

Synthesis and Structural and Microstructural Characterization of Chromium(III) Benzoate Coordination Polymer

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Abstract A chromium (III) benzoate coordination polymer was synthesized and characterized to elucidate its structural and microstructural properties. The crystal structure, chemical functionalities, morphology, and elemental composition were investigated using powder X-ray diffraction (PXRD), Fourier-Transform infrared (FTIR) spectroscopy, scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDS), and X-ray fluorescence (XRF) spectroscopy. The PXRD pattern reveals a highly crystalline phase with numerous reflections, particularly in the low-angle region, indicating a well-organized crystal structure with relatively large interplanar spacings. The absence of a reference phase indicates that the obtained material may correspond to a crystalline Cr(III) benzoate phase for which no corresponding crystallographic structure has been identified in the database consulted. FTIR analysis confirms the deprotonation of the benzoic carboxylic groups and their involvement in coordination with the Cr(III) centers. SEM observations reveal predominantly prismatic crystal morphologies, with particle sizes ranging from 2.26 to 17.21 μm (mean size: 8.15 μm), while EDS and XRF analysis confirm the elemental composition of the material. The microstructural parameters were evaluated using the Scherrer, Monshi-Scherrer, Williamson-Hall (UDM), Size-Strain Plot (SSP), and Halder-Wagner methods. The apparent coherent domain sizes obtained ranged from 49.17 to 550.99 nm, depending on the approach employed, highlighting the influence of the underlying assumptions associated with each diffraction profile analysis method. The SSP and Halder-Wagner methods provided the best statistical fits ($R^2 = 0.823$) and yielded consistent estimates of the coherent domain size (49.17 nm), together with low microstrain values (2.05×10^{-3} and 3.33×10^{-3} , respectively). Overall, the structural and microstructural results confirm the formation of a crystalline Cr(III)-benzoate material and indicate that the SSP and Halder-Wagner approaches provide the most consistent estimates of the microstructural parameters for this system.

Keywords Chromium(III) benzoate coordination polymer, Benzoic acid, Powder X-ray diffraction, Diffraction peak broadening analysis, Microstructural parameters

1. Introduction

Coordination polymers are currently among the most extensively studied classes of crystalline materials in coordination chemistry and materials science, owing to their remarkable structural diversity and outstanding functional properties. The diversity of coordination geometries of metal centers, combined with the wide variety of available ligands, gives rise to a remarkable range of structural architectures, which can extend from one dimensional (1D) chains to two

dimensional (2D) layers and three dimensional (3D) frameworks. This modularity endows coordination polymers with a broad range of physicochemical properties, making them particularly attractive for numerous applications, including adsorption, catalysis, molecular separation, chemical sensing, gas storage, molecular magnetism, and materials science [1-6]. Among the transition metals used in the design of these materials, chromium(III) is of particular interest owing to its d^3 electronic configuration, high thermodynamic stability, and strong tendency to form octahedral complexes. Cr(III)-based coordination polymers are generally distinguished by their excellent chemical and thermal stability, as well as their electronic and magnetic properties, making them

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promising candidates for the development of functional materials [7,8]. Carboxylate ligands occupy a prominent position in this field of coordination chemistry because they exhibit multiple coordination modes, including monodentate, chelating, and bridging modes, thereby enabling the construction of extended networks diverse geometries [9,10]. In particular, the benzoate ion is a simple aromatic ligand capable of bridging multiple metal centers and promoting the formation of crystalline coordination networks [11,12]. The rational design of such materials relies not only on the determination of their crystal structure but also on a thorough understanding of their microstructural organization, as both aspects directly govern their physicochemical properties. Powder X-ray diffraction (PXRD) remains the reference technique for identifying crystalline phases and assessing long-range structural order. However, when the crystallographic structure is unavailable or when no suitable single crystal can be obtained, the interpretation of diffraction patterns should be complemented by spectroscopic and microscopic techniques to establish the nature of the synthesized material [4,13]. In this context, FTIR spectroscopy provides essential information on the coordination modes of the ligands, whereas SEM-EDS and XRF analyses enable the characterization of the solid in terms of its morphology and elemental composition, respectively. Beyond structural identification, diffraction peak broadening contains valuable information on the microstructure of crystalline materials. The sizes of coherently diffracting crystallite domains, microstrain, and crystal defect density directly influence numerous material properties, including stability, reactivity, and functional performance. Several approaches based on diffraction peak-broadening analysis have been developed to estimate these parameters, including the Scherrer, Monshi-Scherrer, Williamson-Hall, Size-Strain Plot, and Halder-Wagner methods. However, the reliable assessment of these microstructural parameters remains challenging because the different analytical methods are based on distinct physical assumptions and may therefore yield divergent results. Despite their widespread application to oxides, nanoparticles, and ceramic materials, comparative studies addressing their applicability to coordination polymers remain relatively scarce. To the best of our knowledge, a comprehensive literature search revealed no reported crystal structure describing a coordination polymer composed exclusively of Cr(III) centers interconnected by benzoate ligands. The absence of a crystallographic reference structure is therefore of particular significance for the characterization of the material obtained and supports the use of a multi-technique analytical approach combining several complementary methods [11]. Beyond the synthesis of the Cr(III)-benzoate coordination polymer, this study also aims to provide a critical assessment of the principal diffraction peak-broadening analysis methods in order to identify those that offer the most representative description of its crystalline microstructure.

2. Experimental

2.1. Hydrothermal Synthesis of the Cr(III)-Benzoate Coordination Polymer

Benzoic acid (1.50 g) was dissolved in 30 mL of distilled water. In parallel, chromium(III) nitrate nonahydrate (1.56 g) was dissolved in 30 mL of distilled water. The resulting aqueous solution of chromium(III) nitrate nonahydrate was then added to the benzoic acid solution under continuous stirring for 30 min at room temperature. The resulting suspension was transferred to a stainless-steel autoclave equipped with a polytetrafluoroethylene (PTFE) inner liner and heated at 120 °C in an oven for 4 days. After the hydrothermal treatment, the autoclave was allowed to cool naturally to room temperature. The resulting solid was recovered by filtration and washed with distilled water. The material was subsequently dried at 100 °C for 30 min. The resulting green powder was collected and stored in a glass vial.

