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SYNTHESIS AND CHARACTERIZATION OF TITANIA (TiO_2) DERIVED BY SOL-GEL AND HYDROTHERMAL PROCESSES

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ABSTRACT

In this research, anatase Titania nanoparticles were synthesized using two methods, i.e. sol-gel and hydrothermal using titanium isopropoxide as a precursor with deionized water as solvents. X-ray diffraction and Fourier-transform infrared spectroscopy were conducted to describe the formation and characterization of crystal structure and functional groups. The particle size was achieved from sol-gel method was 12 nm and 20 nm from the hydrothermal method using the Scherrer equation. The anatase crystal structure was confirmed through X-ray diffraction peaks at 25°, 38°, 47°, 53° and 62.6°.

Key words: Sol-gel, Hydrothermal, Titania, Photovoltaic.

I. INTRODUCTION

Metal oxide nanoparticles are considered as important technological material and frequently synthesized and intensively studied. In compare to bulk compounds metal oxide nanoparticles shows different physiochemical behaviors, with enhanced optical, surface, thermal and electrical properties [1]. Recently; Titanium dioxide (TiO_2) is gained potential interest due to its extra ordinary properties for various applications as shown in table 1 [2]. It is fourth most abundant material, consisting about 0.63% of the earth crust [3]. TiO_2 is non-toxic and biocompatible, inexpensive materials that exhibits excellent photo efficiency and photoactivity. The wide range of its application from common products as sunscreens [4] to advance devices for photovoltaic cells [5], including a range of environmental and biomedical applications [6], including photocatalytic pollutant degradation [7], water purification [8], biosensing [9], and drug delivery [10]. All these outstanding properties of TiO_2 contributed in making model for all metal oxides. Three common polymorphs i.e. anatase, brookite and rutile of titanium dioxide is naturally found in nature. However only rutile is thermodynamically stable at all temperature and pressure. Different methods were reported to synthesis this Titania, including sol-gel, hydrothermal, solvothermal, direct oxidation, sonochemical, electrode position, emulsion , anodization , vapor deposition, sonication and microwave [11-14]. Hydrothermal and sol-gel techniques, unlike other techniques, have an environmentally safe and low-cost way and the ability to monitor homogeneity, chemical structure, morphology, purity, phase composition and powder form under moderate conditions such as pH value, reaction time and temperature effect in the processing of TiO_2 products. The materials final properties are depend entirely on the precursor, synthesis process, the experimental conditions, and the polymorphic structure [15]. Several reports conclude that in the formation of nanoparticles, the hydrothermal treatment is substantially the most important step in comparison to the washing process. Anatase phase TiO_2 has a crystalline structure that follows the tetragonal form as shown in figure 3 and the low recombination frequency of photogenerated electrons and hole makes it more attractive that is why primarily used as a UV irradiation photocatalyst, anticorrosive coatings and antireflection films [16]. Rutile phase TiO_2 has a tetragonal crystal structure as well. This form of titanium oxide is used mainly in paint as a white pigment. The TiO_2 brookite phase has a crystalline orthorhombic structure [17].

The transformation from one phase to another phase is independent to synthesis temperature, since there is no equilibrium between the polymorphs of Titania. Depending upon the preparation condition the anatase polymorph have been achieved at different temperature ranging from 600 to 1100 °C, which shows the influence of several factors on the phase change of Titania. Insight study on the factors that affect growth, phase stability and phase transformation kinetics is important to quantification of materials properties.

The process requires the direct hydrolysis of titanium alkoxide with acetic acid. Compared to other methods of synthesis, the procedure reported in this study presents many aspects that may be an attractive alternative to obtaining Titania anatase nanoparticles with uniform crystalline phase purity within a relatively short period of time. Simplicity and quickness of the process are the main advantages, as only titanium precursor, acid and NaOH need to be used.

Table-1: Properties of Anatase Titania

Phase name	Anatase
Formula	TiO_2
Molecular weight	79.899
Density	3.895
Molecular volume	20.516
Atomic number	4
Crystal structure	Tetragonal
Band gap	3.2 eV

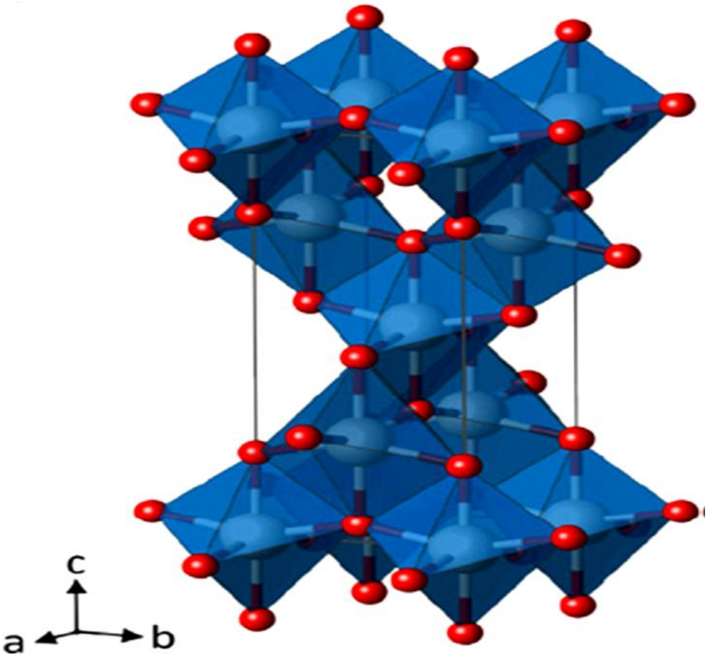

Fig.-1: Tetragonal crystal structure of Anatase phase Titania

II. MATERIALS AND EXPERIMENTAL WORK

Materials

Titanium tetraisopropoxide (DEAJUNG), NaOH (Merck KGaA) and acetic acid (99.5%) (Panreac, Barcelona, Spain) and deionized (DI) water.

