EFFECTS OF COMPOSITE PROCESSING METHODS ON WOOD PARTICLE DEVELOPMENT AND LENGTH DISTRIBUTION: CONSEQUENCES ON MECHANICAL PROPERTIES OF WOOD–THERMOPLASTIC COMPOSITES

*Hassine Bouafif**
Research Associate

Ahmed Koubaa†
Professor
Chaire de recherche du Canada sur la valorisation
la caractérisation et la transformation du bois
Université du Québec en Abitibi-Témiscamingue
445 BD de l'Université Rouyn-Noranda
Québec, J9X5E4, (QC) Canada

Patrick Perre
Professor
AgroParisTech, INRA, UMR1092, LERFOB
14 rue Girardet, 54000
Nancy, France

Alain Cloutier†
Professor
Centre de Recherche sur le Bois
Université Laval, Pavillon Eugène-Kruger
Québec, G1K7P4, (QC) Canada

(Received August 2009)

Abstract. The relationship between structure and properties of high-density polyethylene (HDPE) filled with wood particles and processing techniques—injection molding, compression molding, and extrusion—was investigated. Wood particles were hammer-milled, sieved, and compounded into pellets at 35% by weight with HDPE using a twin-screw extruder. Coupling agent (ethylene-maleic anhydride copolymer) was added at 2% by wood filler weight. The pellets were used to produce test samples using the three processing techniques. The sensitivity of jack pine and several other wood particles (eastern white cedar, black spruce, and jack pine bark) to composite processing was analyzed. Bark particles showed higher propensity to generate fines than wood particles, possibly because of a higher thermal sensitivity. The major reduction in mean particle length was found to occur in the compounding process. Extrusion and injection molding contributed to particle length reduction to a lesser extent. Conversely, compression molding did not cause significant damage to wood particles. Stiffness and strength increased linearly with weight-averaged length.

Keywords: Wood, fiber length, composites, compounding, injection molding, compression molding, extrusion.

INTRODUCTION

The relationships among processing methods, structures, and properties of wood-fiber or flour–polymer composites have been widely studied (Xanthos 1983; Park and Balatinecz 1997; Barbosa and Kenny 2000; Balasuriya et al 2001; Kim et al 2004). Balasuriya et al (2001) investigated the structure–property relationship of wood-flake/high-density polyethylene (HDPE)

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* Corresponding author: hassine.bouafif@uqat.ca
† SWST member

composites in relation to the matrix agent melt-flow behavior and processing technique, namely twin-screw compounding and mechanical blending. It was found that the structure of the twin-screw compounded composites, based on medium melt-flow-index HDPE, consistently achieved better flake wetting and distribution and therefore produced higher mechanical properties than mechanically blended composites or twin-screw compounded composites with a low melt-flow index. Xanthos (1983) reported that the compounding procedure improves adhesion through the use of a coupling agent and at the same time produced minimum degradation of the wood flour. Stark et al (2004) studied the effect of the processing method (ie injection molding, extrusion, or extrusion followed by planing) on the surface and weathering characteristics of wood-flour/HDPE composites. It was concluded that lightness after weathering was not dependent on the manufacturing method. However, composites with more wood component at the surface (ie planed samples) exhibited a larger percentage of total loss in flexural modulus of elasticity and strength after weathering.

Compounding is a preprocess in thermoplastic injection molding, whereby the polymer and wood filler are mixed together by counterrotating screws in an extrusion machine. The compound is then extruded and chopped into pellets, which are ready for injection molding. The compounding process directly influences the compounding quality of wood–polymer blends and the coupling agent performance in the resultant composites (Lu et al 2005). However, the compounding of thermoplastics with wood flour or natural fibers presents many drawbacks because of the temperature and shear sensitivity of the thermoplastic matrix, the presence of moisture in the cellulosic fibers, and the critical process temperature limitations of the wood filler. Ali et al (2003) reported that during mixing and compounding, the higher shear stresses that developed in a counterrotating conical intermeshing twin-screw extruder that was used to disperse the fibers caused a substantial reduction in both fiber diameter and length. Fu and Lauke (1996) verified that the postcompounding processes, namely extrusion and injection molding, also led to fiber damage, and the end result was an asymmetric fiber length distribution with a tail at higher fiber lengths.

