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Evaluation of Methods for Quantification of Transmission Electron Microscopy (TEM) Dispersions

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Transmission electron microscopy (TEM) micrographs are routinely used to evaluate the dispersion of insoluble additives in polymeric systems. For routine TEM analysis, many analysts have relied on a visual analysis of the TEM micrographs to estimate the quality of the additive dispersion. When comparing large numbers of TEM micrographs, the ability to determine or estimate the dispersion quality is often difficult. The objective of this study was to develop a method to quantify dispersions observed in TEM micrographs that both enables a numerical "ranking" to be assigned to individual dispersions as well as enables tabulation of a multitude of images acquired over time. Several methods were reviewed and applied to a set of TEM dispersion images acquired of an insoluble additive in polystyrene.

Projected area diameter [1], particle area [1], and Euclidean distance between particle centroids [2] were chosen from all the particle size distribution and spatial distribution parameters present in the literature to evaluate their effectiveness in yielding a numerical value useful in ranking dispersion quality. Projected area diameter, also called equivalent circular diameter, was chosen because its measurement considers two dimensions of the particle, making it a preferred statistical diameter for use in particle sizing. Particle area was selected to evaluate how weighting particle size distributions by area rather than just number might differentiate the histograms. Euclidean distance between centroids was chosen to characterize spatial distribution because it is the most inclusive method by which to measure interparticle spacing, considering all interparticle distances in the field of view in addition to just nearest and near neighbor distances. In addition, its computation is fairly straightforward and would facilitate a quick evaluation of the method.

The three methods evaluated did not successfully yield a quantitative indicator of dispersion quality for the micrographs in Figure 1. Their inability to generate a "ranking" value suggested that a different parameter is needed, one which quantifies size and distribution differently than these three methods. It appeared that this different parameter should be a three dimensional parameter, considering that Euclidean distance between centroids is a linear parameter, and equivalent circular diameter and particle area both incorporate two dimensions. It followed that a volume distribution may offer a better ability to quantitatively rank TEM micrograph dispersions. This was confirmed by calculating cumulative volume percent curves [1] for the Figure 1 micrographs.

Generating cumulative volume percent curves for different samples appears to be a preferred method of quantifying and comparing dispersions in TEM micrographs. The volume diameter values obtained by this method can be used for "ranking" and tabulation of dispersion quality (Figure 2). This method proved much more successful in quantifying dispersion quality than equivalent circular diameter, particle area, or Euclidean distance between particle centroids, providing a method to account for both "good" dispersions of fine domains and "bad" dispersions of non-uniform domains of additives.

References
[1] T. Allen, *Particle Size Measurement, Volume 1: Powder sampling and particle size measurement*, 5th ed., Chapman and Hall, New York, 1997.
[2] N. Silva and A. Velhinho, *Mater. Sci. Forum* 514-516 (2006) 779.

