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Synthesis of Highly Crystalline Tio2 Nanorod by Using Hydrothermal Method

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Abstract

Highly crystalline TiO2 nanorod was by simple hydrothermal method by using mesoporous anatage TiO2 power. The prepared sample we characterized with SEM, XRD, and FESEM. By the changing in process parameter we get the high crystalline nanorod we discussed. The results show that TiO2 nanorod fact that is highly crystalline, lowest diameter and highly aspect ratio.

Introduction

Titanium semiconductor most useful recherché because performance of TiO_2 depends strongly on its crystallite phase, dimension, and morphology, since they give a decisive influence on the chemical and physical properties of TiO_2 materials. One-dimensional (1D) nanostructure materials, like nanotube and nanofibers, are significance due to their highly physicochemical properties, such as other properties like electronic, magnetic, optical, catalytic, and mechanical properties, and potential applications in environmental purification, nano devices, gas sensors, and high effect solar cell etc[2]. several recent studies indicated that TiO_2 nanotube possessed better properties in photo catalysis and sensing [3].

The combination of the 1D nanostructures and mesoporous structures would endow titania with unique properties and multiple functions. However, much attention was mainly paid to mesoporous TiO2 particles or thin films, while 1D mesoporous TiO2 materials were less studied, mainly due to the difficulties in fabricating such structures with good control over their dimensions [4-9]

Recently, many attempts have been made to prepare TiO_2 1D titanate nanostructure materials with large specific surface area [6–10,21–23]. Crystalline TiO_2 nanotubes were usually synthesized using porous anodic alumina as templates [16,18,19,24,25]. However, the prepared nanotubes by the above method generally had large diameters (>50 nm) due to the confinement and aspect ratio. Using a simple hydrothermal treatment of crystalline TiO_2 particles with NaOH aqueous solutions, high crystalline nanorod with uniform diameter of around 10 nm. Considering their large specific surface area, high pore volume, and unique morphology, the obtained nanotubes will offer another possibility to design various TiO_2-related materials by post-treatment methods, such as hydrothermal post-treatment and well-controlled calcinations.

Hydrothermal synthesis, a low temperature technique for development 1D nanostrcture, which apply to prepare various materials due to high reactivity of reactants, easy control of solution, less air pollution, and low energy consumption under hydrothermal conditions [27]. In the present work, titanate nanorods were first prepared by a hydrothermal reaction using a 10M NaOH aqueous solution and TiO2 powder. Subsequently, the as-prepared nanotubes were hydrothermally. The effects of hydrothermal post-treatment time on the phase structure, crystallization, crystallite size, morphology. This is the report on the preparation of mesoporous anatase TiO_2 nanorod by the hydrothermal method.

Experimental

Preparation of Titanate Nanotubes

Titanate nanotubes were prepared using a chemical process similar to that described by Kasuga et al. [6,7] and Tian et al. [28]. TiO2 source used for the titanate nanotubes was commercial grade TiO_2 powder with crystalline structure of ca. 20% rutile and ca. 80% anatase and primary particle size of ca. 30 n TiO_2 white powder 420 mg mixed with prepared aqueous solution of 10M NaOH in 20 ml DI water, TiO_2 powder was placed into the solution

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and put on 1hr magnetic stirring. The prepared hydrothermal able solution was loaded into a Teflon vessel of 30 ml volume capacity. optimized the temperature of the sealed into stainless oven 200^{0}C than put in autoclave for 24 hr. After we removed autoclave from oven leave for 12 hrs to cool down at room temperature natural cooled at room temperature, filtered and washed with DI water for several times (7-8 times), the inter-mediate metastable product sodium metatitatante ($Na_2Ti_3O_7$). Sodium metatitatante ($Na_2Ti_3O_7$) was Centrifuged at 8000 rpm for 15minutes, the precipitate was separated by filtration and washed with DI water for 6-7 time after hydrothermal reaction and washing than precipitate was separated by filtration and washed with 0.2M HCl solution for 24 hr put it for soaking to get hydrogen titanate, and again wash with distilled water until the pH value of the rinsing solution reached ca. 6.5, approaching the pH value of the distilled water. The washed samples were dried in a vacuum oven at 80^{0}C for 8 h. The collected material was placed in a furnace at 400^{0}C - 500^{0}C for 4 h under air to remove hydrogen which present nanostructure. Then, the sample was allowed to cool naturally inside the furnace to room temperature for further Characterization

