Investigation of the influencing factors on the formation of TiO_2 nanoparticles during the controlled hydrolysis of titanium ethoxide

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TiO_2 particles are used in a variety of applications like cosmetics, fuel cells and photovoltaics because of their physicochemical properties and their high stability against heat and chemicals. One attribute of the synthesized particles critical to their quality for a certain application is often their size, which is controlled in solution phase synthesis by the addition of different stabilizing agents [1]. It has been reported, that spherical TiO_2 particles form during the hydrolysis of titanium ethoxide in the presence of different salts, while particle size depends on the type and the concentration of the added salt [2]. This work aims at finding empirical relations between the factors influencing the reported synthesis and the properties of the obtained product suspensions. Such relations can be used to optimize the process with respect to a high particle yield while at the same time being able to control the particle size distribution.

The parameter space of factors that could possibly influence the synthesis is large and testing this space would usually require a large number of experiments. A Design of Experiments (DoE) approach, which also considers effects that result from the interaction between factors, reduces the number of necessary syntheses. Understanding the influence of a certain factor also requires observing the synthesis process itself. This is addressed in this study by Photon Density Wave (PDW) spectroscopy, which determines the reduced scattering coefficient μ_s' of particle suspensions at volume fractions even above 1 %. μ_s' is influenced, apart from others, by particle size and volume fraction [3]. Hence, PDW spectroscopy gives information about the process, which cannot be accessed without dilution or sampling by other conventional methods.

The results indicate a strong influence of the type of the added salt, the concentrations of the used chemicals, pH and the reaction temperature on the synthesis process, as e.g. particle yields between 30 % and 99 % were achieved. However, some of the factors that give rise to high particle yields cause a loss of control over the particle size distribution. Hence, multiple parameters will be optimized at the same time to obtain particles with the desired properties.

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