



# Nanoparticulate titanium dioxide synthesized by sol–gel and solution combustion techniques

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## Abstract

Nano-sized titanium dioxide is renowned for its prominent photocatalytic properties. Various synthesis techniques have been successfully employed in production of nano-titanium dioxide. Some of the techniques require expensive initial reagents as well as extensive preparation time. A rapid, simple and cost-effective technique capable of producing high quality nano-particles is highly desirable. This study, therefore, aims at examining effects of synthesis techniques and initial reagents on chemical compositions and particle sizes of titanium dioxide. A solution combustion technique as well as a sol–gel method using Titanium (IV) isopropoxide (TTIP) and submicrometer-sized titanium dioxide as initial reagents were employed as synthesis techniques. Experimental results indicated that all synthesized powders contained pure anatase titanium dioxide. Average particle sizes of powders prepared by the solution combustion technique from TTIP and submicrometer-sized titanium dioxide were 44 and 77 nm whereas, powder sizes prepared by the sol–gel method averaged at 48 and 85 nm respectively.

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## 1. Introduction

In the last few decades, nanomaterials have drawn worldwide attention because of their unique properties. Nano-sized titanium dioxide is one of the materials renowned for its prominent photocatalytic properties [1,2]. It has been accepted that titanium dioxide is a polymorph materials, which exists in three main structures, anatase (tetragonal), rutile (tetragonal) and brookite (orthorhombic). Pure anatase or mixture of anatase and rutile at ratio of 80:20 is desirable for photocatalytic applications due to their high reactivity [3]. In addition to crystalline structures, particle size is also a factor contributing to high catalytic activities of the titanium dioxide particles.

Small particles in the range of nanometer have high specific surface area, which greatly enhance reactivity. To attain the desirable characteristics, nano-sized powders with high quantities of the anatase phase needs to be carefully monitored during titanium

dioxide synthesis. Common techniques employed in the synthesis process of nano-sized titanium dioxide include hydrothermal [4], co-precipitation [5], sol–gel [1,6] and solution combustion technique [7]. The solution combustion and sol–gel methods are simple and effective synthesis techniques of obtaining nanometer or submicrometer-sized powders with high compositional homogeneity. In addition, both techniques require cost-effective initial reagents and low energy supplies during the synthesis. The techniques also accommodate doping of various cations and anions [8,9].

As mentioned previously, numerous research works have reported techniques related to the synthesis of nanoparticulate titanium oxide. Some of the techniques, however, require expensive initial reagents as well as extensive preparation time. Rapid, simple and cost-effective techniques capable of producing high quality nano-particles are highly desirable. This study, therefore, aims at examining effects of synthesis techniques and initial reagents on chemical compositions and particle sizes of titanium dioxide. Simple routes that require low energy supply during the synthesis, specifically the sol–gel and the combustion techniques were employed as synthesis techniques of titanium dioxide in this

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study. To reduce processing cost, cost-effective and commercially available titanium (IV) isopropoxide and submicrometer-sized titanium dioxide were used as initial reagents.

## 2. Materials and methods

The solution combustion technique and the sol–gel technique were employed in the synthesis of  $\text{TiO}_2$  powders. To synthesize the powders by the solution combustion technique, aqueous solutions containing titanium ions were prepared. Titanium (IV) isopropoxide (TTIP) (Aldrich chemistry<sup>®</sup>, 97%) and micron-sized titanium dioxide ( $\text{TiO}_2$ ) (Sigma-Aldrich<sup>®</sup>, 99%) were used as initial reagents for the combustion synthesis. For the combustion process prepared from Titanium (IV) isopropoxide, deionized water was added to the TTIP to obtain a 2.5 M concentration solution. For the process prepared by micron-sized titanium dioxide, the titanium dioxide powder was dissolved in sulfuric acid ( $\text{H}_2\text{SO}_4$ , Emsure<sup>®</sup>, 95–97%), and subsequently diluted with deionized water to obtain a 6 M concentration solution. Glycine ( $\text{NH}_2\text{CH}_2\text{COOH}$ , Fluka analytical<sup>®</sup>, 99%), acting as combusting fuels, at a ratio of 1:1 glycine:titanium was added to the prepared TTIP and titanium dioxide solutions. The mixtures were subsequently stirred by a magnetic stirrer and placed in an ultrasonic bath for 10 min. A low heating temperature of less than 400 °C was applied to the solution to initiate combustion. Upon completion of the combustion reaction, the powder products were collected and calcined at 250 °C and 500 °C for 2 h.

