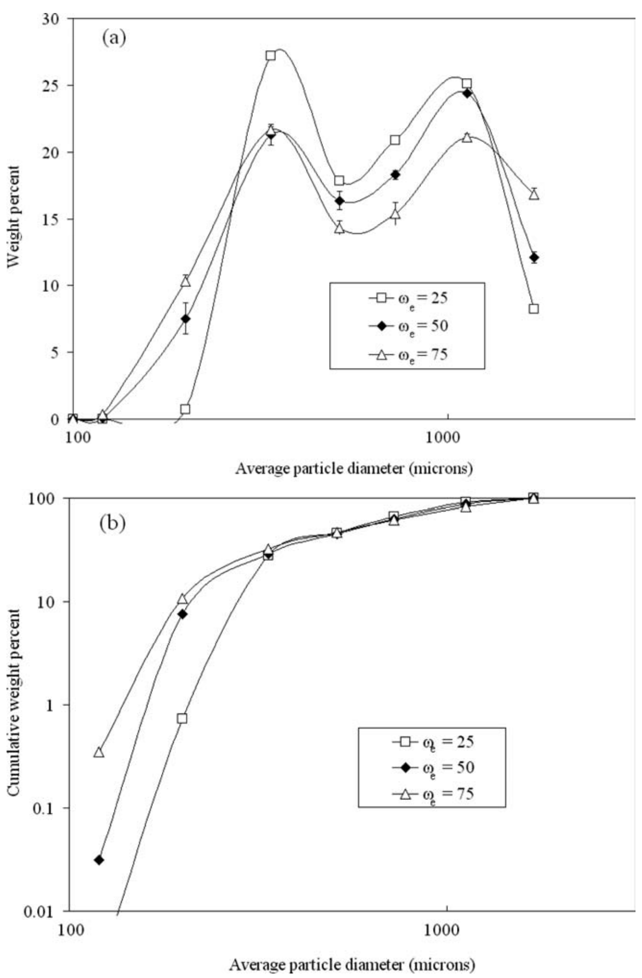

Figure 10 Effect of ω_e on (a) the PSD and (b) the cumulative PSD (tests T108, T111, and T112).

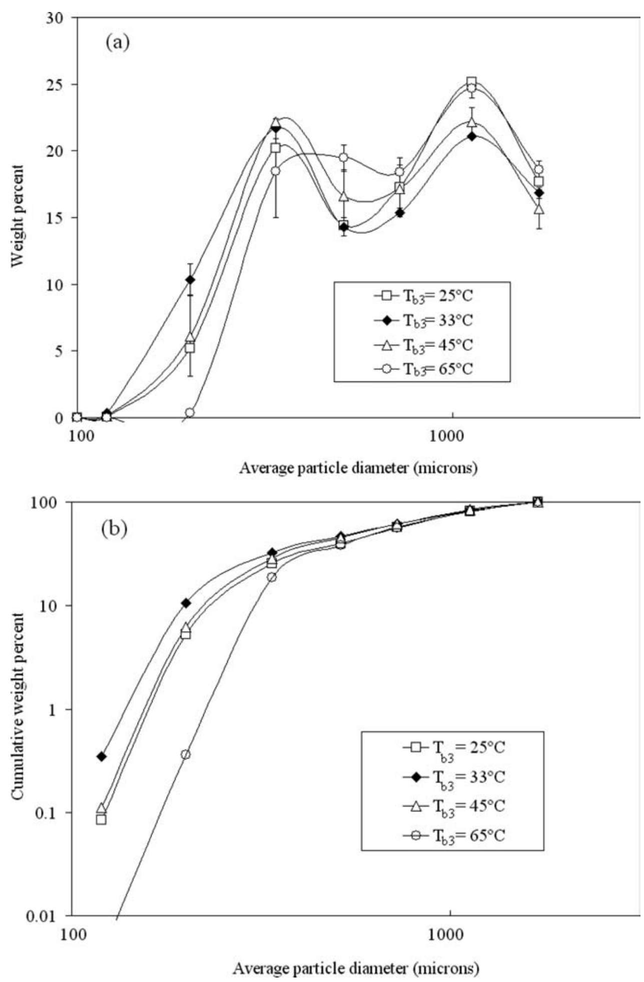

Figure 12 Effect of T_{b3} on (a) the PSD and (b) the cumulative PSD (tests T108 and T113–T115).