In contrast to numerous studies on inorganic fillers, there are few studies that document the effect of processing conditions on fiber-length distribution and mechanical properties of wood-fiber reinforced thermoplastic matrices. Bigg (1985) found that a single-screw compounder did less damage to inorganic filler than a twin-screw compounder under similar processing conditions when the polymer was in pellet form. Conversely, when the polymer was supplied as a powder, the twin-screw compounder did less damage to fiber. Subsequently, Grillo et al (1993) proved that compounding short glass fibers in a corotating twin-screw extruder required process design optimization (ie screw configuration assembly and die) to yield a material with suitable mechanical properties. They also found that certain physical mechanical properties such as notched Izod were highly correlated with the degree of damage to the glass fibers, whereas flexural properties were a function of preserved glass length as well as fiber–matrix adhesion. The study concluded that reduction in glass abrasion requires a very gentle screw configuration and minimal average shear rate in the extruder and die, whereas good adhesion is promoted by complete glass wetting, requiring an effective mixing screw and die restriction. Recently, Yilmazer and Cansever (2002) studied the effect of screw speed and feed rate (in different combinations). Results showed that the average fiber length decreased when the shear rate increased through screw speed and/or feed rate. Consequently, impact strength, tensile modulus, and tensile strength increased, but the elongation at the breaking point decreased with a decrease in the average fiber length. In addition, lower shear history and melt temperature during the primary compounding operations of such composites generally result in better mechanical properties of the finished product (Bledzki and Faruk 2004;

Kasliwal and Jones 2004). Increased screw rotational speed or reduced processing temperature has been shown to reduce fiber dimensions (Fu et al 2001; Ali et al 2003; Bledzki and Faruk 2004).

As mentioned, the separation processes of natural fibers and the composite processing method influence fiber degradation and hence fiber-length distribution (FLD). FLD is controlled by a number of factors, including original fiber length, fiber content, die and mold geometry, and processing conditions (Fu and Lauke 1996; Fu et al 1999, 2001). Because the mechanical properties such as strength, elastic modulus, and fracture toughness of short-fiber reinforced thermoplastics are critically dependent on FLD (Fu and Lauke 1996; Fu et al 1999), several methods have been used to accurately characterize the geometric distribution of fibers. Characterization of a sample for FLD before processing is rather straightforward. Nevertheless, it is much more complicated when fibers are blended into a thermoplastic matrix. To study the relationships between the mechanical properties and FLD of short-fiber reinforced thermoplastics, Fu et al (2001) used a statistical cumulative fiber length distribution model, whereas Suzuki (2003) used a probabilistic approach based on the projection length of the fiber to the composite surface. Clemons et al (1999) successfully characterized the FLD of postprocessing residual fibers using solvent extraction followed by optical measurement techniques. Results of these studies indicated that fiber lengths were reduced by one-half when they were compounded in a high-intensity thermokinetic mixer followed by injection molding. Recently, Balasuriya et al (2001) used an optical microscope with an image analyzer to determine the wood-flake length/width distribution after processing. They also measured length and width distributions before compounding by projecting images of the flakes onto a large screen.

Because the intrinsic characteristics of wood fillers vary with the chemical composition of the species involved, the vulnerability of different wood species to processing conditions can be expected to vary. A few studies have highlighted the relationship between variability in wood fiber structure and damage levels during mixing. Neagu et al (2006) investigated the stiffness contribution of various wood fibers to the resulting composite materials. The study concluded that unbleached fibers are more suitable than bleached fibers for use as stiffening reinforcement. A correlation between lignin content and fiber longitudinal Young's modulus was observed, and the optimal lignin content range at which fiber stiffness attains a maximum was identified for softwood kraft fibers. Moreover, they found that hardwood fibers showed a higher reinforcement potential than softwood fibers.

This article presents the correlations among processing, structure, and properties of wood-particle reinforced HDPE composites. Initially, the study explores the vulnerability of wood filler to the environment prevailing during polymeric processing by comparing the susceptibility of different softwood particles to the damage exerted by high shear stresses that developed during processing. The physical properties of the finished composites were analyzed for the degree of particle damage. Finally, the relationship among processing techniques (compounding, extrusion, and molding), structure, and some mechanical properties of wood-particle reinforced HDPE are highlighted.

