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# Analysis and measurement of carbon nanotube dispersions: nanodispersion *versus* macrodispersion

**Micah J Green***

## Abstract

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**Keywords:** nanotubes; dispersion; rheology; composites

## NANOTUBE BUNDLING AND DISPERSION

The initial discovery of carbon nanotubes (CNTs) generated intense interest because of the possibility of utilizing their extraordinary properties in high-performance devices and materials. The dispersion of CNTs in liquids is one of the most challenging and important issues facing the development of such applications because scalable processing typically requires effective liquid-phase processing. Unfortunately, CNTs naturally form 'bundles' of varying sizes due to strong van der Waals forces; this is particularly true of single-walled nanotubes (SWNTs). These bundles typically cluster to form large aggregates that are insoluble in most liquids. A variety of processing techniques have been developed to solve this problem, which we address in detail.

In 2005, NASA and NIST sponsored a workshop designed to address the issues of CNT purity and dispersion. The broad distinction that emerged from this workshop was the conceptual difference between 'nanodispersion' of individual CNTs and 'macrodispersion' of CNT bundles. The workshop proceedings defined macrodispersion as the dispersion of small CNT bundles and nanodispersion as splitting the bundles into individual, non-bundled CNTs.[1] This distinction is critical because the functionality of a wide range of applications depends on the dispersion of individual CNTs rather than just CNT bundles. Such applications range from sensors and biomolecular markers to polymer nanocomposites and nanoelectronic devices. For example, some CNT-based biosensors relying on photoluminescence are only effective in the case of unbundled CNTs. The percolation threshold for polymer/CNT nanocomposites depends inversely on the average aspect ratio of the dispersed entities.

Potential definitions for the dimensionless degree of nanodispersion, $X$, include the following:

- $X_1$ is the fraction of dispersed entities (bundles or CNTs) that are individual CNTs;[2]
- $X_2$ is the reciprocal of the average number of CNTs per bundle (often assessed via the root-mean-square bundle diameter); and
- $X_3$ is the fraction of CNTs that are unbundled.

These concepts are illustrated in Fig. 1; note that $X_3 = X_1X_2$. In all cases, $X = 1$ indicates complete nanodispersion.[3] These quantities are summarized in Fig. 1 in terms of $p(z)$, which represents the probability that a dispersed entity will have $z$ CNTs. For applications where the only effective CNTs are individuals (such as photoluminescent biosensors), $X_1$ or $X_3$ is more appropriate, while $X_2$ is more useful for applications where desirable properties have a simple inverse correlation with bundle size (such as conductive nanocomposites). The need for these definitions stems from widespread equivocation and imprecision in the nanotube literature. One example is the common use of the phrase 'well dispersed'; this phrase is problematic since one researcher may take this to mean nanodispersion while another may simply take this to mean a lack of large nanotube aggregates, i.e. macrodispersion. The aim of the present article is to carefully examine these two concepts and the controversies that arise over their analysis and measurement. The characterization

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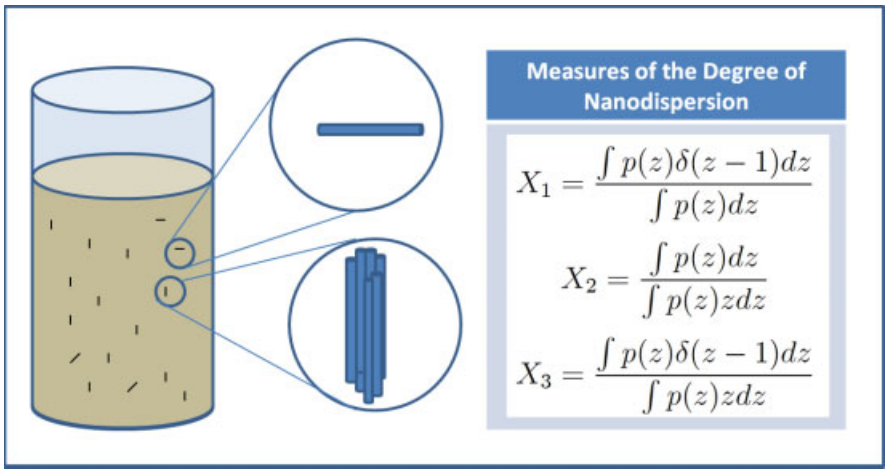


