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Optimization of Sol-Gel-Derived TiO2-Based Thin Films for Methylene Blue Photodegradation: A Systematic Review

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Abstract

TiO_2-based thin films are among the most widely developed photocatalytic materials for degrading organic pollutants because of their chemical stability, low toxicity, relatively low production cost, and compatibility with various deposition methods. However, their photocatalytic performance is strongly influenced by synthesis parameters, crystal phase, particle size, crystallite size, morphology, substrate type, and material modification strategies. This review aims to analyze the relationship between TiO_2 thin-film synthesis parameters and methylene blue degradation efficiency based on 14 articles published between 2015 and 2024. The findings indicate that the sol-gel method is the most dominant synthesis approach, applied through dip coating, spin coating, drop coating, spray coating, and combinations with electrodeposition and magnetron sputtering. A thermal treatment range of 450–600 °C was identified as the most effective condition because it promotes anatase crystallinity while minimizing excessive crystal growth and agglomeration. High photocatalytic performance was generally associated with anatase phase dominance, small to moderate crystallite size, open or uniformly distributed morphology, and material modifications that improve charge separation. Among the reviewed

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modified materials, the optimum condition was achieved in 40PVP/SnO_2/TiO_2 thin films calcined at 600 °C for 2 h, with a crystallite size of approximately 8.6 nm and methylene blue degradation of about 90.4%. For pure TiO_2 thin films, the optimum condition was obtained through heat-assisted sol-gel spin coating with 48 h sol aging and annealing at 600 °C, producing a crystallite size of approximately 10–15 nm and methylene blue degradation of about 92.90%. This review concludes that optimizing TiO_2 thin films should not focus solely on reducing crystallite size but should integrate control of phase composition, crystallinity, morphology, and charge separation efficiency. The study contributes to photocatalytic material development by synthesizing key synthesis-performance relationships that can guide future optimization of TiO_2-based thin films for organic pollutant degradation.

Keywords: TiO_2 Thin Film; Sol-Gel Method; Methylene Blue Degradation; Photocatalysis; Synthesis Optimization.

INTRODUCTION

Water pollution caused by organic dyes remains a significant issue in environmental technology, as dye-containing wastewater exhibits high chemical stability, resistance to natural degradation, the ability to hinder light penetration in aquatic environments, and the potential to cause toxic effects on aquatic organisms as well as human health. Among the various dyes commonly used as model pollutants, methylene blue is frequently selected due to its stable aromatic structure, ease of detection via UV–Vis spectrophotometry, and its representation of cationic dyes commonly found in textile and related industrial effluents (Sarker et al., 2022). Therefore, the development of photocatalytic materials capable of effectively degrading methylene blue has become an important approach in water remediation based on advanced oxidation technologies (El Sharkawy et al., 2025; Hou et al., 2024; Prawiranegara et al., 2025).

Titanium dioxide (TiO_2) is one of the most extensively studied photocatalytic semiconductors due to its high chemical stability, low toxicity, relatively low production cost, resistance to photocorrosion, and its ability to generate electron–hole pairs upon absorption of suitable photon energy (Shao & Kang, 2024). In photocatalytic processes, the electron–hole pairs generated in TiO_2 can interact with water and dissolved oxygen to produce reactive oxidative species, such as hydroxyl radicals and superoxide radicals, which subsequently play a role in decomposing organic molecules into simpler compounds

(Moma & Baloyi, 2019; Wang et al., 2013). However, the application of TiO_2 is still limited by its relatively wide band gap, predominant response in the UV region, and the rapid recombination of electron–hole pairs, which can reduce its photocatalytic efficiency (Agrios & Pichat, 2005; Moma & Baloyi, 2019).

In practical applications, TiO_2 in thin film form offers several advantages over powder-based photocatalysts, as it is easier to separate from solution, can be applied onto substrate surfaces, and is more suitable for self-cleaning systems, water disinfection, glass coatings, and reusable photocatalytic devices (Covei et al., 2023). The use of powder photocatalysts often faces challenges such as post-reaction separation, potential agglomeration in suspension, and limitations in continuous applications (Aziz & Sopyan, 2009). TiO_2-based thin films can mitigate these limitations; however, their performance strongly depends on film quality, thickness, substrate type, surface morphology, crystallite size, porosity, crystal phase, and the deposition method employed (Nematov et al., 2024). Therefore, the successful synthesis of TiO_2 thin films is not only determined by the formation of a coating on the substrate but also by the ability to control structure and surface properties to achieve photocatalytic activity (Covei et al., 2023; Nematov et al., 2024).

The sol-gel method is one of the most widely used synthesis routes for producing TiO_2 thin films due to its simplicity, relatively low cost, applicability at low to moderate temperatures, and its ability to provide control over precursor composition, hydrolysis–condensation rates, and film homogeneity (Rahmani, 2024). This method is also flexible and can be combined with various deposition techniques such as dip coating, spin coating, drop coating, and spray coating (Subhayu Choudhury, 2024). Furthermore, the sol-gel approach enables modification of TiO_2 through metal doping, non-metal doping, heterostructure formation, incorporation of carbon-based materials, porosity control, and core–shell design (Abbas & Bensaha, 2021; Rahmani, 2024; Subhayu Choudhury, 2024). These strategies are generally aimed at reducing the band gap, extending visible-light response, increasing active surface area, and suppressing electron–hole recombination (Abdul Razak et al., 2022).

Although numerous studies have reported successful synthesis of TiO_2 thin films for methylene blue degradation, the obtained results remain highly varied due to differences in calcination temperature, thermal treatment duration, solvent type, raw materials, substrates, deposition methods, particle size, crystallite size, crystal phase, and morphology (Phromma et al., 2020). Some studies indicate that increasing temperature can enhance crystallinity and photocatalytic activity; however, excessively high temperatures may lead to crystal growth, induce agglomeration, and promote excessive transformation from anatase to rutile (Kassahun et al., 2017). Other studies demonstrate that doping or heterostructure formation can improve photocatalytic activity, but non-optimal dopant concentrations may act as new recombination centers and reduce material performance (Moma & Baloyi, 2019). This variability makes it difficult to identify truly effective synthesis parameters when each study is considered independently.

Therefore, a systematic review is necessary to construct a more structured mapping of the relationships among synthesis parameters, morpho-structural characteristics, and methylene blue degradation efficiency in TiO_2-based thin film materials (Doula & Bensaha, 2025). Such a review is important because previous studies often emphasize a single aspect, such as annealing temperature, doping, porosity, or deposition method, whereas photocatalytic performance is actually governed by the interaction of multiple parameters simultaneously (Han et al., 2012). Through a systematic literature review approach, dispersed findings can be compared more systematically to identify general patterns, dominant parameters, trends in crystal phase, effective crystallite size ranges, and the most promising synthesis conditions. In this way, the review not only summarizes previous studies but also provides a rational basis for designing more efficient TiO_2 thin film synthesis (Gómez et al., 2024). Based on the above considerations, this study aims to systematically review articles on TiO_2 and TiO_2-based thin film materials synthesized primarily via the sol-gel approach for methylene blue degradation applications. Specifically, this study analyzes the relationships among calcination temperature, treatment duration, solvent, raw materials, substrate, morphology, particle size, crystallite size, crystal phase, and methylene blue degradation efficiency, and determines the most optimal synthesis parameters to achieve small crystallite size alongside high photocatalytic activity.

