

Effects of Boric Acid (H_3BO_3) Addition on the Physicochemical Properties Properties of TiO_2 Nanocrystals

Iin Lidia Putama Mursal¹

¹ Prodi Farmasi Fakultas Teknologi dan Ilmu Komputer Universitas Buana Perjuangan Karawang, Indonesia

*corresponding email: iin.lidia@ubpkarawang.ac.id

DOI: -

Received:15-02-2026

Accepted: 03-04-2026

Published: 20-06-2026

Abstract:

Nanocrystalline TiO_2 thin films were successfully prepared by electrodeposition from a 1.5 M $TiCl_4$ solution. The films were characterised using scanning electron microscopy (SEM) and X-ray diffraction (XRD) to examine their morphology and crystalline structure. The characterisation results confirmed the formation of TiO_2 nanocrystals. The addition of boric acid (H_3BO_3) during electrodeposition improved the morphological uniformity of the resulting films, producing more consistent grain shapes and sizes, together with a more even grain distribution across the film surface. These findings indicate that boric acid addition influences the morphological characteristics of electrodeposited TiO_2 nanocrystalline thin

Keywords: electrodeposition; boric acid (H_3BO_3); TiO_2 nanocrystals; thin films; surface morphology

INTRODUCTION

The development of nanocrystalline materials has attracted considerable research interest owing to their potential applications in energy conversion, environmental treatment and functional coatings. Among these materials, titanium dioxide (TiO_2) is particularly relevant to the development of semiconductor thin films. Its applications in photocatalytic water and air purification, hydrogen production and solar energy conversion have encouraged continued investigation into suitable fabrication methods. In this context, the preparation of TiO_2 with controlled structural and morphological characteristics remains an important research objective. Wu et al. (2013)

The fabrication of TiO_2 as a thin film offers practical advantages for applications requiring the material to be immobilised on a supporting surface. In photocatalytic systems, for example, immobilisation facilitates material recovery and reuse, although the accessible surface area may be lower than that of dispersed powders. The suitability of a thin film therefore depends on the characteristics of the deposited layer, including its uniformity, surface morphology and stability. Covei et al. (2023) demonstrated that preparation conditions influenced the structure, morphology and stability of TiO_2 coatings, highlighting the importance of controlling film formation. These findings provide a basis for examining how changes in synthesis conditions affect the characteristics of the resulting material. Covei et al. (2023)

Electrodeposition is one of the methods available for preparing TiO₂ thin films. It allows film formation to be investigated under controlled electrochemical conditions, with deposition potential and temperature serving as relevant experimental variables. Previous work using a TiCl₃-based electrolyte showed that changes in these parameters affected the crystalline characteristics and surface morphology of TiO₂ films following annealing. Such observations indicate that the deposition environment requires careful consideration when developing films with reproducible characteristics. They also provide a rationale for investigating electrolyte formulation alongside the electrical and thermal conditions of preparation. Wu et al. (2013)

The present study focuses on the addition of boric acid (H₃BO₃) to a TiCl₄-based electrolyte used for the electrodeposition of TiO₂ thin films. The central question is whether this addition produces identifiable differences in crystalline structure and surface morphology under the selected preparation conditions. In particular, the investigation considers whether the films exhibit changes in grain appearance, size uniformity and spatial distribution. Boric acid is therefore examined as an electrolyte additive whose influence is to be established experimentally. Any interpretation of its role in controlling solution chemistry or film growth requires supporting evidence from the deposition conditions and characterisation results.

A combined structural and morphological assessment is necessary to address this question. X-ray diffraction (XRD) is employed to identify the crystalline phases present and examine the diffraction characteristics of the prepared films. Scanning electron microscopy (SEM) is used to observe surface morphology, with attention to the shape, apparent size and distribution of surface features. The two techniques provide complementary information: XRD supports the assessment of crystalline structure, whereas SEM reveals the organisation of the deposited material at the surface. Their combined use enables a more complete evaluation of the changes associated with boric acid addition.