**Figure 1.** Left: after the removal of insoluble material, a typical CNT dispersion contains both individual CNTs and small bundles. Right: the degree of nanodispersion can be expressed through $p(z)$, the probability distribution function expressing the probability that a dispersed entity will have $z$ CNTs. Three commonly used dimensionless nanodispersion indicators ($X_1$, $X_2$ and $X_3$) can be easily calculated from $p(z)$.

techniques for quantifying $X$ are reviewed and their advantages and disadvantages are discussed.

A variety of methods are used to break bundles into individual CNTs and prevent re-aggregation. These methods fall into three broad categories: (1) the CNTs may be functionalized and rendered soluble in common aqueous or organic solvents; (2) mechanical mixing or ultrasonication breaks the bundles apart such that the CNTs are dispersed in some fluid (typically water) and are often stabilized by some wrapping agent such as a surfactant or polymer; and (3) CNTs may be directly dissolved in a solvent. Methods (2) and (3) are more interesting and controversial since they aim to disperse pristine CNTs; functionalizing CNT sidewalls tends to compromise electrical and other properties. In principle, methods (2) and (3) may be distinguished as 'kinetic dispersion' and 'thermodynamic dissolution', respectively.[4] Kinetic dispersion relies on high-shear mixing and/or ultrasonication to break CNT bundles apart and then relies on centrifugation or interfacial trapping to remove aggregates and some bundles from the dispersion.[5] (Note that ultrasonication invariably cuts the CNTs as well.) Both ultrasonication (bundle-breaking) and centrifugation (bundle-removing) processes are kinetically limited, so it is unrealistic to expect them to yield solutions where $X = 1$.

## COMPLETE NANODISPERSION

In contrast, whenever thermodynamic dissolution or spontaneous debundling of CNTs in a solvent is claimed (method (3) above), the compatibility between CNT and solvent should allow for complete equilibrium nanodispersion such that $X$ approaches 1. This can be proven by showing that no insoluble CNT material 'crashes out' during centrifugation and that the dispersed material contains only individual CNTs with no bundling. A number of CNT solvents have been claimed in the past several years.

Very recently, the author and colleagues claimed superacids as true solvents for CNTs based on several lines of evidence, including spontaneous dissolution, formation of liquid crystals at high concentrations and the lack of insoluble CNT material.[6] Most importantly, an isotropic phase of a CNT/chlorosulfonic acid mixture was imaged using cryogenic transmission electron microscopy (cryo-TEM); the images show CNTs dispersed as individuals with no sign of bundles. This method of direct visualization appears to be the most conclusive test for complete nanodispersion. These findings stand in contrast to the widespread claims that 'all solvents' disperse only a fraction of the nanotubes as individuals.[7] Second, a range of organic solvents such as *N*-methylpyrrolidone (NMP) appear to have favorable solubility parameters in the context of Flory–Huggins theory, and it was claimed that the CNT/NMP dispersions showed spontaneous, thermodynamic dissolution.[8–11] However, several caveats were recently applied to these claims: dissolution in these solvents requires extensive ultrasonication, results in a large number of bundles such that $X < 1$, requires centrifugation and removal of insoluble material, and is incapable of concentrations high enough for the formation of liquid crystals.[7,10] These characteristics are all consistent with kinetic dispersion rather than thermodynamic dissolution. Also, favorable solubility parameters were found for poor solvents; these false positives suggest that more studies are required to understand how surface energies relate to CNT dispersion.[10]

In a similar vein, many of the method (2) techniques claim complete or near-complete nanodispersion after ultrasonication and centrifugation.[12] Again, complete nanodispersion in these top phases is unlikely due to the fact that ultrasonication and centrifugation are kinetically limited processes. Also, when comparing $X$ for two separate dispersion methods, a careful distinction must be made to note whether insoluble material is removed by centrifugation or not.

