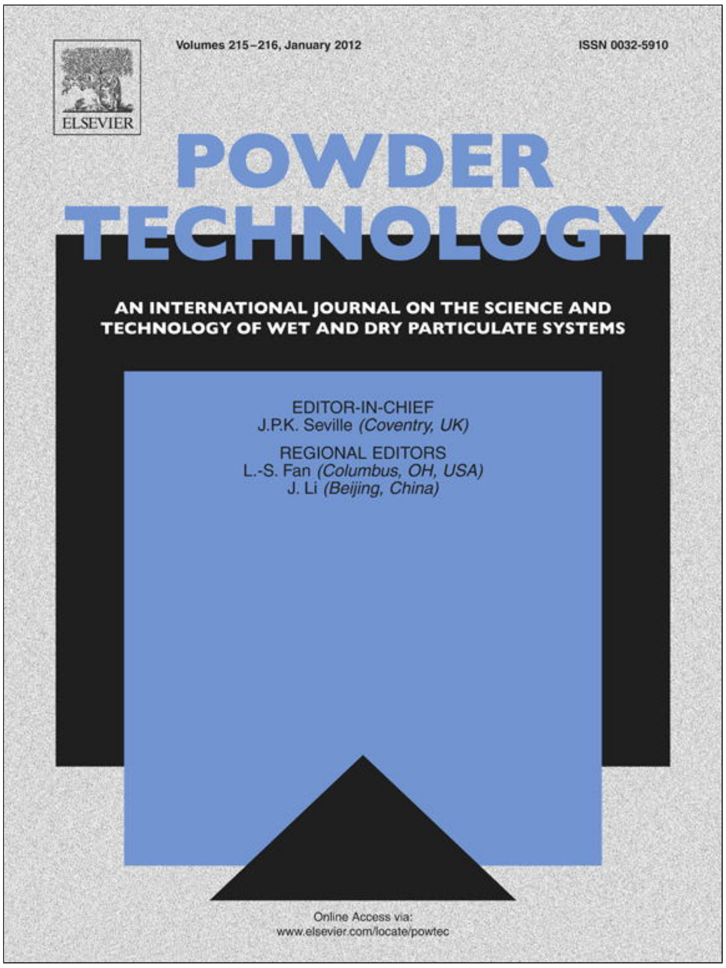
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Short communication

Dispersion of particulate clusters via the rapid vaporization of interstitial liquid

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ABSTRACT

A method for the dispersion of particle clusters driven by the pressure generated from the volatilization of interstitial liquid is described. To demonstrate the method, carbon black agglomerates were heated to induce the rapid evaporation of incorporated water or acetone, which resulted in breakage of the particle cluster. Measurements of the resulting fragment size distribution demonstrate the influence of processing temperature and type of incorporated liquid on the dispersion process.

1. Introduction

The dispersion of clusters of solids is a common step in many chemical or material processing applications, and the ultimate quality and performance of systems incorporating fine particles is directly affected by the degree to which these clusters are dispersed. Dispersion occurs when forces active on the length scale of the cluster or its constituent particles are sufficient in magnitude to overcome the cohesive forces binding the cluster together [1]. The cohesivity of particles clusters can arise from van der Waals or electrostatic interactions between the individual particles, interactions between secondary chemical species (binders or surfactants) added to the cluster to augment the intrinsic interparticle interactions, or capillary forces associated with liquids present within the interstices of the cluster. In order to accomplish dispersion, external forces (e.g. hydrodynamic shear, or shock waves associated with the collapse of ultrasonically induced cavitation bubbles) can be applied to overcome the cohesive forces that bind the particle clusters together.

In this paper, we present an alternate approach to dispersion in which forces are produced internal to the cluster itself. Agglomerates infused with a volatile liquid are introduced to a medium that is maintained at an elevated temperature, into which they are to be dispersed. The agglomerate will experience an increase in temperature and the incorporated liquid will vaporize thereby generating internal pressure. At some point, the confinement of the pressure within the agglomerate by the surrounding medium will break down, at which point the liquid internal to the agglomerate will rapidly vaporize and the resulting vapor seek to escape from the agglomerate. Under proper processing conditions the vapor produced within the agglomerate will generate enough internal pressure to overcome the cohesivity of the cluster, thereby producing multiple fragments and accomplishing the initial stage of dispersion. Subsequent application of external forces (e.g., hydrodynamic shear) could then be used to complete the dispersion process.

