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Structural and Morphological Characterization of Titania Nanostructure Synthesized Using Hydrothermal Method

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***Abstract* - In present work, hydrothermal method was used to synthesize, (n-TiO_2) Titania nanotubes using homemade system. These tubes were prepared at two different reaction times such 10 and 14 hours with fixing reaction temperature at 110 ^{0}CThe final samples structural and morphological characterizations were carried out using XRD technique and SEM. The results show that the hydrothermal route is a simple and low coast way to produce finite Titania nanotubes.**

Keywords - Hydrothermal method and morphological characterizations, Nanotubes, Titania.

INTRODUCTION

Since of their special crispness and structural properties, nanostructured materials have piqued interest as catalysts. Much attention has been paid to important oxides like TiO_2, SnO_2, VO_2, and ZnO. Titania nanostructures have attracted a lot of attention due to their unique catalytic properties, low cost, and lack of toxicity [1]. Titanium powders are widely used in catalysis as both an active component and a catalyst support. Titania's use in medical applications has recently expanded to include artificial heart valves and dental implants [2]. Titania has been used in these various applications due to an increase in interest in the development of porous, high surface area, and complex forms of titania-based materials [3].

As among various methods for producing TiO_2 nanostructures [4,] the alkaline hydrothermal response was widely used due to its high efficiency and dependability. Hydrothermal synthesis is typically carried out in steel vessels known as autoclaves, with or without Teflon liners, at controlled conditions of temperature and/or pressure, with the response taking place in aqueous systems. The temperature could be raised just above boiling temperature, resulting in vapor saturation point. The benefit of this method, which occurs under elevated conditions of temperature and pressure, is that it allows for the creation of materials with highly homogeneous nanoparticles [5].

Kasuga et al. at 1998 [6], were produced tiatnia nanotubes (TNTs) using a hydrothermal method by. For its simplification and capacity to make large-area nanotubes, the hydrothermal route is widely used in the synthesis of TNTs [7]. This method is based on the reaction of TiO_2 nanoparticles with a coordinated NaOH solution at elevated pressure. This method works well for the formation of nanotubes [8]. Titanium powders are widely used in catalysis as both an active component and a catalyst support. Titania's use in medical applications has recently expanded to include artificial heart valves and dental implants [2]. Titania has been used in these various applications due to an increase in interest in the development of porous, high surface area, and complex forms of titania-based materials [3].

Titania nanowires (TNWs) synthesis using several methods including: electrochemical deposition [9], vapor deposition [10] and VSL growth [11].

In overall, alkaline hydrothermal therapies of TiO_2 NPs yielded 1D-TiO_2 nanostructures such as NTs and NWs [12]. They were thermally metastable, allowing them to be quickly converted into steady TiO_2 nanomaterials. As a result, these 1D main TiO_2 nanostructures were suitable titanium precursors and templates for the synthesis of other ancillary TiO2 nanoparticles [13, 14]. In the present work, titania nanotubes (TiO_2 NT's) were synthesized via hydrothermal method under a fixed temperature at (110 ºC) and two different reaction times (10) and (14) hours.

EXPERIMENTAL PART

The solvothermal system (temperature control system, Teflon lined stainless steel exploit a vulnerability with a volume of 120 ml) was built first (homemade). All row materials used in the synthesis of the nanostructure have been of analytical reagent grade. The Teflon was cleaned for 10 minutes in a diluted HCl (20 percent) solution before being washed in DI water. The structures also were ultrasonic assisted cleaned in an ethanol/acetone (1:1) combination, DI watered, and air-dried.

All of the chemicals used in this study came from commercial sources and were not purified before use. The reagent used for the synthesis of TNT's were TiO_2 (Aldrich, 99 % purity) and sodium hydroxide (Aldrich, 99 % purity). Firstly, 2 gm of TiO_2 was added into 4M of sodium hydroxide (NaOH) aqua key through vigorous rousing at room temperature for one hour, then this mixture was put in autoclave for 10 and 14 hours to obtain the final nanopowder at 110 ºC. The resultant powder mixed with H_2SO_4 acid to fix the pH at 7. The autoclave then was left to cool down. To obtain the resultants, the samples were washed with distilled water and centrifuged to remove the organic impurities. It was then dried for 3 hours at 100°C and calcined for 6 hours at 550°C.