MATERIALS AND METHODS

Materials

Four types of wood particles were investigated: eastern white cedar (*Thuja occidentalis*) sawdust, jack pine (*Pinus banksiana*) residue divided into wood sawdust and bark shavings, and black spruce (*Picea mariana*) sawdust. These were supplied by softwood sawmills located in the Abitibi-Temiscaming region of Quebec. Wood sawdust and bark shavings were hammer-milled into particles at FPInnovations–Forintek Division located in Quebec City. The particles were screened and classified into three mesh size groups (Table 1) using an oscillating

Table 1. *Classification of hammermilled particles.*

Mean class	Class interval	
	Mesh	µm
24 mesh	[20, 28]	[850, 600]
42 mesh	[35, 48]	[425, 300]
70 mesh	[50, 100]	[300, 150]

multideck-type screen classifier. At this stage, the MC of the particle filler was about 10.5%. The wood particle fillers were not extracted.

HDPE polymer (Goodfellow Corp) was used as the matrix. This is a semicrystalline material (typically 70 – 80%) with 950 kg/m^3 density, 9.0 g/10-min melt index, and 135°C melting point. Ethylene-maleic anhydride copolymer (MAPE, A-C® 575A), supplied by Honeywell (Minneapolis, MN), was used as the coupling agent. It has a 920-kg/m^3 density and a 104 – 107°C melting point. A modified fatty-acid ester (STRUKTOL® TPW 113) was used as an internal lubricant to facilitate extrusion.

Composites Preparation

Compounding. Similarly sized wood particles were compounded into pellets at 35 wt% HDPE using a Coperion Werner and Pfleiderer ZSK-25 WLE corotating twin-screw extruder. The coupling agent was 2% of the total wt% (HDPE + wood particles) for all experimental design blocks. Wood particles, coupling agent, and polymer were fed into Zone 1 of the extruder separately using a Colortronic GmbH gravimetric feeder. Barrel temperatures of the four zones were 180, 180, 180, and 190°C from infeed to the die zone. Screw speed was 240 rpm and melt pressure at the die varied 1.5 – 2.5 MPa, depending on wood particle content. Vacuum venting (–40 kPa) at Zone 3 was used to minimize volatile extractives. Residence time was 240 s and material feed rate was 15 kg/h. Extrudates were air-cooled and pelletized into a nominal 5-mm pellet length.

Injection molding. Injection molding experiments were carried out using a reciprocating-screw injection molding machine (Toshiba ISE60P) with a maximum clamping force of 530 kN. Machine settings are shown in Table 2. The mold was opened and samples were cooled at room temperature.

Table 2. *Reciprocating-screw injection molding machine settings.*

Mold temperature: fixed/mobile	38/38°C
Injection pressure	900 kPa
Injection pressure time	10 s
Hold pressure	900 kPa
Hold pressure time	4 s
Barrel temperature profile: feed–zone –zone 2–nozzle	160–190–190–190°C
Screw speed	135 rpm
Cooling time	15 s

Extrusion. A single-screw extruder from the AMUT EA48 series (Marano Ticino) was used to extrude plastic and wood particle pellets. Screw diameter was 48 mm and length to diameter ratio was 24:1. The rectangular die was 13 × 6 mm. Temperature profiles and screw speed conditions of the extruder were constant for all sample preparations, as shown in Table 3. Extrudates were quenched in water and samples were collected for analysis.

Compression molding. Pellets were compression-molded on a preheated press (Fontyne TP 1000) into plaques according to ASTM D4703-03 (ASTM 2003c). Machined-cavity flash-molding was used to prepare 3.2-mm-thick plaques. The mold was preheated to 180°C. The material was spread to slightly overfill the mold and the press was closed for 8 min to preheat the material by applying contact pressure. A molding pressure of 0.58 MPa was applied for 5 min. Cooling rate of the mold was maintained at 10 ± 2°C/min. Test specimens were cut using a standard blanking die.

Mechanical Properties Measurement

Specimens were stored under controlled conditions (50% RH and 23°C) for 40 h before testing. Only specimens having 35 wt% of wood fillers and 42-mesh particles were analyzed in this study. Tensile tests and bending tests were conducted according to ASTM D638-03 (ASTM 2003a) and ASTM D790-03 (ASTM 2003b), respectively.

