

Synthesis Of $Mn_{1.3}FeTi_2O_w$ Nanocomposite By Urea Catalyzed Thermal Combustion

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Abstract

This study reports the synthesis and characterization of the multimetallic nanocomposite $Mn_xFe_yTi_z$ via urea-assisted solution combustion synthesis. Stoichiometric amounts of $FeCl_3 \cdot 6H_2O$, $MnCl_2 \cdot 4H_2O$ and TiO_2 were used as precursors, with urea acting as both fuel and complexing agent. The precursor mixture was thermally ignited at 300–400 °C in air and subsequently calcined at 800 °C and 1000 °C. Structural and morphological characterizations were performed using X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), energy-dispersive X-ray spectroscopy (EDS), and X-ray fluorescence (XRF). The results revealed the formation of spinel-type ($MnFe_2O_4$) and perovskite-type ($MnTiO_3$) crystalline phases, especially after calcination at 1000 °C. SEM and TEM analyses showed granular nanostructures with homogeneous elemental distribution and intergranular porosity, while EDS confirmed the presence of Mn, Fe, Ti, and O with high purity and no detectable contaminants. This synthetic route proved to be efficient, cost-effective, and environmentally friendly, offering a promising strategy for producing nanocomposites with tunable physical and chemical properties. The results demonstrate the significant influence of metal molar ratios and thermal treatment on the final structure, supporting the potential application of $Mn_{1.3}FeTi_2O_w$ materials in sensors, catalysis, and magnetic or electronic devices.

Keywords: Multimetallic nanocomposite, Urea-assisted combustion synthesis, Spinel and perovskite, Structural and morphological characterization, Applications in magnetic and electronic devices.

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I. Introduction

Multicomponent metal oxides have gained prominence in the research of new materials due to the wide range of structural, electronic, magnetic, and catalytic properties they can exhibit, making them promising candidates for various technological applications, including heterogeneous catalysis, energy storage systems, sensors, optoelectronic devices, and biomaterials [1–4]. Among these, materials containing manganese (Mn), iron (Fe), and titanium (Ti) exhibit tunable structural and electronic properties, which can be modulated through precise control of composition and synthesis methodology [5,6]. The incorporation of these elements into a single ternary system, such as the compound $Mn_xFe_yTi_z$, may result in crystalline structures with unique characteristics, as well as potentially synergistic magnetic, catalytic, and electrical properties [7,8].

Among the synthesis methods applicable to the production of these compounds, urea-assisted combustion synthesis [10] stands out as a versatile technique that enables the formation of homogeneous nanocomposites through a highly exothermic reaction [2,8]. In this process, urea simultaneously acts as a fuel, reducing agent, and complexing agent, promoting the homogenization of the precursor mixture and facilitating the formation of desired crystalline phases at moderate temperatures [11]. The simplicity of the method,

combined with the low cost of reagents and the potential for scalability, makes this approach particularly attractive, especially in research settings with budgetary or infrastructural constraints [2].

Among the available synthesis methods, thermal combustion stands out as an efficient, low-cost, and fast technique capable of producing crystalline oxides with high chemical homogeneity and controlled morphology [9]. This method is based on an exothermic reaction between metal precursors and an organic fuel, which, when ignited, releases a large amount of thermal energy, promoting the formation of the target material. This approach has shown promise for the production of complex oxides; however, studies focused on obtaining the Mn_xFe_yTi_z compound through this synthetic route remain scarce. The specialized literature still presents a significant gap regarding the synthesis and systematic characterization of the Mn_xFe_yTi_z system obtained via thermal combustion, particularly with respect to the influence of the molar ratio between the metal cations on phase formation, crystallinity, and the final material's morphology [12]. Understanding these aspects is essential for optimizing the functional properties of the resulting materials and for exploring their potential applications in advanced devices [13].

Thus, the present research proposes the synthesis of the Mn_xFe_yTi_z compound via urea-assisted thermal combustion, with an emphasis on the structural and morphological characterization of the resulting materials, as well as the analysis of compositional variation effects on the phases formed.

In this context, the central question of this study is: does the thermal combustion technique enable the effective synthesis of the Mn_xFe_yTi_z compound with structural and morphological characteristics suitable for functional applications?

The relevance of this research lies in the potential to develop new materials through an accessible and environmentally friendly synthetic route, contributing to advances in the production of functional oxides with compositional and structural control. Furthermore, investigating the influence of molar composition and synthesis parameters on the material's properties may provide valuable insights for process optimization and the targeting of specific applications.

II. General Objective

To investigate the synthesis of the ternary compound Mn_xFe_yTi_z using the urea-assisted combustion method, evaluating the influence of the molar ratio between the metallic ions on the crystalline structure and morphology of the obtained materials.