2.2. Structural Characterization Method for the Synthesized Powder

The synthesized product was characterized using several complementary techniques. Powder X-ray diffraction (PXRD) measurements were performed using an X'Pert³ Analytical powder diffractometer with CuK α radiation, with wavelengths of $\lambda_{K\alpha1} = 1.54059$ Å and $\lambda_{K\alpha2} = 1.54441$ Å. The diffraction data were collected over the 2θ range of 5 - 50 °, with a step size of $\Delta 2\theta = 0.02$ °, at an accelerating voltage of 45 kV and a current of 100 mA. A systematic search for crystallographic structures of Cr(III) benzoate compounds was conducted in the CCDC and COD databases using the keywords 'chromium(III) benzoate', 'chromium benzoate', 'Cr(III) benzoate', 'chromium(III) benzoate complex', 'chromium(III) benzoate coordination compound', 'chromium(III) benzoate coordination polymer' and 'chromium(III) benzoate crystal structure', supplemented by searches based on the chemical formulas $\text{Cr}(\text{C}_6\text{H}_5\text{COO})_3$ et $\text{Cr}(\text{O}_2\text{CPh})_3$. Fourier-transform infrared spectroscopy was performed using a Thermo Scientific Nicolet iS50 FTIR spectrometer over wavenumber range of 500 - 4000 cm^{-1} . The morphology and elemental composition of the synthesized material were investigated by scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDS). The analyses were carried out using an SU8100 Regulus scanning electron microscope equipped with an energy-dispersive X-ray spectroscopy detector. SEM observations were performed at an accelerating voltage of 2 kV, using magnifications of $\times 5000$, $\times 2000$, $\times 1000$, and $\times 500$, with a working distance of 7.8 mm. Elemental analysis by EDS was conducted at a magnification of $\times 10000$, with an acquisition time of 7.68 μs , a real analysis time of 47.4 s, and an energy resolution of 123.8 eV. The elemental composition was further determined using a Thermo Fisher Scientific X-ray fluorescence (XRF) spectrometer. The coherent diffraction domain size and lattice microstrain were estimated using the

Scherrer, Monshi-Scherrer, Williamson-Hall, Halder-Wagner, and Size-Strain Plot methods. Only the reflections attributed to the coordination complex were considered for the determination of the coherent crystallite domain size and microstrain. The instrumental broadening of the XRD peaks was determined using a crystalline silicon standard, for which an instrumental FWHM, $\beta_{\text{instrumental}}$, of 0.071° was measured for the (1 1 1) reflection at $2\theta = 28.4^\circ$. The instrumental contribution was corrected using a Gaussian deconvolution procedure according to $\beta_{\text{corrected}}^2 = \beta_{\text{obs}}^2 - \beta_{\text{instrumental}}^2$, where β_{obs} is the observed FWHM. The profiles of the individual XRD reflections were fitted in OriginPro 2024 software using a Voigt function to determine the peak positions and FWHM values. Only well-resolved reflections without significant overlap with neighboring peak were considered for the microstructural analysis and subsequent fitting procedures.

2.3. Microstructural Characterization Methods of the Synthesized Powder

2.3.1. Scherrer et de Monshi-Scherrer Methods

The Scherrer method is used to estimate the average coherent crystallite domain size from XRD peak broadening. This approach assumes that peak broadening arises exclusively from the finite size of the coherent crystallite domains and does not account for the contribution of crystal lattice microstrain. The coherent crystallite domain size is determined using Equation (1) [14,15].

$$D = \frac{K \cdot \lambda}{\beta \cos \theta} \quad (1)$$

The Monshi-Scherrer method was used to estimate the average coherent crystallite domain size from XRD peak broadening using a linearized form of the Scherrer equation. This from given by Equation 2 [16,17].

$$\ln \beta = \ln \left(\frac{K \cdot \lambda}{D} \right) + \ln \left(\frac{1}{\cos \theta} \right) \quad (2)$$

With D: average coherent crystallite domain size (nm); K: Scherrer constant ($K = 0.9$); λ : X-ray wavelength (nm); β : Full width at half maximum (FWHM) corrected for instrumental broadening in radians; θ : Bragg's angle in radians.

2.3.2. Williamson-Hall Method: Uniform Deformation Model (UDM)

The UDM model was employed to distinguish the contribution of the coherent crystallite domain size and microstrain (ϵ) to XRD peak broadening. This approach assumes that the microstrain is uniform along all crystallographic directions, allowing the total peak broadening to be resolved into a size-related contribution associated with lattice distortion (Equation 3) [18].

$$\beta = \beta_{\text{size}} + \beta_{\text{strain}} \quad (3)$$

Consequently, the UDM approach is expressed by Equation 4 [19,20]:

$$\beta_{\text{hkl}} \cos \theta = \frac{K \cdot \lambda}{D} + 4\epsilon \sin \theta \quad (4)$$

2.3.3. Size-Strain Plot Method

The Size-Strain Plot (SSP) method was employed to simultaneously estimate the coherent crystallite domain size and microstrain by accounting for their respective contributions to XRD peak broadening (Equation 5). This approach assumes that size-induced broadening follows a Lorentzian distribution, whereas strain-induced broadening is Gaussian in nature [15,21,22].

$$\beta_{\text{hkl}} = \beta_L + \beta_G \quad (5)$$

Where β_L et β_G are the full widths at half maximum (FWHM) of the Lorentzian and Gaussian functions, respectively. The relationship used to estimate the coherent crystallite domain size and lattice strain by SSP method is expressed by Equation 6 [15,23].

$$(d_{\text{hkl}} \cdot \beta_{\text{hkl}} \cdot \cos \theta)^2 = \frac{K \cdot \lambda}{D} (d_{\text{hkl}}^2 \cdot \beta_{\text{hkl}} \cdot \cos \theta) + \frac{\epsilon^2}{4} \quad (6)$$

2.3.4. Halder-Wagner Method

This method was applied to simultaneously estimate the coherent crystallite domain size and crystal lattice microstrain from X-ray diffraction peak broadening. The approach is based on a Voigt function, assuming that the diffraction profiles result from the convolution of Lorentzian and Gaussian components, which are generally attributed to the effects of coherent crystallite domain and microstrain, respectively (Equation 7) [21,24-26].

