Summary

Nanocrystalline titanium dioxide has been widely experimented as catalyst and photo catalyst. The photocatalytic property of titania was effectively used in applications such as water and air purification, water splitting, solar cells and in fabricating self cleaning surfaces. The thesis entitled “Aqueous sol-gel process for nanocrystalline photocatalytic titania, transparent functional coatings and ceramic membrane” investigates the plausibility of an aqueous sol-gel process for the preparation of nanocrystalline titania for functional applications.

The present thesis describes a new aqueous sol-gel method for the preparation of nanocrystalline titania using titanyl sulphate as precursor through precipitation-peptisation reactions. The sol stability was monitored using zeta potential measurement and was highly dependant on peptised pH. The lower particle size was obtained in the acidic region. Titania sol having average particle size of 27 nm and a zeta potential value of 35 mV stable for months, could be prepared. The anatase to rutile transformation was found to occur in the region 500-800°C, which is in accordance with earlier reports for nano crystalline titania. The nano crystalline nature was evident from X-ray diffraction patterns and the crystallite size calculated from Scherrer equation was ~20 nm for the titania powders after calcining at 600 °C. Transmission electron micrographs also confirm the result.

The prediction of Shanon and Pask was used to increase the temperature stability of anatase derived from aqueous sol-gel process. Accordingly +3 dopants with size higher than that of Ti occupying lattice sites and +5 dopants with size similar to that of Ti occupying interstitial sites were used which should decrease oxygen deficiency concentration in titania. Oxygen vacancy acts as the nucleation centre for anatase to
rutile phase transformation and also as charge recombination centers. Further process was optimized to investigate other parameters which influence photoactivity such as high specific surface area, small crystallite size and higher anatase to rutile phase transformation temperature. In the present study the influence of +5 dopant tantalum, and +3 dopants, gadolinium and ytterbium on the photocatalytic activity of the titanyl sulphate derived sol-gel titanium oxide is presented. The addition of dopant salts was made to the sol in the process for the preparation of doped titania. Photocatalytic activity of titania was measured by following degradation measurements of methylene blue solution under UV exposure. In the case of tantalum addition, the photoactivity was found to increase and compositions with activity better than commercially available Hombikat UV100 could be prepared. The crystallite size, anatase to rutile phase transformation, specific surface area and photo catalytic property are explained. In this study, we observed that the selected dopants in the titania matrix increase the phase stability of the titania. In the case of tantala doped titania sample the phase stability was obtained above 1000 °C. In gadolinium and ytterbium doped sample also the phase stability is obtained upto 900 °C but in higher concentration of dopant additional phase such as Gd$_2$TiO$_7$ and Yb$_2$TiO$_5$, these phases increase the rate of anatase to rutile phase transformation at higher temperatures. We have also observed that the anatase to rutile phase transformation happens only when the crystallite size reaches a critical value between ~12-20 nm in tune with reported literatures. Transmission electron micrographs also indicate that the doping with tantalum, gadolinium and ytterbium effectively hinder the crystallite growth. The specific surface area enhanced in the doped system and it shows higher value than the undoped one in the whole range of temperatures studied. The catalytic sites (lewis and brownsted sites) also increased in doped titania.
Gadolinium and ytterbium doped titania show higher photoactivity than the pure titania calcined at 700 and 800 °C.

The aqueous sol-gel derived titania was further used for the preparation of titania films on the surface of porous alumina and glass surfaces. The porous alumina tubular substrate 10 mm diameter had an average pore size of 0.2 μm and was coated with an intermediate layer of alumina using a boehmite sol (average particle size 165 nm) followed by calcinations at 800 °C for 5h. This was followed by coating a titania layer using titania sol (average particle size 27 nm). The coated samples were further characterised using scanning electron microscope, which showed that the alumina intermediate layer has a thickness of ~1-2 μm. The total thickness of alumina and titania layer was ~4-5 μm. The coated membrane was used for the filtration of organic compound such as congo red. A 99.3% of rejection of dye was observed in the permeate. Further nano titania thin films doped with alumina were formed on glass surface using titania sol derived from titanyl sulphate. Titania coatings having transmittance between 85 to 95 %were developed on the glass surface using dip coating method. The UV visible spectral characteristics indicate a blue shift in the absorption spectrum. Scanning electron microscopic and atomic force microscopic investigations indicate that a thin layer of titania was formed on the glass surface. BET measurement indicates that a mesoporous layer was formed on the glass surface. The alumina addition increases the surface area, total pore volume and mesopore volume of the system. The alumina doped titania thin film shows higher photoactivity than that made using the pure titania.
List of Publications


Presentations and Posters at Symposia