METHODS

1. Research design

This study employs a Systematic Literature Review (SLR) approach to analyze the relationship between sol-gel-based synthesis parameters, morpho-structural characteristics, and the photocatalytic activity of TiO_2-based thin films. The review process was conducted systematically following the PRISMA framework, which includes identification, screening, eligibility assessment, and final inclusion of the analyzed articles. This approach was adopted because studies on TiO_2 thin films are still dispersed across various parameters, including calcination temperature, treatment time, solvent, substrate, morphology, particle size, crystallite size, phase, and methylene blue degradation efficiency. The initial dataset and selection flow were structured based on compiled literature search results.

2. Data Sources and Literature Search Strategy

The literature was collected from Scopus, ScienceDirect, Springer, MDPI, and Taylor & Francis databases, covering publications from 2015 to 2024. The search focused on research articles discussing the synthesis of TiO_2 or TiO_2-based materials in thin film form using the sol-gel method or its combinations, and reporting morpho-structural characteristics and photocatalytic activity toward methylene blue. The search strategy was conducted using Boolean keyword combinations as presented in Table 1.

Table 1. Boolean Search Used in Literature Search

No	Search Focus	Boolean Search
1	Main search for sol-gel-based TiO_2 thin films for photocatalysis	("TiO2" OR "titanium dioxide") AND ("sol-gel" OR "sol gel method" OR "sol gel synthesis") AND ("thin film" OR "thin films") AND (photocatalytic OR "methylene blue") AND (crystallinity OR "crystal size" OR morphology)
2	Specific search on anatase phase and methylene blue degradation	("sol-gel" AND "anatase" AND "photocatalytic" AND "methylene blue" AND "synthesis" AND "thin films")
3	Additional search for morphology and crystallite size variations	("TiO2 thin film" AND "sol-gel" AND ("particle size" OR "crystallite size" OR morphology) AND "photocatalytic activity")
4	Additional search for TiO_2-based material modification	("doped TiO2" OR "TiO2 composite" OR "TiO2 heterostructure") AND ("thin film" OR coating) AND ("methylene blue" OR photocatalytic)

Following the initial search, articles were filtered based on document type, language, full-text accessibility, relevance of title and abstract, and completeness of synthesis and characterization data.

3. Inclusion and exclusion criteria

Inclusion and exclusion criteria were applied to ensure that the selected articles aligned with the review focus, namely the synthesis of TiO_2 or TiO_2-based materials in thin film form via sol-gel methods or their combinations. The detailed criteria are presented in **Table 2**.

Table 2. Article Inclusion and Exclusion Criteria

Aspect	Inclusion Criteria	Exclusion Criteria
Publication year	Articles 2015–2024	Articles outside 2015–2024
Document type	Research article	Review articles, book chapters, conference papers, proceedings
Language	English	Non-English
Article acces	Full-text or PDF available	Full-text or PDF not available
Materials	TiO_2 or TiO_2 based materials	Non-TiO_2 materials without relevance
Materials form	Thin film or coating	Powder or bulk materials without thin film context
Synthesis methode	Sol-gel or sol-gel combined with other deposition methode	Non-sol-gel methods
Synthesis data	Reports temperature, time, solvent, raw materials, substrate, or deposition method	Unclear synthesis parameters
Characterization	Reports morphology, particle size, crystallite size, phase, or band gap	No relevant material characterization
Photocatalytic activity	Reports methylene blue degradation or related photocatalytic activity	No relevant photocatalytic data

4. Articles selection process

The article selection process was conducted in four stages: identification, screening, eligibility, and inclusion. During the identification stage, articles were collected from databases using the search strategy described in Table 1. Duplicate records, non-research documents, non-English articles, and those without accessible PDFs were removed. In the screening stage, titles and abstracts were examined to assess relevance to TiO_2 thin films, sol-gel methods, and photocatalysis. Articles that passed this stage were then evaluated through full-text reading to ensure completeness of synthesis data, material characterization, and methylene blue degradation results. Articles that satisfied all criteria in Table 2 were included as the final dataset for analysis.

5. Data analysis and extraction

Data were systematically extracted from the selected articles, including article identity, research objectives, materials, synthesis methods, calcination temperature, treatment time, solvent, raw materials, substrate, morphology, particle size, crystallite size, phase, and methylene blue degradation efficiency. The extracted data were then analyzed using a descriptive-comparative approach to identify relationships between synthesis parameters, morpho-structural characteristics, and photocatalytic performance. The determination of optimum conditions was not based solely on the highest degradation value but also considered small crystallite size, anatase phase dominance, morphology that supports surface accessibility, and high methylene blue degradation efficiency.

RESULTS

1. Selection Results, Literature Distribution, and General Characteristics of the Study

The article selection process was conducted systematically using the PRISMA flow to ensure that the analyzed articles were truly relevant to the focus of the study, namely the synthesis of TiO_2-based thin films through the sol-gel approach and their relationship with morpho-structural characteristics and photocatalytic activity toward methylene blue. The identification, screening, eligibility assessment, and final inclusion stages of articles used in this review are presented in Figure 1. Figure 1 shows the stages of article selection used in the systematic literature review. In the identification stage, literature searching was carried out through five scientific databases, namely ScienceDirect, Scopus, Springer, MDPI, and Taylor & Francis. From the initial search process, 7,420 articles were obtained, consisting of 6,003 articles from ScienceDirect, 136 articles from Scopus, 1,193 articles from Springer, 3 articles from MDPI, and 85 articles from Taylor & Francis. Before the screening process, a total of 130 duplicate articles were removed using an automated tool, resulting in 7,290 articles entering the screening stage.

In the screening stage, articles were selected based on document type. A total of 4,294 articles were excluded because they did not meet the specified document type, namely 64 conference abstracts, 2,057 review articles, 63 encyclopedia entries, 1,644 book chapters, and 466 other documents. After screening based on document type, 2,996 articles remained for further examination. Of these, 2,612 articles were further excluded because 2,603 were closed access and 9 were not in English. Thus, 384 articles proceeded to the eligibility assessment stage. Furthermore, the 384 articles were screened through title and abstract reading. At this stage, 348 articles were excluded because they did not match the focus of the study, particularly due to not specifically addressing TiO_2 thin films, sol-gel methods, morpho-structural characteristics, or photocatalytic activity toward methylene blue.

After this process, 36 articles remained and were evaluated in more detail through full-text eligibility assessment. At the final eligibility stage, the 36 articles were assessed based on the completeness of experimental data and their relevance to the review variables. A total of 22 articles were excluded because they did not meet the final criteria, namely 3 articles lacked SEM/TEM analysis, 13 articles reported inconsistent data, 4 articles did not have complete PDFs, and 2 articles lacked XRD analysis. After completing all selection stages, 14 articles met the inclusion criteria and were used as the main sources in the analysis. The articles were then categorized based on their bibliometric characteristics, as presented in Table 3.