Preparation of highly crystalline TiO_2 nanorods

To prepare TiO_2, 0.42 g of TiO_2 powder which is perched by sigma Aldrich, that was dissolved in 20 mL NaOH (10M) solution under continuous magnetic stirring for 30 min in solution at room temperature and stirred continuously, therefore TiO_2 powder uniformly dispersed in solution at room temperature and stirred continuously for another 30 min to get homogeneous solution. The prepared hydrothermal able solution was loaded into a Teflon vessel of 30 mL volume. The oven temperature we already set at 200^{0}C. The solution was put into Teflon lined autoclave than in oven for 24 hrs after removed autoclave from oven leave for 12hrs to cool down at room temperature. the precipitate was separated by filtration and washed with DI water for 6-7 time than soaked with 0.2M HCl solution put for soaking different time12hr, 18hrs, 24hrs, 48hrs, 72hr also add shaker 35rpm it for soaking to get hydrogen titanate, and again wash with distilled water until the pH value of the rinsing solution reached ca. 6.5, approaching the pH value of the distilled water. The washed samples were dried in a vacuum oven at 80^{0}C for 8 h. The collected material was placed in a furnace at 550^{0}C for 4 h under air to remove hydrogen which present nanostructure. Then, the sample was allowed to cool naturally inside the furnace to room temperature for further use and characterization.

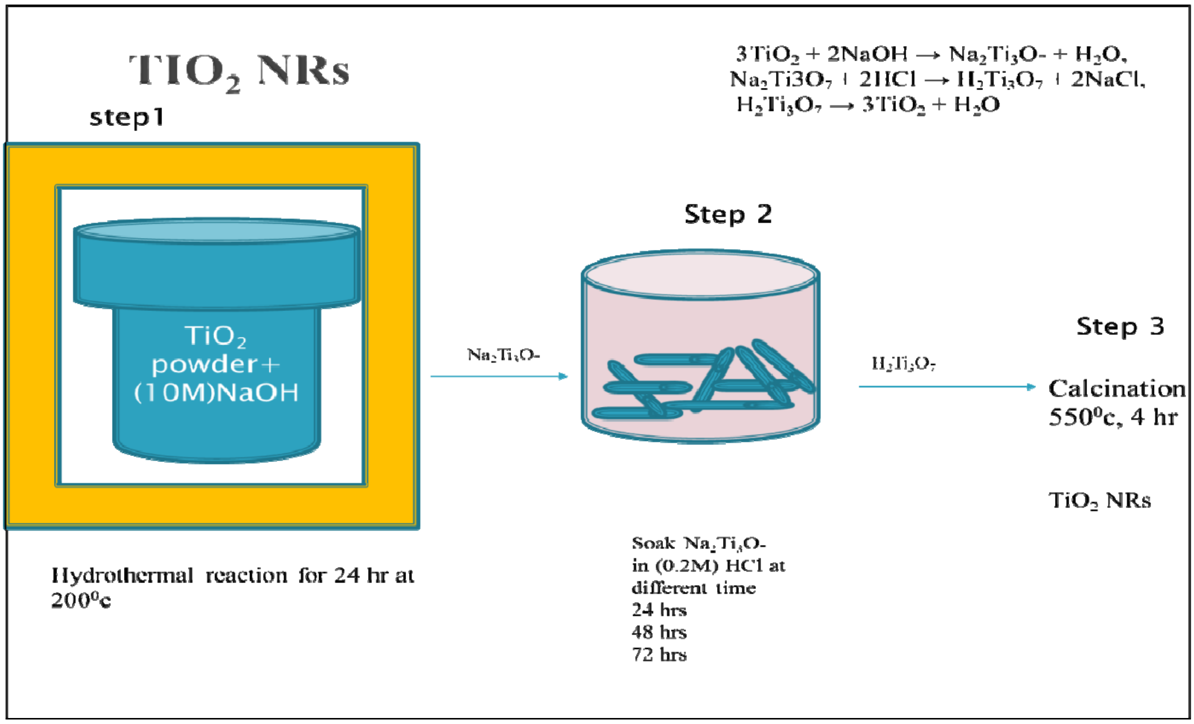


Fig :- Symmetric diagram of the synthesis if TiO_2nanorods

Characterization

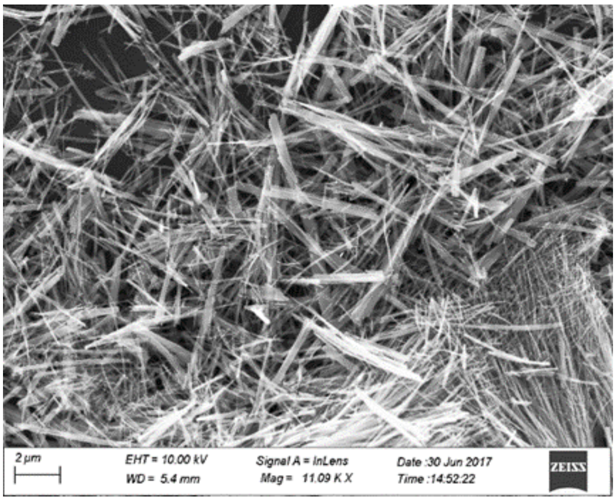
The X-ray diffraction (XRD) patterns were obtained on a XRD patters were recorded using a Panalytical Bruker D8FOCOUS X-ray diffractometer equipped with Cu-Kα radiation (λ = 1.540598 Å) radiation source at a scan rate of 0.05◦ 2θ s−1 and were used to determine the identity of crystalline phase and the crystallite size. Finally, the XRD patterns were compared with standard XRD peaks given by Xpert High score Database software, which was useful to find out the crystalline peaks of the original sample. The accelerating voltage and the applied current were 40 kV and 40 mA, respectively.

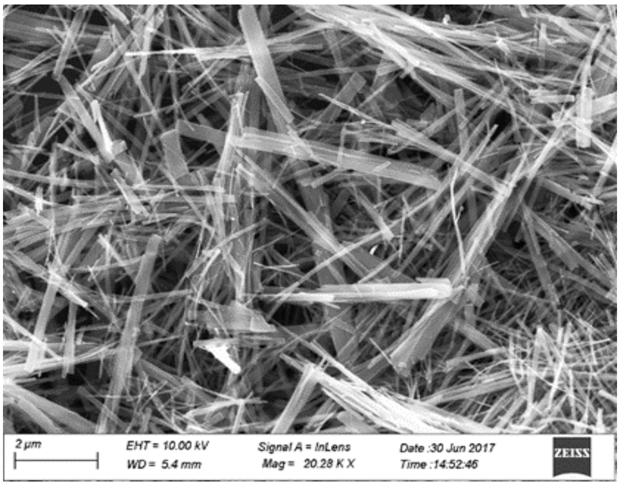
The surface morphologies of the samples were observed using a scanning electron microscope MIRA3 TESCAN was used at a pressure below 10-8 Torr with a probe current of 100 mA and beam voltage of 10 - 20 kV (SEM). The smallest beam cross section present between cathode and anode, the spot size is large to produce a sharp image by e beam.

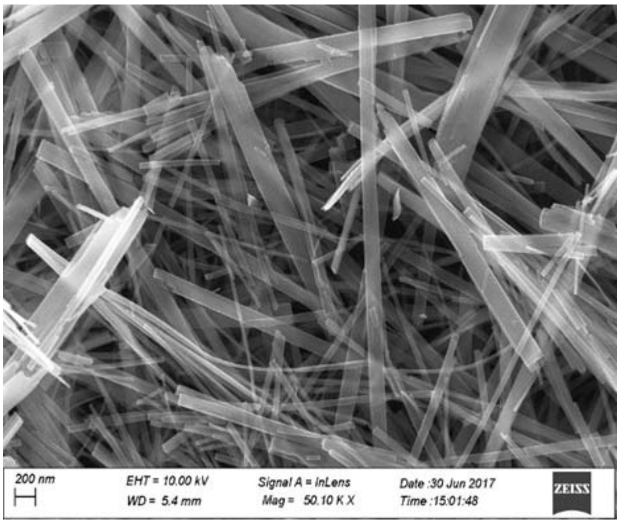
Results and discussion

Morphology and structure of titanate nanotubes

TiO_2 nanopowder commercial available for growth of 1D nanostructure by using hydrothermal method. These titania nanostructures are potentially dependent on the concentration of the base (NaOH) for 48 hrs and duration of acid (HCl) washing for 4 h only as well as the calcination temperature for 4 h employed. The effect of the alkali hydrolysis is concentration of NaOH of 10 M for 48h. The nanorods diameter is obtain their morphology and geometry with an average diameter of ∼ 150 nm. Typical diameter of the one-dimensional nanostructures from 20 nm to 100 nm. The lengths of the nanorods are greater than 3µm though longer lengths of up to 20µm are also observed. At even 48 hr hydrothermal reaction, a mixture of nanorods and highly agglomerated networks of one-dimensional produced. The nanorod produced is shorter (3–8) and sparsely distributed amongst the sample. The nanorods is an unstable structure due to its high surface-to-volume ratio or high energy system and also effected by process parameter. At specific hydrothermal reaction condition, there are possible ways in which the use construction of the nanorods may occur transforming either the nanosheets into nanorod structures by folding up. In our case, we observed that the nanorods amongst the nanosheet-like structures may have aggregated through physical attraction due to the presence of high surface energy. It is postulated that the dangling bonds and large surface area of the to one-dimensional nanorods can strongly interact with their adjacent nanorods via weak van der Waals forces leading to the formation of larger network of randomly aligned nanorods. The entire chemical processes that take place are as follows:







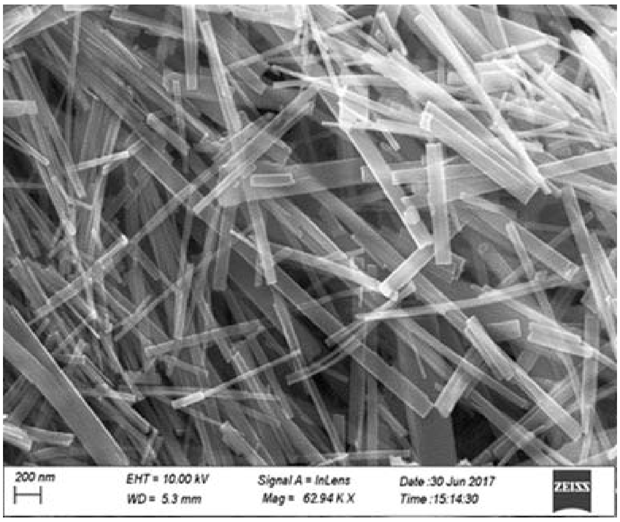
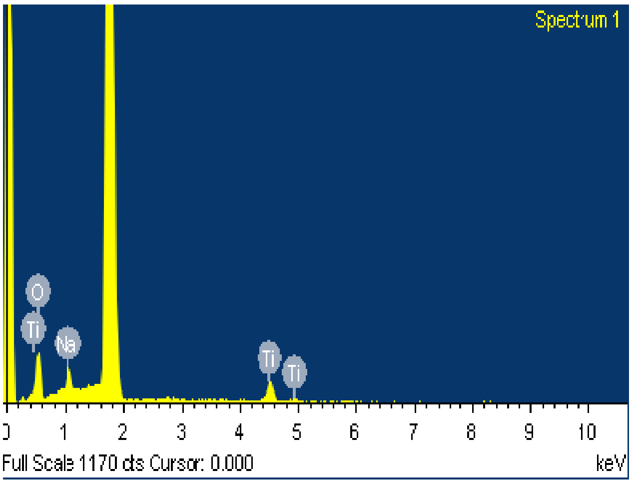
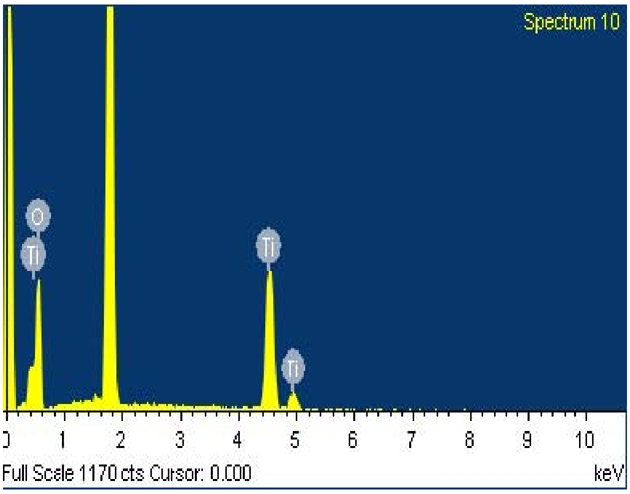
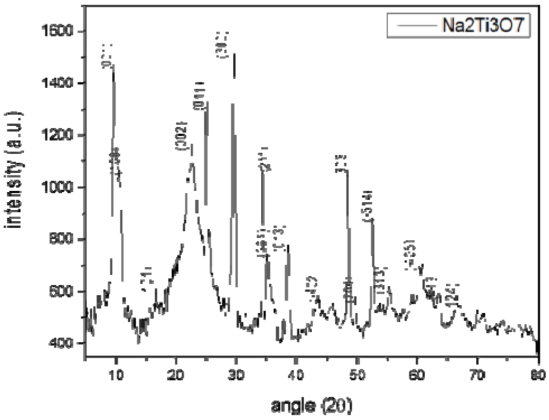


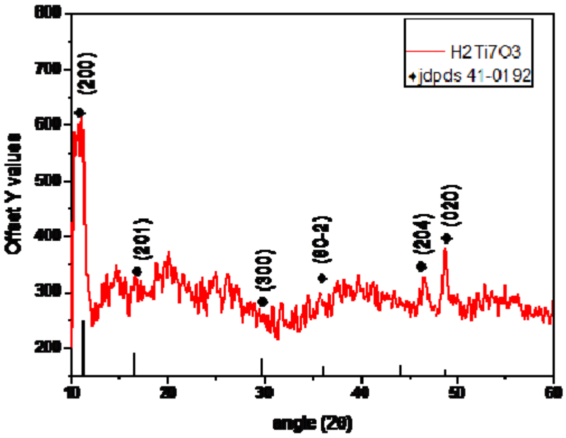
Figure :- (a), (b), (c), (d) SEM image of the uniform TiO_2nanorods

