

SYNTHESIS, CHARACTERIZATION, AND CATALYTIC ACTIVITY OF ZnZrO_3
MIXED METAL OXIDE FOR METHYLENE BLUE OXIDATION

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ABSTRACT

Zinc zirconate (ZnZrO_3) is a mixed-metal oxide with potential applications in heterogeneous catalysis and advanced oxidation processes. In the present study, ZnZrO_3 was synthesized by the sol-gel method under the optimized experimental conditions previously established and reported by the authors.^[1] The synthesized material was subjected to thermal treatment at different temperatures and characterized using differential thermal analysis (DTA), X-ray diffraction (XRD), and Fourier-transform infrared spectroscopy (FT-IR) to investigate its thermal behavior, phase evolution, and structural characteristics. The catalytic activity of ZnZrO_3 was evaluated for the oxidative degradation of methylene blue (MB) in the presence of hydrogen peroxide (H_2O_2) under heterogeneous catalytic conditions. For comparison, NiZrO_3 , which had previously been synthesized and characterized by the sol-gel method according to the authors' earlier work^[2], was evaluated under the same catalytic conditions. The results showed that the ZnZrO_3 phase was formed at high calcination temperatures, with the ZnZrO_3 phase obtained at 1000 °C. The catalytic experiments demonstrated that ZnZrO_3 exhibited higher catalytic efficiency toward MB oxidation than NiZrO_3 , with catalytic efficiencies of 58.33% and 28.57%, respectively. These findings indicate that ZnZrO_3 possesses promising catalytic activity for the heterogeneous oxidative treatment of methylene blue using hydrogen peroxide and demonstrate its potential as a mixed-metal oxide catalyst for advanced oxidation applications.

KEYWORDS: Zinc zirconate, ZnZrO_3 , Methylene blue, Heterogeneous catalysis, Hydrogen peroxide, Advanced oxidation process.

1. INTRODUCTION

Metal oxides, whether in bulk form, thin films, or nanostructures, exhibit a wide variety of functional properties. This versatility makes them ideal candidates for a broad range of applications, including solar cells, gas sensors, optoelectronic devices, catalysis, corrosion protection, and environmental remediation.

The functional performance of a metal oxide depends heavily on its crystal structure, chemical composition, and defects, which ultimately govern its optical, chemical, physical, and mechanical properties. Processing methods and growth parameters significantly dictate the morphological and structural features, and consequently, the chemical and physical behavior of the

metal oxide. Furthermore, the electronic structure and bandgap of these oxides can be precisely controlled and tuned by altering their size and dimensions, thereby enabling an even wider array of technological applications.^[3]

Scientists have discovered that many natural materials possess unique properties, leading to the development of what are known as mixed oxides. These are chemical compounds formed by blending two or more metal oxides in specific, fixed stoichiometric ratios. Consequently, they exhibit novel properties that distinctively differ from and often surpass those of their individual constituent metal oxides.

Mixed oxides have been known since antiquity and have been extensively utilized since the beginning of the 19th century (circa 1800 AD) in the ceramic and pottery industries, owing to their exceptional color stability and thermal resistance. These industries utilize kilns that reach extremely high temperatures a range in which most conventional chemicals and natural colorants decompose. Transition metals are the most commonly employed components in these applications, where the coloration of the resulting mixed oxide is dictated by the specific metal used, the synthesis method, and the generated crystal structure.

Typically, mixed oxides are synthesized via the calcination of a mixture of metal hydroxides or their respective oxides within a temperature range of 300°C to 1300°C. This thermal treatment is the primary reason behind the remarkable chemical stability of the structure, rendering it highly resistant to various aggressive media (both acids and bases) in addition to ensuring its thermal stability.^[4]

These compounds exhibit a diverse range of crystalline phases, including perovskite, spinel, and rutile structures, among others. Within this framework, zirconates (M₂ZrO₃) emerge as a prominent class of mixed oxides adopting the perovskite crystal structure, which endows them with a multitude of unique physical and chemical properties.^[5]

1.1 The Role of Mixed Oxides in Catalytic Oxidation Applications

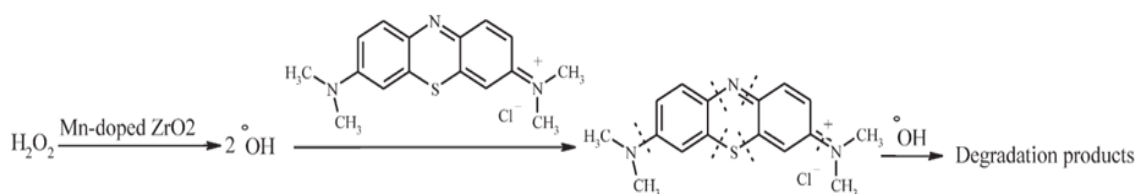
Catalytic oxidation using hydrogen peroxide (H₂O₂) represents one of the most vital Advanced Oxidation Processes (AOPs) employed in treating wastewater contaminated with non-biodegradable organic compounds. This technique relies on the activation of (H₂O₂) by a solid catalyst to generate hydroxyl radicals

(•OH). Characterized by their remarkably high oxidation potential, these radicals oxidize organic pollutants, converting them into less complex compounds or achieving complete mineralization into carbon dioxide (CO₂) and water (H₂O).