$$\beta_{\text{hkl}}^2 = \beta_L \beta_{\text{hkl}} + \beta_G^2 \quad (7)$$

The relationship used to determine the coherent crystallite domain size and lattice microstrain according to the Halder-Wagner methods is given by Equation 8 [19,25,27]:

$$\left(\frac{\beta_{\text{hkl}}}{2 \tan \theta} \right)^2 = \frac{K \cdot \lambda}{D} \left(\frac{\beta_{\text{hkl}}}{4 \tan \theta \cdot \sin \theta} \right) + 4\epsilon^2 \quad (8)$$

3. Results and Discussion

3.1. XRD Data Analysis

The powder X-ray diffraction pattern of the synthesized solid (Figure 1) reveals a highly crystalline compound, with the main intense reflections distributed over the $5 - 30^\circ (2\theta)$ range. The principal reflections are located at approximately $2\theta(^\circ) = 8.08, 8.22, 8.42, 8.6, 8.88, 9.56, 10.16, 10.84, 11.7, 11.86, 15.16, 16.24, 16.52, 16.74, 17.28, 17.84, 18.46, 18.96, 19.82, 20.26, 20.54, 22.44, 22.9, 23.66, 23.8, 24.92, 25.66, 26.80, 28.00$. The presence of numerous well-defined and well-resolved reflections is consistent with a highly ordered crystalline phase exhibiting long-range periodicity. This structural organization is consistent with the nature of coordination polymers, in which metal centers and organic ligands are assembled in a repetitive manner to form periodic crystalline networks [28-30]. In particular, carboxylate

ligands can act as coordinating and bridging groups between metal centers, thereby promoting the propagation of structural order throughout the coordination network [28,29,31]. The reflections observed at low angles correspond, according to Bragg's law, to relatively large interplanar spacings. They therefore indicate the presence of crystallographic planes characterized by significant interplanar distances [32]. In the context of the coordination polymer investigated, these results are consistent with an extended structural organization involving coordination units interconnected through Cr-O-carboxylate interactions. In its worth nothing that the reflections at 2θ ($^\circ$) = 8.08, 16.24, 17.28, 18.96, and 26.8 $^\circ$ also coincide with reflections attributed to residual benzoic acid (CCDC 2533705). The experimental diffraction pattern does not reproduce the entire diffraction pattern of benzoic acid. Several major reflections of the synthesized material occur at different positions or exhibit markedly altered relative intensities, particularly around 2θ ($^\circ$) = 8.22, 8.42, 8.60, 8.88, 9.56, 10.16, 10.84, 11.7, 11.86, 15.16, 16.52, 16.74, 18.46, 19.82, 20.26, 20.54, 22.44, 22.90, 23.66, 23.80, 24.92, 25.66, and 28.00. These differences indicate that the crystalline structure of the synthesized material does not correspond to that of either the free ligand or chromium(III) nitrate nonahydrate, whose principal (-111) reflection occurs at 2θ = 13.40 $^\circ$ (JCPDS card n $^\circ$ 31-0409). These changes are consistent with deprotonation of the carboxylic group followed by its coordination to the Cr(III) centers, resulting in a new structural arrangement. A comprehensive literature search did not identify any CIF file corresponding to a coordination polymer composed exclusively of Cr(III) centers and benzoate ligands. Consequently, direct indexing of the experimental diffraction pattern could not be performed by comparison with a reference crystal structure. The absence of a reference phase suggests that the obtained material may correspond to a crystalline Cr(III)-benzoate phase for which no corresponding crystallographic structure was identified in the databases consulted. The PXRD analysis therefore suggests the formation of a crystalline Cr(III)-benzoate coordination network; however, the lack of an available CIF structure necessitates an interpretation based on the overall diffraction signature of the material rather than on an unambiguous crystallographic identification.