To investigate the phase and crystalline structure, a (Rigaku XRD with the standard Cu-K radiation source, = 0.15418 nm) operated at 40 kV and 40 mA at room temperature in the 2 range from 10° to 90° at a rate of 0.01°s-1 was used. The samples' structure and shape were determined using (SEM, Hitachi S-4800 microscope).

RESULTS AND DISCUSSION

The hydrothermal time plays paramount lead on the TiO_2 NT's formation. The period of hydrothermal was showed at 10 hs and 14 h.

Two dimensions TiO_2 Nano sheet was the firstly produce of alteration from 3D TiO_2 NP's to 1D TiO_2 NT's under high alkaline condition. A TiO_2 NP's reacted with great attention of H2Ti3O7 Nano crystals form a highly disordered phase in a NaOH solution. TiO_2 NPs are exposed into thin lamellar remains under hydrothermal conditions. Huge quantities of Ti-O bonds have been cracked to form H2Ti3O7 that after removal procedure has progressed to a certain extent. It's worth noting that the transformation happened gradually, starting at the surface and working its way up to the core. As a result, the surface Ti-O bonds have been cracked slightly earlier on. During in the breaking of Ti-O bonds, the O atoms were distorted or repositioned, resulting in an angle here is between Ti and O atoms.

The intensity of the rutile peaks increases in an XRD analysis performed between 10 and 14 hours (Figures 1 and 2, respectively). These findings suggest that anatase crystallites are primarily found at the metal-oxide interface, while the rutile layer is located at the external interface.

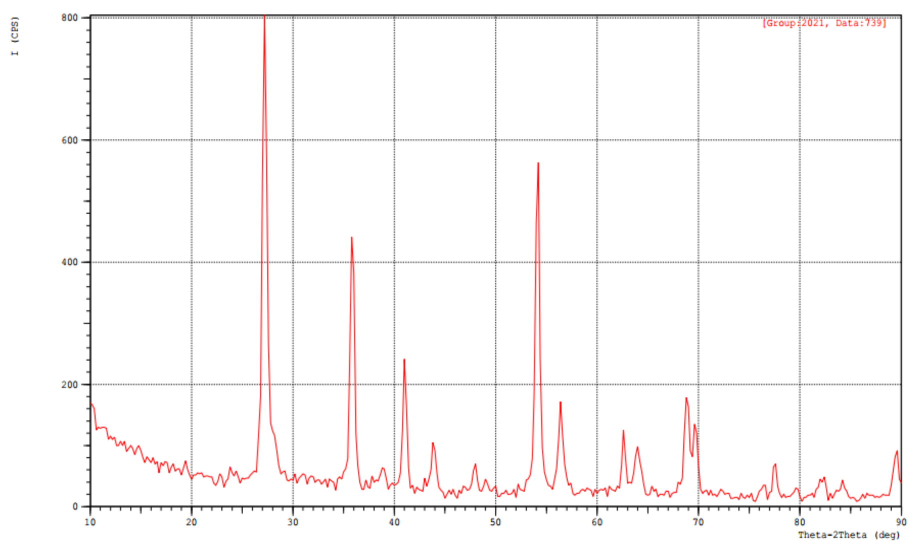

FIGURE 1
XRD PATTERN OF TiO_2 NT'S HYDROTHERMAL TREATED AT 10H

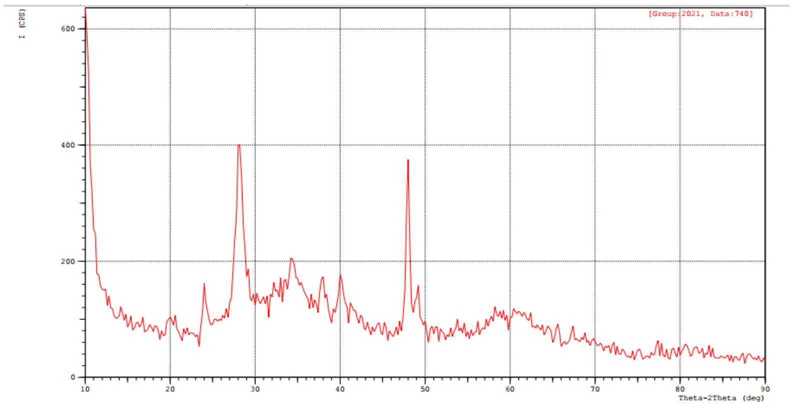

FIGURE 2
XRD PATTERN OF TiO_2 NT'S HYDROTHERMAL TREATED AT 14H

As shown in figures 1 and 2, XRD pattern represents the peaks at 2θ = 25.3°, 38.0°, 48.18°, 54.8°, 55.8°, 62.58°, 67.21°, 68.79°, 75.18° consistent to (101), (004), (200), (105), (211), (204), (116), (220), (215) phases, individually. After calcining the samples, it was discovered that only anatase phase formed. rutile .The peak 28.8° is indicates to the rutile phase , it was shown only at TiO_2 after 14 hs hydrothermal treatment. Same results were reported by other researchers that lone anatine phase was shaped after calcinations of TiO_2 NT's at 500°C. This shape is alike to that TiO_2 NT's prepared by anodic oxidation [14], sol gel method [15].