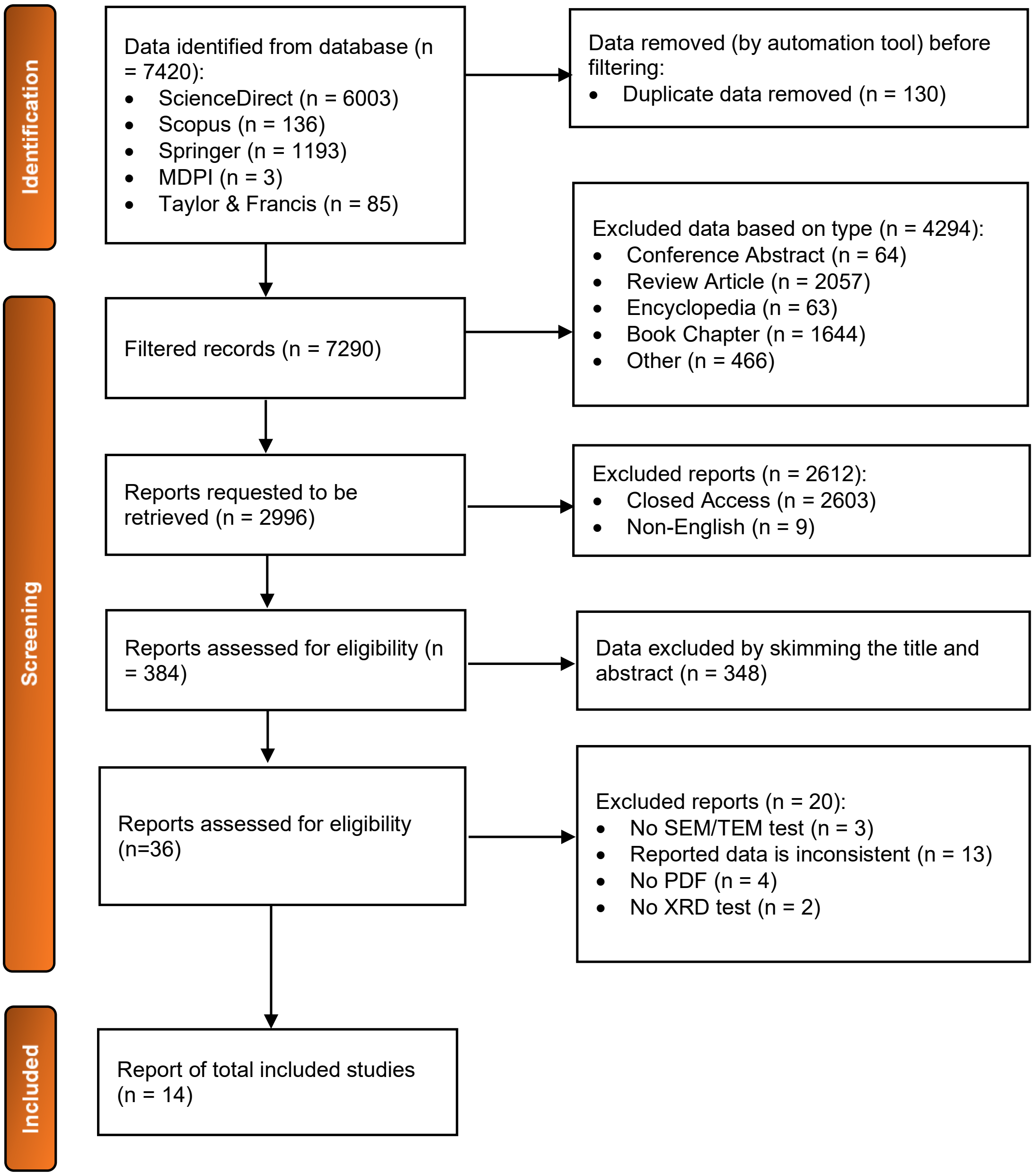

Figure 1. PRISMA Flowchart of the Article Selection Process in a Systematic Literature Review

Table 3. Description of Articles by Year, Country, Journal, Publisher, Keywords, Citations

No	Author (Year)	Country	Journal Name	Publisher	Keywords	Cit.
1	(Abbas & Bensaha, 2021)	Algeria	Optik	Elsevier	TiO_2, Hg- TiO_2, Thin films, Optical properties, Photocatalysis	15
2	(Yazid et al., 2019)	Malaysia	Journal of Materials Research and Technology	Elsevier	Sol–gel dipping, TiO_2 thin film, TTIP molarity, solvent-free, photocatalytic activity, green route	53
3	(Pinton et al., 2024)	Brazil	Materials Chemistry and Physics	Elsevier	Titanium dioxide (TiO_2) films, Sol-gel solution deposition, Spray coating, Environmental solutions	11
4	(Lukong et al., 2022)	South Africa	Heliyon	Elsevier	Annealing, Self-cleaning, Spin coating, Thin film, Titanium dioxide	47
5	(Sarker et al., 2022)	Bangladesh and USA	Cleaner Engineering and Technology	Elsevier	Doping, MWCNTs, Photocatalyst, Band gap, Sol-gel drop coating, Thin films	51
6	(Pérez-González et al., 2019)	Mexico	Journal of Alloys and Compounds	Elsevier	Oxide materials, Thin films, Catalysis, Sol-gel process, X-ray diffraction, Optical spectroscopy	68
7	(Lukong et al., 2021)	South Africa	Materials Research Express	IOP	Sol-aging, Thin films, Self-cleaning, Photovoltaic	20
8	(Assaker et al., 2015)	Tunisia	Applied Surface Science	Elsevier	Heterojunction, $ZnIn_2S_4/TiO_2$, Electrodeposition of thin film, Photoelectrochemical properties, Photocatalytic applications	70
9	(Kongsong et al., 2018)	Thailand	Songklanakarin Journal of Science and Technology	Prince of Songkla University	PVP doped SnO_2/ TiO_2, Glass fiber, Water disinfection, Escherichia coli, Staphylococcus aureus	3
10	(Sunil et al., 2024)	India	Journal of Sol-Gel Science and Technology	Springer	Sol-gel dip coating method, TiO_2 thin films, Photocatalysis, Antimicrobial activity, Hydrophilicity	12
11	(Nakahara et al., 2015)	Japan	Bulletin of the Chemical Society of Japan	Oxford University Press	SiO_2@ TiO_2, Core-shell nanoparticles, Anatase, Aqueous dispersibility, Photocatalysis	2
12	(Nursam et al., 2015)	Australia	Journal of Materials Chemistry A	RSC	Titania thin films, Porous films, Photocatalysis, Methylene blue, High-throughput screening	30
13	(Zhang et al., 2015)	China	Materials Science in Semiconductor Processing	Elsevier	Ag substrates, Cu-doped, TiO_2, Visible light	19
14	(Zhu et al., 2017)	China	Journal of Advanced Oxidation Technologies	De Gruyter	Nd-doped, TiO_2 thin film, Sol-gel method, Photocurrent, Photocatalytic activity	12

Based on Table 3, the reviewed articles consist of 14 studies with a publication range from 2015 to 2024. Temporally, the highest number of articles originated from 2015, with 4 articles or approximately 28.57% of the total, namely Assaker et al., (2015)., Nakahara et al., (2015).,Nursam et al., (2015)., and Zhang et al., (2015). The years 2019, 2021, 2022, and 2024 each contributed 2 articles, while 2017 and 2018 each contributed only 1 article. This pattern indicates that research on TiO_2 thin films, sol-gel methods, doping, and photocatalytic activity has developed relatively consistently over the past decade, with a renewed increase during the 2021–2024 period. In terms of country distribution, the studies are spread across Algeria, Malaysia, Brazil, South Africa, Bangladesh and USA, Mexico, Tunisia, Thailand, India, Japan, Australia, and China. The countries with the highest contributions are South Africa and China, each with 2 articles, while the others contributed 1 article each. This distribution indicates that research on TiO_2-based thin films and photocatalysis is not concentrated in a single region but has developed globally across Asia, Africa, Latin America, and Australia.