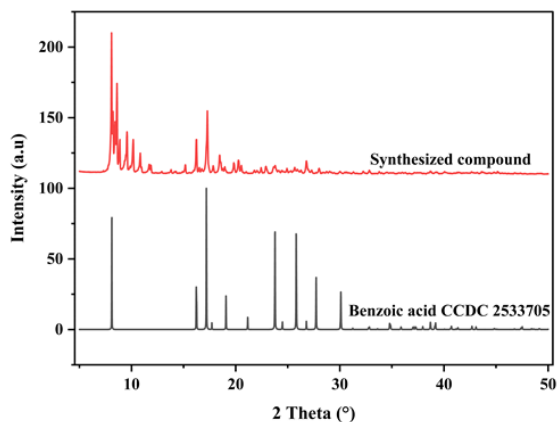


Figure 1. XRD pattern of synthesized compound

3.2. Fourier –Transform Infrared Spectroscopy

The FTIR spectrum of the synthesized material (Figure 2) exhibits several characteristic bands consistent with the presence of benzoate ligands coordinated to chromium(III). The band located at around 3605 cm^{-1} is assigned to $\nu(\text{O-H})$ vibrations, suggesting the presence of hydroxyl groups weakly involved in hydrogen bonding within the material. The band observed at 3066 cm^{-1} is attributed to $\nu(\text{C-H})$, stretching vibrations, of the aromatic hydrogens atoms of the benzene ring of the benzoate ligand. The weak band observed at 1710 cm^{-1} is assigned to the stretching vibration, of residual carboxylic acid groups, suggesting that a small fraction of benzoic acid may remain in the non-deprotonated or uncoordinated form. The band observed around 1610 cm^{-1} is primarily assigned to the stretching vibration of the aromatic C=C bonds of the benzoate ring; a contribution from the asymmetric stretching vibration of the carboxylate group, $\nu_{\text{asym}}(\text{COO}^-)$, cannot be excluded because of the possible overlap of vibrational bands in this spectral region. The bands observed at 1572 and 1529 cm^{-1} are assigned to the asymmetric stretching vibrations, $\nu_{\text{asym}}(\text{COO}^-)$, whereas those at 1410 and 1332 cm^{-1} correspond to the symmetric stretching vibrations, $\nu_{\text{sym}}(\text{COO}^-)$, of the carboxylate groups [33,34]. The appearance of these carboxylate bands, together with the decrease in the contribution associated with the protonated carboxylic group, is consistent with the deprotonation of benzoic acid and the formation of benzoate groups associated with the Cr(III) centers. The differences in wavenumber, $\Delta\nu$, are 162 and 197 cm^{-1} , obtained from (1572 – 1410 cm^{-1}) and (1529 – 1332 cm^{-1}), respectively. The $\Delta\nu$ values obtained indicate that the carboxylate groups are involved in the coordination environment of chromium. This interpretation is consistent with the coordination modes reported for certain Cr(III)-carboxylate complexes. Sydora and collaborators notably demonstrated that benzoate ligands can adopt different coordination modes around Cr centers, including a bridging mode in the complex $\text{Cr}_2(\mu\text{-O}_2\text{CPh})_4(\text{THF})_2$, in which the benzoate groups bridge two Cr(III) centers [34]. Likewise, Vlachos and collaborators reported, by single crystal X-ray diffraction, the trinuclear oxo-carboxylate complex $\text{Cr}_3\text{O}(\text{O}_2\text{CPh})_6(\text{MeOH})_3(\text{NO}_3)_2\cdot 2\text{MeOH}$, in which the benzoate ligands participate in the connection of the Cr(III) centers through a $\mu_2\text{-}\eta^1\text{:}\eta^1$ bridging mode [35]. Thus, these values are consistent with the presence of coordinated benzoate groups involved in an extended coordination framework. However, in the absence of direct structural determination, the $\Delta\nu$ values alone do not allow the coordination mode (monodentate, bridging, or chelating) to be unambiguously established. The bands at 1218, 1179, 1115, 1072, 1025, 1002, 975 cm^{-1} are mainly assigned to vibrations of the aromatic ring and C-H deformation modes of the benzoate ligand [36]. The bands observed at 937, 817, and 713 cm^{-1} are attributed to the out-of-plane C-H bending vibrations, $\delta(\text{C-H})$, of the substituted aromatic ring, which are characteristic of benzoate ligand. The bands located at 659 and 510 cm^{-1} are consistent with metal-oxygen stretching vibrations,

$\nu(\text{Cr-O})$, and may therefore be associated with Cr-O interaction involving the oxygen atom of the carboxylate groups. These low-wavenumber vibrational features provide additional spectroscopic support for the interaction between the Cr(III) centers and the oxygen donor atoms of the carboxylate groups.

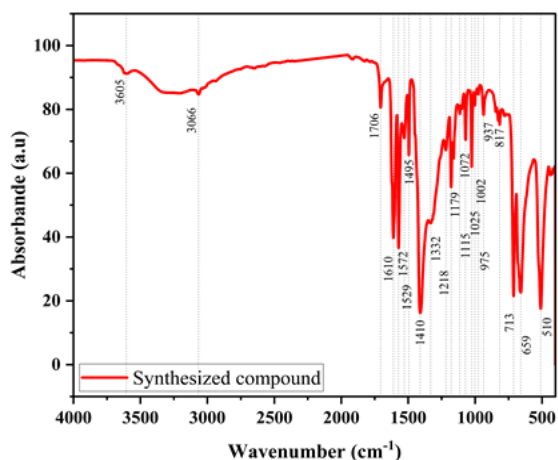
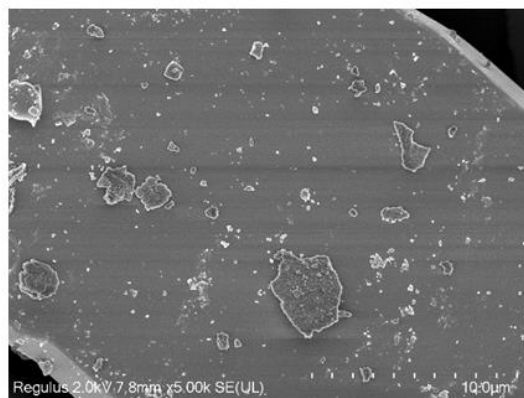


Figure 2. FTIR spectrum of the synthesized product

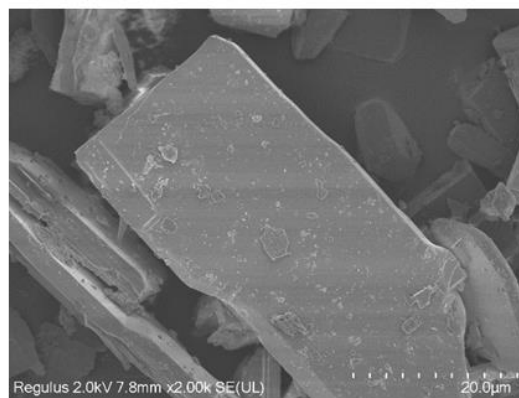
3.3. Scanning Electron Microscopy Coupled with Energy Dispersive X-Ray Spectroscopy (SEM-EDS)

The SEM micrographs acquired at different magnifications (figure 3) show that the material consists of an assembly of

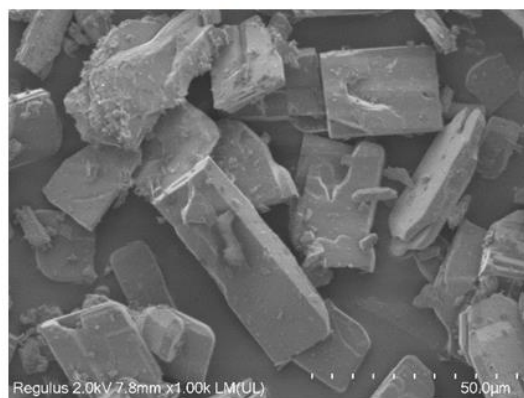
micrometre-size crystalline particles exhibiting predominantly a prismatic morphology. The image acquired at $\times 500$ highlights the overall organization of the synthesized powder, whereas the $\times 1000$ magnification reveals faceted particles with planar surfaces and clearly defined edges. The particle size ranges from 2.26 to 17.21 μm , with an average size of 8.15 μm (Figure 4). These observations also reveal partial agglomeration of the crystals, with several particles being in direct contact with one another. The particle surfaces appear generally compact, and no clearly discernible open macroporosity is observed at the scale investigated. However, this observation does not rule out the presence of microporosity or mesoporosity, the assessment of which requires specific techniques, such as nitrogen adsorption-desorption measurements [37]. The presence of planar faces and well-defined edges is consistent with relatively ordered crystal growth and the preferential development of specific crystallographic faces [38,39]. However, crystal morphology results from the interplay of several factors associated with nucleation and growth processes, as well as the physicochemical conditions prevailing during crystallization [38,39]. In carboxylate-based coordination polymer systems, the synthesis conditions can notably affect the crystal growth kinetics, resulting in variations in particle morphology and size [40]. Therefore, the observed morphology should be regarded as the outcome of the specific synthetic conditions employed in the study [38,39,40].