This technique offers several distinct advantages compared to conventional oxidation processes, including:

1. **Operation under mild conditions:** The process can be conducted under ambient temperature and pressure operating conditions.
2. **No external energy requirements:** It eliminates the need for a light source or electrical energy.
3. **Catalyst recyclability:** The catalyst can be recovered and reused when utilizing heterogeneous catalysts.
4. **Prevention of sludge formation:** It avoids the generation of large quantities of iron sludge, which is a major drawback associated with the conventional Fenton process.^[6]

The intrinsic nature of the catalyst plays a fundamental role in determining the efficiency of the catalytic oxidation process. Specifically, the specific surface area, the density of active sites, and the electronic properties of the catalyst directly dictate the activation rate of hydrogen peroxide and the subsequent generation of free radicals. For this reason, mixed metal oxides have garnered increasing attention. This interest is driven by their superior chemical and thermal stability, as well as the synergistic effects between their individual components, which enhance electron transfer and boost overall catalytic activity. Ultimately, the efficiency of these processes depends fundamentally on the type of catalyst utilized, as it accelerates (H₂O₂) decomposition to yield reactive hydroxyl radicals. The following mechanism illustrates the typical reaction pathway:



Following the generation of free radicals, they vigorously attack the Methylene Blue molecules by cleaving the chemical bonds, aromatic rings, and nitrogen- and sulfur-containing functional groups within their molecular structure. This degradation pathway leads to the formation of low-molecular-weight organic intermediates, which undergo progressive oxidation until complete mineralization is achieved.

Consequently, this process results in a gradual decrease in color intensity and a concomitant reduction in absorbance at the maximum absorption wavelength (λ_{max}), which serves as a key indicator for evaluating the catalytic efficiency.^[7]

Mixed metal oxides represent one of the most promising classes of materials in catalytic oxidation applications, owing to their distinctive structural and electronic properties compared to simple oxides. The integration of two metallic elements within a single crystalline lattice increases the density of active sites, enhances electronic charge transfer, and improves the thermal and chemical stability of the catalyst. Consequently, this synergistic effect directly amplifies the efficiency of hydroxyl radical generation.

Zirconium (Zr) occupies a prominent position in this field due to its high chemical stability, corrosion resistance, and suitable amphoteric (acid-base)

properties. Furthermore, its capacity to form mixed oxides with various transition metals such as zinc, nickel, vanadium, and iron significantly enhances catalytic activity and boosts the material's efficiency in removing organic pollutants.^[8]

Numerous recent studies have demonstrated that zirconium-based mixed oxides exhibit superior catalytic activity compared to their single-oxide counterparts in the removal of organic dyes. This enhanced performance is primarily attributed to the improved activation efficiency of H_2O_2 , a reduced recombination rate of free radicals, and an increased density of active sites on the catalyst surface.^[9]

2. Zirconate compounds (MZrO_3)

Zirconate compounds crystallized in the perovskite structure, which bear the general chemical formula (MZrO_3) where (M) represents a divalent cation such as barium (Ba), calcium (Ca), or zinc (Zn) constitute a pivotal class of mixed metal oxides in solid-state chemistry and modern materials science.^[10]

These compounds attract extensive research and technological interest due to their high thermal and chemical stability, elevated melting points, and characteristically wide bandgaps. The properties of these materials are directly influenced by the nature of the M cation and its associated structural distortion within the crystal lattice. Consequently, their physical and chemical behaviors span a broad spectrum, including high electrical insulation, distinct optical properties, and antiferroelectric behavior. In addition to their electronic features, these compounds have demonstrated high catalytic efficiency and excellent structural durability when employed as heterogeneous catalysts.^[11]

This functional versatility renders (MZrO_3) compounds a cornerstone in the development of high-performance electronic ceramics, ceramic capacitors, and gas sensors, as well as in environmental catalysis and fuel cell applications. This positions them at the forefront of smart materials driving clean energy technologies and environmental sustainability.^{[10],[11],[12],[13]}

2.1. Catalytic Activity of Zirconate-Based Compounds

The treatment of organic pollutants generated by industrial activities represents one of the most critical contemporary environmental challenges, with a particular emphasis on wastewater contaminants derived from textile dyes. Significant quantities of organic dyes are consumed during textile manufacturing processes, a substantial fraction of which is discharged into industrial effluents due to low fixation efficiency during the dyeing stage. These pollutants critically degrade water quality by reducing light penetration within the aquatic medium, thereby inhibiting the photosynthetic processes of resident organisms. Furthermore, certain degradation by-products of these dyes possess toxic or hazardous effects

on both human health and the ecosystem. Consequently, the remediation of these organic compounds from wastewater prior to environmental discharge is imperative. Although conventional methodologies such as adsorption, filtration, and biological treatment have been widely deployed, they often suffer from notable limitations. These include an inability to completely degrade recalcitrant pollutants or the mere translocation of contaminants from one phase to another without achieving chemical mineralization.^[14]

In recent years, advanced oxidation processes (AOPs) have garnered escalating interest as highly effective technologies for the remediation of organic pollutants, demonstrating exceptional efficiency owing to the development and implementation of specialized catalysts.

Among the most critical organic contaminants are the synthetic dyes widely utilized in textile, plastic, and various other industrial sectors. Approximately 100,000 distinct commercial dyes exist globally, with annual production capacities reaching up to one million tons. A significant portion of these compounds is characterized by complex chemical architectures that impart high structural stability and exceptional resistance to natural biodegradation pathways. Consequently, these persistent pollutants endure for extended periods in aquatic environments, exerting severe, long-term detrimental effects on ecosystems and living organisms.^[15]

Methylene blue (MB), whose chemical structure is illustrated in Figure (1), represents one of the most widely utilized dyes across various industrial processes, and its removal from industrial wastewater poses a formidable challenge. Consequently, the primary objective of this research is to investigate the catalytic activity of zinc zirconate and nickel zirconate compounds, synthesized via the sol-gel methodology, in the oxidation of methylene blue using hydrogen peroxide. This study aims to evaluate their efficacy in removing organic pollutants from aqueous solutions and to assess their potential viability as highly effective catalysts for environmental remediation applications.