Accordingly, this study aims to investigate the influence of H₃BO₃ addition on the structural and morphological characteristics of nanocrystalline TiO₂ thin films prepared by electrodeposition from a 1.5 M TiCl₄ solution. Through XRD and SEM characterisation, the study seeks to assess the relationship between electrolyte modification and the resulting film characteristics, particularly morphological uniformity. The findings are intended to inform the selection of preparation conditions for TiO₂ thin films with more consistent structural and surface characteristics.

MATERIALS AND METHODS

Materials and Equipment

The materials used in this study were titanium tetrachloride (TiCl₄; Merck, 99.99% purity), boric acid (H₃BO₃), distilled water, 96% alcohol and indium tin oxide (ITO)-coated conductive glass. The ITO substrates measured 2.5 × 0.5 cm and served as the supporting material for thin-film deposition. A platinum plate was used as the anode. The laboratory equipment comprised beakers, measuring cylinders, spatulas, tweezers, disposable syringes, crucibles, an OHAUS GALAXY™ 160 digital balance, a magnetic stirrer, a hot plate, an ultrasonic

cleaner, a hair dryer, an electrodeposition apparatus and an electric furnace. Masks and gloves were used during sample preparation and handling.

Preparation of ITO Substrates

Prior to electrodeposition, the ITO substrates were cleaned in an ultrasonic bath. Approximately 200 mL of 96% alcohol was transferred into a beaker, and the substrates were immersed in the solution. The ultrasonic cleaner was filled with distilled water to the appropriate operating level, after which the beaker containing the alcohol and substrates was placed in the bath. Ultrasonic cleaning was performed for 15 min to remove surface contaminants before deposition. The substrates were subsequently removed using tweezers and dried with a hair dryer before being mounted in the electrodeposition apparatus.

Preparation of the Electrolyte

An aqueous TiCl_4 electrolyte with a concentration of 1.5 M was prepared using distilled water. Boric acid was incorporated into the electrolyte at a concentration of 0.241 M. The solution was mixed using a magnetic stirrer until homogeneous and then transferred to the electrodeposition vessel.

Electrodeposition of Thin Films

Electrodeposition was carried out using a two-electrode configuration. The ITO substrate was connected to the negative terminal of the power supply and used as the cathode, whilst the platinum plate was connected to the positive terminal and used as the anode. Both electrodes were immersed in the electrolyte within the deposition vessel.

A constant voltage of 5 V was applied across the electrodes for 1 h. Deposition was conducted at room temperature, approximately 27 °C. Following deposition, the coated ITO substrate was disconnected from the cathode holder and removed from the electrolyte for subsequent treatment.

Post-deposition Heat Treatment

The deposited films were rinsed with distilled water to remove residual electrolyte and then dried. The coated substrates were subsequently heat-treated in an electric furnace at 500 °C for 5 h. This treatment constituted the final preparation stage before characterisation of the resulting films.

Film Characterisation

The heat-treated films were characterised using ultraviolet-visible (UV-Vis) spectroscopy and scanning electron microscopy (SEM). UV-Vis spectroscopy was used to examine the optical response of the films, whilst SEM was employed to investigate their surface morphology. The morphological assessment focused on the appearance, apparent size and spatial distribution of the surface features, with particular attention to film uniformity

RESULTS AND DISCUSSION

Sample Preparation Conditions

Two samples, designated A and B, were examined to assess the influence of boric acid (H_3BO_3) addition on the surface morphology of electrodeposited TiO_2 thin films. Sample A was prepared without boric acid, whereas sample B was prepared with boric acid added to the electrolyte. Both samples were

deposited using a 1.5 M TiCl_4 solution at an applied voltage of 5 V for 1 h, at approximately 27 °C. The preparation conditions are summarised in Table 1.

Table 1.

Electrolyte composition and electrodeposition conditions for samples A and B.

Sample	TiCl_4 concentration (M)	H_3BO_3 concentration (M)	Deposition temperature (°C)	Applied voltage (V)	Deposition time (h)
A	1.5	Not added	Approximately 27	5	1
B	1.5	0.2451	Approximately 27	5	1

This comparison provides a basis for examining the morphological differences associated with boric acid addition under otherwise comparable preparation conditions. The assessment focuses on the shape, uniformity and distribution of the surface features observed by SEM.

Surface Morphology

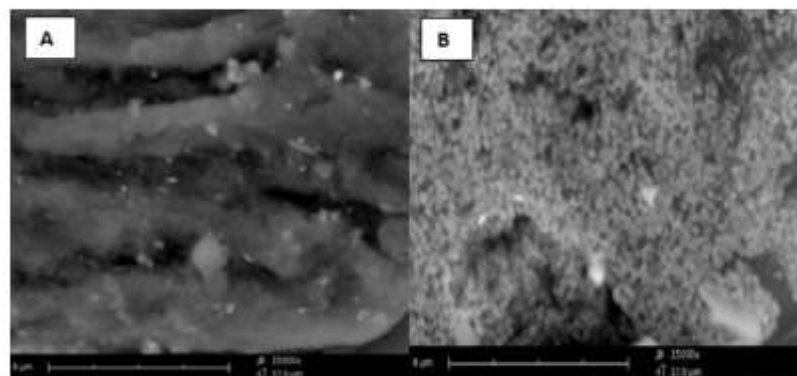
Figure 1 presents SEM micrographs of samples A and B at a magnification of 15,000×. According to the qualitative observations, the samples exhibited distinct surface morphologies despite being prepared at the same nominal deposition temperature and applied voltage.

Sample A, prepared without boric acid, exhibited a heterogeneous surface containing relatively large, irregular features. These features appeared unevenly distributed and may represent agglomerates of smaller particles. Their presence suggests localised clustering within the deposited layer. However, the identification of these features as agglomerates remains tentative, as their internal structure cannot be established from surface appearance alone. In contrast, sample B, prepared with boric acid, exhibited more rounded surface features with a more uniform appearance. The observed features were more evenly distributed across the examined region, and the surface appeared comparatively smoother. Relative to sample A, the morphology described for sample B therefore indicates greater uniformity in the deposited layer.

The difference between the samples is consistent with an influence of boric acid addition on film formation under the conditions investigated. In particular, the more even distribution of rounded features in sample B suggests that the additive may favour a more uniform deposition process. Nevertheless, SEM observations alone do not establish the mechanism responsible for this difference. Explanations involving changes in electrolyte chemistry, nucleation or growth behaviour would require additional experimental evidence.

The apparent improvement in surface uniformity should also be distinguished from a measured reduction in particle size or surface roughness. Although the SEM observations permit a qualitative comparison, quantitative conclusions would require image analysis across representative regions and, for roughness, an appropriate surface measurement technique. Likewise, the features visible in the micrographs should not automatically be identified as individual crystallites, since a surface feature may contain more than one crystalline domain.

Overall, the reported SEM observations indicate that sample B exhibited a more homogeneous surface morphology than sample A. Boric acid addition was associated with more rounded features and a more even surface distribution, whereas the film prepared without the additive contained larger, irregular features suggestive of agglomeration. These findings support a qualitative association between H_3BO_3 addition and improved morphological uniformity under the preparation conditions examined.

