



# XPS and FTIR investigation of the surface properties of different prepared titania nano-powders

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## Abstract

Surface studies of nano-sized TiO<sub>2</sub> powders prepared by different methods showed that the preparation method had great impact on the surface properties. XPS measurements showed that the oxygen composition was related to the preparation method. The chloride method yielded the lowest amount of surface oxygen (29%) and sol–gel prepared powder showed the greatest amount of surface oxygen (66%) in the form of surface hydroxyl groups. The remaining oxygen was identified as lattice oxygen. The powder prepared by the sol–gel method contained carbon impurities originating from residual alkoxy groups. Supercritical sol–gel prepared powder and powder prepared by the sulphate method revealed same trends regarding oxygen composition with 44–47% being surface oxygen; neither contained carbon impurities. The results obtained from XPS were confirmed by FTIR measurements.

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## 1. Introduction

Titanium oxide nano-particles with high specific surface area are of considerable interest in photocatalytic applications [1]. Several commercially available nano-sized powders are prepared by either the sulphate or the chloride process [2], and during the last two decades the sol–gel process has been developed. The sol–gel process offers several advantages for

producing nano-particles including a simple low cost technology, lower process temperatures [3], and flexible control of the structure and size of final products using many operating parameters [4]. The possibility to fine tune the particle properties by the sol–gel process is particularly well suited for photocatalytic applications, where small particle size, high surface area, and high crystallinity are often considered as key parameters for high photocatalytic activity of nano-particles [5–10]. At the same time, the need for defining as many surface properties as possible is important, since the final level of photoactivity is determined not

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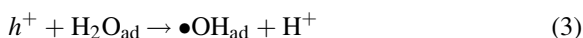
only by semiconducting properties but also by the physicochemical ones, such as the ability of light absorption, the amount of adsorbed reactant species, and the nature of the interface [11].

A great deal of work has been done in describing the photocatalytic activity of nano-sized TiO<sub>2</sub> as a function of surface properties [12–14]. Recently, it has been recognized that the presence of defects in the surface of TiO<sub>2</sub> [15] are of great importance. However, to our knowledge no one has successfully described the effect of defects, hydroxyl groups, surface oxygen, and impurities on the photocatalytic activity of nano-particles.

Kumar et al. investigated nano-crystalline TiO<sub>2</sub> by XPS and FTIR [16]. They observed that two different preparation methods yielded different surface properties and surface states of Ti for nano-sized TiO<sub>2</sub>. The different chemical states of titanium, Ti<sup>4+</sup> and Ti<sup>3+</sup>, and oxygen are essential in photocatalysis. The primary step in photocatalysis is the generation of electrons and holes:



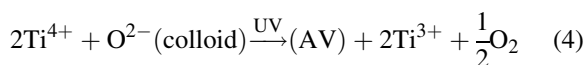
In the presence of dissolved oxygen and an electron donor •OH radicals are formed by the reaction between valence band holes and the TiO<sub>2</sub> surface active OH<sup>-</sup> groups or H<sub>2</sub>O:



The electron is trapped by dissolved oxygen or defects.

According to Eq. (2), increasing surface hydroxyl content will enhance photocatalytic activity. As mentioned above, the presences of defects also have

a positive effect on the photocatalytic activity due to electron capturing, that diminish recombination of electron and holes. Ti<sup>3+</sup> defects are believed to be created by the removal of surface oxygen atoms as [17]:



where AV are anion vacancies.

In this paper, the surface properties of four titanium dioxide photocatalysts prepared by different techniques are investigated. Industrial powders Degussa P25, Hombikat UV100, TiO<sub>2</sub>\_5 nm, and a homemade powder were investigated by X-ray photoelectron spectroscopy (XPS) and Fourier transformed infra-red (FTIR) spectroscopy to determine if the preparation method had an impact on the surface properties. XPS and FTIR spectroscopy are well suited for such physicochemical investigations, due to the ability to define not only the elements present at the surface, but also the chemical state of the elements.

The presented XPS investigation was focused on XPS spectra peaks corresponding to titanium, oxygen, and carbon to distinguish powder prepared by different methods by the different chemical state of surface species. The results obtained from XPS were compared to FTIR.

## 2. Experimental

### 2.1. Materials

Degussa P25 is a commercially available powder from Degussa Company. It is produced by the chloride

Table 1  
Measured particle properties from [23,24]

	Degussa P25	Hombikat UV100	TiO <sub>2</sub> _5 nm	SSEC78
Crystallinity (%)				
Anatase	72.6	67.2	60	62.6
Rutile	18.4	–	–	–
Amorphous	9	32.8	40	37.4
Crystallite and particle size (nm)				
XRD	35.1	12.3	13.7	10.1
SSA	30 (50 m <sup>2</sup> /g)	4.2 (360 m <sup>2</sup> /g)	7.0 (220 m <sup>2</sup> /g) <sup>a</sup>	6.4 (236 m <sup>2</sup> /g)
Photocatalytic activity (min)				
<i>t</i> <sub>1/2</sub> (chloroform)	5.7	18.3	8.1	12.4

<sup>a</sup> From: [www.nanoamor.com](http://www.nanoamor.com).

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