Laser Diffraction Particle Size Analysis of Non Spherical Particles Synthesized by Hydrothermal Method

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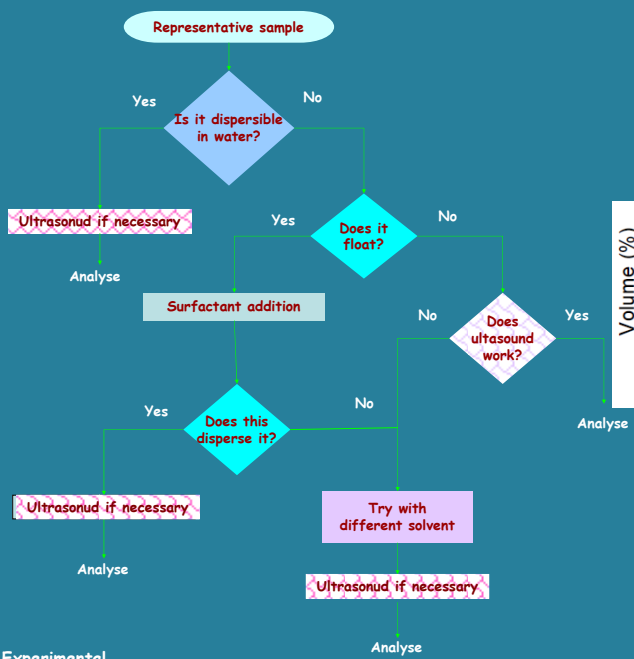
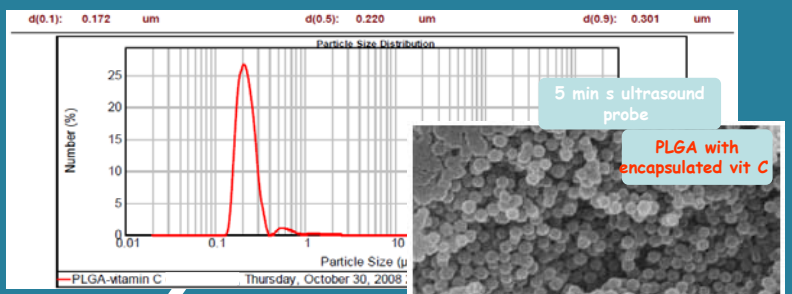
Introduction

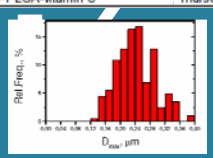
Laser diffraction is nowadays the most used granulometric technique mostly because it is easily used and reproducibile. Modern instruments for measuring light scattering patterns from particles in dispersions can detect particles in the range from 10 nm to 3 mm. Calculations of particle size distributions are based on the Mie approximation, which is the correlation of the scattered light intensity, the angle and the diameter of the spherical particle. The type of scattering depends on the diameter-to-wave length ratio. For larger optically inactive particles the approximate calculation of PSD can be made using the Fraunhofer scattering model. In case $D/\lambda \leq 1$, the optical properties of particles, i.e. the refractive and absorptive indices, must be taken into consideration. Problems related to laser diffraction, such as overestimation of small particles (less than 1 micrometer) and inability to solve nonisometric particles, occur due to poor choice of optical parameters and assumptions that particles are spheres and that they are randomly oriented. Other false results may occur because of pure sample preparation.