Specific Objectives

- To synthesize samples of the compound Mn_xFe_yTi_z with varying molar ratios between Mn, Fe, and Ti, as well as different fuel-to-oxidizer proportions;
- To evaluate the efficiency of the urea-assisted solution combustion synthesis in forming the final product;
- To characterize the obtained materials using techniques such as X-ray diffraction (XRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDS);
- To identify and analyze the presence of secondary phases or impurities resulting from compositional variations;
- To correlate the synthesis conditions with the structural and morphological properties of the compound, aiming at future applications in functional materials.

III. Theoretical Framework

Advances in materials science and engineering have stimulated the research and development of multimetallic compounds with adjustable physicochemical properties, especially those based on transition metals such as manganese (Mn), iron (Fe) and titanium (Ti). These elements, alone or in combination, have wide applicability in fields such as catalysis, electrochemical devices, sensors and magnetic materials, due to their varied valences, dense electronic structure and ability to form complex oxides [14,15].

Multimetallic Materials Based on Mn, Fe and Ti

Mn is widely recognized for its redox capacity and for participating in structures with remarkable catalytic and magnetic behavior. Fe, in turn, is one of the most explored elements in materials for energy storage and environmental catalysis, while Ti stands out for its chemical stability, photocatalytic activity and biocompatibility [16,17]. The combination of these three metals in ternary systems allows the formation of compounds with perovskite, spinel or pyrochlore structures, whose final configuration depends strongly on the molar ratio between the elements and the synthesis conditions.

The literature describes the production of binary and ternary oxides by different routes, such as coprecipitation, sol-gel, hydrothermal and combustion. However, there is a relevant gap regarding the controlled synthesis of ternary Mn_xFe_yTi_z systems and the detailed understanding of the phase formation mechanisms and

the correlations between structure and functional properties [10].

Urea Catalyzed Thermal Combustion Synthesis

The thermal combustion synthesis technique is a consolidated route for the production of ceramic and metallic materials with porous structure, high surface area and homogeneous distribution of elements. It is based on a redox reaction between an oxidant (usually metal nitrates) and an organic fuel. Urea (CO(NH₂)₂) has been widely used in this approach due to its dual function: it acts as a fuel, promoting the release of thermal energy during combustion, and as a complexing agent, contributing to the homogenization of the precursor solution [18].

During combustion, urea decomposes, releasing gases such as CO₂, NH₃ and N₂, which favor the formation of highly porous structures and prevent the agglomeration of metal particles. The energy generated is sufficient to induce the formation of crystalline oxides in a few minutes, which makes the process fast, low cost and highly efficient [19]. Additionally, the stoichiometry of the reactant mixture (fuel/oxidant ratio) is a determining factor in the morphology, crystallinity and purity of the phases obtained [20].

Influence of Composition and Synthesis Conditions

Previous studies indicate that variations in the molar ratio between metals in the precursor solution directly influence the crystal structure and the formation of secondary phases. In similar systems, such as NiFe₂O₄ or MnTiO₃, it was observed that small changes in the molar ratios result in significant changes in the crystal symmetry and in the magnetic or catalytic properties [14,18]. However, there is a lack of systematic studies involving the Mn_xFe_yTi_z system that explore, in detail, the relationship between composition, structure and properties.

Furthermore, different experimental conditions, such as reaction time, heating rate, synthesis atmosphere, and urea concentration, also affect the combustion reaction efficiency and the quality of the final product [10]. Strict control of these parameters is essential to avoid the formation of unwanted impurities or amorphous phases.

Gaps and Potential Contributions

Although thermal combustion synthesis is a well-established technique, the systematic application of this method to obtain ternary compounds involving Mn, Fe and Ti is still incipient. There is a lack of studies that quantitatively correlate the initial elemental composition with the final products formed, as well as their morphology, degree of crystallinity and phase composition. Furthermore, little is known about the influence of synergistic interactions between Mn, Fe and Ti on the final structure of the materials.

In this context, this study seeks to contribute to filling these gaps by proposing a systematic investigation of the synthesis of the compound Mn_xFe_yTi_z via urea-catalyzed thermal combustion, focusing on the structural and morphological analysis of the materials produced and the correlations between composition and properties.

Comparative Table between Synthesis Methods

The choice of synthesis methodology directly affects the structure, morphology, particle size and final properties of the material. Table 1 presents a comparison between different methods applied to the synthesis of multimetallic oxides, focusing on feasibility, product purity and morphological control.