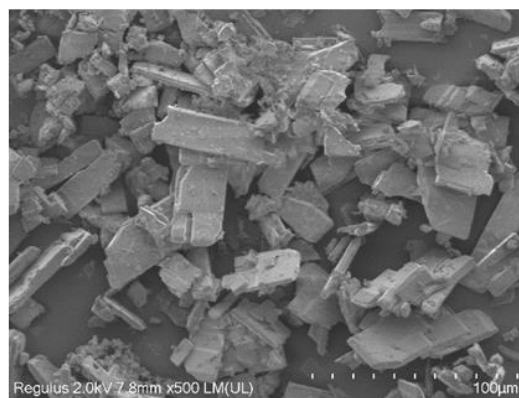
a) Magnification: $\times 5000$



b) Magnification: $\times 2000$



c) Magnification: $\times 1000$



d) Magnification: $\times 500$

Figure 3. SEM of synthesized compound

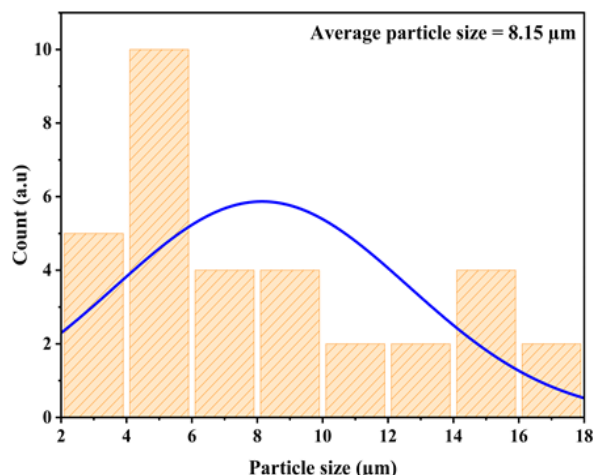


Figure 4. Particle size of the synthesized compound

The SEM-EDS spectra (Figure 5) reveal exclusively the presence of C, O, and Cr, as evidenced by the C K α (0.27 keV), O K α (0.52 keV), and Cr K α /K β (5.42 and 5.95 keV) emission lines. The simultaneous detection of these elements is consistent with the expected chemical composition of the synthesized material and confirms the incorporation of chromium into the polymeric matrix. The absence of other major elements further suggests the formation of

a chemically homogeneous material. Although EDS is primarily a quantitative technique and does not provide direct information on the coordination environment of chromium, it unequivocally confirms the presence of Cr within the material, thereby providing essential evidence supporting the proposed composition of the coordination polymer.

The combined analysis of the different characterization provides complementary evidence supporting the formation of a chromium(III)-benzoate coordination polymer. XRD analysis reveals a crystalline phase with several low angle reflections, indicative of an extended periodic framework, whereas FTIR spectroscopy confirms the chemical nature of the building units involved in this organization. The transformation of protonated benzoic acid into coordinated carboxylate groups is evidenced by the decreased contribution of the $\nu(\text{C}=\text{O})$ band characteristic of the free acid and by the appearance of the characteristic $\nu_{\text{asym}}(\text{COO}^-) / \nu_{\text{sym}}(\text{COO}^-)$ vibrational pair. The EDS results are in excellent agreement with the FTIR findings. Indeed, while EDS confirms the coexistence of C, O, and Cr within the material, the FTIR spectrum demonstrates the conversion of the carboxylic acid functionalities of benzoic acid into coordinated carboxylate groups, together with the appearance of $\nu(\text{Cr}-\text{O})$ vibrations in the 659 – 510 cm^{-1} region.

