PREPARATION OF NANOCRYSTALLINE TITANIUM DIOXIDE PARTICLES FROM NEW-ZEALAND ILMENITE

by

RANJEETH KUMAR RAJASHEKAR R

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Abstract

Titanium dioxide being one of the most important composite precursors has wide range of application due to the unique properties that it exhibits. TiO_2 with varying amount of anatase and rutile phases were prepared by controlled hydrolysis of dissolved liquor (Ti—Fe—Cl solution) from dissolution of New Zealand ilmenite followed by calcination of the hydrate sample at different temperatures. The kinetics of ilmenite digestion is examined based on the factors affecting the ilmenite dissolution rate such as acid/ilmenite ratio, additive (iron powder) and optimum dissolution temperature. In hydrolysis, the use of structure determining agents (SDA) that alters the morphology of TiO_2 fine particles is analyzed. Samples without SDA have resulted in rutile phase formation at 110°C, while samples with SDA (phosphoric acid/tri-sodium citrate/citric acid) resulted in either anatase phase or mixed phase (both anatase and rutile) at 110°C. The phosphate and citrate ions (0.35% P_2O_5 and 0.4% citrate) helps in promoting an anatase phase of TiO_2 particles. Along with SDA, parameter such as hydrolysis temperature and percentage seed also affects the intermediate product.

The influence of calcination temperature ranging from 925°C—1000°C on anatase-rutile phase transformation and variation in crystallite size was studied. X-ray diffraction (XRD) and scanning electron microscopy (SEM) were employed to characterize the resultant TiO_2 phase, crystallite size and particle size and shape. The degree of conversion to rutile was higher at higher calcination temperature. Introducing potassium additive (0—2 mass% K_2O) in the hydrate sample enhanced the anatase-rutile phase transformation at higher calcination temperature. However, the potassium content in the hydrate sample has a negligible effect on the crystallite

size of anatase and rutile after calcination. The XRD pattern shows an increase in the rutile peak intensity and a decrease in the anatase peak intensity with higher calcination temperature. SEM images show that the particle size of the calcined product at 975°C with 1% K_2O ranges from 230nm—300nm.

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List of Abbreviations

1D- one dimensional

2D- two dimensional

$2\theta_A$ – Braggs angle of anatase peak

$2\theta_R$ – Braggs angle of rutile peak

3D- three dimensional

AAM - anodic alumina membrane

ALD - atomic-layer deposition

CF-CVC – combustion flame-chemical vapor condensation

CMC – critical micelle concentration

CVD – Chemical vapor deposition

d_A – crystallite size of anatase sample

d_R – crystallite size of rutile sample

DSSC – dye-sensitized solar cell

EPA – Environmental protection agency

ESEM – Environmental scanning electron microscopy

Et-OH – Ethanol

FWHM – Full width at half maximum

GENS – Green earth nano science

HF – Hydrogen fluoride

I_A – Intensity of anatase peak

I_R - Intensity of rutile peak

MOCVD – metal organic chemical vapor deposition

OA - oleic acid

PDMAEMA-b-PFOMA - dimethyl amino ethyl methacrylate-block-1H,1H,2H,2H-perflourooctyl methacrylate

PTCB – peroxo-titanium solution chemical bath

PVC – Poly vinyl chloride

PVD – physical vapor deposition

RBF – Round bottom flask

SDA – Structure determining agent

SEM - scanning electron microscopy

TEOA – triethanolamine

THyCA – transfer hydrolytic crystallization in alcohols

TOPO – trioctylphosphine oxide

TTIP - titanium tetraisopropoxide

UV – Ultra violet

XRD – X-Ray powder diffraction

β_A – FWHM of anatase peak

β_R – FWHM of rutile peak