Table 1: Comparison between synthesis methods for multimetallic oxides

Method	Advantages	Disadvantages	References
Co-precipitation	Simplicity; low cost	Possible formation of amorphous phases or impurities	Liu et al. (2017), [14]
Sol-gel	Excellent control of morphology and stoichiometry	High reaction time; use of organic solvents	Dong et al. (2018), [16]
Hydrothermal	High crystallinity; formation of controlled morphologies	Requires autoclaves; long synthesis time	Singh e Mohan (2016), [20]
Thermal combustion (urea)	Fast; high purity; good metal dispersion	Difficult to control particle size; formation of gases	Rajeshwari et al. (2014); Varma & Vaghasiya (2020), [18,10]

Simplified Theoretical Model of Combustion with Urea

The thermal combustion reaction is based on a redox reaction between metal chlorides (oxidants) and urea (fuel). The stoichiometry of the reaction and the nature of the metal ions directly influence the heat generated, the combustion rate and the formation of the crystalline phases. The overall schematic equation (1) is given by:



In this case the hypothesis is that:

- Urea acts as a fuel and complexing agent.
- Metal chlorides (MnCl₂, FeCl₂) provide the reactive metal ions.
- TiO₂ can be added as a precursor or as a stable oxide.
- Combustion generates gases such as N₂, CO₂, H₂O, HCl, NH₂ and isocyanate (HNCO) in the ambient atmosphere.

IV. Metodology

Materials, Reagents and Equipment

The following reagents were used as precursors: FeCl₃·6H₂O (99%, Sigma-Aldrich PA), MnCl₂·4H₂O (99%, Synth PA), TiO₂ (98%, Sigma-Aldrich PA) and the urea catalyst (CH₄N₂O) (98%, Synth PA). The reagents were used without further purification. A muffle furnace was used in this work. JUNG-2895, an energy-dispersive fluorescence x-ray of the TXRF S4 T-STAR (Brunker Nano Analytics) a D2 PHASER Brunker diffractometer and a Scanning Electron Microscope (SEM), model Jeol JSM – 6610, with EDS equipment.

Synthesis steps

Preparation of the reaction mixture: The precursor reagents were prepared in different mass proportions of Mn: Fe: Ti: Urea (1:1:1:3), homogenized in a pounder.

Thermal combustion: The solid mixture was stored in a muffle furnace in an ambient atmosphere of 300 to 400 °C until autoignition, characterized by the release of gases and the formation of the spongy solid product.

Heat treatment: The obtained nanocomposite was calcined in the ambient atmosphere at temperatures of 400, 800 and 1000 °C for 1 hour to promote greater crystallinity of the nanocomposite (see Table 2).

Table 2: Thermal synthesis steps and expected chemical transformations

Step	Conditions	Expected Transformations
Initial mixture	Manual maceration (pounder)	Homogenization of salts with TiO ₂ and urea (CH ₄ N ₂ O)
Heat treatment 1	300 to 400°C, 1 h, air	Decomposition of urea; release of NH ₃ , CO ₂ ; dehydration of salts; partial oxidation
Calcination 1	800°C, 1 h, air	Initial oxide formation (Fe ₂ O ₃ , Mn ₃ O ₄), doping in TiO ₂ , beginning of the formation of MnFe ₂ O ₄
Calcination 2	1000°C, 1 h, air	Consolidated training of MnFe ₂ O ₄ ; grain growth; TiO ₂ converted to rutile

Characterizations

The nanocomposite represented by a sample with a mass of 5 grams was characterized using the methods and techniques below:

- Scanning Electron Microscopy (SEM) with EDS and Transmission Electron Microscopy (TEM): For analysis of morphology and elemental composition.
- Fluorescence X-rays.
- X-ray Diffraction (XRD): For identification of crystalline phases.

V. Results And Discussions

Here we present the synthesis was carried out using the urea-assisted combustion methodology, with the aim of obtaining metal oxides and possible spinel phases containing iron, manganese and titanium 1 g of ferric chloride hexahydrate were used as precursors (FeCl₃·6H₂O), 1g manganese chloride tetrahydrate (MnCl₂·4H₂O), 1 g of titanium dioxide (TiO₂, analytical degree) and 3 g of urea (CH₄N₂O), homogenized by maceration in a porcelain mortar until a fine, uniform powder is obtained.

The mixture was initially subjected to heat treatment in a preheated muffle furnace at 300°C to 400°C for 1 hour in an open atmosphere (atmospheric air). This step promoted the dehydration of the hydrated salts, the thermal decomposition of the urea and the beginning of the oxidation reactions of the metallic salts. The release of gases such as H₂O, NH₃, CO₂ and traces of Cl₂ contributed to the formation of a porous solid matrix, composed of partially oxidized precursors dispersed over the TiO₂.

Subsequently, the mixture was calcined at 800 °C e 1000 °C for 1 hour in order to promote the crystallization of the desired phases (see Table 3). The increase in temperature intensified the ionic diffusion processes and allowed the formation of more ordered structures. The following physical-chemical transformations are expected:

- Total conversion of TiO₂ for the rutile phase, especially the 1000 °C.
- Formation of spinel phases of the type MnFe₂O₄, due to the reaction between Mn²⁺ e Fe³⁺ in the presence of oxygen, characteristic of high calcination temperatures $Mn^{2+} + 2Fe^{3+} + 4O^{2-} \rightarrow MnFe_2O_4$.

- Presence of individual oxides, such as Fe₂O₃ (hematite) and Mn₂O₃ or Mn₃O₄, in the stoichiometric ratio and in the presence of the atmosphere and calcination.
- Doping of the TiO₂ by ions Fe³⁺ e Mn²⁺, promoting possible changes in the crystal structure and electronic properties, especially if the ions are incorporated into substitutional sites of the network.

Visually, the transformation of the solid into a dark ceramic powder, denser and with sintered particles, was observed, indicating high crystallization. The color change and the loss of initial mass corroborate the decomposition of urea and precursor salts during the process. Here we present a possible stoichiometric equation as the measures of reactant and product masses in the following structure (equation 2): $1.3\text{MnCl}_2 + 1\text{FeCl}_3 + 2\text{TiO}_2 + 8\text{CO}(\text{NH}_2)_2 + \text{O}_2(\text{g}) \rightarrow \text{Mn}_{1.3}\text{FeTi}_2\text{O}_w + (\text{HCl}, \text{CO}_2, \text{H}_2\text{O}, \text{N}_2, \text{NH}_3)$ (2)

The calculated redox factor was $\Phi=0.97$, where $\Phi \approx 1$ indicates a slightly oxidizing stoichiometric combustion, ideal for synthesis via urea-assisted thermal combustion. This means that its 1:1:1:3 mass ratio is adequate, and combustion occurs efficiently, with good energy release and without excessive residues.

Table 3: Nanocomposite phases Mn_{1.3}FeTi₂O_w after calcination

Sample	Expected Main Phases	Relative Intensity	Crystallinity	Observations
Post-heat treatment	TiO ₂ (anatase/rutile), amorphous oxides of Fe/Mn	Low	Partial/low	Porous and dark product, low structural order
Calcination at 800°C	Fe ₂ O ₃ , Mn ₃ O ₄ , TiO ₂ (rutile), beginning of MnFe ₃ O ₄	Average	Moderate	Increasing crystallinity, presence of multiple phases
Calcination at 1000°C	MnFe ₂ O ₄ (spinel), Fe ₂ O ₃ , TiO ₂ (rutile)	High	High	Clear spinel formation and total oxidation of precursors

Figure 1 is a micrograph of the nanocomposite sample Mn_{1.3}FeTi₂O_w obtained by scanning electron microscopy (SEM) synthesized at a temperature of 800°C, were recorded with a voltage of 10 kV at resolutions of 5µm and 1µm respectively.

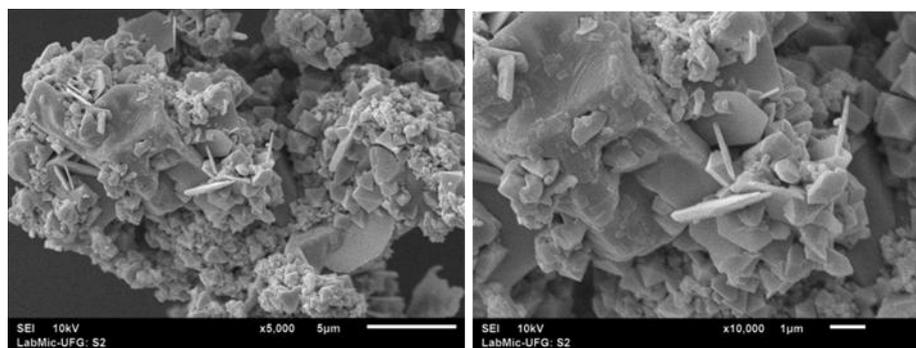


Figure-1: Micrograph obtained by scanning microscope of the nanocomposite Mn_{1.3}FeTi₂O_w obtained at a temperature of 800 °C, at 10 kV at resolutions of 5 µm and 1 µm respectively.

In the image obtained with a resolution of 5 µm (Figure 1), an irregular and heterogeneous surface morphology is observed, characterized by well-defined and agglomerated particles. This structure is indicative of accentuated granular growth, with evidence of coalescence, typical of materials obtained by high temperature synthesis routes [21,22]. The grain size distribution is broad, suggesting a nonuniform nucleation process during calcination. In addition, the presence of intergranular porosity can be noted, which directly impacts the transport properties of the material, such as electrical conductivity and ionic diffusion [23].

In the image with greater magnification (1 µm resolution), it is possible to identify more precise details of the grain surface. Well defined contours and the presence of surface roughness can be noted, which may be associated with the formation of multiple crystalline phases or heterogeneity in the elemental distribution [24]. Some particles present visible facets, which can be interpreted as evidence of oriented growth, associated with crystalline anisotropy [25]. A degree of interconnectivity between the grains is also observed, which favors the formation of continuous paths for the transport of electrical charge, an essential factor in electroceramic applications [26].

The morphological results presented here are consistent with previous studies on mixed oxide nanocomposites containing manganese, iron and titanium, in which the surface structure is directly related to the synthesis method and calcination temperature [27–29].

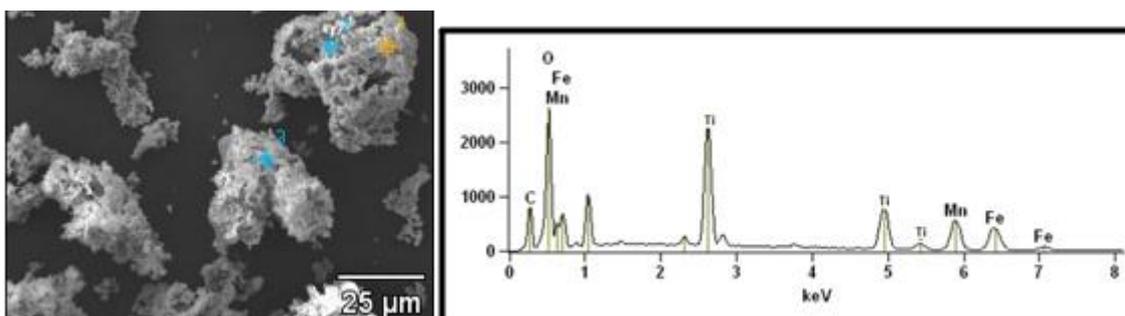


Figure-2: Scanning electron microscopy (SEM) image of the sample Mn_{1.3}FeTi₂O_w, accompanied by the corresponding energy dispersive X-ray (EDS) spectrum. The SEM image, with a scale of 25 μm, highlights the analysis areas marked in blue and orange, representing selected points of interest. The EDS spectrum illustrates the intensity peaks of the detected chemical elements, including oxygen (O), iron (Fe), manganese (Mn) and titanium (Ti).

Figure 2 shows the scanning electron microscopy (SEM) image of the nanocomposite Mn_{1.3}FeTi₂O_w calcined to 800 °C, together with the energy dispersive X-ray spectrum (EDS) obtained in the highlighted regions. The image, with a scale of 25 μm, reveals a heterogeneous morphology composed of clusters of irregular particles, typical characteristics of materials subjected to heat treatment at high temperatures [30,31]. The presence of grains with different contrasts suggests possible local variations in the electron density and, consequently, in the elemental composition or in the surface topography.

The regions marked in blue and orange indicate points of interest where specific analyses were performed by EDS. The spectrum obtained shows the presence of the elements oxygen (O), iron (Fe), manganese (Mn) and titanium (Ti), compatible with the theoretical composition of spinel. Mn_{1.3}FeTi₂O_w [32]. The detection of these elements at both analyzed points confirms the successful incorporation of the metallic precursors into the nanocomposite structure.

Additionally, no peaks attributable to undesirable elements were observed, indicating the absence of significant contamination and suggesting high purity of the material. However, small variations in the intensity of the peaks between the two points suggest a slight heterogeneity in the distribution of the constituents, possibly related to the dynamics of the calcination process and the formation of microdomains with slight local enrichment of certain elements, such as Mn or Ti [33].

The morphological analysis associated with the chemical composition corroborates the effectiveness of the synthetic protocol used, showing that calcination at 800 °C favors the formation of coalesced particles with efficient incorporation of metal ions. To complement this characterization, additional analyses such as X-ray diffraction (XRD) and EDS mapping in scanning mode can be used to confirm the crystalline phases and evaluate the elemental homogeneity on a larger scale [34].

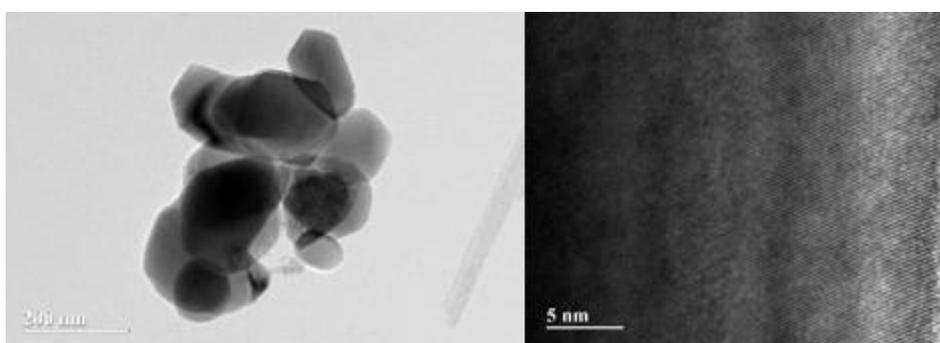


Figure-3: Micrograph obtained by transmission microscopy (TEM) of the nanocomposite Mn_{1.3}FeTi₂O_w obtained at a temperature of 800 °C, at resolutions of 200 nm and 5 nm respectively.

Figure 3 shows the micrograph obtained by transmission electron microscopy (TEM) of the nanocomposite Mn_{1.3}FeTi₂O_w synthesized at 800°C. Images were acquired at two different resolutions: 200 nm and 5 nm, allowing a comprehensive morphological analysis of the material.

In the 200 nm resolution image, a homogeneous distribution of nanoparticles is observed, indicating a good dispersion of the nanocomposite constituents. This homogeneity is essential to ensure consistent physicochemical properties, such as magnetic response and electrical conductivity, especially in technological and biomedical applications [35].

In the image with a resolution of 5 nm, the presence of well-defined crystalline structures becomes evident, suggesting that the heat treatment at 800 °C was effective in promoting the crystallization of the metal oxides present. This structural organization on a nanometric scale is a positive indication for the functional performance of the material, for example, in applications such as catalysts, sensors or contrast agents for magnetic resonance imaging [36,37].

Additionally, the morphology analysis reveals the formation of granular domains with average dimensions of less than 50 nm, which is in line with other works in the literature that describe similar syntheses of nanocomposites containing Mn, Fe and Ti [38]. The presence of such domains can favor electronic transport mechanisms by quantum tunneling, which directly impacts the electrical response of the material [39].

The results observed by TEM are consistent with the literature regarding thermal stability and the formation of homogeneous phases in multielement compositions obtained by ceramic routes assisted by high-energy milling [40].

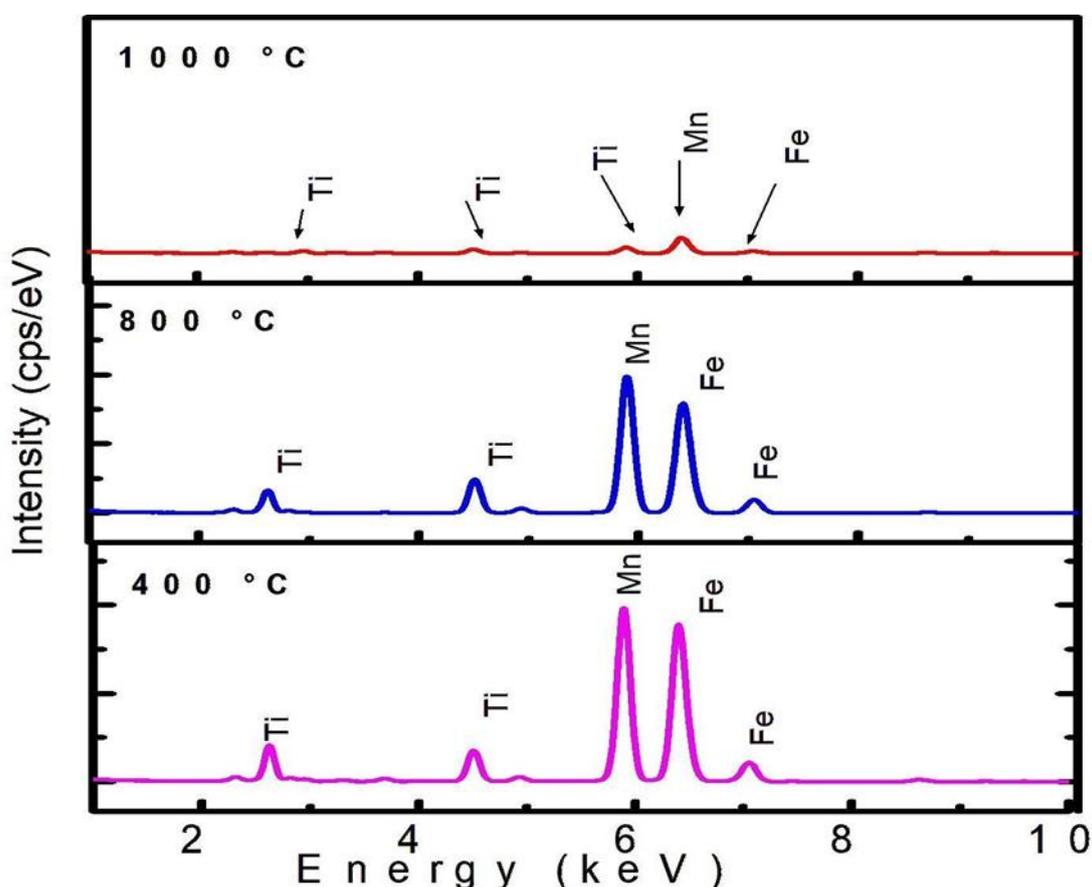


Figure-4: XRF spectrum of the nanocomposite Mn_{1.3}FeTi₂O_w obtained at temperatures of 400 °C (magenta spectrum), 800 °C (blue spectrum) and 1000 °C (red spectrum).

Figure 4 shows the X-ray fluorescence (XRF) spectra of the nanocomposite Mn_{1.3}FeTi₂O_w heat treated at 400 °C, 800 °C and 1000 °C. It is observed that all spectra exhibit characteristic peaks of the elements Mn, Fe, Ti and F, evidencing the presence of these elements in the final composition of the material.

In the sample calcined at 400 °C (lower spectrum), it is possible to identify well-defined peaks for Mn (~5.9 keV), Fe (~6.4 and 7.0 keV) and Ti (~4.5 keV), in addition to two peaks in the range of 0.6 to 1.0 keV attributed to carbon (C), possibly remaining from precursors such as metallic fluorides used in the synthesis as impurities. The intensity of the signals indicates good incorporation of the metallic elements in the nanocomposite matrix.

In the sample calcined at 400 °C (lower spectrum), it is possible to identify well-defined peaks for Mn (~5.9 keV), Fe (~6.4 and 7.0 keV) and Ti (~4.5 keV), in addition to two peaks in the range of 0.6 to 1.0 keV attributed to carbon (C), possibly remaining from catalyst precursors used in the synthesis as impurities. The intensity of the signals indicates good incorporation of the metallic elements in the nanocomposite matrix.

Finally, the spectrum corresponding to the heat treatment at 1000 °C (upper spectrum) shows a considerable reduction in the intensity of all detected elements. This behavior may be related to the possible

volatilization of carbon-containing phases and/or phase transitions that promote the segregation or encapsulation of metallic elements, making their detection by XRF difficult [42]. Still, it is possible to identify residual signals of Ti, Mn and Fe, although quite attenuated.

These results show that heat treatment plays a fundamental role in the compositional stability and analytical response of the nanocomposite, with 800 °C being the temperature that provides the greatest intensity of peaks without significant losses due to volatilization, which may indicate an ideal thermal condition for synthesis and subsequent application of the material.

Figure 5 shows the X-ray diffractograms of the nanocomposite Mn_{1.3}FeTi₂O_w calcined at 400 °C, 800 °C and 1000 °C. A significant evolution in crystallinity and in the phases formed is observed as the calcination temperature increases.

Samples treated at 400 °C exhibit peaks of low intensity and considerable width, indicating the presence of a predominantly amorphous structure or incipient crystallinity. This characteristic is common in nanocrystalline materials obtained at low calcination temperatures, as also reported in mixed oxides containing Mn, Fe and Ti [43].

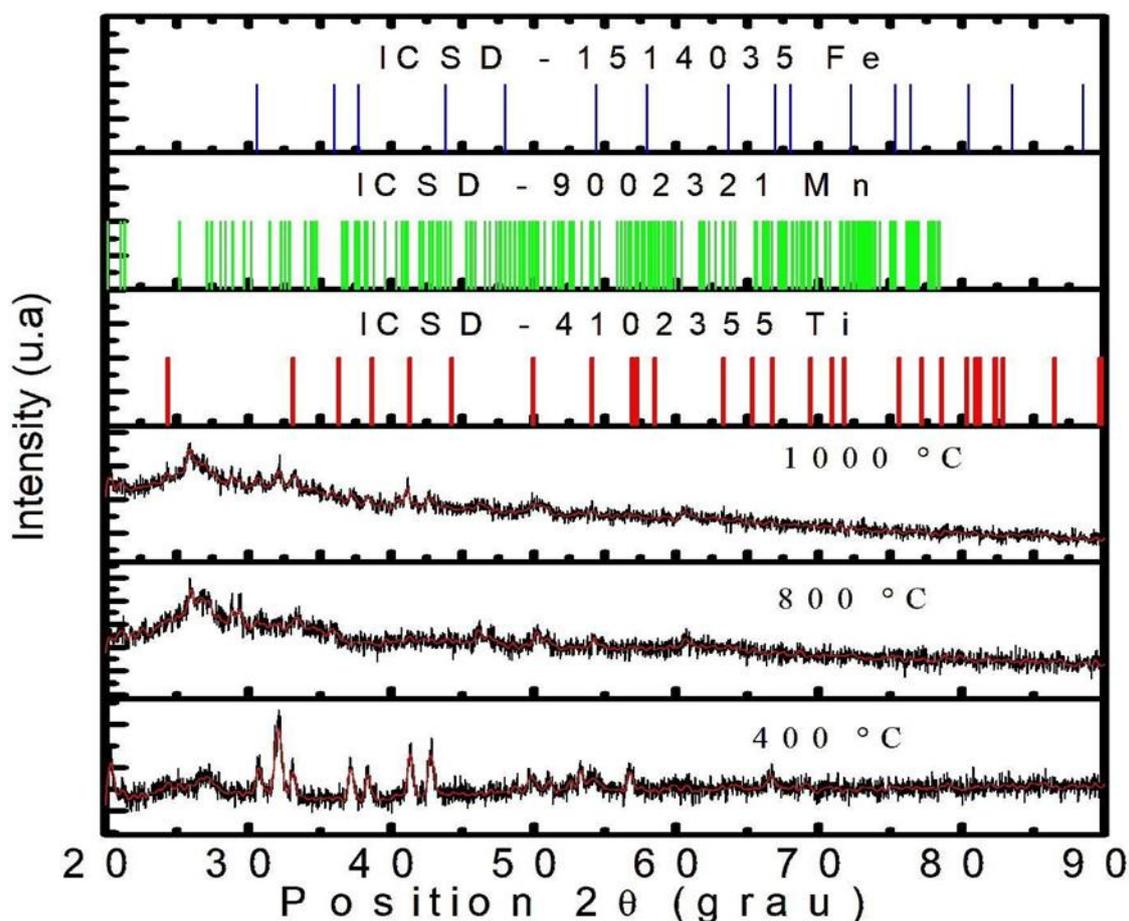


Figure 6 - We present the X-ray diffractogram (XRD) of the nanocomposite Mn_{1.3}FeTi₂O_w (black line) and Rietveld refinement (red line) heat-treated at temperatures of 400 °C, 800 °C and 1000 °C. The patterns with the crystalline phases of Fe (ICSD - 1514035), Mn (ICSD - 9002321) and Ti (ICSD - 4102355) are shown in the upper part of the graph, according to the ICSD database.

As the temperature increases to 800 °C, the diffractograms reveal more defined and intense peaks, indicating the development of crystalline phases. Comparison analysis with the PDF (Powder Diffraction File) patterns suggests the formation of phases such as pseudobrookite (Fe₂TiO₅) and perovskite (MnTiO₃), as noted by Costa, J. R. et al. [44] and corroborated by structural studies of Fe–Ti–Mn composites obtained by sol-gel and Pechini methods [45–49]. This structural transition is accompanied by an increase in crystallite size, indicating a greater degree of ordering.

In the sample calcined at 1000 °C, the peaks become even more intense and narrow, evidencing a high degree of crystallinity. Majority phases such as MnFe₂O₄ (spinel) and MnTiO₃ are clearly observed, compatible with crystallographic data from ICSD. The formation of the spinel phase in this temperature range is in agreement

with other studies indicating the thermal and structural stability of these mixed oxides at temperatures above 900 °C [50-52]. The presence of multiple phases may indicate complex interfacial reactions between the constituent oxides during the thermal process, resulting from solid diffusion between the Mn²⁺, Fe³⁺ and Ti⁴⁺ cations and the stability of the spinel and perovskite phases [53].

Therefore, the XRD results demonstrate that the calcination temperature plays a determining role in the structural evolution of the nanocomposite Mn_{1.3}FeTi₂O_w, directly affecting crystallinity, phase formation and, potentially, their functional properties.

VI. Conclusion

The urea assisted combustion synthesis method proved to be an effective and versatile approach for obtaining the Mn_{1.3}FeTi₂O_w nanocomposite. The process enabled the formation of well-defined crystalline phases, such as MnFe₂O₄ and MnTiO₃, with high crystallinity and nanoscale particle size. Structural and morphological analyses confirmed homogeneous incorporation of metal ions and successful control of grain size and distribution. The ability to tune the material's properties through compositional and thermal parameters highlights the suitability of this method for the development of functional materials for use in catalysis, sensors, and magnetic applications.

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