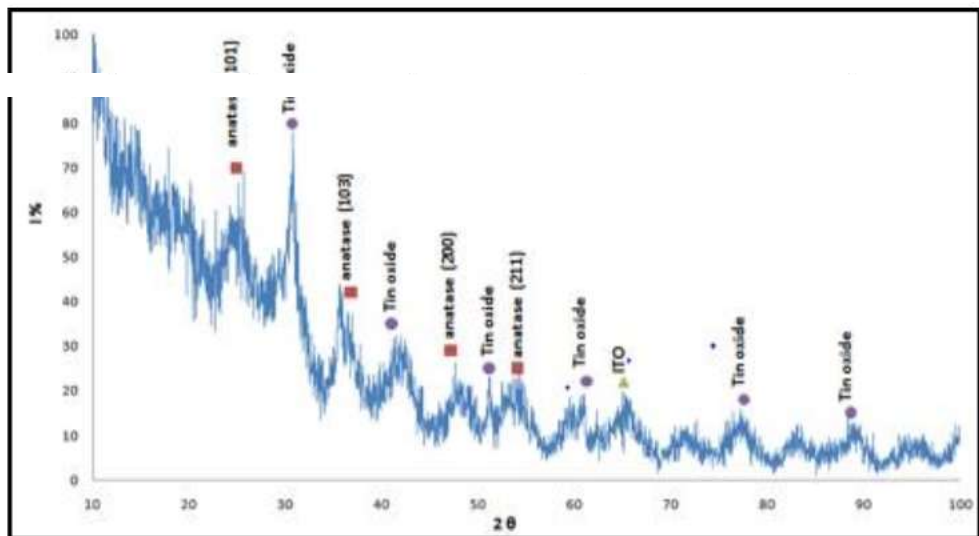


Gambar 1. Morfologi permukaan sampel A dan B

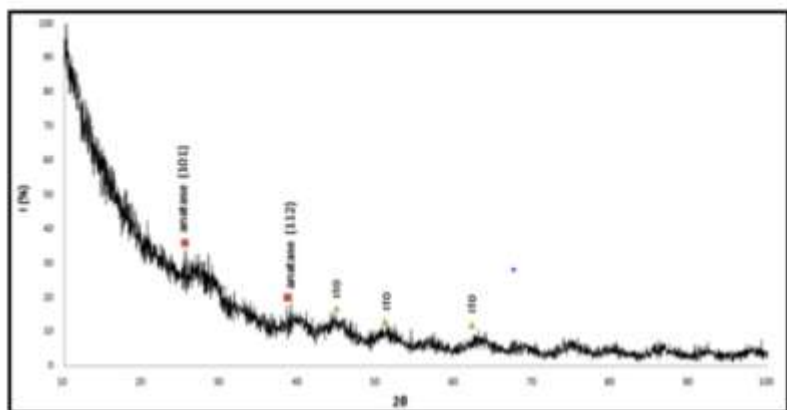
A comparison of the surface morphologies of the two samples suggests that the observed differences are associated with the addition of H_3BO_3 . During electrodeposition, boric acid addition may suppress hydrogen (H_2) gas evolution, thereby promoting the incorporation of titanium species at the cathode and facilitating more effective deposition.

XRD Analysis

X-ray diffraction (XRD) analysis was performed using a PANalytical X'PERT PRO powder X-ray diffractometer to investigate the crystalline structure of the thin films. Diffraction patterns were recorded over a 2θ range of 10° – 100° using Cu K α radiation with a wavelength of $1.5405 \text{ \AA}$. The XRD results indicated the formation of TiO_2 with a tetragonal crystal structure in both samples A and B. The diffraction patterns of the two samples were compared with standard reference data from the Joint Committee on Powder Diffraction Standards (JCPDS) to identify reflections corresponding to the anatase phase. The characterisation results are presented in the following figure and table.