The shape of TiO2 NT panels produced by hydrothermal process at 110 oC with two different response time of 10 and 14 hours was studied (SEM). Figures 3 and 4 show reflective SEM imageries of a wide range of samples.

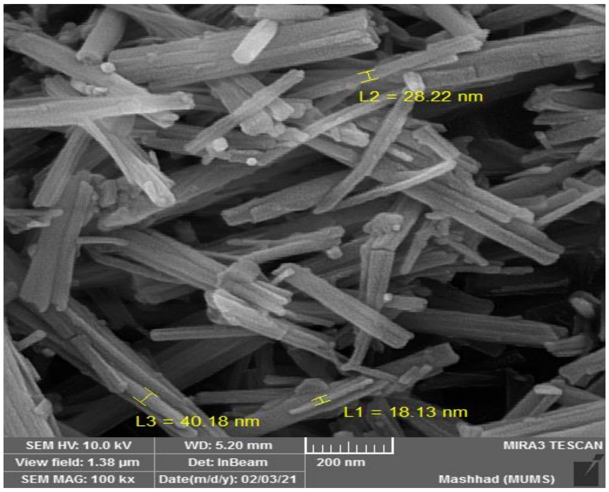

FIGURE 3
SEM IMAGE OF TiO_2 NANTUBES AT 10HRS

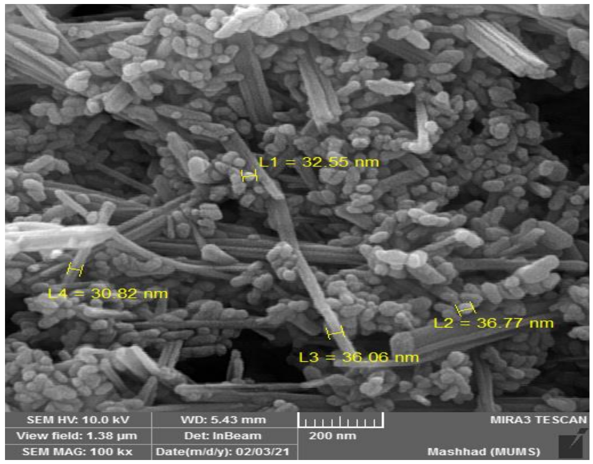

FIGURE 4
SEM IMAGE OF TiO_2 NANTUBES AT 14 HRS

The NT diameters of samples obtained after 14 hours were more homogeneous and larger than those obtained after 10 hours. To observe the formation of nanotubes, the reaction time must be balanced with other conditions such as the type and concentration of electrolyte and the anode potential. The morphology of NT has not yet formed after 10 hours of treatment. After 14 hours of hydrothermal treatment, the morphology of NT has been formed uniformly, as shown in Figure 4. The longer the treatment time, the more well-shaped TiO_2 NTs can be produced. It was discovered that the hydrothermal duration influenced the formation of TiO_2 NTs. Because of the response with NaOH, the Ti-O-Ti words were fragmented as during duration of the treatment [16]. The edges of the free octahedral figures will share after calcining the samples, it was discovered that only anatase phase formed. Limits with the Ti ions to form hydroxyl bonds, resulting in a choppy structure. As a result of saturating these dangling bonds from the ground, the crystalline sheets rolled up. As a result of the lower total energy, TiO_2 NTs have been founded [17, 18]. Has also reported similar findings.

CONCLUSION

The hydrothermal method is a good way to make TiO_2 nanostructures, particularly nanotubes. This method was successful in producing TiO2 NTs with high crystallinity and surface area. The shape and structure of the product are strongly influenced by the preparation conditions, particularly the time of hydrothermal treatment, which has an impact on the formation of TiO_2 NTs as well as the structure and crystallinity of TiO_2 NTs. The optimal hydrothermal treatment time for producing TiO_2 NTs was 14 hours.

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