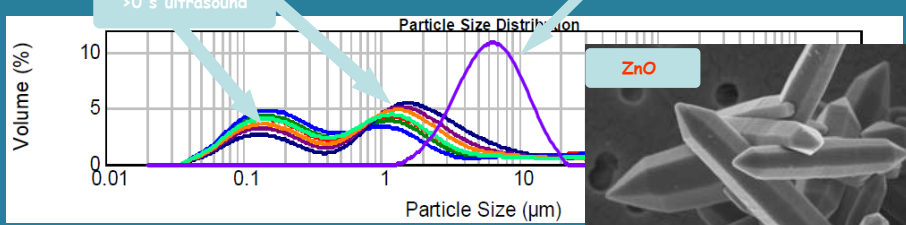


Experimental

For the particle size analysis we employed Malvern Mastersizer 2000 (633 and 455 nm light sources). The measured samples of polylactic-co-glicolic acid as referent spherical and hydrothermaly synthesized zinc oxide, hydroxyapatite and lithium iron phosphate were first dried on air and then redispersed in suitable solvent according to the above scheme. For the measurement we used wet dispersion units Hydro S and Hydro μP. In order to check the LD analysis, SEM and TEM photographs were compared with PSD results. For large $LiFePO_4$ particles Fraunhofer approximation was used. The refractive indices for ZnO, HAp and PLGA used were 2.08, 1.649 and 1.44, respectively. Ultrasound was applied for ZnO, HAp and PLGA. The time of the ultrasound treatment depended on the extent of agglomeration.

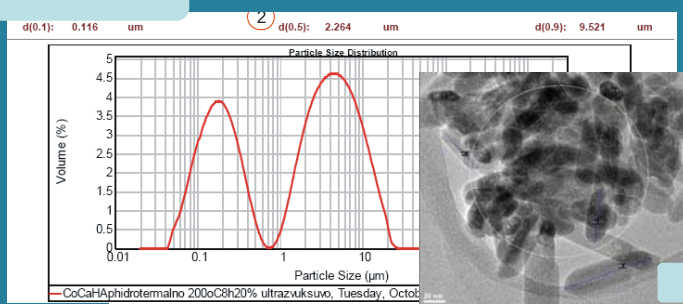


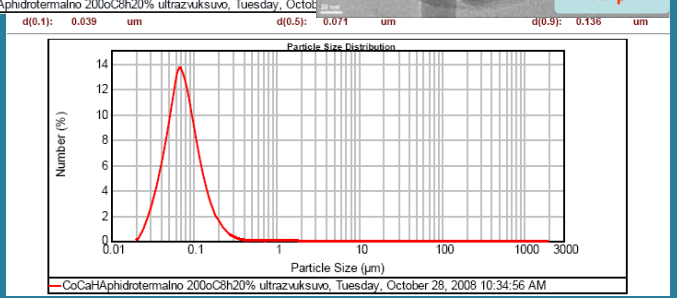


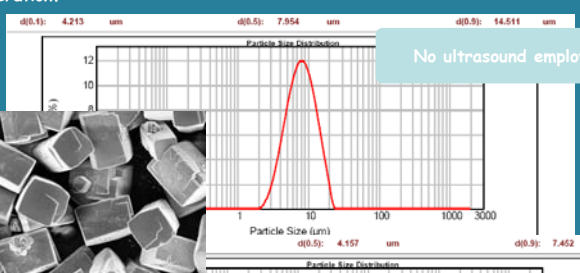


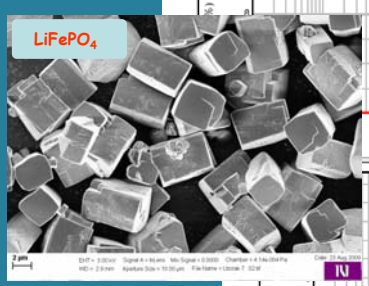
30 min ultrasound dry

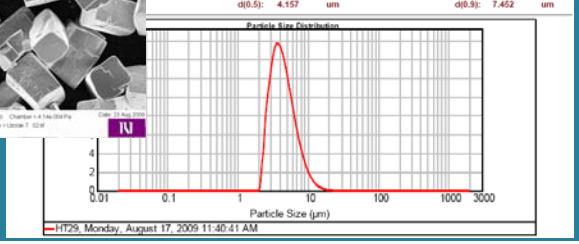
5 min ultrasond in water











Conclusion

In the particle size analysis using laser diffraction, the sample preparation and the choice of optical parameters are essential for reliable results, as it is shown on PLGA and HAp samples. For the analysis of particle sistems containing larger particles ($D/\lambda >> 1$) optical properties are not essential, as it is shown for $LiFePO_4$ particles. In case of non isometric particles, such as rods, wiskers or fibers, the image analysis must be involved in setting up the appropriate LD PSA methodology, as it is shown on the example of ZnO rods.