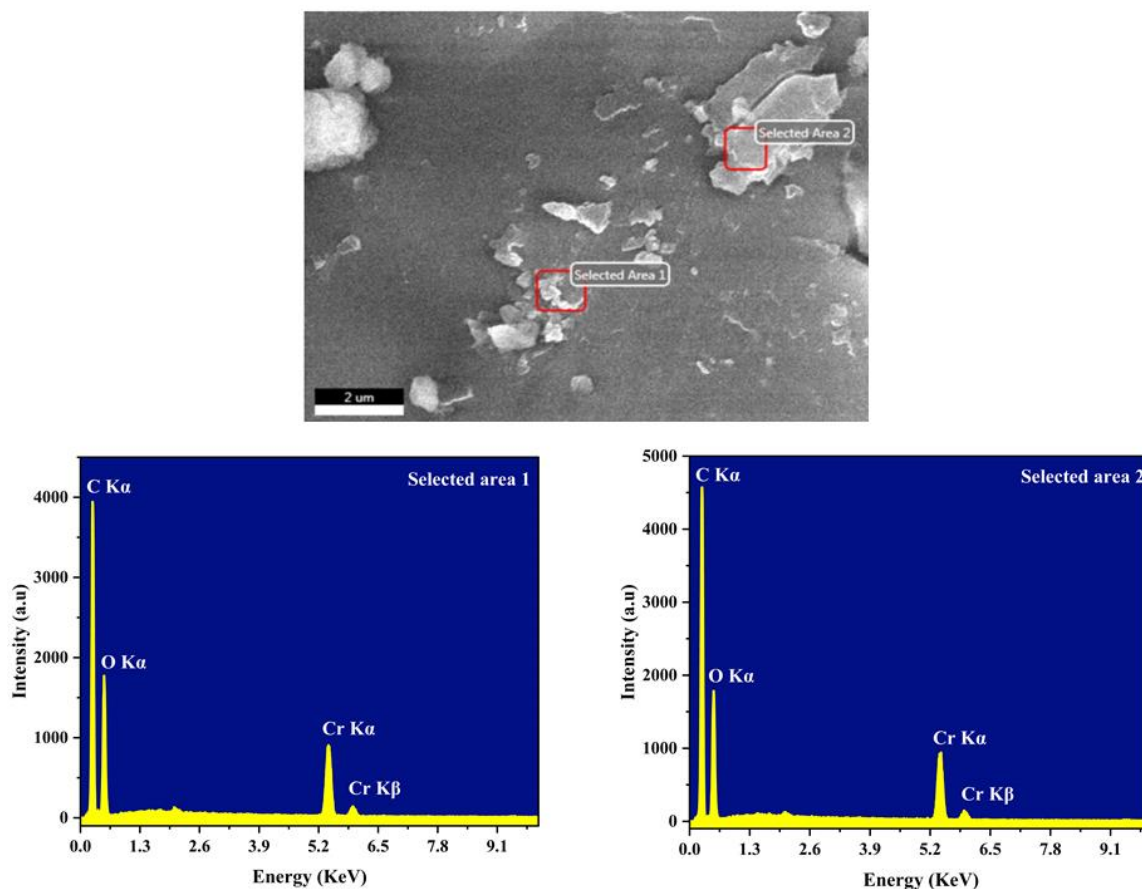


Figure 5. SEM-EDS analysis of the synthesized compound

The complementary information provided by EDS and FTIR supported the involvement of the carbon and oxygen containing functionalities of the ligand in the coordination of Cr(III) centers, leading to the information of a coordination polymer in which carboxylate ligands act as bridging linkers. In the absence of a corresponding crystallographic CIF structure in the databases consulted, the exact structural assignment – including the crystal symmetry, and network topology cannot be established solely from powder XRD data. Nevertheless, the convergence of the XRD signatures, characterized by a crystalline phase with numerous low angle reflections, and the FTIR features, indicating bridging carboxylate groups and Cr-O vibrations, provides compelling evidence for the formation of a crystalline Cr(III)-benzoate coordination polymer.

3.4. X-ray Fluorescence (XRF)

XRF analysis revealed the presence of Cr as the metallic center, whereas the elements constituting the ligand (C, O, and H) were not detected owing to the intrinsic limitations of XRF for the analysis of low atomic number elements [41,42]. The apparent Cr content was 97.97%. This value does not directly represent the actual chromium content of the material, as the contribution of light elements is not adequately accounted for during quantitative analysis. Accordingly, the remaining 2.03% cannot be considered representative of the actual C, O, and H content, but rather corresponds to the unassigned fraction under the analytical conditions employed. XRF therefore provided only partial information on the composition of the synthesized material. In contrast, the presence of the organic ligand is corroborated by EDS analysis, which detected carbon and oxygen, as well as by FTIR spectroscopy, whose characteristic bands are consistent with the coordination of carboxylate groups to metal center, in agreement with the behavior reported for Cr(III) benzoate complexes [11].

3.5. Microstructural Study on the Synthesized Cr-Benzoate Coordination Polymer

The angular positions and corrected FWHM values of the Cr-MOF diffraction peaks used to estimate the coherent crystallite domain size and lattice microstrain are listed in Table 1.

3.5.1. Scherrer and Monshi-Scherrer Methods

Figure 6 represents the coherent crystallite domain sizes determined using the Scherrer equation for the different

crystallographic reflections, together with their average value. The calculated coherent crystallite domain size exhibit significant variation, ranging from 39.66 to 297.57 nm, indicating a dependence on the crystallographic direction considered. The average size, estimated at 120.23 nm, therefore provides a representative overall value for the material. In contrast, Figure 7 presents the experimental data and the corresponding linear fit obtained from the plot of $\ln\beta$ versus $\ln(1/\cos\theta)$ according to the Monshi-Scherrer method. This fitting procedure yielded an estimated coherent crystallite domain size of 168.54 nm.

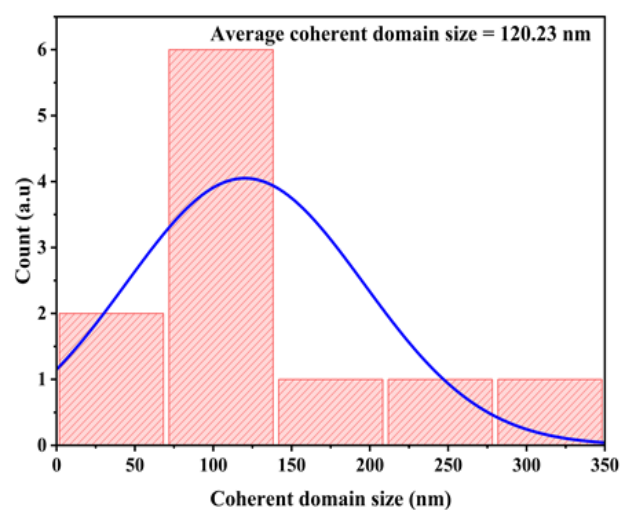


Figure 6. Scherrer method

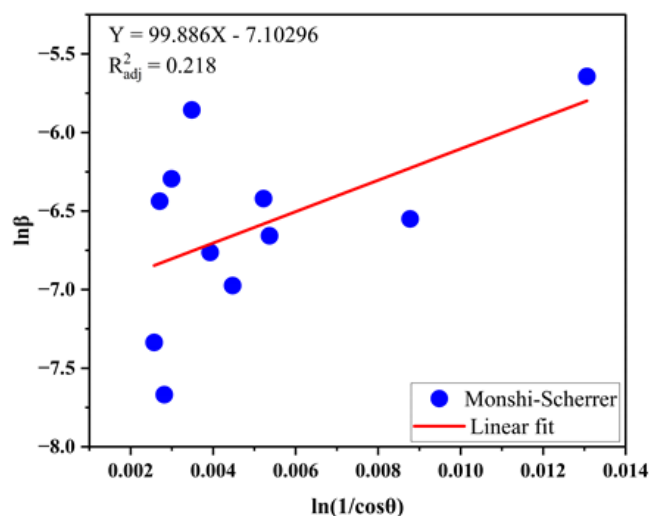


Figure 7. Monshi-Scherrer method

Table 1. Angular positions and corrected FWHM, β

2θ	8.21	8.42	8.59	8.86	9.56	10.15	10.83	11.70	11.86	15.16	18.48
$\beta(^{\circ})$	0.0802	0.116	0.0759	0.1274	0.1786	0.0971	0.0889	0.1172	0.1023	0.1085	0.2149

3.5.2. Williamson-Hall Method: Uniform Deformation Model (UDM)

Figure 8 presents the plot of $\beta \cos \theta$ versus $4 \sin \theta$ according to the uniform deformation model (UDM) of Williamson-Hall. The experimental data were fitted by linear regression, with the intercept providing the coherent crystallite domain size and slope yielding the microstrain of the material. The resulting values were 550.99 nm for the coherent crystallite domain size and 4.73×10^{-3} for the microstrain.

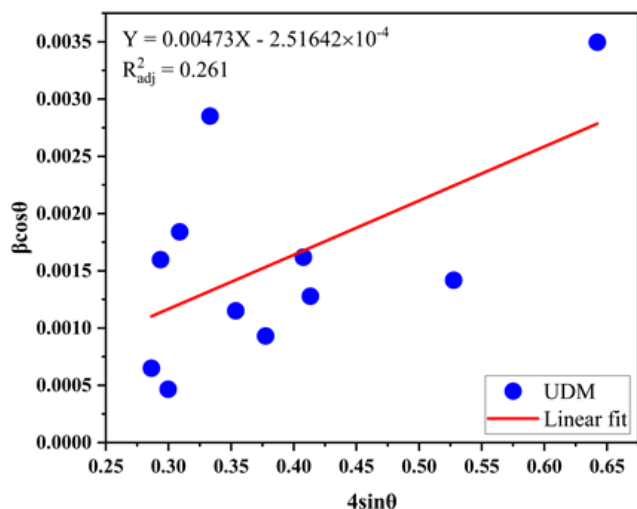


Figure 8. UDM

3.5.3. Size-Strain Plot Method

Figure 9 presents the experimental data and the corresponding linear fit obtained using the SSP method, based on the plot of $(d_{hkl}^2 \cdot \beta_{hkl} \cdot \cos \theta)$ versus $(d_{hkl} \cdot \beta_{hkl} \cdot \cos \theta)^2$. The linear fitting yielded an estimated coherent crystallite domain size of 49.17 nm and a microstrain of 2.053×10^{-3} .

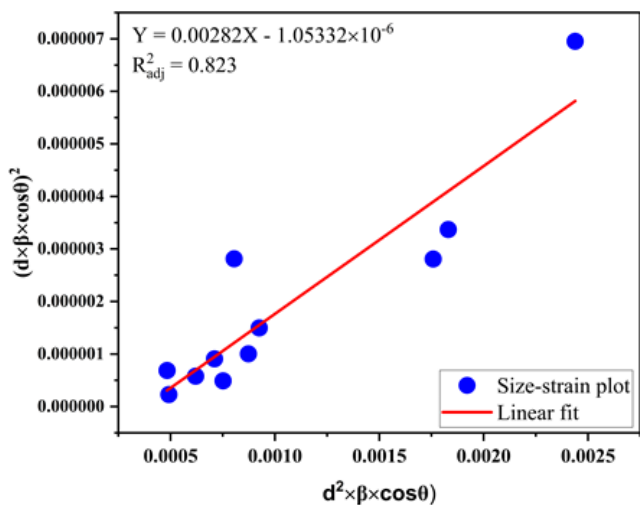


Figure 9. Size-strain Plot method

3.5.4. Halder-Wagner Method

Figure 10 presents the Halder-Wagner plot and the corresponding linear of the experimental data, obtained by

plotting $(\beta_{hkl}/2 \tan \theta)^2$ versus $(\beta_{hkl}/4 \tan \theta \cdot \sin \theta)$. The regression line parameters enabled the simultaneous determination of the coherent crystallite domain size and microstrain. The resulting values were 49.17 nm for the coherent crystallite domain size and 3.331×10^{-3} for the microstrain.

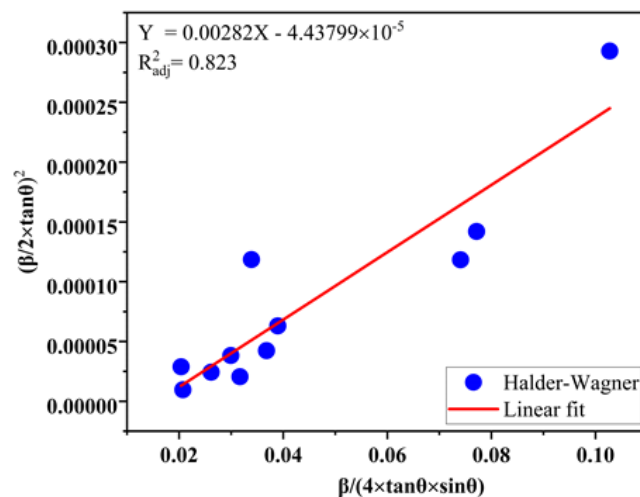


Figure 10. Halder – Wagner method

The microstructural parameters of the synthesized chromium(III) benzoate coordination polymer were evaluated using several methods based on diffraction peak broadening analysis. The application of several independent formalisms to the same dataset allows the consistency of the microstructural estimates to be assessed and their dependence on the assumptions inherent to a particular model to be reduced. The different approaches yielded significantly different estimates of the coherent crystallite domain size and microstrain (Table 2), Thereby highlighting the influence of the physical assumptions and mathematical formalisms underlying each model.

The linear regressions obtained using the UDM, SSP, and Halder-Wagner models yielded slightly negative intercepts. The negative sign of these intercepts has no physical significance for the parameters considered. Therefore, the absolute values of the intercepts were used to estimate the coherent crystallite domain size and microstrain, in accordance with the approach reported in several previous studies [15,43-45].

Application of the Scherrer equation yielded an average apparent coherent crystallite domain size of 120.30 nm. This method provides a first estimate of the coherent domain size, assuming that diffraction peak broadening arises exclusively from the finite size of the coherently diffracting domains. However, it does not allow the contributions arising from microstrain, crystal defects, or internal stresses to be separated [46,47]. Consequently, the obtained value should be interpreted as an apparent estimate of the crystallographic coherent length rather than as an absolute measure of the actual crystallite size.

Application of the Monshi-Scherrer model yielded an apparent coherent crystallite domain size of 168.54 nm, which is higher than obtained using the conventional Scherrer equation. However, the low adjusted coefficient of determination ($R^2 = 0.218$) indicates a poor correlation between the experimental data and the linearized model. Therefore, this value should be interpreted with caution, as the limited goodness of fit indicates that the Monshi-Scherrer model does not adequately describe the experimental data for the material investigated [48-50].

The Williamson-Hall model based on the uniform deformation model (UDM) yielded a markedly higher apparent coherent crystallite domain size of 550.99 nm, associated with an estimated microstrain of 4.73×10^{-3} . However, the low coefficient of determination ($R^2 = 0.261$) indicates a poor fit between the experimental data and the linearized model [49-51]. This result suggests that the assumption of uniform and isotropic lattice strain may not adequately describe the contributions responsible for diffraction peak broadening in the coordination polymer investigated. The relatively large coherent domain size may therefore arise from the coupling between size and microstrain contributions during the fitting procedure, particularly when the diffraction peak broadening exhibits anisotropic characteristic or cannot be adequately described by a simplified model [49].

In contrast, the SSP and Halder-Wagner models yielded consistent estimates of the coherent crystallite domain size (49.17 nm) and exhibited the highest coefficients of determination ($R^2 = 0.823$). This agreement, together with the superior quality of the fits, indicates the greater ability of these approaches to describe the experimentally observed diffraction peak broadening by simultaneously accounting for the contributions arising from coherent domain size and lattice microstrain [22,25]. The resulting microstrain values remained relatively low (2.05×10^{-3} for SSP and 3.33×10^{-3} for Halder-Wagner), indicating that lattice distortions make only a limited contribution to peak broadening in the coordination polymer investigated.

The discrepancy between the coherent crystallite domain sizes estimated using the Scherrer equation and those obtained from the SSP and Halder-Wagner models primarily arises from the different assumptions underlying each approach

[49,51]. The Scherrer method assumes that diffraction peak broadening originates predominantly from the finite size of the coherently diffracting domains, whereas the SSP and Halder-Wagner models allow the relative contributions of size and lattice microstrain to be considered separately. In the Cr(III)-benzoate coordination polymer, the connectivity of the Cr-carboxylate units, the diversity of coordination environments, and potential local structural distortions may induce variations in structural parameters and lattice microstrain, which can contribute to the observed broadening of the diffraction peaks.

Table 3 presents the apparent dislocation densities calculated using the relationship $\delta = 1/D^2$ [52,53], based on the coherent crystallite domain sizes obtained from the different analytical approaches. The calculated values range from 3.294×10^{12} to $4.14 \times 10^{14} \text{ m}^{-2}$, reflecting the strong dependence of this parameter on the model used to estimate the coherent domain size. Accordingly, the methods yielding larger coherent domain sizes (Scherrer, Monshi-Scherrer, and Williamson-Hall) result in lower apparent dislocation densities, whereas the SSP and Halder-Wagner approaches, which yield smaller coherent domain sizes, produce higher values. This trend directly follows from the inverse-square relationship between the apparent dislocation density and the coherent domain size. Nevertheless, the interpretation of these values should take into account the specific model employed to separate size and microstrain contributions to diffraction peak broadening.

Overall, this comparative analysis highlights the complementary nature of the different approaches employed to evaluate the microstructural parameters, each of which relies on specific assumptions regarding the relative contributions of coherent crystallite domain size and microstrain to diffraction peak broadening. The low coefficient of determination obtained for the Monshi-Scherrer and Williamson-Hall (UDM) models suggest that these simplified formalisms do not fully describe the experimental profiles of the material investigated. In contrast, the good agreement between the results obtained using the SSP and Halder-Wagner methods, together within their superior fitting criteria and relatively low microstrain values, indicates that these approaches provide a more consistent description of the microstructural state of the chromium(III)-benzoate coordination polymer.

Table 2. Microstructural parameters of the synthesized Cr-benzoate coordination polymer

Scherrer	Monshi-Scherrer	Williamson-Hall (UDM)		Size-Strain Plot		Halder-Wagner	
D (nm)	D (nm)	D (nm)	$\varepsilon (10^{-3})$	D (nm)	$\varepsilon (10^{-3})$	D (nm)	$\varepsilon (10^{-3})$
120.23	168.542	550.99	4.73	49.17	2.05	49.17	3.33

Table 3. Dislocation densities of the synthesized Cr-benzoate coordination polymer

Scherrer	Monshi-Scherrer	Williamson-Hall (UDM)	SSP	Halder-Wagner
$\delta (\text{m}^{-2})$				
6.917×10^{13}	3.52×10^{13}	3.294×10^{12}	4.14×10^{14}	4.14×10^{14}

The particle size observed by SEM and the coherent crystallite domain size estimated from XRD should not be considered equivalent quantities. SEM provides the morphological dimensions of the observed particles, whereas XRD broadening analysis estimates coherent crystallite domain size. A single SEM visible particle may consist of several coherent crystalline domains; consequently, the XRD derived values can be significantly smaller than the particle dimensions observed by SEM. Accordingly, these two parameters are reported and interpreted as distinct microstructural characteristics.

4. Conclusions

In this study, a chromium(III)benzoate coordination polymer was synthesized and characterized using a multi technique approach combining XRD, FTIR, SEM-EDS, and XRF. The XRD results reveal the formation of a highly crystalline phase exhibiting numerous low angle reflections, indicative of an extended periodic organization. Although no crystallographic reference structure could be identified, the structural signatures obtained suggest the formation of a crystalline Cr(III)-benzoate coordination network distinct from both the benzoate ligand and the chromium precursor. FTIR analysis confirms the transformation of the benzoic acid ligand into carboxylate groups coordinated to the Cr(III) centers, with a coordination mode consistent with bridging carboxylates, while the bands assigned to Cr-O vibrations further support the establishment of metal-ligand interactions. SEM-EDS observations revealed a prismatic crystalline morphology with micrometre-size particles, while EDS and XRF analyses confirmed the presence of chromium and an elemental composition consistent with the formation of the target material. The convergence of the results obtained from these complementary techniques therefore provides consistent experimental evidence for the formation of a Cr(III)-benzoate coordination polymer. Analysis of diffraction peak broadening using the Scherrer, Monshi-Scherrer, Williamson-Hall, Size-Strain Plot, and Halder-Wagner methods highlighted the influence of the underlying assumptions of each model on the estimation of microstructural parameters. Among these approaches, the SSP and Halder-Wagner methods exhibited the best agreement with the experimental data ($R^2 = 0.823$), yielded consistent values for the coherent crystallite domain size (49.17 nm) and microstrain. