RESEARCH PAPER

Synthesis and Optimization of Nanostructured TiO_2 by Variation in Reaction Parameters for Future Application in Biomedical Applications

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Received: 08/09/2026 Revised: 12/10/2026 Accepted: 19/10/2026 Published: 22/10/2026

Abstract

Titanium dioxide (TiO_2) nanostructures were examined by X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The XRD patterns compare hydrothermal synthesis temperatures of 80, 100, 120 and 140 °C and reaction concentrations of 0.05M, 0.1M and 0.15M. Diffraction peak corresponds to anatase and rutile associated phases provide the basis for qualitative structural comparison. SEM images shows changes in surface texture, packing and aggregation across the reaction-temperature series, while TEM images reveal clustered and elongated fnanorod with selected dimensions annotated as 17.4, 28.7 and 41.8 nm. The combined characterization illustrates how reaction conditions relate to the observed structural and morphological features of TiO_2. The synthesized TiO_2 nanopowder can be used for further anitimicrobial activity and bilogical activity study in future scope.

Keywords

How to cite this article: Ashwini Dattatray Jadhav12 Ashok Pralhad Acharya3 & Ramchandra Tukaram Sapkal14*. Synthesis and Optimization of Nanostructured TiO_2 by Variation in Reaction Parameters for Future Application in Biomedical Applications. Int J Drug Deliv Technol. 2026;16(87s):1227-1231. DOI: 10.25258/ijddt.16.87s.128.

1. Introduction

Titanium dioxide (TiO_2) is widely investigated as a functional metal oxide because of its chemical stability, optical response and potential in environmental, energy and sensing technologies . Its performance is closely related to crystal phase, crystallite dimensions, surface morphology and defect structure. Anatase, rutile and brookite are the principal TiO_2 polymorphs, with distinct crystal structures and properties; their relative formation can be influenced by precursor chemistry, solution acidity and thermal history [1, 6, 13, 25].

Hydrothermal synthesis enables crystal nucleation and growth in a heated solution contained in a sealed vessel. The method is useful for preparing TiO_2 powders and one-dimensional nanostructures because reaction temperature, duration, acidity and precursor chemistry can be adjusted to influence phase development and morphology [2, 5, 17, 24]. Published studies using XRD together with SEM and TEM show that temperature and reaction time can affect crystal growth, agglomeration and the development of nanostructured features [3, 7, 18].

Reaction temperature influences hydrolysis, condensation, nucleation and the mobility of species involved in crystal growth. A change in temperature may therefore affect diffraction-peak visibility and the shape, packing or aggregation of the resulting particles. Reaction duration and concentration variation provides additional time for nucleation, growth and structural reorganization, although the response depends on the specific chemical environment and precursor system [9-10, 18-19]. Recent study also emphasize that the relationship between synthesis conditions, phase composition and morphology should be interpreted through complementary structural and microscopy evidence [17, 22-23, 25]. In this study, the supplied temperature-dependent and time-dependent XRD patterns are considered alongside the SEM and TEM images.

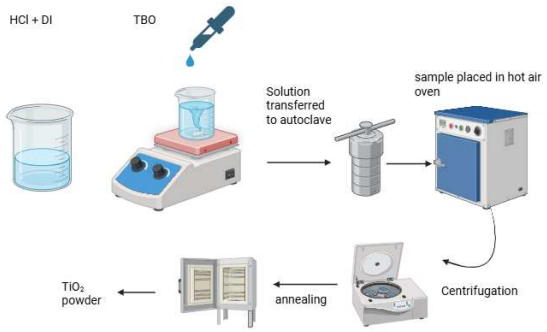
The objective is to compare the structural signatures across both XRD series and relate them to the surface and local-scale features observed by SEM and TEM. Considering these techniques together provides a coherent qualitative account of temperature- and time-associated changes while distinguishing aggregate-scale morphology from XRD study of crystalline structure.Further more the synthesized TiO_2 can be used for biological applications like antimicrobial study.

2. Materials and Methods

All chemicals were of analytical reagent grade and used without further purification. TBO Titanium(IV) butoxide(Sigma Aldrich), HCl(35-37% V/V ,Loba chemicals) and DI water

In this synthesis of TiO_2 a 60 ml mixture of deionised water and hydrochloric acid (1:5) followed by magnetic stirring in a beaker for 15 min. After that 2 ml titanium butoxide (TBO) was added dropwise and mixture was stirred for another 15 min. Prepared solution was transferred into a Teflon-lined stainless steel autoclave of 100 mL volume. The hydrothermal synthesis was carried out at 80°C , 100°C,120°C and 140°C for 4 hr in a hot air oven. After the reaction, the autoclave was allowed to cool naturally. The synthesized powder was centrifuged at 4000 rpm for 10 min and thoroughly rinse with deionized water and acetone followed by drying in hot air oven at at 60°C. Prepared TiO_2 nanoparticles were annealed in furnace at 400°C for 1 hr as shown in scheme 1. Simillar procedure was carried out by keeping 100°C

fixed temparature and varying concentration from 0.05 M, 0.1M and 0.15M at 4hr This precursor and acid-assisted route is consistent with published hydrothermal approaches for TiO_2 nanostructures [11, 17, 24]. The temperature-dependent XRD series compares 80, 100, 120 and 140 °C, while the concentration-dependent series compares 0.05 M, 0.1M and 0.15M. A SEM images described the four reaction durations, and TEM images provide local-scale morphology and selected feature dimensions.The experimental workflow and characterization are summarized from the synthesis schematic, XRD patterns and microscopy images.



Scheme 1. Schematic of the hydrothermal synthesis workflow

2.1. Materials Characterization:

Crystal structures were studied using X-Ray Diffraction diffractometer of Rigaku miniflex 600(Japan) with Cu Ka radiation (k = 1.54 A˚). The angular range of diffraction angle 2θ was 10° - 80° with scanning step was 0.01°. The morphological study of sample was performed by using Scanning electron microscopy (JEOL/MP JSM-IT200,20 KV). Transmission electron microscopy was used for analysis of microstructure of sample (JEOL JEM 2100 plus, Japan)

3. Chemical Reactions

For a titanium(IV) butoxide precursor, hydrolysis replaces alkoxy groups with hydroxyl groups, followed by condensation and dehydration to form the Ti–O–Ti network of titanium dioxide. Hydrochloric acid provides the acidic reaction environment and can influence the hydrolysis and growth pathway. The following equations represent the idealized hydrolysis and net oxide-formation steps for a titanium alkoxide precursor [11, 17, 24].

Hydrolysis: $Ti(OC_4H_9)_4 + 4H_2O \rightarrow Ti(OH)_4 + 4C_4H_9OH$

Condensation/dehydration: $Ti(OH)_4 \rightarrow TiO_2 + 2H_2O$

Overall reaction: $Ti(OC_4H_9)_4 + 2H_2O \rightarrow TiO_2 + 4C_4H_9OH$

4. Results and Discussion

4.1 XRD: Effect of Reaction Temperature

The temperature-dependent XRD plot compares patterns recorded for 80, 100, 120 and 140 °C. The supplied figure assigns selected reflections to anatase and rutile associated phases of diffraction peak at an angle 25.53°, 36.32° including hkl planes of (101), (103), and diffraction peak at 27.61° 41.52°,54.65 at hkl planes of (110), (111) and (211) respectively (JCPDS card No. 21-1272 and 21-1276)[26-27]. Several features appear more clearly resolved in some higher-temperature traces than in the 80 °C trace. This difference suggests that the recorded diffraction response changes with synthesis temperature and may reflect differences in the development of crystalline domains [4-5, 9-10, 12, 14]. Recent hydrothermal studies similarly report that synthesis temperature can affect phase development and crystal dimensions [18-19, 23].

The comparison considers peak positions, relative prominence, peak shape and the appearance of labelled reflections together. Features in the lower-angle region help discuss the phases assigned in the source figure, while higher-angle reflections provide additional structural information. Peak prominence can reflect structural development and is also influenced by sample loading with temperature. For this reason, the present discussion emphasizes consistent qualitative features across the plotted patterns [25].

The labelled (101) feature is an important point of comparison because it is commonly used when discussing TiO_2 anatse phase structure. Phase interpretation is strongest when this reflection is considered together with the complete diffraction pattern, phase at 120 °C was transformed into complete mix phase with elemination of (101) peak [1, 18, 25]. Across the temperature series, the distribution of features over the plotted 2θ range provides more information than any one peak: low-angle reflections and higher-angle features together form the pattern used for structural comparison.Together, these observations provide a basis for comparing temperature-dependent structural features with the surface morphology visible in SEM,

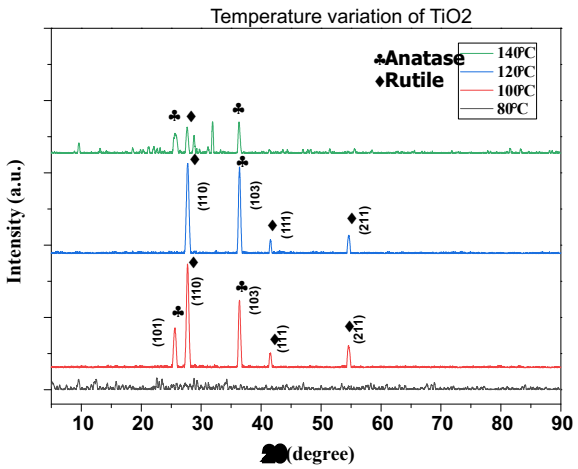


Figure 1. Temperature-dependent XRD pattern of TiO_2

4.2 XRD: Effect of Reaction concentration

The concentration dependent XRD plot compares samples prepared for 0.05M, 0.1M and 0.15M TBO. The source figure assigns selected features to anatase- and rutile-associated phases and marks reflections including (101), (110), (103), (111), (211) at corresponding 2θ

shwon in figure 2 . The 0.05M and 0.1M peak showed mixed phases of TiO_2 and a peak at 0.15M converts mix phase into single anatase phase when increased concentration. These observations indicate that reaction concentration corresponds to changes in the recorded diffraction response and may influence the development of detectable crystalline order [2- 4, 7-8].The XRD plots allow structural differences to be discussed alongside visible aggregation. This combined interpretation distinguishes the dimensions of surface aggregates from the coherent crystalline domains reflected in diffraction.

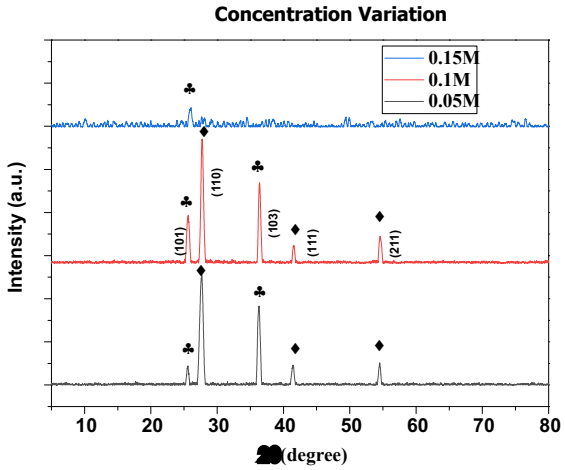


Figure 2. Reaction-concentration-dependent XRD patterns

4.3 SEM: Morphology

The SEM montage presents samples labelled 80, 100, 120 and 140 °C from a-d respectively. At 80 °C, the surface appears heterogeneous, with irregular clusters and a broad range of visible feature sizes. At 100°C, the image shows more spherical and closely packed aggregates. The 120°C image appears comparatively granular and densely covered, while the 140°C image contains larger, rough, cauliflower-like agglomerates assembled from finer-looking subunits. These differences indicate variation in surface texture and aggregation across the reaction-temperature series. Related studies have reported morphology changes with hydrothermal temparature, precursor concentration and post-treatment conditions [7, 17, 20, 21].

The observed differences are consistent with concurrent nucleation, growth and aggregation during hydrothermal treatment. Similar sensitivity of TiO_2 morphology to hydrothermal conditions has been reported in studies of controlled nanostructure formation and morphology tuning [2, 5, 11, 17 -18]. The compact appearance in some fields and the larger agglomerates in others indicate variation in surface architecture among the samples. SEM is especially useful for comparing surface coverage, aggregate shape and packing [7, 20-21].

The coarser agglomerated features visible in the 140°C image may reflect assembly of smaller subunits. Comparing these features with TEM and XRD helps distinguish surface aggregation from the size of individual crystallites [17-18, 24].Overall, the SEM series shows visible surface reorganization across the reaction durations. Image analysis of multiple representative fields can extend this qualitative comparison to particle-size and aggregate-size distributions.Panels (a)–(d) correspond to 80, 100, 120 and 140 °C respectively. The series illustrates changes in aggregation and surface texture across the reaction durations; these features are interpreted together with the XRD and TEM observations [3, 7, 17-18].

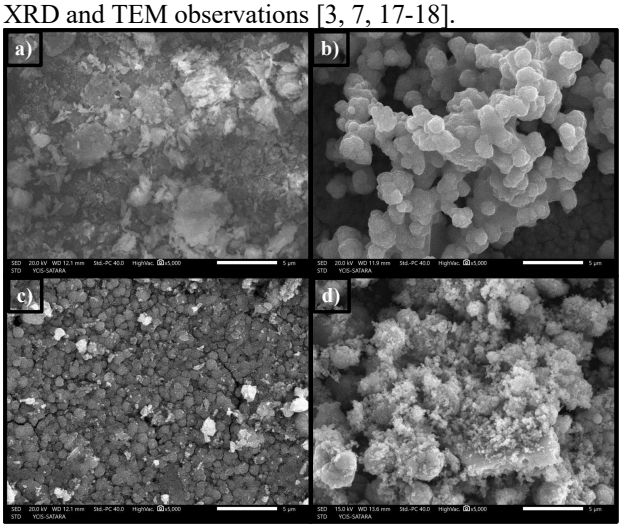


Figure 3. SEM images at 80, 100, 120 and 140 °C

4.5 TEM: Local Structure and Size Annotations

The TEM panel contains two micrographs with distinct local appearances. Figure 5a showsTEM image of TiO_2 with average size 29.3 nm. These values represent individual dimensions marked in the source image. Figure 5b shows elongated, nanorod extending from a darker aggregate. Together, the views display both compact clusters and elongated nanoscale features.The TEM observations complement SEM by revealing local features at finer scale. The annotated dimensions fall in the nanoscale range and illustrate variation among the selected features. Published studies similarly combine TEM and diffraction to examine hydrothermally grown TiO_2 nanostructures [5, 9, 11, 18, 24].

When considered alongside XRD and SEM, TEM provides complementary local-scale evidence; phase assignments and growth-direction interpretations are strengthened by integrating morphology with diffraction information.

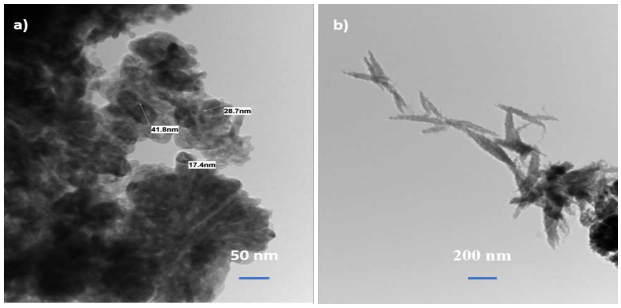


Figure 5. TEM micrographs and the size annotations shown in the source images

5. Conclusions

The supplied figures provide a comparative overview of TiO_2 prepared at different hydrothermal temperatures and reaction concentration at 4h. The temperature-dependent XRD patterns compare samples prepared at 80, 100, 120 and 140 °C, while the concentration-dependent patterns compare 0.05M, 0.1M and 0.15M TBO treatments. Both series contain reflections assigned in the source figures to anatase- and rutile-associated phases, with visible differences in the prominence in phases. The SEM images show changes in surface texture, packing and aggregation across the reaction-temperature series and TEM reveals clustered and elongated nanorod structures with selected dimensions of 17.4, 28.7 and 41.8 nm. The combined observations show that the selected processing conditions are associated with differences in structural and morphology, consistent with published studies of hydrothermal TiO_2 [17–19, 22–25]. XRD, SEM and TEM contribute complementary information and together provide a useful basis for further quantitative analysis of phase composition, crystallite dimensions and particle-size distributions.Synthesized nanoparticles can be used in bilogical applications like antimicrobial applications and antifungal properties, particularly for developing antimicrobial coatings, wound dressings, and infection-resistant medical surfaces. Further studies can explore the use of functionalized TiO_2 nanostructures in controlled drug delivery and bioimaging, with particular attention to their cellular uptake, toxicity, and biological safety.

Acknowledgements

This work received financial support from the Department of Physics and Center for Invention, Innovation and Incubation (CIII) of Mudhoji College, Phaltan-415523 , Satara, Maharshtra, India on Materials synthesis and characterization. Besides, this work was supported by the Department of Physics, Tuljaram Chaturchand College of Arts, Science, and Commerce, Baramati 413102, MH (India)

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