Proof-of-concept experiments have been performed with carbon black agglomerates infused with either water or acetone, and processed within oil as the dispersion medium. In our work, the oil provides a convenient substitute for an actual dispersion medium (e.g., a polymer melt). The degree of dispersion was measured as a function of processing temperature and liquid content within the agglomerate through optical microscopy and image analysis.

To the best of our knowledge, the use of forces associated with the volatilization of internal liquid is a new approach for breaking agglomerates; however, analogous phenomena are used in some industrial and food processing practices. For example, a process known as explosive disintegration has previously been used as a way to reduce wood to splinters for use in particle board. The Masonite process [2–4] involves fully permeating a piece of wood with moisture while it is under pressure at elevated temperature. When the pressure is suddenly dropped, the expanding vapors cause the wood to disintegrate into splinters. Analogous processes are used for the production of gun-puffed cereals, except the conditions are controlled so that the solid structure is expanded rather than shattered. A related process is the production of popcorn. The steam contained within the kernel expands once the outer shell of the kernel can no longer contain the internal pressure (typically 9.3×10^5 Pa) which develops when the kernel is heated to around 177 °C [5].

In some cases, dispersion via rapid volatilization may have an advantage over conventional dispersion processing in terms of energy

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Table 1
Compositions of the distilled water (DW)- and acetone-infiltrated agglomerates used in the proof-of-concept experiments. Also shown are the various temperatures used in the dispersion experiments, as well as the vapor pressure (P_{vap}) of the infused liquid at the corresponding temperature. Here T_b corresponds to the boiling point of the particular solvent used to wet the agglomerate.

Relative T	T (°C)	P_{vap} (Pa)	Oil (g)	CB (mg)	DW (mg)
Distilled water					
Room	22	2.64×10^3	30.36	4.5	16.1
T_b-50 °C	50	1.24×10^4	31.58	5	14.6
T_b-20 °C	80	4.74×10^4	32.65	3.7	13.6
T_b+10 °C	110	1.40×10^5	31.69	4.8	17.3
T_b+50 °C	150	4.74×10^5	32.05	3.9	14.8
Acetone					
Room	22	2.69×10^5	32.72	3.3	37.6
T_b-10 °C	45	6.81×10^5	30.34	4.2	51.8
T_b+10 °C	65	1.36×10^5	33.68	7.2	65.2
T_b+30 °C	85	2.49×10^5	31.02	5.3	55.9

requirements. When agglomerates are to be dispersed in polymer melts, for example, energy for the dispersion may come from the melt itself, if the processing occurs at a temperature above that required to volatilize the interstitial liquid. The dispersed particles would then already reside within the host polymer matrix.

2. Experimental

Agglomerates of carbon black (Black Pearls 120V-424 manufactured by the Cabot Corporation) were used as-received for the dispersion studies. These agglomerates are comprised of nominally 75-nm carbon black particles. To aid interpretation of the dispersion results, carbon black agglomerates in the size range 2–2.5 mm in diameter were manually chosen from the wider range of agglomerates provided by the manufacturer. Distilled water (boiling point = 100 °C) or acetone (boiling point = 56.5 °C, supplied by Fisher Scientific) were used to wet the agglomerates. Light mineral oil (Fisher Scientific, Lot #101970) was used as the host fluid for heating of the agglomerates and played the role of the medium in which dispersion is intended to occur.

For experiments with distilled water, one drop of water was added to the carbon black cluster and its weight recorded. After allowing 5 min for the water to be absorbed into the cluster, excess water was removed by dabbing with tissue paper. For experiments with acetone, the weight was recorded after one drop was added. Additional drops of acetone were added at 60-s intervals. After the fifth drop of acetone was added, the excess liquid was absorbed using tissue paper.

Approximately 30 g of mineral oil was placed into a 150 mL beaker, which was equilibrated in heated oil bath set to various temperatures ranging to upwards of 50 °C higher than the boiling point of the infused liquid. Temperatures were controlled to within ±5 °C. The wetted clusters of carbon black were placed in the mineral oil for 5 min, at which point the beaker was removed and allowed to cool for at least 30 min before the contents were analyzed. Table 1 provides details of the parameters used in the proof-of-concept experiments.