To synthesize the powders from TTIP by the sol–gel technique, ethanol at the volumetric ratio of 1:2 ethanol:titanium isopropoxide (TTIP), was added to TTIP. High acidic condition (pH 2) was generally required for synthesis of titanium oxide. This could be achieved by adding nitric acid (Univar<sup>®</sup>, 70%) into the prepared solution. The solution mixture was subsequently stirred by a magnetic stirrer for 30 min. To synthesize titanium dioxide nanoparticles from the micron-sized titanium oxide, the titanium dioxide powder was dissolved in sulfuric acid, and subsequently diluted with

deionized water to obtain a 2.3 M concentration solution. Polyacrylic acid ( $\text{C}_3\text{H}_4\text{O}_2$ )<sub>n</sub> (Carbopol<sup>®</sup>), acting as gelling agent was added to the prepared solution. Starting when gelation of the titanium (IV) isopropoxide and micron-sized titanium dioxide mixtures occurred, the process was allowed to continue for 24 h. Heating the gels to 300 °C, the gels were transformed into fine powders, which were then collected and calcined at 250 °C and 500 °C for 2 h.

The composition of the calcined powders were investigated using an x-ray diffractometer (Bruker, D8 Advance), over angles ranging from 20° to 80° in  $2\theta$ , at a step size of 0.02° and a scanning rate of 1.3°/min. A scanning electron microscope (FEI Quanta 450) was employed in the morphological examination of the particles. Particle sizes were determined using Image J Software and the Scherrer equation.

## 3. Results and discussion

### 3.1. Chemical compositions

X-ray diffraction analysis of the calcined powders, as shown in Figs. 1 and 2, indicated that all powders exhibited complete formation of anatase phase  $\text{TiO}_2$  (JCPDS 01-086-1157) with the predominant peak at  $2\theta=25.4$ , 37.8, 48.0, and 54.5 which corresponded to the planes (101), (004), (200), and (105), respectively. Neither rutile nor brookite  $\text{TiO}_2$  phase was evident in the diffraction patterns obtained.

Single phase of the anatase titanium dioxide appeared in the x-ray diffraction patterns of the powders prepared by the solution combustion technique. Pure anatase phase obtained from the solution combustion technique might be attributed to appropriate quantities of combustion fuels and appropriate calcination temperature used in this experiment. It has been reported that inadequate amount of fuels led to incomplete reaction, which consequently resulted in powders with initial reagents still remaining. On the contrary, excessive quantity of fuel could promote formation of undesired carbon compounds [10].

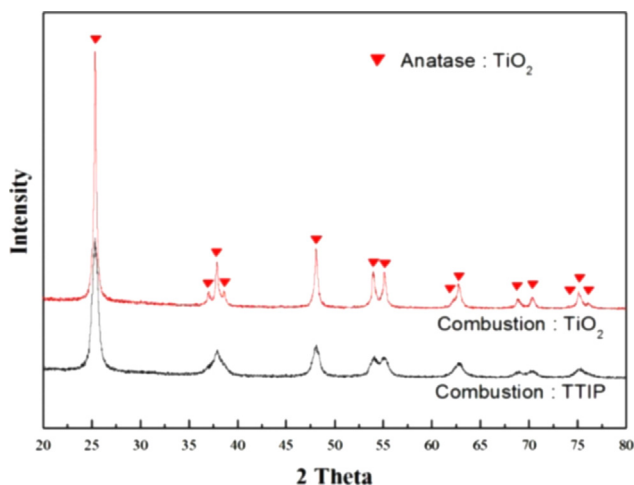


Fig. 1. X-ray diffraction patterns of powders synthesized by the solution combustion technique, prepared from titanium (IV) isopropoxide (TTIP) and sub-micrometer titanium oxide.

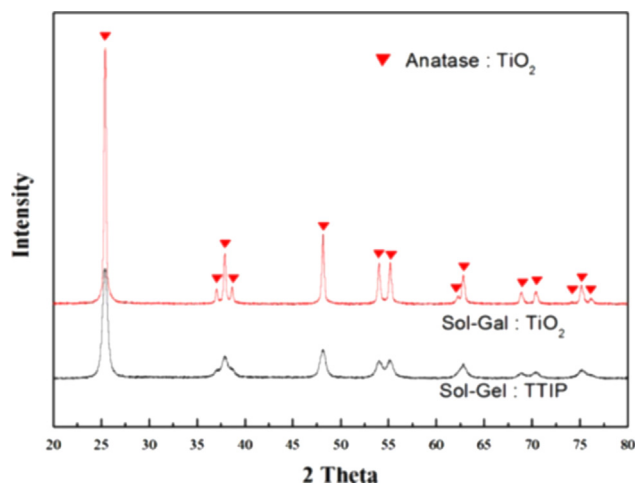


Fig. 2. X-ray diffraction patterns of powders synthesized by the sol–gel technique, prepared from titanium (IV) isopropoxide (TTIP) and sub-micrometer titanium oxide.

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