Preserving Particle Size Volume Distribution Data At Low Sample Masses Using Low Angle Laser Light Scattering (Lalls) Analysis

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Introduction: Particle size analysis of implant debris (ASTM 1877) has typically been conducted by particle counting using Scanning Electron Microscopy (SEM). Technologies such as Low Angle Laser Light Scattering (LALLS) provide both volume and number distribution information as opposed to the number-only distribution of SEM analysis. LALLS is the preferred standard in many industries for characterization and quality control, although it typically requires large amounts of particles/powder (>100mg). Can this technology be used to analyze the small amounts of particles associated with implant simulator testing and in vivo use (i.e. typically <10mg)? Will LALLS analysis be affected by decreased amounts of sample? We hypothesize that as the percent of the sample used for analysis gets smaller (less mass) and approaches the method detection limit, the measured average size of the particles will decrease proportionally due to the statistical sampling bias of "collecting" and preferentially analyzing smaller more numerous particles. We tested this hypothesis by analyzing decreasing amounts of different Co-alloy particle samples using LALLS and SEM analysis.

Materials And Methods: *Materials:* Vapor sprayed spherical Cobalt alloy (ASTM F-75) particles of two different size distributions were analyzed: (1) "Powder 1-500", with particles ranging from 0.5 to 500 microns and (2) "Powder 20", with a single size of particles in the 20 micron range. *Methods:* Low Angle Laser Light Scattering (MicroTrac-X-100) analysis was used to measure progressively smaller masses of each powder at 10, 5, 1, 0.2, 0.1, 0.05 and 0.01mg additions to a LALLS machine custom adapted to analyze small sample masses. Each mass constitutes a separate analysis. Additonally, SEM analysis of particles was conducted using a Scanning Electron Microscopy Hitachi 3000-SN (SEM/EDS) after filtration (0.1 micron alumina ceramic filter).(1,2)

Results: Particle diameter was measured for each sample at each mass run using three criteria: a volume basis (mv), a surface area basis (ma) and a number basis (mn). A volume-based histogram of particle diameter was generated for each sample at each mass run. Fig 1 shows the results for "Powder 1-500". The mv, ma, and mn for each of the two powders are compiled in Fig 2 (Powder 20) and Fig 3 (Powder 1-500). For Powder-20, mv increases from 3 to 8 microns when the particle sample mass was reduced from 10mg to 0.1 mg (Fig 2); whereas Powder 1-500 demonstrates an increase of mv from 146 to 185 microns and an increase in mn from 1.5 to 2.2 microns when the sample mass was decreased from 5mg to 0.01mg. For Powder 20, a sample size of 0.01mg was not detectable, thus, surprisingly the method detection limit for metal powders with an average of a small mean size was less than that of larger averaged size samples, such as Powder 1-500.

Discussion: These results surprisingly refute our original hypothesis. Instead of showing a proportional decrease in particle size with decreasing amount of sample, a slight increase in particle size was demonstrated, as illustrated in Fig 1 where the peak of the largest size particles, at approx 300microns (arrows), represents a greater percentage of the total volume the lower the sample mass. We expected this peak (Fig 1 arrows) to virtually disappear at 0.1mg. This demonstrates the ability of LALLS to "find" the relatively few large particles within the sample and include them in the volume distribution and analysis. This inclusion, implicates size variation may be associated with the physical sampling of progressively smaller amounts of the total (5-10mg) rather than measurement error. LALLS works on the principle that diffraction angle is inversely proportional to particle size when passing in front of fixed wavelength He-Ne gas laser (λ=0.63µm), and thus directly measures millions of particles to calculate volume and number distribution data; however, it lacks the capability to yield morphologic data, e.g. aspect ratios. The preservation of volume distribution information with tiny amounts of particles (i.e. 0.01mg) with wide-ranging distributions demonstrates the utility of such technology to analyze implant debris. There was less change in particle size with sample mass variation for the powder with a wider size range distribution (Powder 1-500) than for more homogeneous micron sized samples (Powder 20). The preservation of volume distribution data (i.e. the larger particles included in an mv analysis) is critical an understanding, that gravimetric weight loss from an implant is not entirely due to the predominantly smaller particles in a number based analysis of particles (nm generated by LALLS or by SEM analysis, Fig 4). This understanding is essential to accurate animal testing (dosing) and any analysis of implant wear mechanisms that engineers seek to improve.

Acknowledgments: BioEngineering Solutions Inc.

References: 1. Campbell P, et al *JBMR* 29:127-131, 1995.
2. Scott M et al *JBMR-B Appl:* 73:325-337, 2005.

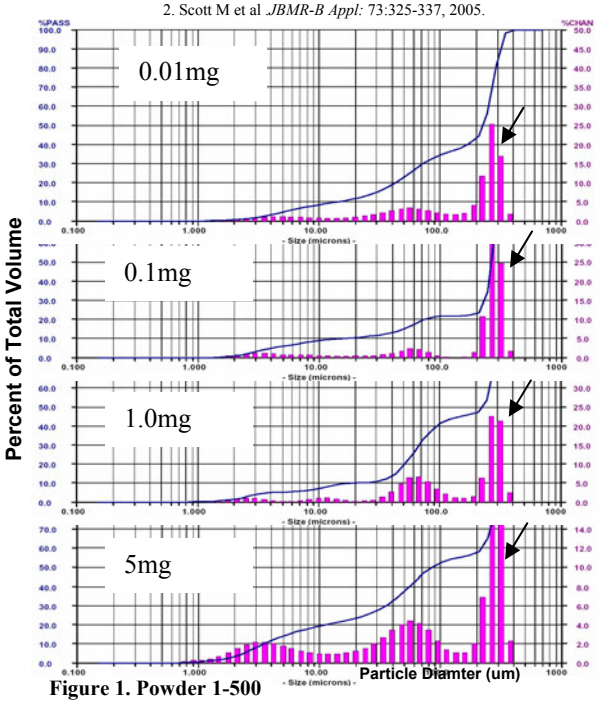

Figure 1. Powder 1-500

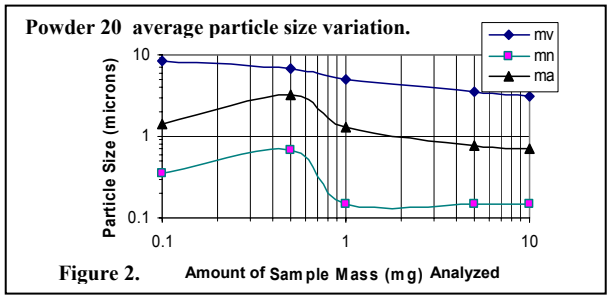

Figure 2.

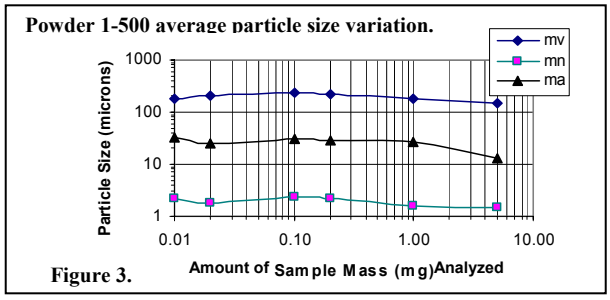

Figure 3.

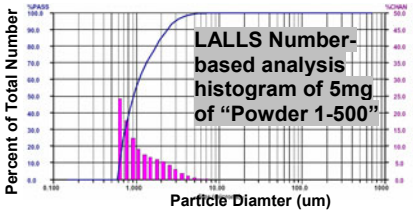

Figure 4.