Images of the results of the dispersion experiments were obtained using an Olympus BX51 Optical Microscope. A micropipette (having an enlarged tip) was used to transfer the contents from the dispersion to glass slides. At least three, and usually six, slides were made per experiment. In order to get a good representation of the fragment size distribution, 6–15 images were taken at different locations on each slide. *MATLAB* code was written to analyze the images. Each image was converted to an 8-bit grayscale image and the individual area of each fragment was then used to find the equivalent diameter, assuming each fragment to be approximately spherical. These results were then used to produce histograms of the fragment size distributions.

3. Results and discussion

In order for this approach to lead to successful dispersion, it is essential that the infused liquid remains within the cluster during the heating period. The possibility that the infused liquid could be forced out of the carbon black cluster was investigated by placing a drop of the mineral oil onto the wetted carbon black cluster. In this case, the oil encapsulated the cluster, trapping the water inside. This encapsulation is believed to be useful in enhancing the pressurization that could occur within the carbon black cluster, and is therefore useful in improving the dispersion effect.

All carbon black clusters treated with distilled water showed some dispersion upon simply being placed within the mineral oil. However, there was a visible difference between the experiments performed at temperatures below and above the boiling point of water. Agglomerates processed above 100 °C appeared to explode, and for trials performed well above the boiling point, there were audible popping sounds immediately after the agglomerate was dropped into the heated oil. Fig. 1 shows representative images of the fragments produced at three different operating temperatures.

For trials in which acetone was used, there was also some minor erosion associated with dropping the sample into the oil. Here too, there was an appreciable difference between experiments performed about the boiling point of acetone. However, large differences were not seen until a temperature of 85 °C, (30 °C above the boiling point) was tested. As was the case with the water-infused agglomerates, the

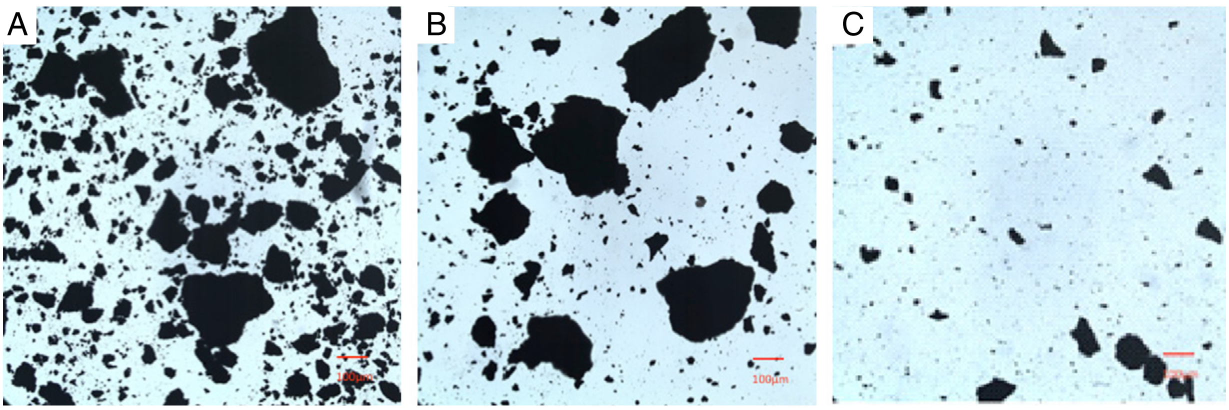

Fig. 1. Optical micrgraphs of the fragments produced from carbon black agglomerates infused with distilled water, processed at different temperatures: (A) 50 °C; (B) 80 °C; (C) 110 ° C. Note the the scale bar depicts 100 μm.

quantity and size of large fragments decreased with increased processing temperature.

Normalized fragment size distributions were computed from the microscopy images. Figs. 2 and 3 show the results for the dispersion of water-infused and acetone-infused carbon black agglomerates obtained at various processing temperatures. Note the production of greater quantities of small fragments, and the reduction in the number of large fragments, as the processing temperature was increased.

The difference in behavior seen for the water-infused and acetone-infused carbon black agglomerates can be attributed to the different effects of the two liquids on the cohesivity of the cluster. For the case of water, the cohesivity is apparently reduced to the point where less internal pressure is needed to break the cluster. For example, comparison of the results from processing at 10 °C above the boiling point for the two liquids (i.e., 110 °C for water, 65 °C for acetone) which produces similar internal pressures (see Table 1) in the two cases, show that there are fewer large fragments for the case of water.