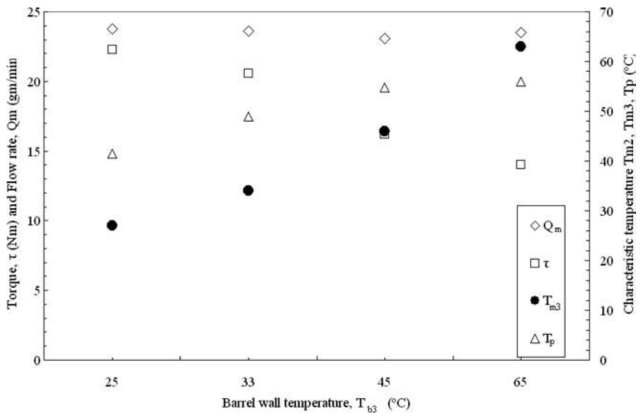

Figure 13 Effect of T_{b3} on the material temperatures in the extrusion and pulverization sections, Q_m of rubber particles from the extruder, and τ for the original-design screw (tests T108 and T113–T115).

residence time; this resulted in lower τ and higher Q_m values. An increase in T_p due to higher frictional forces caused by a higher rotation rate was noticed. Although the extent of pulverization seemed high at 25 rpm from the PSD curve with a high weight fraction toward the left in the bimodal distribution, the cumulative PSD showed that the extent of pulverization was very high at 75 rpm. Figure 10 shows that the fine particles were produced at 75 rpm because of higher compression and shear forces. τ for the extrusion section screw was high at 25 rpm because of the accumulation of rubber particles at low rotation rates, as shown in Figure 11. Higher accumulation could have caused agglomeration of finer particles. T_{m3} did not change significantly at different ω_e values because of efficient water cooling of the third zone.

Effect of T_{b3} of the original design

To investigate the effects of T_{b3} on the PSD of rubber and other dependent variables of the SSSE process, tests T108 and T113–T115 were conducted, and the results are shown in Figures 12 and 13. T_{b3} was varied from 25 to 65°C. Figure 12(a) shows that the extent of pulverization did not change significantly with changes in T_{b3}. Each experiment was conducted three times, and the error bars in the graphs indicate the standard deviations of the average PSDs. The error bars for the PSDs show that the effect of T_{b3} was not significant in changing the compression or shear forces on the rubber particles. Rubber particles experience chemical and physical stress relaxation effects at higher temperatures.[3] The cumulative PSD showed nonmonotonic behavior with finer particles produced at 33°C versus other temperatures. Figure 13 shows that an increase in T_{b3} from 25 to 65°C reduced τ for the extrusion section screw because of stress relaxation of the rubber particles. T_p was consistent with T_{b3} of the original-design screw and increased with increasing temperatures. Changing T_{b3} did not change Q_m of the rubber particles significantly.

CONCLUSIONS

We studied the pulverization of rubber with Shahidi et al.'s[5] modified SSSE process. The original SSSE design has been modified to use a low-CR screw in conjunction with an external screw that can be rotated and controlled independently of the original extruder. The original design acts as an extrusion section, and the extended design acts as a pulverization section. The new approach reduces erosion and maintenance costs and yields lower power consumption and τ requirements for the original-design screw and the extended-design screw. The modified design of the SSSE process also increases the throughput of the entire SSSE process while producing finer particle size fractions and improved pulverization efficiencies. The residence times of the rubber particles in the pulverization section and the extruder section are crucial in controlling the PSD of the pulverized rubber particles. The residence time is controlled by the variation of L_e, ω_e, and ω_p. Increasing the residence time of rubber particles increases the compression and shear forces and produces finer fractions. However, optimum control of the residence time of rubber particles in the pulverization section is needed to avoid agglomeration.

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