## CHARACTERIZATION METHODS FOR CNT DISPERSION QUALITY

For most applications, complete nanodispersion ($X = 1$) is not necessary; however, the targeted properties for the application typically improve with increasing $X$. Such properties may include reinforcement in nanocomposites, photoluminescence for imaging applications, conductivity in thin CNT films and separation of nanotube by type and diameter.

Here we assess a number of experimental methods for characterizing $X$, with a distinction between bulk techniques and techniques that only investigate a small sample. This discussion is summarized in Table 1. Cryo-TEM imaging (non-bulk) allows for dispersed bundles and individual CNTs to be directly observed and



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**Table 1.** Techniques for characterizing CNT dispersion quality

| Dispersion quality measurement | Bulk/non-bulk | Assessment |
|---|---|---|
| Cryo-TEM | Non-bulk | Effective, allows for clear visualization; underutilized |
| AFM | Non-bulk | Sample preparation can cause aggregation; time-consuming |
| TEM | Non-bulk | Sample preparation can cause aggregation; time-consuming |
| Dynamic light scattering | Bulk | Effective, rapid; underutilized |
| Photoluminescence | Bulk | Effective, rapid, qualitative; limited to aqueous dispersions |
| UV-visible spectroscopy | Bulk | Rapid, versatile; only qualitative |
| Freeze-drying | Bulk | Qualitative supplementary technique for assessing performance of stabilization; underutilized |
| Rheology | Bulk | Promising for the dilute limit, but has difficulties in resolution and in assessment of bundle diameter; underutilized |
| Liquid-crystalline phase transitions | Bulk | Promising, but limited to high-concentration dispersions |
| Rheological percolation | Bulk | Ambiguous – low threshold can indicate attractive interactions rather than excellent dispersion |
| Electrical percolation (composites) | Bulk | Ambiguous - low threshold can indicate attractive interactions rather than excellent dispersion |
| SEM (composites) | Non-bulk | Unable to detect difference between bundles and individuals |
| TEM (composites) | Non-bulk | Helpful; time-consuming |

has been used for surfactant- and polymer-stabilized dispersions[13] as well as superacid solutions.[6] A more common technique is AFM, a non-bulk method that can be used to assess $X_1$ or $X_2$ by measuring the root-mean-square bundle diameter.[9] (Prior data[12] suggest that in a sonicated and surfactant-stabilized dispersion, $X_1$ may be as high as 0.5–0.8.) Although this is the most commonly used method, the time-consuming preparation, the small sample size and the subjective elements of AFM have led many to seek alternatives. An interesting bulk technique is the use of dynamic light scattering, which has been used to characterize the dimensions of dispersed SWNTs and SWNT bundles.[14,15] For surfactant- and polymer-stabilized dispersions, the photoluminescence intensity is a common bulk indicator for individual CNT dispersion such that the ratio of fluorescence to concentration can be qualitatively correlated with $X$. Similarly, the heights of certain UV-visible absorbance peaks increase with increasing $X$; this bulk technique was used to qualitatively monitor exfoliation during ultrasonication.[16] The freeze-drying of a dispersed sample followed by simple re-dispersion is an interesting supplementary technique for characterizing dispersion quality; samples that display the same photoluminescence after freeze-drying and re-dispersion are indicative of small bundles or individually dispersed CNTs that are well stabilized against aggregation.[13,17,18]