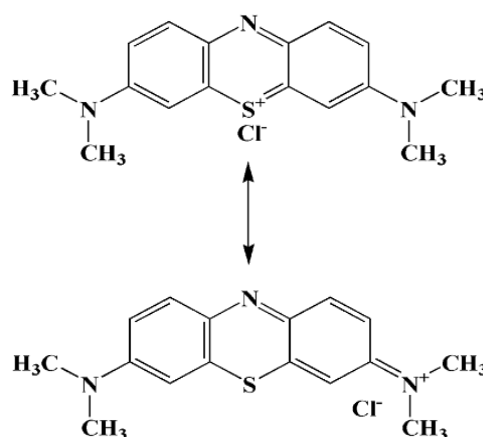


Figure 1: Chemical structure of methylene blue dye.

3. Toxicity of Methylene Blue

Textile industries usually release a large amount of MB dyes in natural water sources, which becomes a health threat to human beings and microbes. MB dye is harmful to human health above a certain concentration due to its substantial toxicity. MB is toxic, carcinogenic, and non-biodegradable and can cause a serious threat to human health and destructive effects on the environment. MB causes several risks to human health such as respiratory distress, abdominal disorders, blindness, and digestive and mental disorders. It also causes nausea, diarrhoea, vomiting, cyanosis, shock, gastritis, jaundice, methemoglobinemia, tissue necrosis, and increased heart rate, causing the death of premature cells in tissues and skin/eye irritations. MB contacts with skin may result in

skin redness and itching. The no observed adverse effect level (NOAEL) for the MB in rats was observed to be 25 mg kg⁻¹. Some of the toxic effects of MB on humans and other animals are represented in Figure (2). MB discharge into the environment is a significant threat for aesthetical and toxicological reasons. It also reduces light penetration and is a toxic supply to food chains for organisms. MB presence in water bodies, even at a very low concentration, makes highly coloured sub-products. Owing to its high molar absorption coefficient ($\sim 8.4 \times 10^4$ L mol⁻¹ cm⁻¹ at 664 nm), which reduces sunlight transmittance, it decreases oxygen solubility, affects the photosynthetic activity of aquatic life, and decreases the diversity and aesthetics of the biological community.^[16]

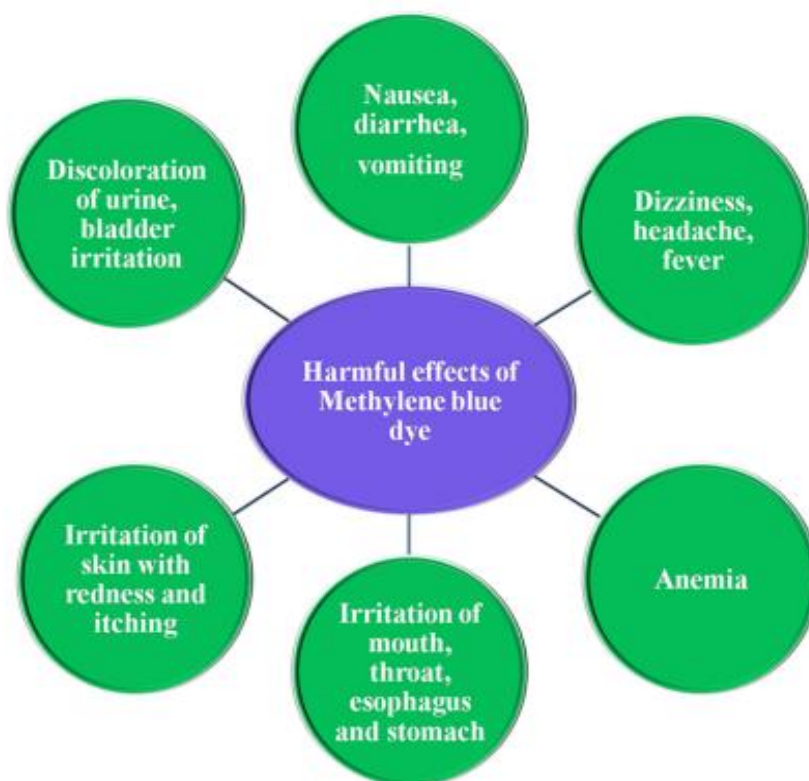


Figure 2: Harmful effects of the MB dye.

4. EXPERIMENTAL

4.1 Materials and Apparatus

Zinc zirconate (ZnZrO₃), Methylene Blue (MB), Hydrogen peroxide (H₂O₂), X-ray Diffraction (XRD), Differential Thermal Analysis (DTA), Fourier Transform Infrared Spectroscopy (FT-IR), Ultraviolet-Visible Spectroscopy (UV-VIS).

4.2. Characterization of the (ZnZrO₃) Compound

After determining the optimal conditions for gel preparation and stabilization, as described in reference^[1], the formed gel was separated by filtration, washed with distilled water, and dried at 105 °C for 2 hours.

The resulting solid sample was divided into four equal portions and calcined at different temperatures (400, 550,

800, and 1000 °C) for 6 hours. The obtained samples were subsequently characterized using FT-IR, XRD, and DTA.

4.3. Catalytic Oxidation of Methylene Blue

The core objective of this study is to evaluate the catalytic efficiency of the synthesized compounds in the oxidation of methylene blue using hydrogen peroxide (H₂O₂). The experimental procedure was conducted according to the following steps:

4.3.1. Preparation of the Methylene Blue Calibration Curve

A series of increasing concentrations (2–64 mg/L) of methylene blue (MB) was prepared. The absorbance of

each concentration was measured at the maximum absorption wavelength ($\lambda_{\max}$) of 665 nm.

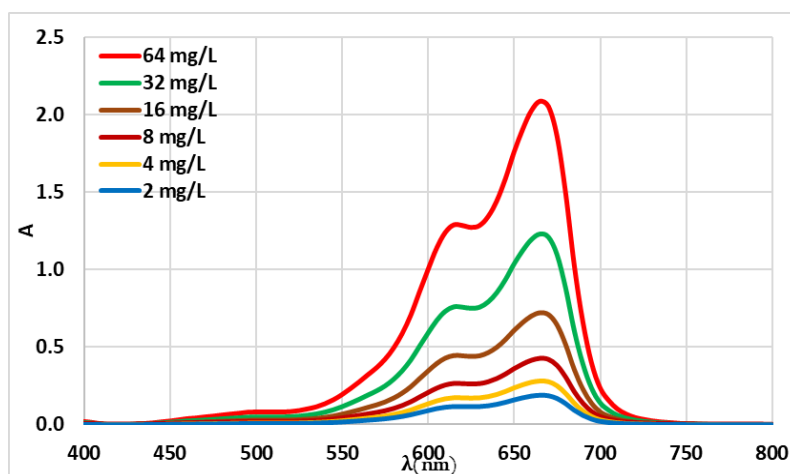


Figure 3: UV-Vis absorption spectra of methylene blue at various concentrations.

Table 1 presents the maximum absorbance values corresponding to each concentration:

Table 1: Maximum absorbance values of methylene blue at various concentrations.

C (mg/L)	2	4	8	16	32	64
A	0.1889	0.2833	0.4250	0.7225	1.2282	2.0880

The linear relationship between absorbance and the prepared concentrations is illustrated in the figure below:

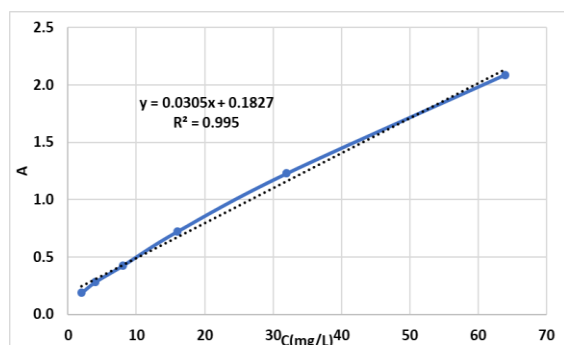


Figure 4: Absorbance of methylene blue as a function of concentration.

A linear relationship is observed between absorbance and concentration, with a correlation coefficient of $R^2=0.995$.

4.3.2. Oxidation of Methylene Blue in the Presence of Mixed Oxides

Methylene blue solutions were prepared at a constant concentration of 64 mg/L. To these solutions, a fixed amount of the mixed oxides (0.05 g) was added, followed by the addition of a constant volume of 30% hydrogen peroxide (H_2O_2), (10 mL). The mixtures were stirred for a fixed duration of 20 min, after which the absorbance of the solutions was measured using a UV-Vis spectrophotometer.

The figure below displays the absorption spectra of methylene blue in the presence of hydrogen peroxide and the added catalytic compound

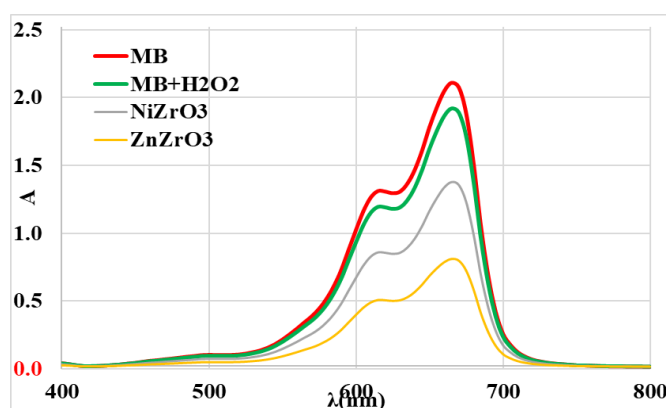


Figure 5: Changes in the UV-Vis absorption spectra of methylene blue in the presence of mixed oxides.

5. RESULTS AND DISCUSSION

5.1. Characterization of the ZnZrO_3 Compound via Differential Thermal Analysis (DTA)

Differential Thermal Analysis (DTA) is a crucial technique used to investigate the thermal behavior of materials when subjected to a specific temperature range. This technique is based on measuring the temperature difference between the sample and a thermally inert reference material. Consequently, any thermal evolution (endothermic or exothermic) is exclusively attributed to the studied system.^[17]

The following figure illustrates the DTA curve of the studied (ZnZrO_3) compound, scanned within a temperature range of 0–800 °C. The curve exhibits a first endothermic peak at 170.4 °C, which is attributed to moisture loss and the decomposition of the organic stabilizer. The second peak, an exothermic one observed at 352 °C, corresponds to the formation of zinc oxide (ZnO). Another exothermic peak at 519.2 °C is related to the formation of zirconium dioxide (ZrO_2). Finally, the peak at 745.6 °C is assigned to the formation of the investigated compound.

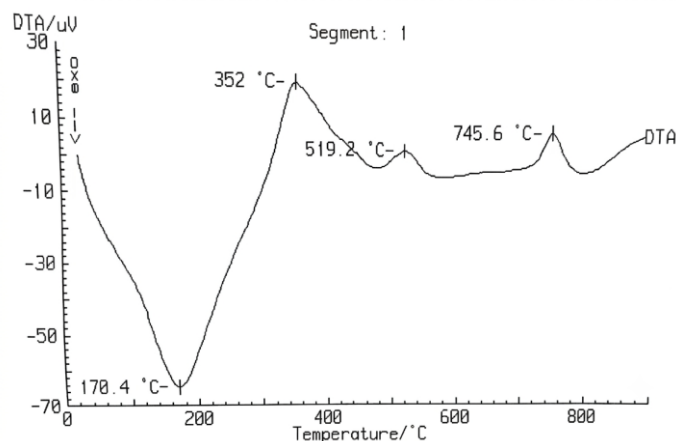


Figure 6: DTA curve of the zinc zirconate system.