Based on the publisher, Elsevier is the most dominant publisher with 8 out of 14 articles or approximately 57.14%. These articles are published in journals such as Optik, Journal of Materials Research and Technology, Materials Chemistry and Physics, Heliyon, Cleaner Engineering and Technology, Journal of Alloys and Compounds, Applied Surface Science, and Materials Science in Semiconductor Processing. Other publishers each contributed 1 article, namely IOP, Prince of Songkla University, Springer, Oxford University Press, RSC, and De Gruyter. The dominance of Elsevier indicates that most of the reviewed literature originates from reputable international journals in the fields of materials, thin films, photocatalysis, and environmental technology. In terms of citations, the total citations from the 14 articles amount to 413, with an average of 29.5 citations per article. The most cited article is Assaker et al., (2015) with 70 citations, followed by Pérez-González et al., (2019) with 68 citations, Yazid et al., (2019) with 53 citations, Sarker et al., (2022) with 51 citations, and Lukong et al., (2022) with 47 citations. In contrast, the lowest citations are found in Nakahara et al., (2015) with 2 citations, followed by Kongsong et al., (2018) with 3 citations. The median citation value is 19.5, indicating that most articles have a moderate level of influence, while several articles serve as key references in topics such as heterostructures, Ag-loaded TiO_2-ZnO, green sol-gel, and thin-film photocatalysis.

Table 4. Focus of objectives, materials, synthesis methods, research results

No	Author (Year)	Research purposes	Material	Synthesis Method	Research result
1	(Abbas & Bensaha, 2021)	To investigate the effect of annealing time on the structural, optical, and photocatalytic properties of Hg^{2+}-doped TiO_2 thin films.	Hg^{2+}-doped TiO_2 thin films on silicon substrate.	Sol-gel and dip-coating	Longer annealing enhanced anatase-rutile-$HgTiO_3$ phase formation, reduced band gap from 3.47 to 2.95 eV, and improved methylene blue degradation up to 56.72% after 120 min.
2	(Yazid et al., 2019)	Menganalisis pengaruh ariasi molaritas titanium (IV) isopropoxide (TTIP) terhadap kristalinitas dan aktivitas fotokatalitik TiO_2 thin film menggunakan green sol–gel tanpa pelarut	TiO_2	sol–gel and dip-coating	A higher TTIP molarity, particularly 0.5 M, produces TiO_2 thin films with higher crystallinity, a mixed anatase–rutile phase, small crystallite size, porous/cracked morphology, and the highest methylene blue degradation activity of 95% under UV and 86% under visible light.
3	(Pinton et al., 2024)	To develop a low-cost automated spray-coating apparatus for depositing TiO_2 photocatalytic films.	TiO_2 thin film	Sol-gel solution deposition using automated spray coating	The apparatus successfully produced uniform anatase TiO2 thin films with good wettability and photocatalytic activity, degrading methylene blue and methyl orange under UV irradiation up to about 90%.
4	(Lukong et al., 2022)	To evaluate the effect of annealing temperature on the self-cleaning properties of TiO_2 thin films.	TiO_2 thin film	Sol-gel spin coating	Annealing improved crystallinity and reduced band gap from 3.39 to 3.20 eV; the film annealed at 600 °C showed the best self-cleaning performance.
5	(Sarker et al., 2022)	To enhance visible-light photocatalytic activity of TiO_2 thin films using P doping and MWCNTs.	P-doped TiO_2 /MWCNTs thin films on glass.	Sol-gel drop coating followed by annealing at 500 °C.	P-doped TiO2/MWCNTs showed anatase phase and higher MB degradation than TiO_2 and TiO_2 /MWCNTs.
6	(Pérez-González et al., 2019)	To evaluate Ag loading and low ZnO content on TiO_2-ZnO thin-film photocatalysis.	Ag-loaded TiO_2 -ZnO thin films.	One-step sol-gel deposition followed by annealing at 500 °C.	The best photocatalytic response was obtained for TiO_2 -ZnO with 2 mol% Ag.
7	(Lukong et al., 2021)	To investigate the effect of TiO_2 sol aging on self-cleaning thin-film performance.	TiO_2 thin film on glass.	Heat-assisted sol-gel spin coating with sol aging for 24, 48, and 72 h.	Aging improved crystallinity and MB degradation, with the best performance at 48 h

					aging.
8	(Assaker et al., 2015)	To fabricate $ZnIn_2S_4/TiO_2$ heterostructure for photocatalytic and photoelectrochemical applications.	$ZnIn_2S_4/TiO_2$ heterostructure on ITO glass.	TiO_2 sol-gel spin coating followed by $ZnIn_2S_4$ electrodeposition.	$ZnIn_2S_4/TiO_2$ showed stronger visible-light activity than TiO_2, reaching 91% MB degradation.
9	(Kongsong et al., 2018)	To assess photocatalytic antibacterial performance of PVP-doped SnO_2/TiO_2 films.	PVP-doped SnO_2/TiO_2 thin films on glass fibers.	Sol-gel dip coating followed by calcination at 600 °C.	$40PVP/SnO_2/TiO_2$ showed the best photocatalytic and antibacterial performance.
10	(Sunil et al., 2024)	To study multifunctional TiO_2 thin films for photocatalytic, antimicrobial, and self-cleaning applications.	Pure TiO_2 thin films.	Non-aqueous sol-gel dip coating followed by annealing at various temperatures.	TiO_2 annealed at 723 K showed the highest MB degradation of 79.35%.
11	(Nakahara et al., 2015)	To synthesize uniform $SiO_2@TiO_2$ core-shell nanoparticles for photocatalytic and thin-film applications.	$SiO_2@TiO_2$ core-shell nanoparticles.	Sol-gel coating of TiO_2 shell on SiO_2 core followed by mild crystallization.	Spherical anatase $SiO_2@TiO_2$ nanoparticles showed good dispersibility and MB photocatalytic activity.
12	(Nursam et al., 2015)	To analyze the effect of pore architecture on titania thin-film photocatalytic activity.	Macro-/mesoporous titania thin films.	One-pot sol-gel soft templating with phase separation.	Hierarchical porous anatase films improved MB photocatalytic activity; optimum pore structure was PEG:PVP:F127 = 1:1:0.
13	(Zhang et al., 2015)	To examine the effect of Ag substrate thickness on Cu-doped TiO_2 visible-light photocatalysis.	Cu-doped TiO_2 thin films on Ag substrates.	Sol-gel method combined with magnetron sputtering.	Ag substrate thickness of 30 nm gave the best dispersion and MB degradation of about 86%.
14	(Zhu et al., 2017)	To study the effect of Nd doping on TiO_2 photocurrent and photocatalytic activity.	Nd-doped TiO_2 thin films on ITO glass.	Sol-gel dip coating followed by calcination at 450 °C.	1 at.% Nd-doped TiO_2 showed the highest photocurrent and about 60% higher MB degradation than undoped TiO_2.

Thematically, the most frequently appearing keywords are related to TiO_2 thin films, sol-gel method, photocatalysis, methylene blue degradation, doping, self-cleaning, optical properties, and antimicrobial activity. This indicates that the main focus of the reviewed articles is the development of sol-gel-based TiO_2 thin films to enhance photocatalytic activity, particularly through control of deposition methods, annealing temperature, crystal structure, metal/non-metal doping, heterostructure formation, and modification of morphology and porosity. Based on Table 4, all reviewed articles indicate that TiO_2 and TiO_2-based materials remain the primary focus in the development of photocatalytic, self-cleaning, antibacterial thin films, and environmental applications. Of the 14 articles, all employed sol-gel-based approaches, including dip-coating, spin coating, drop coating, spray coating, or combinations with other methods such as electrodeposition and magnetron sputtering. This demonstrates that the sol-gel method is the most dominant due to its simplicity, low cost, ease of control, and suitability for thin film deposition on various substrates.

In terms of materials, 6 articles used pure TiO_2 or titania without major dopants, namely Yazid et al., (2019), Pinton et al., (2024), Lukong et al., (2022), Lukong et al., (2021), Sunil et al., (2024), and Nursam et al., (2015). Meanwhile, 8 articles used modified materials, such as Hg^{2+}-doped TiO_2, P-doped TiO_2/MWCNTs, Ag-loaded TiO_2-ZnO, $ZnIn_2S_4$/ TiO_2, PVP-doped SnO_2/ TiO_2, SiO_2@ TiO_2, Cu-doped TiO_2, and Nd-doped TiO_2. This composition shows that most studies do not rely solely on pure TiO_2 but also modify it through doping, heterostructures, core–shell design, or pore engineering to enhance photocatalytic activity.

In general, the research results show that improvement in TiO_2 performance can be achieved through three main strategies, namely process parameter control, material composition modification, and morphology engineering. Process parameters such as annealing, TTIP molarity, and sol aging have been proven to influence crystallinity, phase, band gap, and degradation activity. Abbas & Bensaha, (2021) showed that longer annealing reduced the band gap from 3.47 to 2.95 eV and increased methylene blue degradation up to 56.72%. Yazid et al., (2019) showed that a TTIP molarity of 0.5 M produced the highest degradation, reaching 95% under UV and 86% under visible light. Lukong et al., (2022) also reported a decrease in band gap from 3.39 to 3.20 eV, with the best self-cleaning performance at 600 °C annealing.

Table 5. Synthesis Parameters, Morphostructural Characteristics, Crystal Phase, and MB Degradation Efficiency on TiO_2-Based Materials

No	Temp. (°C)	Time (hours)	Solvent	Raw Material	Substrat	Morphology	Particles Size	Crys. Size	Phase	Deg. (%)	Ref.
1	1000° C	4-6	Butanol, acetic acid, ethanol, distilled water	Tetrabutyl orthotitanate dan mercuric acetate	Silicon	Agglomerated particles; nanotube-like structures	20–500 nm	41.19 – 95.87 nm	Anatase-rutile-$HgTiO_3$	22.74 – 56.72	(Abbas & Bensaha, 2021)

2	500° C	1	DI Water dan HCl	TTIP	Glass	Solid dengan retakan & berpori	0.5-1 µm	11-34 nm	Anatase, Rutile, & Brookite	65-86	(Yazid et al., 2019)
3	400° C	24	Xylene	Titanium (IV) butoxide, Triton X-100, distilled water	Glass	Uniform thin film; slight cracks at edges	741 nm	20-34 nm	Anatase	90	(Pinton et al., 2024)
4	400-800° C	1	Ethanol	Titanium isopropoxide, nitric acid, ionized water	Glass	Snowflake at 400 °C; agglomerated at 600 and 800 °C	1-2 µm	4.77–34.59 nm	Anatase, dan Rutile	47-56	(Lukong et al., 2022)
5	500	2	Absolute ethanol	Titanium isopropoxide, phosphoric acid, FMWCNTs, TEA, HCl, water	Soda-lime silica glass	Composite thin film	1-8 µm	10.75 -11.3 nm	Anatase TiO_2	55	(Sarker et al., 2022)
6	500	NR	2-propanol	Titanium isopropoxide, zinc acetate dihydrate, silver source	Glass	Aggregated particles; smaller particles after Ag loading	20–25 nm	15.74 - 18.63	Anatase TiO_2	80	(Pérez-González et al., 2019)
7	600	NR	Ethanol	TTIP, nitric acid, water	Glass	Uniformly distributed TiO2 particles	3.61-22.77 nm	3.61–21.77 nm	Anatase; possible rutile transition	72.50 – 92.90	(Lukong et al., 2021)
8	450	1	Isopropanol, methanol, water	Titanium isopropoxide, Zn/In/S precursors	ITO glass	Full surface coverage after ZnIn2S4 deposition	10-100 nm	34-78 nm	Anatase TiO_2 and rhombohedric $ZnIn_2S_4$	91	(Assaker et al., 2015)
9	600	2	Ethanol	TTIP, TEOS, tin chloride pentahydrate, PVP, HCl	E-glass fiber	Thin film coating on glass fibers	29-78 nm	8.4 nm	Anatase TiO_2	71.9-90	(Kongsong et al., 2018)
10	350–950	1	Isopropanol, methanol	Titanium tetra isopropoxide, acetic acid	Soda-lime glass, quartz	Spherical morphology	16.73-74.28 nm	13.45 – 69.24 nm	Anatase; mixed phase at high temperature	79.35	(Sunil et al., 2024)
11	100	48–72	Ethanol, n-butanol, aqueous HNO3	Colloidal SiO2, TBOT	Glass	Monodisperse spherical core-shell particles	23.5-72 nm	20.7nm	Anatase TiO_2 shell	70-94	(Nakahara et al., 2015)
12	450	1	Ethanol	Titania precursor, PEG, PVP, Pluronic F127	Borosilicate glass coverslip	Hierarchical macro-/mesoporous film	75-200 nm	25.8 nm	Anatase TiO_2	5-32	(Nursam et al., 2015)
13	500	NR	NR	Cu-doped	Ag-	Well-	200-	18.7-	TiO_2-based;	86	(Zhang

				TiO_2 sol, Ag target/substrate	coated glass	dispersed nanoparticles at 30 nm Ag; agglomerated at excess Ag	300 nm	24.6 nm	Ag-TiO_2formation		et al., 2015)
14	450	2	NR	Ti precursor, Nd precursor	ITO glass	Homogeneous spherical nanoparticles; agglomeration at 0.5 at.% Nd	45-89 nm	10.4-12 nm	Anatase TiO_2	17-25.9	(Zhu et al., 2017)

Articles using modified materials generally show more significant performance improvements. $ZnIn_2S_4$/ TiO_2 in Assaker et al., (2015) achieved 91% methylene blue degradation, while Cu-doped TiO_2 on Ag substrate with 30 nm thickness in Zhang et al., (2015) reached approximately 86% degradation. In Sunil et al., (2024), TiO_2 annealed at 723 K achieved 79.35% methylene blue degradation. Meanwhile, Zhu et al., (2017) showed that 1 at.% Nd-doped TiO_2 produced the highest photocurrent and increased methylene blue degradation by approximately 60% compared to undoped TiO_2. Based on Table 5, the calcination temperatures used in the reviewed articles span a wide range, from 100 to 1000 °C. The lowest temperature was used by Nakahara et al., (2015), namely 100 °C, while the highest was used by Abbas & Bensaha, (2021), namely 1000 °C. Among the 14 articles, 500 °C is the most frequently used temperature, appearing in 4 articles or approximately 28.57% of the total. This temperature is used in Yazid et al., (2019), Sarker et al., (2022), Pérez-González et al., (2019), and Zhang et al., (2015). Meanwhile, 450 °C is used in 3 articles, namely Assaker et al., (2015), Nursam et al., (2015), and Zhu et al., (2017). This indicates that the range of 450–500 °C is the most common thermal condition for producing TiO_2 thin films with good crystallinity without causing excessive phase transformation.