Synthesis of TiO_2 by Sol-gel and Hydrothermal methods

In a beaker containing a magnetic bar, 40 ml of cold DI water was placed. Suddenly, 4 ml of TTIP was added to the beaker, covered with a closure and stirred for 30 minutes. Then, 9 ml of acetic acid was included and left at room temperature under continuous stirring for 3 h. The suspended particles were then precipitated with 20 ml of 1M NaOH solution by adjusting the pH of the suspension. When a pH of 7 was obtained, the precipitated particles were washed with DI water. Particles were finally permitted to settle and the residual water was extracted. For hydrothermal method, there was additional step that sample was transferred into Teflon steel lined in an oven at 180 ^{0}C for 12 hrs. The Titania was dried out in an air oven at 100 ^{0}C for 12 h. Samples were label by SG-Titania for sol-gel and HT-Titania for hydrothermal methods.

III. RESULTS

The XRD pattern of the synthesized Titania from sol-gel and hydrothermal methods are shown in figure 1. The peaks are found at 25°, 38°, 47°, 53° and 62.6° which are corresponded to crystal planes (101), (112), (200), (105) and (204). All peaks are distinct and are perfectly assigned to the tetragonal anatase Titania. The crystalline size of the prepared samples is found 12 nm from the sol-gel method and 20 nm from the hydrothermal method, calculated by Scherer equation [18].

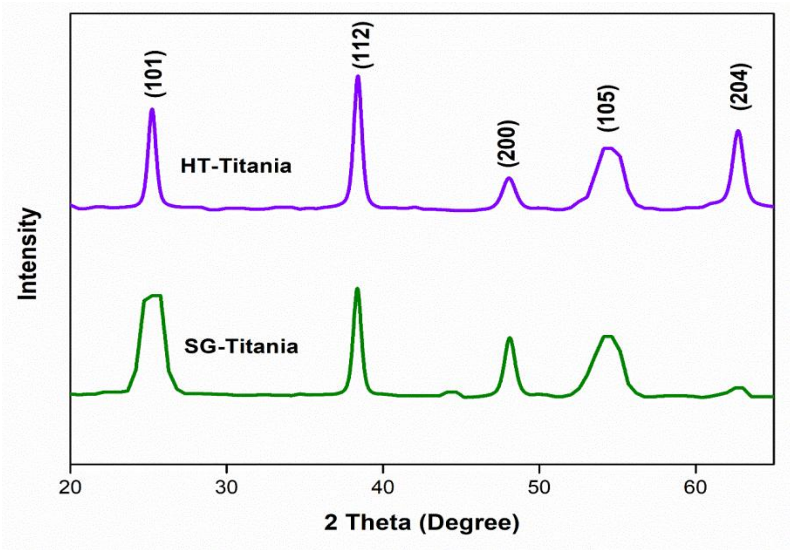

Fig-2: XRD pattern of Titania from sol-gel and hydrothermal methods

The FTIR spectra of both methods of Titania are shown in figure 2. The bands are obtained at 3323 cm^{-1}, 1632 cm^{-1}, 1428 cm^{-1} and 876 cm^{-1}. The band at 3323 cm^{-1} is attributed to physisorption of O-H stretching. The band at 1632 cm^{-1} resembles to the bending of O-H group. The band at 1428 cm^{-1} is due to carbon vibrations. The broad band at 876 cm^{-1} is of Ti-O-Ti group [19].

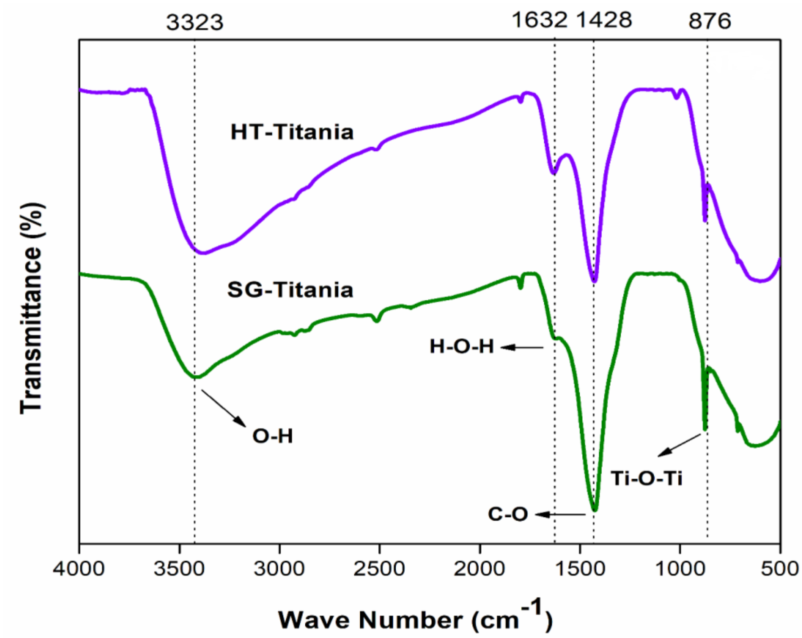

Fig.-3: FTIR spectra of Titania from sol-gel and hydrothermal methods

IV. CONCLUSION

From both sol-gel and hydrothermal methods, pure anatase phase was achieved. The advantage of this process that requires only TTIP, acetic acid and NaOH which simplifies the method and makes it an eco-friendly process and can be used for bulk production of Titania. From Scherrer equation, 12 nm and 20 nm particle size were calculated from sol-gel and hydrothermal samples respectively.

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