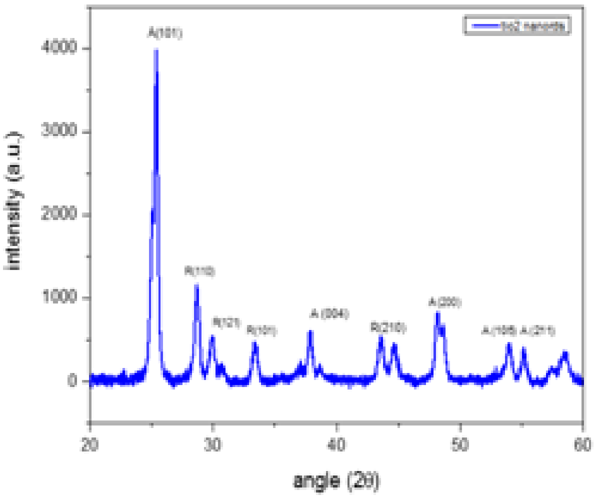


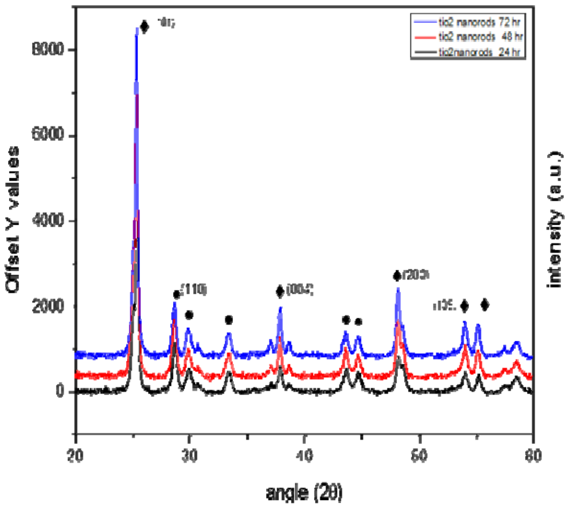


These titania nanostructures are potentially dependent on the concentration of the base (NaOH) for 24 hrs and duration of acid (HCl) washing for 4 h only as well as the calcination temperature for 4 h employed. The effect of the alkali hydrolysis is concentration of NaOH of 10 M for 48h. The nanorods diameter is obtain their morphology and geometry with an average diameter of ~ 100 nm. We produced nanorod uniformly but we did not get exact XRD pattern which we match with the JCPDS (01-072-0148) for sodium titanate, JCPDS (047-0561) for hydrogen titanate, JCPDS (21-1272) for TiO2.that show we optimised more to obtain pure titania nanorods.









Which resulted in the high flexibility of these high aspect ratio 1 D nanostructures? In the alkali-hydrothermal reaction with the acid (NaOH), the zigzag chain-like structural units of TiO_2 nanobelts remain relatively unchanged other than lattice rotation and rearrangement of the $[TiO_6]$ octahedra to form anatase lattice.

Thus, it is proposing that anatase titania phase formed on the basis of the parent titanate nanoparticles. It is interesting to note that the produced titania and titanate nanostructures retain essentially the same morphology, even after intervention of different chemical processing steps, such as acid washing and high temperature treatment. This observation confirms that the titanate nanostructure hosts modified are easily intercalated with alkali metal with slight lattice rotation and rearrangement of crystal structures without modifying its final morphology. Cross-sectional SEM image of figure 6(b) shows that the nanorods diameter average 20-40 nm and the length is 3-5 um. As per aspect ratio definition we can say 1 D nanostructure is nanorods.

Conclusions

Hydrolysis and ion exchange of titania particles via hydrothermal reaction at 200 ◦C has generated one-dimensional (1D) anatase titania nanorods. The chemical composition of sodium titanate nanorods or hydrogen titanate nanorods is highly dependent on the concentration and duration of the NaOH and HCl soaking, respectively. The product of the hydrolysis and ion exchange reaction, proceeding to HCl acid soaking, is sodium titanate nanorods. Therefore, HCl leaching and neutralization process yield hydrogen titanate nanorods which retain the nanorods-like morphology.

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