From the aspect of calcination time, the duration of thermal treatment ranges from 1 hour to 72 hours. A duration of 1 hour is the most dominant, used in 5 articles, namely Yazid et al., (2019), Lukong et al., (2022), Assaker et al., (2015), Sunil et al., (2024), and Nursam et al., (2015). A duration of 2 hours is used in Sarker et al., (2022), Kongsong et al. (2018), and Zhu et al., (2017). The longest treatment is found in Nakahara et al., (2015), namely 48–72 hours, while Pinton et al., (2024) uses 24 hours. This variation indicates that calcination time not only serves to improve crystallinity but also plays a role in controlling morphology, crystal size, and film stability.

The most commonly used solvent is ethanol, either as the main solvent or combined with other solvents. Ethanol appears in several articles, such as Abbas & Bensaha, (2021), Lukong et al., (2022), Sarker et al., (2022), Lukong et al., (2021), Kongsong et al., (2018), Nakahara et al., (2015), and Nursam et al., (2015). In addition to ethanol, some studies use isopropanol, methanol, xylene, n-butanol, DI water, HCl, and aqueous HNO_3. This shows that solvents play an important role in controlling precursor hydrolysis, sol viscosity, film homogeneity, and surface morphology formation.

In terms of raw materials, the most commonly used titanium precursor is TTIP or titanium isopropoxide, both in pure TiO_2 and modified materials. In addition, several studies use titanium butoxide, titanium tetra isopropoxide, tetrabutyl orthotitanate, TBOT, zinc acetate dihydrate, mercuric acetate, phosphoric acid, MWCNTs, TEOS, tin chloride pentahydrate, PVP, and Nd or Zn/In/S precursors. This variation indicates two main trends in TiO_2 development, namely the use of pure TiO_2 for process optimization and the use of composite or doped materials to enhance photocatalytic activity. The most dominant substrates are glass-based substrates. Of the 14 articles, most use glass, soda-lime glass, borosilicate glass coverslip, E-glass fiber, Ag-coated glass, or ITO glass. Glass-based substrates are used because they are transparent, stable, easy to coat, and suitable for photocatalytic coating, self-cleaning, and water disinfection applications. A different non-glass substrate is silicon used by Abbas & Bensaha, (2021), while ITO glass is used by Assaker et al., (2015) and Zhu et al., (2017) to support photoelectrochemical applications.

In terms of morphology, the reviewed materials show a wide variation, ranging from agglomerated particles, nanotube-like structures, porous cracked films, uniform thin films, snowflake morphology, spherical morphology, core–shell particles, to hierarchical macro-/mesoporous films. More open, porous morphologies or well-distributed particles are generally associated with increased photocatalytic activity because they provide a larger contact area between the catalyst surface and methylene blue. However, excessive agglomeration can reduce effectiveness by decreasing active surface area and hindering light penetration. Particle size in the reviewed articles ranges from 10 nm to 8 µm, depending on synthesis method, calcination temperature, dopant type, and substrate. The smallest particle size is reported by Assaker et al., (2015), namely 10–100 nm, while larger sizes are found in Sarker et al., (2022), namely 1–8 µm. Crystallite size also varies, ranging from approximately 3.61–95.87 nm. The smallest crystallite size is reported by Lukong et al., (2021), namely 3.61–21.77 nm, while the largest is found in Abbas & Bensaha, (2021), reaching up to 95.87 nm. In general, smaller crystallite size tends to support photocatalytic activity by increasing surface area, but excessively low crystallinity may reduce charge separation efficiency.

The most dominant crystal phase is anatase TiO_2. Almost all articles report the presence of anatase, either as a single phase or in combination with rutile, brookite, $HgTiO_3$, $ZnIn_2S_4$, or Ag-TiO_2. The anatase phase is important because it generally exhibits higher photocatalytic activity compared to other phases. Some studies report mixed phases, such as anatase-rutile-$HgTiO_3$ in Abbas & Bensaha, (2021), anatase-rutile-brookite in (Yazid et al., 2019), anatase-rutile in Lukong et al., (2022), and anatase TiO_2 with rhombohedral $ZnIn_2S_4$ in Assaker et al., (2015). These mixed phases can enhance photocatalytic activity if they improve electron–hole separation. Methylene blue degradation efficiency shows a wide variation, ranging from 5% to 94%. The highest degradation is reported by Nakahara et al., (2015) with a range of 70–94%, followed by Lukong et al., (2021) with 72.50–92.90%, Assaker et al., (2015) with 91%, Pinton et al., (2024) with 90%, and Kongsong et al., (2018) with 71.9–90%. In contrast, the lowest degradation is reported by Nursam et al., (2015), namely 5–32%. This variation indicates that photocatalytic efficiency is not determined solely by the anatase phase but also by particle size, porosity, substrate type, film thickness, dopant type, and photocatalytic testing conditions.

DISCUSSION

The distribution of articles in Table 3 indicates that studies on TiO_2-based thin films for photocatalysis, self-cleaning, antibacterial, and environmental applications have developed within the publication range of 2015–2024, with a dominance of Elsevier as the publisher. The dominance of articles from journals in materials science, physical chemistry, and environmental technology shows that TiO_2 thin films are no longer positioned solely as conventional semiconductor materials, but as functional platforms that can be engineered through composition, crystal phase, morphology, and substrate configuration. The relevance of TiO_2 as a photocatalytic material is rooted in its chemical stability, relatively low cost, non-toxicity, and its ability to generate electron–hole pairs upon receiving sufficient photon energy (Fujishima & Honda, 1972; Hoffmann et al., 1995; Linsebigler et al., 1995; Mills & Le Hunte, 1997; Diebold, 2003; Carp et al., 2004; Hashimoto et al., 2005; Chen & Mao, 2007; Fujishima et al., 2008). In this context, the variation in citations in Table 3 also indicates that topics such as heterostructures, green sol-gel, doping, and surface engineering have strong appeal because they are directly related to improving the efficiency of organic pollutant degradation.