Table 3. *Single-screw extruder conditions.*

Screw speed	80 rpm
Feed zone temperature	170°C
Compression zone temperature	180°C
Metering zone temperature	190°C
Die temperature	200°C

Characterization of Particle Length Distribution after Processing

Samples of composites were chosen randomly. The procedure used to separate the wood particles from the polymer matrix was as follows: 250 mL of xylene was placed in a 500-mL reactor and gently stirred with a magnetic stirrer while heating to 130 – 140°C. After reaching this temperature, pieces of composites (25 g) were placed in the reactor and allowed to react for 2 h. After the reaction, the mixture was filtered to recover the wood particles. The obtained material was soxhlet-extracted with xylene for 12 h to remove the unreacted anhydride and oven-dried at 70°C for 24 h. Finally, a dilute suspension of recovered particles was analyzed with a Fiber Quality Analyzer (FQA) (OpTest, Hawkesbury, Canada). FQA is an optical device used to characterize the length distribution and aspect ratio of wood fibers. Weight-averaged particle lengths were calculated according to the following equation (Robertson et al 1999):

$$L_W = \frac{\sum_{i=1}^{n} l_i}{\sum_{i=1}^{n} l_i^2} \qquad (1)$$

where,

L_W = weight-averaged length;
l_i = effective length of i^{th} particle; and
n = total number of particles analyzed.

RESULTS AND DISCUSSION

Development of Wood-Particle Reinforced High-Density Polyethylene

To eliminate processing variables, all wood particles (except bark particles) were produced under nearly identical hammermilling conditions. As shown in Fig 1, the vulnerability of wood to a hammermilling process depends on species. Eastern white cedar showed the lowest mean length (0.8 mm) after hammermilling. This apparent weakness of eastern white cedar during processing became more evident when compared with the structural properties of solid wood. Table 4 shows some mechanical properties of solid wood. It confirms that the weakness of white cedar is mainly because of its low density (320 kg/m^3).

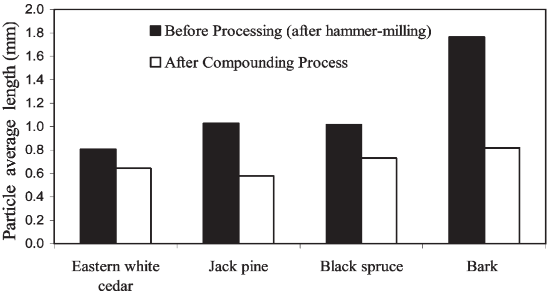

Figure 1. Effect of compounding process on the effective mean length of various wood particles.

In good agreement with previous reports (Hernandez et al 2002; Ali et al 2003; Kasliwal and Jones 2004), the higher shear stresses that developed in the counterrotating conical intermeshing twin-screw extruder during mixing and compounding caused a reduction in both particle length (Fig 1) and diameter (Fig 2). The final products, after the compounding process, appear to have comparable geometric means regardless of wood species or initial dimensions. Apparently, wood particle mean length and aspect depend solely on compounding process conditions. Visual examination of the compounded samples showed that after a residence time of 240 s and a screw speed of 240 rpm, the blend appeared to have been homogenized with no aggregated particles present. At this stage, the composites showed pseudoplastic behavior, which can be explained as follows. Initially, particles that are randomly oriented are subjected to shear. Particle length continues to diminish progressively through friction between the material and both the screw and barrel. After reaching the shortest length, the

Table 4. *Strength properties of the used wood species (Green et al 1999).*

	Density (kg/m^3)	Modulus of rupture (kPa)	Modulus of elasticity (MPa)	Work to maximum load (kJ/m^3)
Eastern white cedar	320	47	6.4	41
Jack pine	430	68	9.3	57
Black spruce	460	74	11.1	72

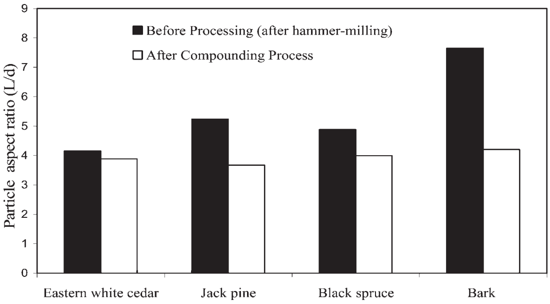

Figure 2. Effect of compounding process on the mean aspect ratio of various wood particles.

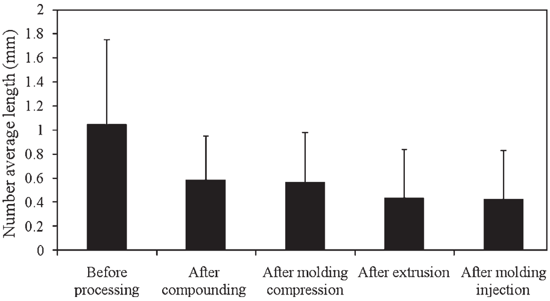

Figure 3. Development of the effective length of jack pine particles in relation to composite processing techniques.

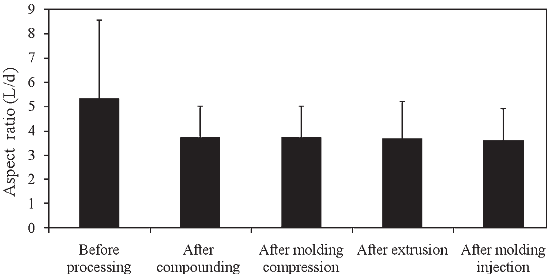

Figure 4. Development of the aspect ratio of jack pine particles in relation to composite processing techniques.

particles tend to align themselves in the direction of flow. Therefore, the extent of particle degradation decreases and averaged particle length appears to be constant regardless of species or initial dimension.

The development of the effective length and aspect ratio of jack pine particles in relation to processing methods are shown in Figs 3 and 4, respectively. The particles are dramatically shortened after the compounding process. The particle damage that occurs in injection molding or in single-screw extrusion is much lower than in twin-screw compounding. Similar effects have been reported by Yilmazer and Cansever (2002) and Bigg (1985), in which short inorganic fibers were used. After injection molding, neither the number average length nor the aspect ratio appears to be influenced by the process. This is primarily from the lack of shear stress and friction during compression.

Characterization of Particle Length Distribution

Figures 5 and 6 depict the number-average particle-size distribution for jack pine after each stage of processing. It was assumed that molding compression did not have a major impact on the size distribution. The analyzed particles were sieved through mesh 35 to 48 before compounding. In Fig 5, the longest particles (greater than 2.4 mm) completely vanished after compounding. Moreover, particles with lengths greater than 1.2 mm were severely damaged from compounding. The aspect ratio showed a symmetric distribution with 4 being the modal value (Fig 6). After compounding, the particle length distribution skewed toward the shortest particle length without modifying the original distribution before processing. However, the average length diminished slightly after the single-screw extrusion and molding injection processes, and particle length distribution exhibited a dramatic shift toward an asymmetric character with a tail on the longer fiber sizes.

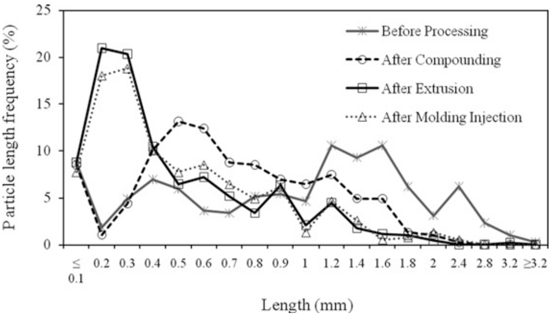

Figure 5. Length distribution of processed Jack pine particles.

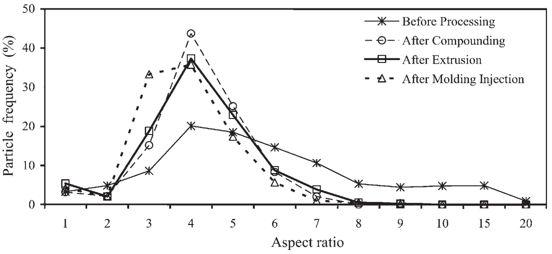

Figure 6. Aspect ratio distribution of processed Jack pine particles.

Table 5. *Fines frequency ($0.1 < \text{length} \leq 0.5$ mm) before and after compounding process.*

Species	Before processing	After compounding	Change (in %)
Jack pine	19.59	28.6	46.00
Eastern white cedar	26.04	38.65	48.42
Black spruce	21.13	30.93	46.37
Bark	10.48	21.91	109.06

Higher shear stresses developed in the compounding process directly attack the wood particles, which consist of wood fibers. In this process, the particle structure is disrupted, resulting in more refined particles. Injection molding and single-screw extrusion attack shives by breaking the extremities of the disintegrated fibers. As a consequence, the number of fines is greatly increased, as shown in Fig 5 and Table 5.