Table 2. Interpretation of the DTA absorption peaks for the uncalcined zinc zirconate system.

Table 2: DTA thermal effects and their corresponding assignments.

Explanation	Shape	Peak
Moisture loss and decomposition of the organic stabilizer	Endo	170.4°C
ZnO formation	Exo	352°C
ZrO_2 formation	Exo	519.2°C
Formation of zinc zirconate	Exo	745.6°C

5.2. Characterization of the (ZnZrO_3) Compound via X-Ray Diffraction (XRD)

X-ray diffraction (XRD) is one of the most vital analytical techniques utilized in materials science, chemistry, and applied physics. It is widely employed to investigate the crystalline structure and structural

properties of solid materials. This technique is based on the interaction of X-rays with the regular atomic planes within the crystal lattice, leading to diffraction phenomena that provide precise information regarding atomic arrangement, crystalline phases, and lattice parameters of the material.^[18]

5.2.1. XRD pattern of the sample prepared at (400 °C)

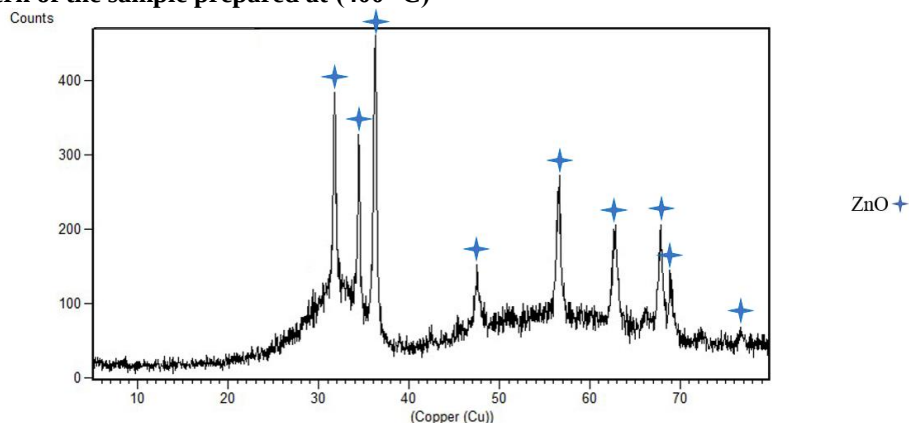


Figure 7: XRD pattern of the sample treated at 400 °C.

The following table presents the diffraction angles, their corresponding intensities, and the crystalline phases to which they belong. Based on both the XRD pattern and the accompanied table, it is observed that all diffraction

peaks are assigned to zinc oxide (ZnO), which is in excellent agreement with the reference card number (ZnO: 96-230-0113).

Table 3: Diffraction angles, Miller indices, and their corresponding crystalline phases for the samples prepared at 400 °C.

Compound	Pos. [°2 θ]	Height	d-spacing [Å]	Rel. Int. [%]	hkl
ZnO	31.75	385	2.8164	83.04	100
ZnO	34.41	327	2.6045	70.7	002
ZnO	36.23	463	2.4776	100	101
ZnO	47.51	152	1.9123	32.9	102
ZnO	56.54	279	1.6264	60.18	110
ZnO	62.73	213	1.4800	46.07	103
ZnO	67.81	216	1.3809	46.58	112
ZnO	68.93	143	1.3611	30.96	201
ZnO	76.81	78	1.2400	16.92	202

5.2.2. XRD pattern of the sample prepared at (550 °C)

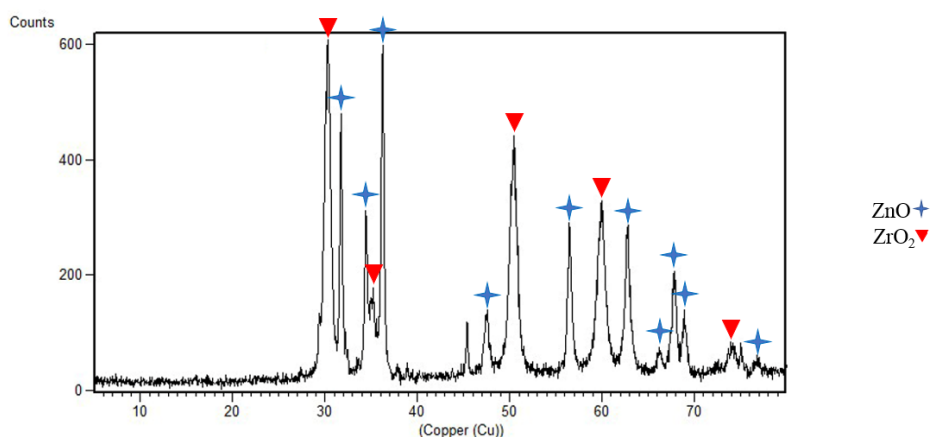


Figure 8: XRD pattern of the sample treated at 550 °C.

The following table presents the diffraction angles, their corresponding intensities, and the crystalline phases to which they belong. Based on both the XRD pattern and the accompanied table, the presence of diffraction peaks

assigned to both zinc oxide (ZnO) and zirconium dioxide (ZrO₂) is observed, which is in excellent agreement with the reference card numbers (ZnO:96-230-0113) (ZrO₂:96-210-1235).

Table 4: Diffraction angles, Miller indices, and their corresponding crystalline phases for the samples prepared at 550 °C.

Compound	Pos. [°2 θ]	Height	d-spacing [Å]	Rel. Int. [%]	hkl
ZrO ₂	30.37	611	2.9411	100	111
ZnO	31.95	483	2.7990	79.05	100
ZnO	34.4	311	2.6050	50.9	002
ZrO ₂	35.17	173	2.5498	28.31	020
ZnO	36.26	609	2.4756	99.67	101
ZnO	47.51	134	1.9123	21.93	102
ZrO ₂	50.42	448	1.8086	73.32	022
ZnO	56.5	288	1.6275	47.14	110
ZrO ₂	60.05	327	1.5395	53.52	131
ZnO	62.72	284	1.4802	46.48	103
ZnO	66.32	68	1.4083	11.13	200
ZnO	67.83	204	1.3805	33.39	112
ZnO	69.01	134	1.3598	21.93	201

ZrO ₂	74.09	83	1.2787	13.58	040
ZnO	76.83	59	1.2397	9.66	202

5.2.3. XRD pattern of the sample prepared at (800 °C)

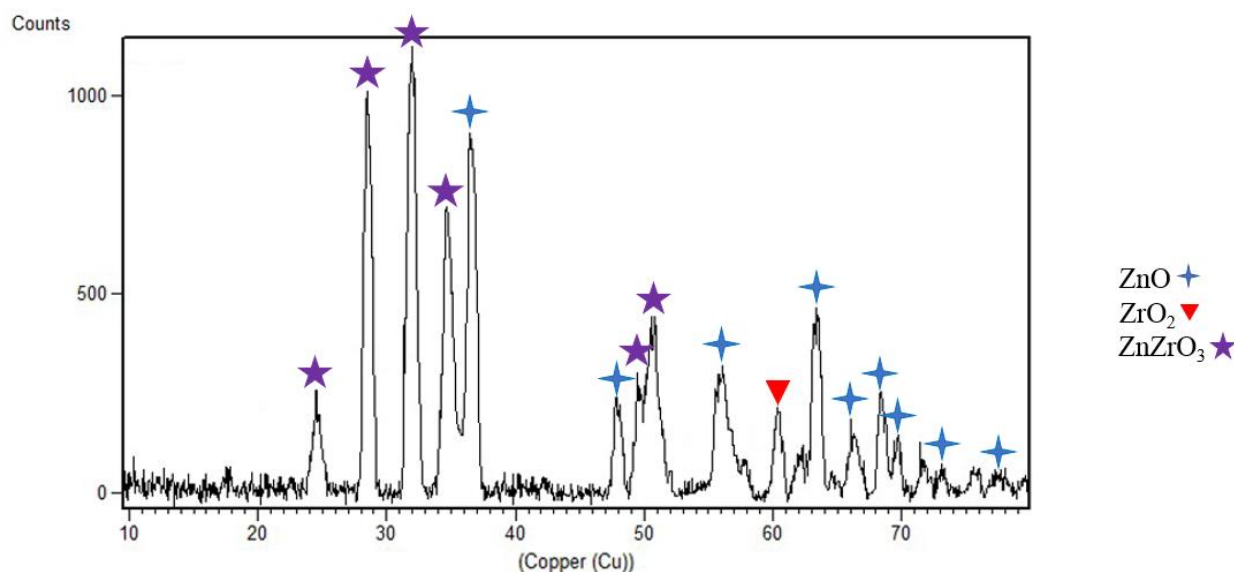


Figure 9: XRD pattern of the sample treated at 800 °C.

The following table presents the diffraction angles, revealed the formation of the ZnZrO₃ phase, as evidenced by the appearance of characteristic diffraction peaks corresponding to ZnZrO₃. The diffraction peaks of ZnO were still observed, indicating the presence of residual ZnO in the sample. In addition, a single diffraction peak attributable to ZrO₂ was detected at $2\theta =$

60.34°, indicating that a small amount of the zirconia phase remained after calcination at 800 °C., which is in excellent agreement with the reference card numbers.
(ZnO:96-230-0113)
(ZrO₂:96-210-1235)
(ZnZrO₃:00-032-1482)

Table 5: Diffraction angles, Miller indices, and their corresponding crystalline phases for the samples prepared at 800 °C.

Compound	Pos. [°2 θ]	Height	d-spacing [Å]	Rel. Int. [%]	hkl
ZnZrO ₃	24.61	256	3.6147	20.6	-
ZnZrO ₃	28.35	1027	3.1460	82.62	-
ZnZrO ₃	31.89	1243	2.8041	100	-
ZnZrO ₃	34.61	713	2.5896	57.36	-
ZnO	36.94	893	2.4315	71.84	101
ZnO	47.74	237	1.9036	19.07	102
ZnZrO ₃	49.52	294	1.8393	23.65	-
ZnZrO ₃	50.81	443	1.7955	35.64	-
ZnO	56.8	315	1.6196	25.34	110
ZrO ₂	60.34	214	1.5327	17.22	302
ZnO	63.62	486	1.4613	39.1	103
ZnO	66.45	208	1.4059	16.73	200
ZnO	68.27	251	1.3728	20.19	112
ZnO	69.83	187	1.3458	15.04	201
ZnO	73.04	84	1.2944	6.76	004
ZnO	77.53	73	1.2303	5.87	202

5.2.4. XRD pattern of the sample prepared at (1000 °C)

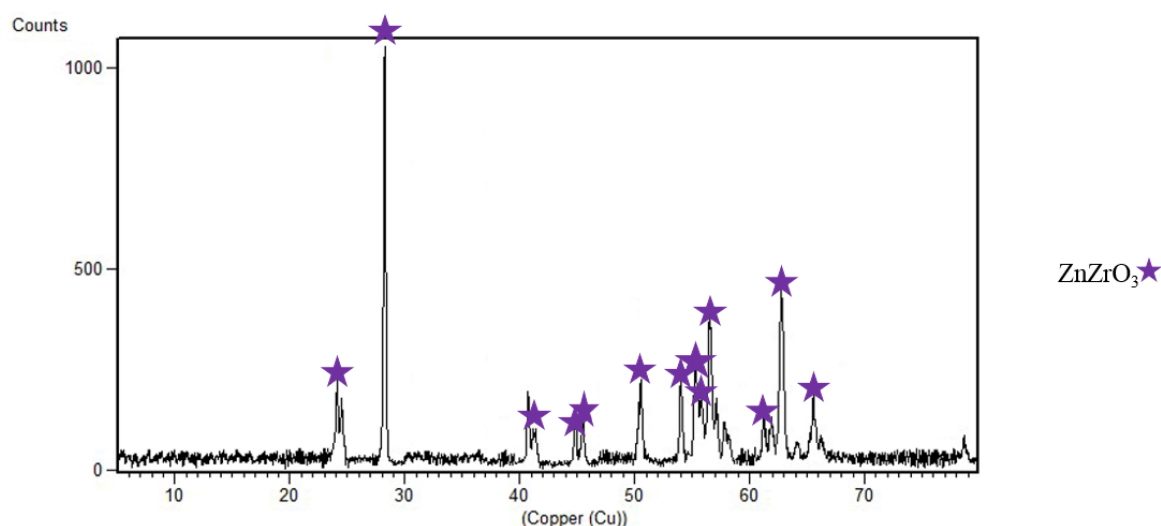


Figure 10: XRD pattern of the sample treated at 1000 °C.

The crystalline phases to which they belong. Based on both the XRD pattern and the accompanied table, the complete disappearance of the peaks corresponding to the starting oxides is observed, and all diffraction peaks are exclusively assigned to the target system (zinc zirconate). This is in excellent agreement with the

reference card number (ZnZrO₃: 00-032-1482). Consequently, it can be concluded that 1000 °C is the optimal temperature for synthesizing the desired compound.

Table 6: Diffraction angles and their corresponding crystalline phases for the samples prepared at 1000 °C.

Compound	Pos. [°2Th]	Height	d-spacing [Å]	Rel. Int. [%]	hkl
ZnZrO ₃	24.26	197	3.6663	18.12	-
ZnZrO ₃	28.21	1087	3.1615	100	-
ZnZrO ₃	41.31	176	2.1840	16.19	-
ZnZrO ₃	44.74	109	2.0241	10.03	-
ZnZrO ₃	45.53	114	1.9906	10.49	-
ZnZrO ₃	50.21	182	1.8156	16.74	-
ZnZrO ₃	54.02	203	1.6964	18.68	-
ZnZrO ₃	55.37	209	1.6579	19.23	-
ZnZrO ₃	56.54	416	1.6264	38.27	-
ZnZrO ₃	57.99	115	1.5891	10.58	-
ZnZrO ₃	61.21	127	1.5130	11.68	-
ZnZrO ₃	62.76	473	1.4793	43.51	-
ZnZrO ₃	65.58	171	1.4224	15.73	-

5.3. Structural Investigation of the Synthesized System

The crystal structure of the synthesized ZnZrO₃ was investigated using powder X-ray diffraction (XRD). Comparison of the experimental diffraction pattern with the reported reference pattern for ZnZrO₃ confirmed that the observed reflections are consistent with a tetragonal perovskite-type structure.^[19] The lattice parameters calculated from the XRD data were $a = b = 3.981$ Å and $c = 4.113$ Å, with a corresponding unit-cell volume of 65.1974 Å³. These results are in agreement with the reported structural characteristics of tetragonal ZnZrO₃ and support the formation of the tetragonal perovskite-type phase under the investigated preparation conditions.

5.4. Characterization of the (ZnZrO₃) Compound via Fourier-Transform Infrared Spectroscopy (FT-IR)

Fourier-transform infrared (FT-IR) spectroscopy represents one of the most vital analytical spectroscopic techniques utilized for investigating and determining the chemical composition of diverse materials. The fundamental principle of this technique relies on the absorption of infrared radiation by the chemical bonds within a compound, where the absorbed energy directly corresponds to the vibrational and rotational transitions of the constituent atoms. Consequently, this method yields highly precise insights into the specific chemical bonds and functional groups present within the synthesized structures. This capability establishes FT-IR as an indispensable tool across numerous scientific and

industrial sectors, where it is routinely deployed to monitor structural and chemical alterations, as well as to elucidate the fundamental molecular characteristics of materials.^[20]

5.4.1. FT-IR Spectroscopic Analysis of the System Synthesised at 1000°C

The infrared (IR) spectrum of the zinc zirconate compound was investigated to verify the structure of the

synthesised product and to validate the findings previously obtained from the X-ray diffraction (XRD) patterns.

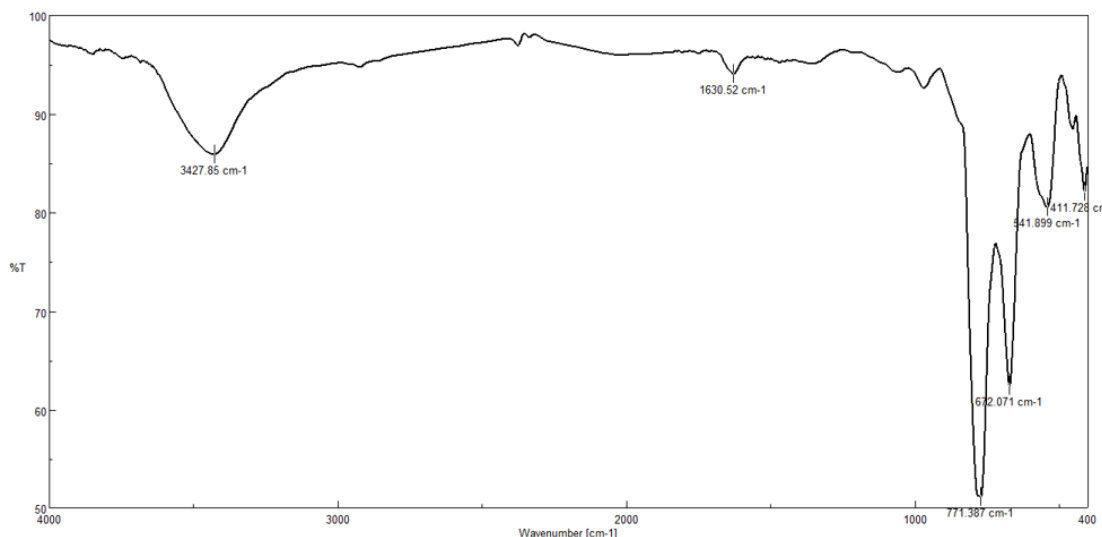


Figure 11: Illustrates the FT-IR spectrum of the zinc zirconate system prepared at 1000 °C.

Table 7: Absorption bands and their corresponding wavenumbers in the IR spectrum of the sample prepared at 1000 °C.

Wavenumber cm ⁻¹	Vibrational mode	Absorption band
3427.85	Bond stretching O-H	1
1630.52	Bond bending H-O-H	2
771.387	Bond stretching Zr-O-Zr	3
672.071	Bond stretching O-Zr-O	4
541.899	Bond stretching Zn-O-Zn	5
411.728	Bond stretching Zr-O-Zn	6

5.5. catalytic activity

The catalytic activity of ZnZrO₃ toward the oxidative degradation of methylene blue (MB) was evaluated in the presence of H₂O₂ and compared with that of NiZrO₃ under the same experimental conditions. The MB/H₂O₂ system without a catalyst was used as the reference control to evaluate the contribution of the mixed-metal oxide catalysts to the observed decrease in MB absorbance. As shown in Table 8, the absorbance of the MB/H₂O₂ solution was 1.898, whereas the addition of NiZrO₃ and ZnZrO₃ decreased the absorbance to 1.355 and 0.790, respectively. Based on the calculated catalytic efficiency, the MB/H₂O₂ system in the absence of a catalyst showed no catalytic efficiency, while NiZrO₃ and ZnZrO₃ exhibited efficiencies of 28.57% and 58.33%, respectively. These results indicate that both mixed-metal oxides enhanced the removal of MB in the

presence of H₂O₂, with ZnZrO₃ showing a higher catalytic efficiency than NiZrO₃ under the investigated experimental conditions. The higher activity observed for ZnZrO₃ may be related to differences in the physicochemical and structural characteristics of the two mixed-metal oxides.

The catalytic efficiency (CA) of the (ZnZrO₃) and (NiZrO₃) compounds was evaluated using the following mathematical relationship:

$$CA = \frac{A_{H_2O_2} - A_{Oxide}}{A_{H_2O_2}} \times 100 \%$$

where ($A_{H_2O_2}$) represents the absorbance of the methylene blue and hydrogen peroxide mixture (MB + H₂O₂) in the absence of a catalyst, and (A_{Oxide})

denotes the absorbance of the target system (Oxide+MB+H₂O₂) after the catalytic reaction. This calculation is predicated on the assumption that the

baseline reaction between methylene blue and hydrogen peroxide without any catalyst serves as the control reference.

Table 8: Catalytic efficiency of the methylene blue oxidation reaction in the presence of mixed oxides.

Solution No	1	2	3
Added Materials	MB +H ₂ O ₂	MB +H ₂ O ₂ +NiZrO ₃	MB +H ₂ O ₂ +ZnZrO ₃
Absorbance	1.898	1.355	0.790
Catalytic Efficiency, CA (%)	0	28.57	58.33

6. CONCLUSION

ZnZrO₃ mixed-metal oxide was successfully synthesized by the sol-gel method under the optimized preparation conditions previously reported by the authors. The thermal and structural evolution of the synthesized material was investigated using DTA, XRD, and FT-IR analyses. The XRD results indicated the formation of the ZnZrO₃ phase at elevated calcination temperatures, with the crystalline ZnZrO₃ phase obtained at 1000 °C. The experimental diffraction pattern was consistent with a tetragonal perovskite-type ZnZrO₃ structure based on comparison with the reported reference pattern and the calculated lattice parameters. The catalytic activity of ZnZrO₃ toward methylene blue (MB) oxidation in the presence of H₂O₂ was evaluated and compared with that of NiZrO₃ previously prepared by the sol-gel method. Under the experimental conditions employed, ZnZrO₃ showed a higher catalytic efficiency (58.33%) than NiZrO₃ (28.57%). These results indicate that ZnZrO₃ exhibits promising catalytic activity for the oxidative treatment of methylene blue in a heterogeneous H₂O₂-based system. Overall, the findings demonstrate that ZnZrO₃ is a potentially useful mixed-metal oxide catalyst for the degradation and decolorization of methylene blue under the investigated experimental conditions. However, further studies involving additional control experiments, repeated catalytic measurements, surface characterization, and analysis of degradation products would be required to clarify the contribution of adsorption and oxidative processes, evaluate catalyst stability and reusability, and provide a more comprehensive assessment of the catalytic performance of ZnZrO₃.

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