4. Conclusions

The ability to accomplish the dispersion of particle agglomerates via the rapid evaporation of an incorporated liquid has been demonstrated. Dispersion results demonstrated that the number of large fragments diminished when the cluster was processed at temperatures that exceeded the boiling point of the incorporated liquid.

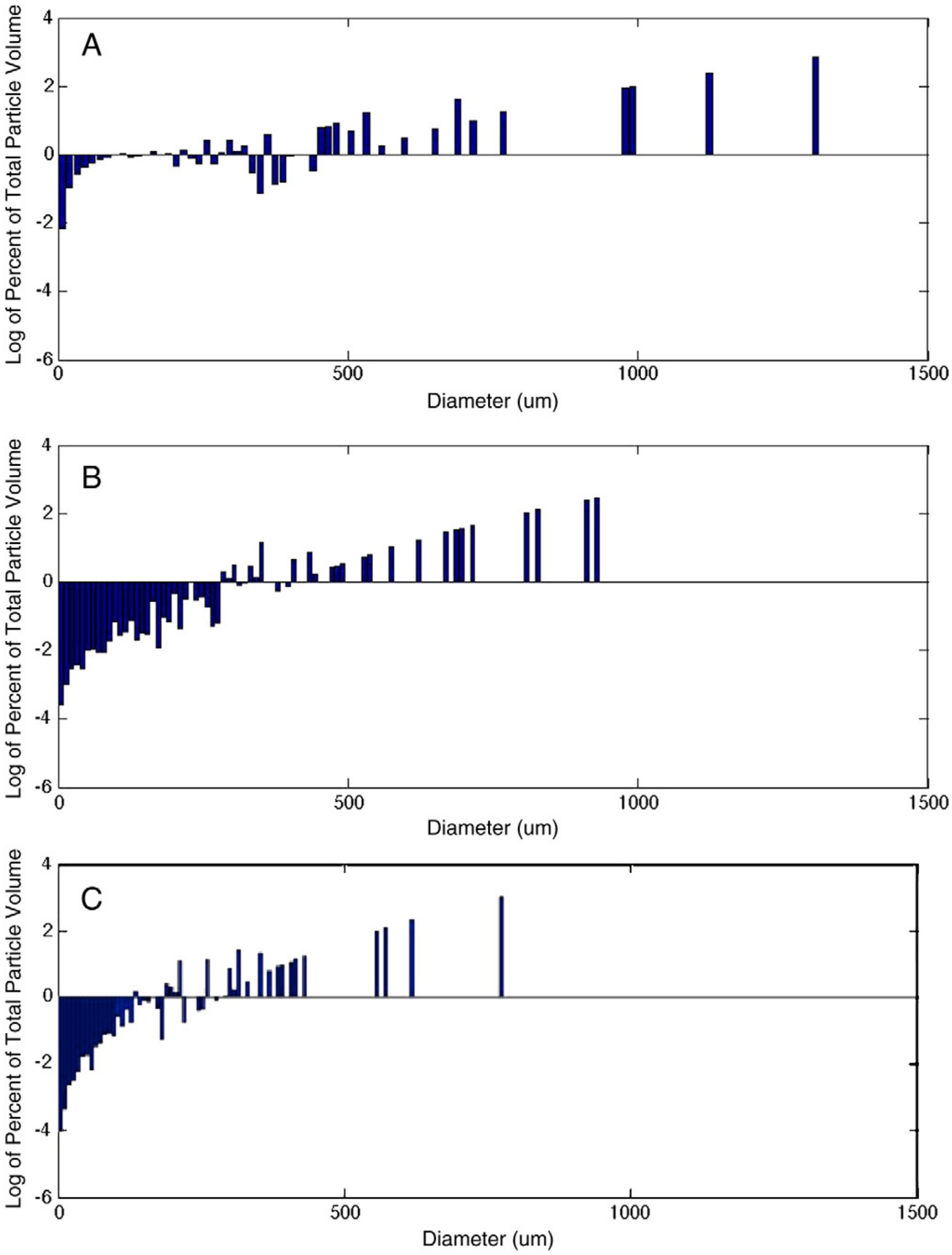

Fig. 2. Volume size distribution vs. fragment diameter for water-infused carbon black agglomerates processed at: (A) 50 °C; (B) 110 °C; (C) 150 °C. Note the decrease in frequency of large fragments at the elevated temperature.