The research focus summarized in Table 4 shows that all articles employ sol-gel-based approaches, either as the main method or as an initial step combined with electrodeposition, magnetron sputtering, spray coating, drop coating, spin coating, or dip coating. This trend indicates that sol-gel remains the most adaptive synthesis route for TiO_2 thin films because it enables control over hydrolysis-condensation, precursor homogeneity, chemical composition, film thickness, and crystal phase formation at relatively moderate temperatures. In photocatalysis, synthesis control is essential because TiO_2 activity is not solely determined by the presence of a semiconductor material, but by the combination of crystallinity, energy band structure, surface area, surface accessibility, and charge separation dynamics (Schneider et al., 2014; Nosaka & Nosaka, 2017; Chong et al., 2010; Nakata & Fujishima, 2012; Pelaez et al., 2012; Daghrir et al., 2013; Khaki et al., 2017; Chen et al., 2020). Therefore, the synthesis of TiO_2 thin films should be interpreted as an integrated engineering process rather than merely the formation of a thin layer on a substrate.

The data in Table 5 confirm that calcination temperature is the most critical parameter in controlling phase, crystallite size, morphology, and MB degradation efficiency. The temperature range used is very broad, from 100 to 1000 °C, but the highest performance is not always achieved at the highest temperature. Increasing temperature indeed enhances crystallinity, but excessively high temperatures accelerate crystal growth, increase agglomeration, reduce effective surface area, and promote the transformation of anatase into rutile. This is consistent with the understanding that the anatase–rutile transformation is influenced by temperature, particle size, surface energy, atmospheric conditions, and the presence of dopants or impurities (Hanaor & Sorrell, 2011; Dambournet et al., 2010; Zhang et al., 2014; Phromma et al., 2020; Kim et al., 2021). In the Hg-doped TiO_2 system, calcination at 1000 °C indeed results in the formation of anatase–

rutile–$HgTiO_3$ phases, but the crystallite size increases up to 95.87 nm and MB degradation remains only 22.74–56.72%. This condition indicates that high crystallinity does not necessarily correspond to high photocatalytic activity when accompanied by grain growth and reduced active surface area.

The temperature range of 450–600 °C is more rational as an optimum region because it provides sufficient crystallinity without excessively increasing crystallite size. This is evident from several data points in Table 5, such as green sol-gel TiO_2 at 500 °C achieving MB degradation up to 95% under UV and 86% under visible light, PVP/SnO_2/TiO_2 at 600 °C achieving degradation up to approximately 90%, sol-aged TiO_2 at 600 °C achieving 72.50–92.90% degradation, and Cu-doped TiO_2 on Ag substrate at 500 °C achieving approximately 86% degradation. Conceptually, this range supports the formation of crystalline anatase with small to moderate crystallite size, maintains active surface area, and suppresses excessive grain growth. The relationship between temperature, porosity, crystallinity, and activity is further supported by studies on mesoporous thin films and sol-gel TiO_2, which show that pore accessibility, crystallite size, film thickness, and surface openness strongly influence photocatalytic activity (Yu et al., 2003; Sakatani et al., 2006; Scarpelli et al., 2018; Pant et al., 2019; Vahl et al., 2019; He et al., 2023).

The anatase phase is the phase most consistently associated with high photocatalytic activity. In Table 5, nearly all articles report the presence of anatase, either as a single phase or in combination with rutile, brookite, $HgTiO_3$, $ZnIn_2S_4$, or Ag-TiO_2. Anatase is generally more active due to its electronic structure and charge carrier dynamics, which favor the formation of oxidative species on the surface. However, mixed phases are not always detrimental. Anatase–rutile mixtures, anatase–brookite systems, or semiconductor heterostructures can enhance charge separation if good interfacial contact is formed and the phase ratio is well controlled. This effect is widely discussed in the literature on heterostructures and interface engineering, emphasizing that activity enhancement arises from suppressed electron–hole recombination rather than solely increased crystallinity (Asahi et al., 2001; Serpone, 2006; Etacheri et al., 2015; Low et al., 2017; Marschall, 2014; Tong et al., 2012; Rajput et al., 2022). Therefore, mixed phases in TiO_2 are beneficial only when they function as effective charge transfer pathways, rather than as a result of uncontrolled crystal growth.

Particle size and crystallite size in Table 5 indicate that MB degradation activity does not follow a linear trend with decreasing size. Smaller crystallite size can increase surface area and the number of active sites, but excessively small size with low crystallinity may introduce structural defects that act as recombination centers. Conversely, excessively large crystallite size reduces active surface area and limits contact between the photocatalyst and MB molecules. This pattern is clearly observed in sol-aged TiO_2: aging for 24 hours produces the smallest crystallite size, but the degradation efficiency is lower than that of 48 hours aging; meanwhile, 72 hours aging increases crystallite size and begins to show phase transition tendencies. Thus, the optimum size is not the smallest absolute size, but the size that provides a balance between crystallinity, surface area, and charge separation. This principle is consistent with studies showing that crystallite size, pore distribution, surface area, and charge transport pathways simultaneously determine photocatalytic rates (Ohtani, 2010; Zhang et al., 2014; Schneider et al., 2014; Nosaka & Nosaka, 2017; Chen et al., 2020).

The morphology summarized in Table 5 shows a wide variation, ranging from agglomerated particles, nanotube-like structures, cracked porous films, snowflake morphology, spherical morphology, core–shell particles, to hierarchical macro-/mesoporous films. Open and porous morphologies in principle enhance contact between MB and the catalyst surface, but the review results indicate that porosity alone is not sufficient. Macro-/mesoporous titania in Nursam et al. only achieved 5–32% degradation despite having a well-defined pore architecture. This indicates that pores must be accessible to both target molecules and light; if pores are too deep, too closed, or if the film is too thick, light penetration and molecular diffusion become limited. Literature on mesoporous TiO_2 thin films also emphasizes that activity is determined by a combination of crystallinity, surface-to-volume ratio, pore openness, film thickness, roughness, and electron transport (Sakatani et al., 2006; Scarpelli et al., 2018; Pant et al., 2019; Vahl et al., 2019). Therefore, the optimum morphology must provide an accessible active surface, not merely visible pores or cracks.

Material modification strategies in Tables 4 and 5 show that doping, heterostructures, core–shell design, and carbon composites can improve performance, but they still have optimum limits. P-doped TiO_2/MWCNTs improve activity compared to pure TiO_2 because MWCNTs can act as electron transport pathways, while P can assist in forming new electronic states or electron traps. $ZnIn_2S_4/TiO_2$ achieves 91% MB degradation because the heterostructure extends visible light response and improves charge separation. Cu-doped TiO_2 on Ag substrate reaches approximately 86% when the Ag thickness is 30 nm; excessive Ag thickness can instead trigger agglomeration and unfavorable phase formation. This principle is consistent with literature showing that dopants and heterostructures can narrow the band gap, prolong charge carrier lifetime, and enhance light absorption, but excessive dopant concentration may act as recombination centers (Yu et al., 2011; Khaki et al., 2017; Low et al., 2017; Marschall, 2014; Rajput et al., 2022).

Visually, Figure 2 summarizes the cause–effect relationships derived from Tables 4 and 5. The initial part of Figure 2 positions temperature, time, solvent, precursor, deposition method, modification strategy, and substrate as the main synthesis variables. These variables then control structural responses in the form of phase evolution, crystallite size, surface morphology, band gap, and charge separation. The final consequence is the change in MB degradation efficiency. Thus, Figure 2 confirms that the photocatalytic performance of TiO_2 thin films cannot be explained by a single parameter, such as temperature or crystallite size alone, but must be understood through the interconnection between synthesis parameters, structure, morphology, and charge mechanisms. The figure also clarifies that optimum conditions are achieved under moderate thermal treatment, anatase phase dominance, controlled crystallite size, and surface engineering that enhances MB accessibility while suppressing electron–hole recombination.

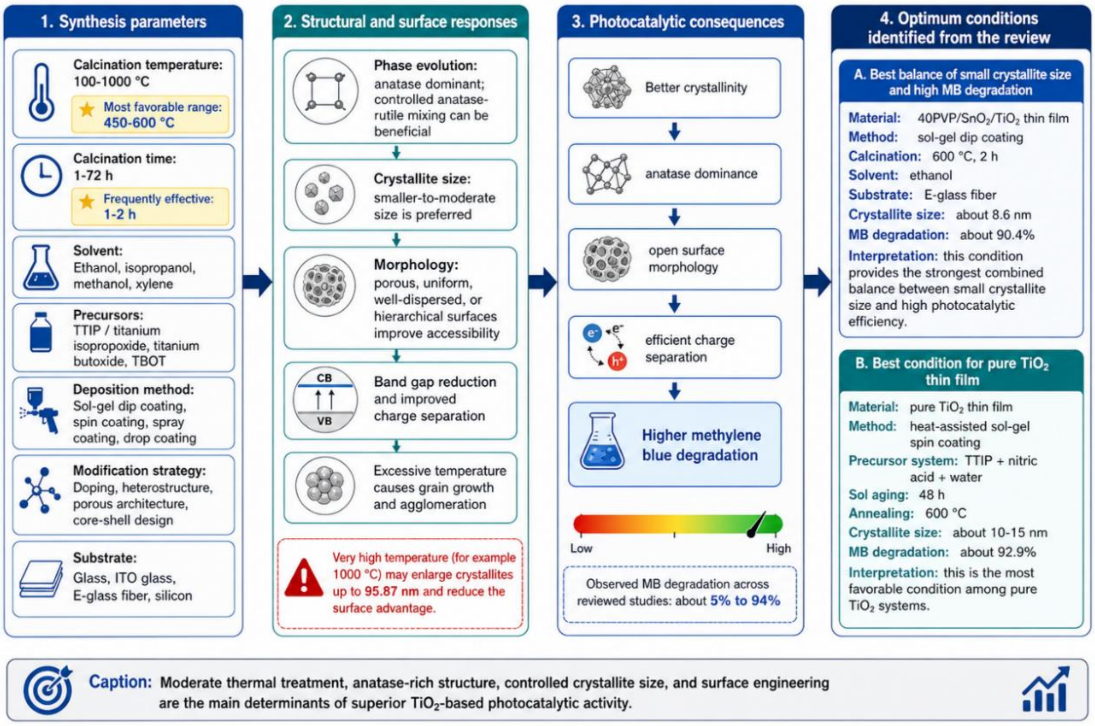

Figure 2. Schematic of the relationship between synthesis parameters, morphostructural characteristics, and photocatalytic activity of TiO_2-based thin films

Based on the combination of small crystallite size and high MB degradation, the most optimum parameters within the modified material group are found in 40PVP/SnO_2/TiO_2 thin films. The synthesis conditions include sol-gel dip coating, ethanol as solvent, raw materials consisting of TTIP, TEOS, tin chloride pentahydrate, PVP, and HCl, E-glass fiber as the substrate, and calcination at 600 °C for 2 hours. These conditions produce a crystallite size of approximately 8.6 nm and MB degradation of about 90.4%. This value represents the best balance between very small crystallite size and high degradation efficiency. Although some studies report slightly higher MB degradation, their crystallite sizes are generally larger; conversely, materials with smaller crystallite sizes do not always achieve comparable degradation performance. Therefore, 40PVP/SnO_2/TiO_2 can be positioned as the optimum condition based on combined criteria rather than a single parameter.

The evaluation is limited to pure TiO_2 without dopants or composites, the most optimum condition is heat-assisted sol-gel spin coating using a precursor system of TTIP, nitric acid, and water, ethanol as the solvent, sol aging for 48 hours, and annealing at 600 °C. This condition produces a crystallite size of approximately 10–15 nm with MB degradation of about 92.90%. Aging for 24 hours indeed yields a smaller crystallite size, but with lower degradation efficiency; meanwhile, aging for 72 hours results in high degradation but increased crystallite size and the onset of a transition toward rutile. Therefore, aging for 48 hours at 600 °C can be considered the optimum point for pure TiO_2 because it provides the best compromise between crystallinity, crystallite size, and photocatalytic activity.

The comparison of MB degradation values across articles must be interpreted with caution because each study employs non-uniform testing conditions, including initial MB concentration, irradiation time, light intensity and type, film surface area, layer thickness, solution pH, substrate type, and reactor configuration. These differences can affect the reported degradation values; therefore, the optimum claims in this discussion are comparative based on available data rather than universal standards. However, the general trend remains strong: TiO_2-based thin films exhibit better performance when synthesized at moderate temperatures, dominated by the anatase phase, possessing small to medium crystallite sizes, open morphology, and compositional or surface modifications that enhance charge separation. Thus, the most promising synthesis direction is not merely reducing particle size, but optimizing crystallinity, phase, morphology, film thickness, and electron transfer pathways in an integrated manner.

CONCLUSION

Based on the review of 14 articles, TiO_2-based thin films demonstrate strong potential as photocatalytic materials for methylene blue degradation, particularly when synthesis parameters are controlled in an integrated manner. The sol–gel method emerges as the most dominant synthesis route due to its compatibility with various deposition techniques, such as dip coating, spin coating, drop coating, spray coating, electrodeposition, and magnetron sputtering. The analysis shows that calcination or annealing temperature is the most influential parameter affecting phase formation, crystallite size, morphology, and photocatalytic efficiency. The temperature range of 450–600 °C is the most effective condition, as it enables the formation of sufficiently crystalline anatase without causing excessive crystal growth and agglomeration. The anatase phase is consistently identified as the primary phase supporting high photocatalytic activity, although mixed phases such as anatase–rutile or semiconductor heterostructures may provide additional advantages when they enhance electron–hole pair separation.

The efficiency of methylene blue degradation is not determined by a single factor, but by the combined influence of calcination temperature, thermal treatment time, solvent type, raw materials, substrate, morphology, particle size, crystallite size, crystal phase, and material modification strategies. Excessively large crystallite sizes tend to reduce the active surface area, while very small but poorly crystalline structures do not necessarily yield the highest activity. Therefore, the optimum size lies within the small-to-medium range that still maintains crystallinity and efficient charge transfer. Based on the combined criteria of small crystallite size and high methylene blue degradation, the best condition for modified materials is achieved in PVP/SnO_2/TiO_2 thin films synthesized via sol–gel dip coating, using ethanol as the solvent, E-glass fiber as the substrate, and calcination at 600 °C for 2 hours. This condition produces a crystallite size of approximately 8.6 nm and methylene blue degradation of about 90.4%. For pure TiO_2, the most optimum condition is obtained through heat-assisted sol–gel spin coating with 48-hour sol aging and annealing at 600 °C, resulting in a crystallite size of approximately 10–15 nm and methylene blue degradation of about 92.90%. Thus, an effective synthesis of TiO_2 thin films should be directed toward the simultaneous optimization of anatase phase, crystallinity, surface morphology, crystallite size, and charge separation capability, rather than merely reducing particle size alone.

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