Preparation of Titanium Dioxide Nanoparticles via a Non-Aqueous Sol-Gel Method

Christopher Cadman*, Nicola Tirelli, Francesco Cellesi

Laboratory of Polymers and Biomaterials, School of Pharmacy and Pharmaceutical Sciences, The University of Manchester, Oxford Road, Manchester, M13 9PT, United Kingdom
*e-mail: christopher.cadman@postgrad.manchester.ac.uk

TiO$_2$ is currently used for many applications including water decontamination, sunscreen, photovoltaics etc. All these applications rely on the high photo-active capability that TiO$_2$ exhibits. TiO$_2$ is highly biocompatible, being in effect non-toxic under normal conditions, this biocompatibility coupled with the enhanced photoactivity makes TiO$_2$ an interesting material to study for biomedical applications, particularly for photodynamic therapy.

The main aim of this study to date has been to optimize a method for synthesizing TiO$_2$ nanoparticles, whilst exacting fine control over their size, morphology and surface composition. It is desired for the nanoparticles to have dimensions >5 nm and <100 nm thus taking full advantage of the increased surface area to volume ratio, optimizing the availability of photoactive surface sites. It is desirable for the nanoparticles to have an anatase morphology as anatase is the most photoactive crystal structure of TiO$_2$.

The synthesis of the nanoparticles has been achieved by employing a non-aqueous sol-gel synthetic route. This method of synthesis is preferred over aqueous sol-gel synthesis as no further heat treatment is required, therefore the nanoparticles retain their small size.

In short the synthesis involves the addition of a titanium alkoxy precursor to a benzyl alcohol solution containing small molar equivalents of H$_2$O and/or HCl followed by heating to 80°C. Subsequent to synthesis the particles may be re-dispersed in an acidic aqueous environment. The effect of changing reaction conditions on the growth kinetics and final physical properties of the nanoparticles has been studied in relation to the content of H$_2$O, HCl and the change in hydrophilicity of the titanium alkoxy precursor.

TiO$_2$ nanoparticles have been synthesized in a range of sizes from 8-30 nm with low polydispersity values 0.1-0.2. The resulting particles all have the desired anatase morphology and are stable in an aqueous environment at pH=1.5, having zeta values of over +25 mV. After synthesis the surface of the nanoparticles are highly alkoxylated, however upon re-dispersion these alkoxy ligands are rapidly hydrolyzed. This hydrolyzation produces particles which are effectively ‘naked’ with only hydroxy ligands covering the surface, this allows the surface of the nanoparticles to be easily modified to yield desired characteristics and to enable dispersion at physiological pH.