Bark particles showed higher propensity to produce fines than wood particles (Table 5). The fraction of bark fines having effective lengths of 0.1 – 0.5 mm doubled as a result of compounding (Table 5). Among the three wood types, the fines fraction increased almost equally by approximately 46%.

Effect of Weight-Average Length on Mechanical Properties of Wood-Particle/High-Density Polyethylene Composites

Weight-average particle length was taken into consideration, because it emphasizes the longer particles in the sample with less emphasis on the fines. Linear regression was used to model the relationship among weight-averaged length of wood particles, MOE (Fig 7a), and MOR (Fig 7b) of the composites. As expected, both exhibited good correlation with weight-averaged length of particles at 35% wood particle content. Maximum flexural strength (MOR [flexure]) showed the best correlation ($R^2 = 0.90$) with the L_W (Fig 6b). Maximum tensile strength (MOR [tensile]) was also highly correlated with L_W ($R^2 = 0.83$). These results are consistent with the literature. Indeed, Bledzki and Faruk (2003) observed that the tensile, flexural, charpy impact, and impact properties of wood-fiber reinforced polypropylene composites were positively influenced by fiber length. Lee et al (2001) found the same behavior when studying tensile and flexural modulus of wood-fiber/polypropylene prepreg sheets.

Effective average length, in contrast to weight-averaged length, is a widely used factor when characterizing mechanical properties of wood-fiber reinforced polymer composites (Fu and Lauke 1996; Fu et al 1999; Lee et al 2001; Juhlin et al 2002). However, after undergoing the processing cycle, particles in the polymer matrix are crushed and squeezed. This significantly affects particle length distribution, although the effective mean length is not changed. Therefore, weight-averaged length, which is directly related to the occurrence of long particles, is a more suitable parameter to consider than the effective mean length when characterizing the effect of a reinforcing material on the mechanical properties of the resultant polymer composite.

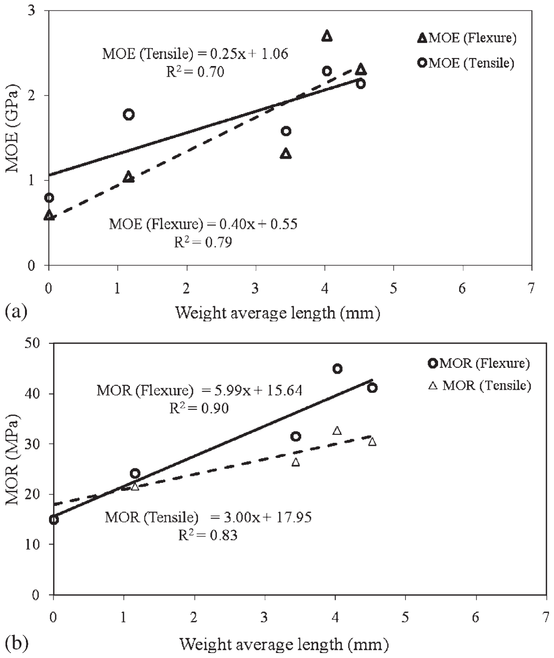

Figure 7. Relationships among (a) stiffness, (b) strength, and weight-average length in wood-particle based composites.

CONCLUSIONS

The vulnerability of wood particulates to composite manufacturing processes depends mainly on the intrinsic characteristics of the wood species. The major reduction in the effective mean length of the wood particles was found to be associated with the compounding process. Subsequent processes such as extrusion, molding injection, and molding compression had relatively small effects on particle-size reduction. This observation explains the greater fraction of fines in the finished composite compared with the original wood particle-size distribution.

Bark particles showed greater propensity to generate fines than wood particles. This may be because of its lower intrinsic strength and higher thermal sensitivity. Bark and eastern white cedar wood-based composites, containing high extractives content, are well known for their low stiffness and strength.

It is suggested that weight-averaged particle length and particle length distribution, primarily in terms of fines content, must be simultaneously considered in characterizing the effect of composite manufacturing processes on particle length attrition. This would also be helpful in determining the mechanical properties of the resultant composite.

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