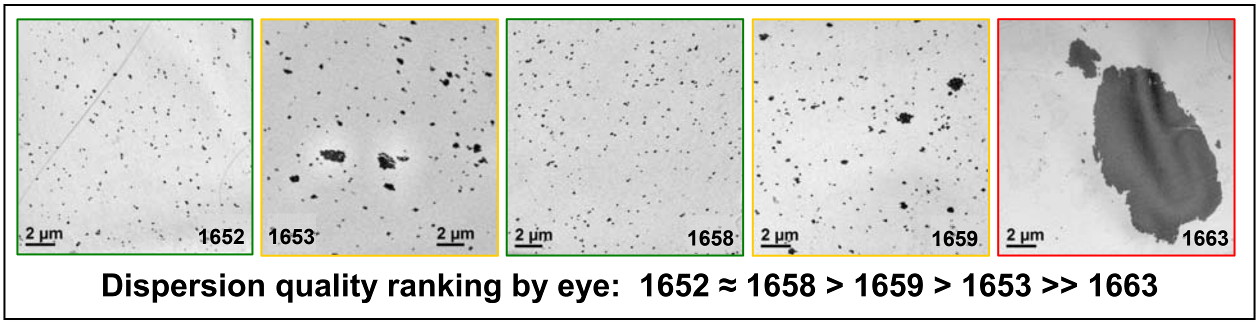

Figure 1. TEM micrographs of dispersions of an insoluble additive in polystyrene

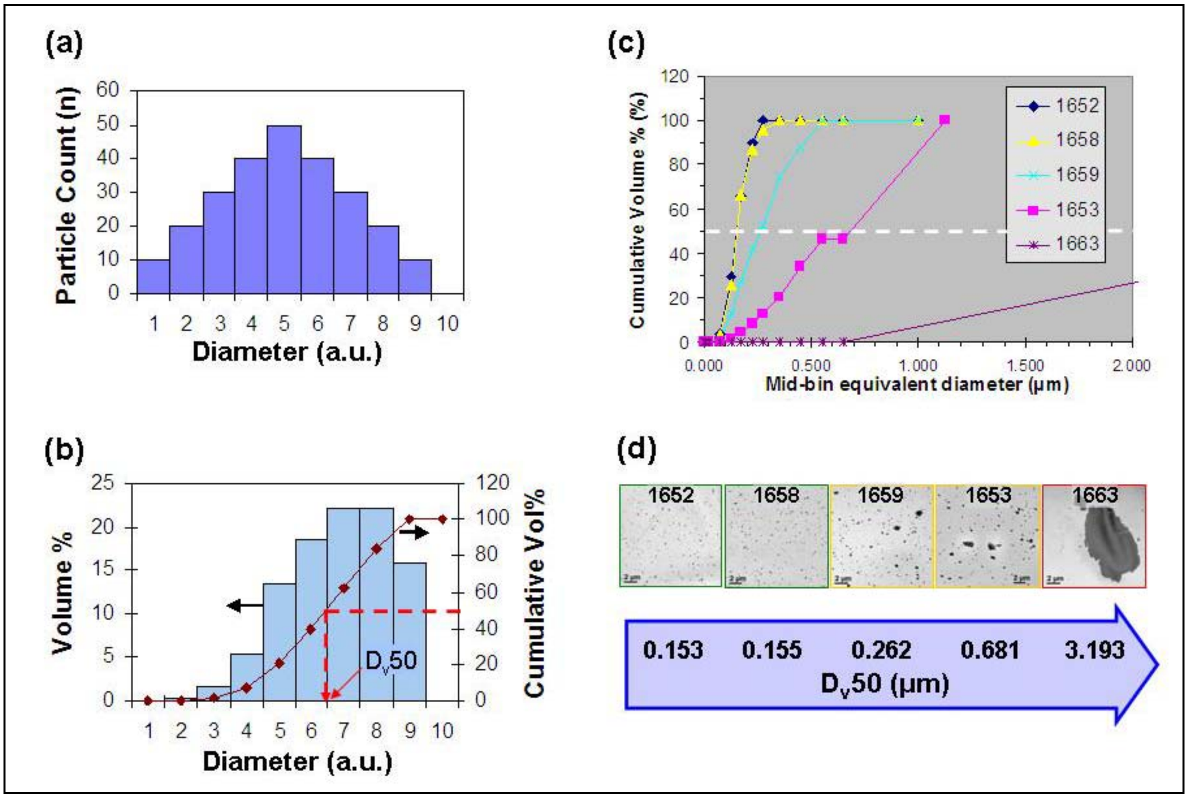

Figure 2. Cumulative volume percent method of providing a "ranking" of dispersions: (a) a histogram of particle number vs. particle diameter is generated, (b) the histogram in (a) is tranformed into a histogram of volume % vs. diameter from which a cumulative volume percent curve is generated. A volume diameter corresponding to 50 cumulative volume percent can be read from the curve, (c) cumulative volume percent curves are generated for the dispersions in Figure 1, and (d) the D_v values obtained for each dispersion can be used to rank them by quality.