An underutilized bulk avenue for nanodispersion characterization is the comparison of experimental phase behavior and rheology data against theories for dispersed rigid rods. For instance, comparison of experimental liquid-crystal transitions against the Onsager or Flory theory estimates for rigid rods provides a reasonable estimate of the degree of nanodispersion.[19,20] Similarly, the dilute rheology of CNT dispersions can provide a bulk measurement of the average aspect ratio of a CNT sample;[21] such a technique can be extended to provide an estimate of the average bundle size. Note that there has been some disagreement in the field of CNT rheology because of the complex relationship between viscosity and dispersion quality. For instance, the claim was recently made that suspensions of 'well-dispersed SWNTs' should have lower viscosities compared with agglomerated or bundled SWNT dispersions.[22] Of course, this is not necessarily true. Although large aggregates may indeed result in a higher viscosity, theoretical predictions show that a dilute solution of SWNT bundles will have a lower intrinsic viscosity than the corresponding dilute solution of individual SWNTs due to the decreased aspect ratio in SWNT bundles.[21] This uncertainty ultimately stems from the fact that there are multiple ways for SWNTs to be poorly dispersed. At non-dilute concentrations, a variety of structured networks may form and display unusual rheological responses, depending on the dispersion. Despite these difficulties, rheological measurements can be used to probe small deviations from complete nanodispersion. (Note that although the rheology of low-viscosity aqueous CNT dispersions is often difficult to probe due to torque noise, the torque resolution for most modern rheometers is accurate enough to allow measurement of the effects of small CNT loadings in high-viscosity liquids.)

For nanotubes dispersed in polymer solutions or melts, the liquid is typically cast to form a solid composite film, and the dispersion quality is assessed by measuring the percolation threshold for the system or by using SEM to assess aggregate size. The use of a low electrical or rheological percolation threshold as a marker of dispersion quality is problematic because CNT dispersions with mild attractive interactions and bundling will have a substantially lower percolation threshold than nanodispersed CNTs in an athermal solvent.[23] (Often, a low percolation threshold is the targeted material property rather than a dispersion quality indictor alone.)

The use of SEM (non-bulk) to assess dispersion quality is also limited. A large number of studies use melt mixing, *in situ* polymerization or solution blending to attempt to disperse CNTs in a polymer melt, and then nanodispersion is claimed after SEM is used to investigate whether any large aggregates are visible.[24] This is problematic since SEM typically cannot distinguish between bundles and individually dispersed nanomaterials, i.e. SEM can confirm macrodispersion but not nanodispersion.[25] (In some cases, these claims do admit the possibility of small bundles.[26,27]) Furthermore, it is unlikely that these mixing techniques can yield a high degree of nanodispersion, particularly in the case of melt mixing, since these are kinetically limited processes with limited bundle-breaking ability.

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Despite being time-consuming, conventional TEM imaging (non-bulk) of nanocomposite samples can give an accurate snapshot of the degree of CNT dispersion by directly visualizing the CNTs or bundles embedded in polymers.[28,29] A more convenient method is the estimation of the fraction of individual CNTs through absorbance measurements, but Grady argues in his recent review that this technique is limited due to the possibility of polymer absorption in the same range.[3]

## CONCLUSIONS

The degree of nanodispersion is commended as a standardized dispersion quality indicator that can be utilized in a wide range of CNT research areas. Several points of controversy are discussed where dispersion quality has been overstated due to difficulties in nanodispersion definition or due to problematic characterization techniques. Finally, the advantages and disadvantages of experimental characterization methods are reviewed, with an emphasis on new possibilities for bulk, quantitative characterization. Bulk techniques such as rheological characterization or spectroscopic measurements yield an ensemble average value of $X$. Unequivocal identification of complete nanodispersion ($X = 1$) is difficult, but the use of cryo-TEM for CNT/solvents and conventional TEM for CNT/polymer composites has proved promising.[6,29] One final consideration is the influence of sample preparation on measurements of $X$; sample preparation can induce bundling and decrease the apparent value of $X$.

Many of the characterization techniques and controversies described here have analogs in the graphene dispersion community, with nanodispersion corresponding to dispersion of individual graphene sheets and macrodispersion corresponding to dispersion of multilayer graphene nanoplatelets. Many of the most successful techniques for dispersing graphene (particularly those that avoid oxidation) are directly adapted from methods used to disperse CNTs.[7,30]

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