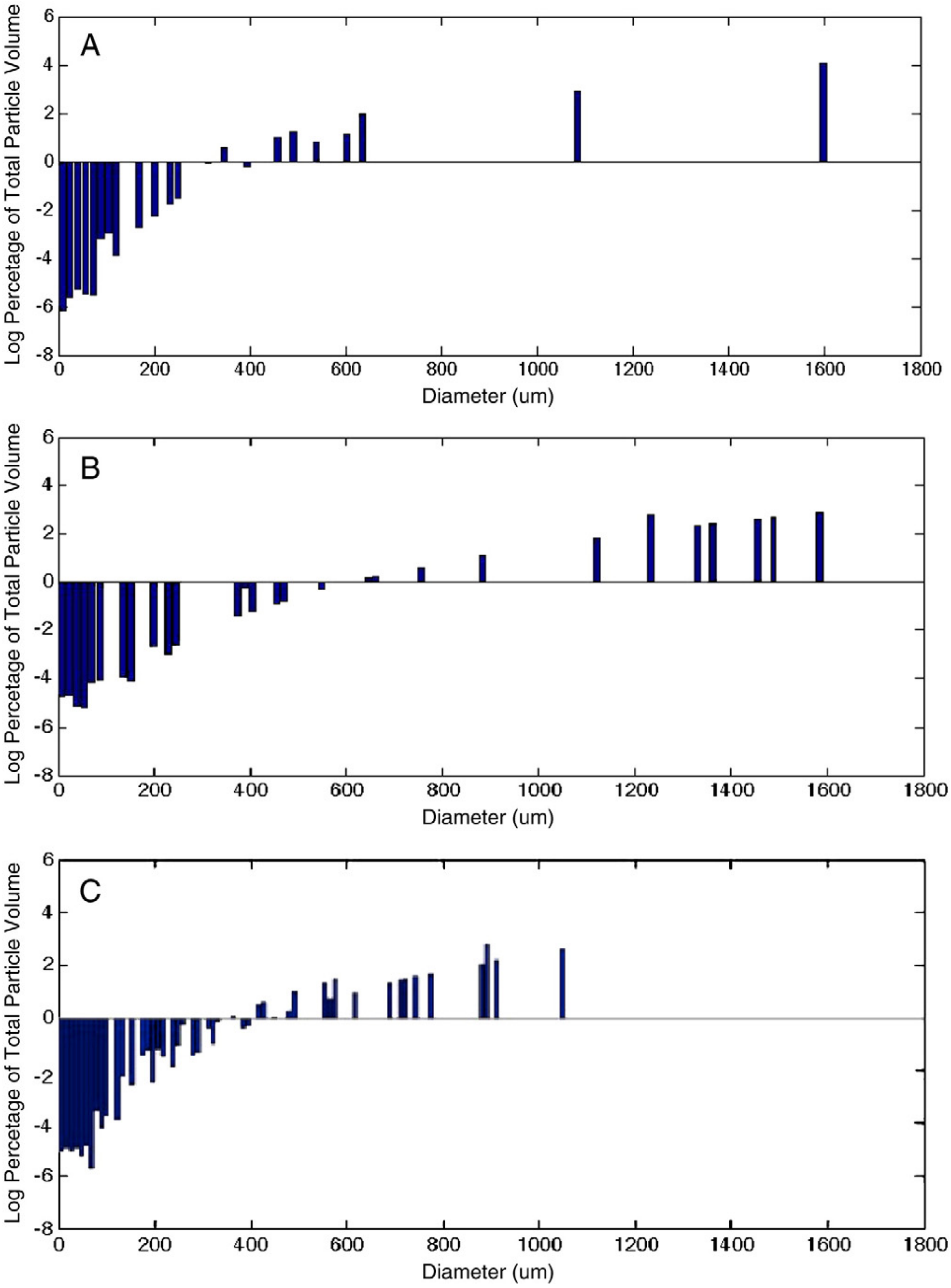

Fig. 3. Volume size distribution vs. fragment diameter for acetone-infused carbon black agglomerates processed at: (A) 45 °C; (B) 65 °C; (C) 85 °C.

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