

Studying the low-temperature crystallization of TiO₂ using design of experiments

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Coating an orthopaedic implant with an osteoinductive material is a common technique to improve bone integration. TiO₂ is one of such materials, as it has proven osteoinductive properties and naturally occurs *in-situ* on titanium-based implants. It is also known that crystallizing TiO₂, namely to its anatase phase, further improves osteoinduction. Anatase can generally be obtained by sintering amorphous TiO₂ at temperatures within the 400-450 °C range, which can be damaging to some substrates. For example, PEEK, a polymer that is used in spinal implants, has a working temperature of 250 °C and a melting point of 343 °C. It is possible, however, to decrease the crystallization temperature of TiO₂ to anatase by carefully controlling the conditions in which it has been synthesised.

Using Design of Experiments, this work aimed to study the sol-gel synthesis of TiO₂ capable of crystallizing into anatase at 250 °C. A definitive screening design was used, with reaction temperature, ethanol volume, H₂O volume, acetic acid volume, sintering heating rate and sintering time selected as factors. Titanium Isopropoxide was used as the TiO₂ precursor. Samples were analysed by TGA and XRD. All but one factors were shown to affect the ability of TiO₂ to crystallize at 250 °C, with the best synthesis conditions obtained by maximizing reaction temperature and sintering time, minimizing ethanol volume, and setting H₂O and acetic acid volumes to an optimum value.