Gambar 3. Pola difraksi sampel B



Gambar 2. Pola difraksi sampel A

Tabel 1. Nilai 2θ fasa anatase pola difraksi XRD sampel A

uncak	2θ	intensitas	d (Å)	enyawa (hkl)
1	25,4831	33,5610	3,4940	anatase (101)
2	38,431	17,3136	2,3384	anatase (112)

The diffraction patterns of samples A and B were compared with reference patterns PDF 01-073-1764 and PDF 01-075-2551, respectively. The XRD results indicated the formation of TiO_2 with a tetragonal crystal structure in both samples, with reflections corresponding to the anatase phase. Additional reflections were attributed to the indium tin oxide (ITO) coating of the conductive glass substrate. Sample B, prepared with boric acid (H_3BO_3), exhibited more clearly defined anatase reflections and sharper diffraction peaks than sample A,

which was electrodeposited without boric acid. These differences suggest that the addition of H_3BO_3 to the TiCl_4 electrolyte influenced the crystalline characteristics of the resulting films.

However, sharper diffraction peaks do not, in themselves, demonstrate a reduction in particle size. Within the Scherrer framework, narrower peaks are associated with larger coherent crystalline domains when other contributions to peak broadening are accounted for. Quantitative crystallite-size estimation therefore requires an analysis of peak widths, with consideration of instrumental broadening and lattice strain. Consequently, neither an increase in specific surface area nor an improvement in photocatalytic performance can be established from peak sharpness alone; these properties require separate experimental evaluation

CONCLUSION

The addition of boric acid (H_3BO_3) to the TiCl_4 electrolyte favourably influenced the morphology and diffraction characteristics of electrodeposited TiO_2 thin films under the conditions investigated. SEM observations indicated that the film prepared with H_3BO_3 exhibited more rounded surface features and a more uniform distribution than the film prepared without the additive. XRD analysis identified tetragonal anatase TiO_2 in both samples, with more clearly defined diffraction peaks in the boric-acid-containing sample. These findings support the use of H_3BO_3 as an electrolyte additive for improving film uniformity, although an optimum concentration has yet to be established. Further research should systematically vary the H_3BO_3 concentration to determine its effects on film morphology and crystalline characteristics and identify suitable conditions for TiO_2 thin-film preparation

REFERENCES

- Grätzel, M. (2003). Dye-sensitized solar cells. *Journal of Photochemistry and Photobiology C: Photochemistry Reviews*, 4(2), 145–153. [https://doi.org/10.1016/S1389-5567\(03\)00026-1](https://doi.org/10.1016/S1389-5567(03)00026-1)
- Jitputti, J., Pavasupree, S., Suzuki, Y., & Yoshikawa, S. (2008). Synthesis of TiO_2 nanotubes and its photocatalytic activity for H_2 evolution. *Japanese Journal of Applied Physics*, 47, 751–756. <https://doi.org/10.1143/JJAP.47.751>
- Kolmakov, A., & Moskovits, M. (2004). Chemical sensing and catalysis by one-dimensional metal-oxide nanostructures. *Annual Review of Materials Research*, 34, 151–180. <https://doi.org/10.1146/annurev.matsci.34.040203.112141>
- Kong, F.-T., Dai, S.-Y., & Wang, K.-J. (2007). Review of recent progress in dye-sensitized solar cells. *Advances in OptoElectronics*, 2007, Article 75384. <https://doi.org/10.1155/2007/75384>
- Misra, S. (2015). Titania nanocrystals as active host for safe drug delivery. *Applied Science and Advanced Materials International*, 1(6), 169–170.

- Nadeak, S. M. R., & Susanti, D. (2012). Variasi temperatur dan waktu tahan kalsinasi terhadap unjuk kerja semikonduktor TiO_2 sebagai dye sensitized solar cell (DSSC) dengan dye dari ekstrak buah naga merah [Effects of calcination temperature and holding time on the performance of TiO_2 semiconductors in dye-sensitised solar cells using red dragon fruit extract]. *Jurnal Teknik ITS*, 1(1), F81–F86. Full text
- Pandey, A., & Samaddar, A. B. (2006). Dye sensitized photovoltaic devices: An answer to the daunting challenge of future energy crisis. In *Advances in energy research* (pp. 497–502).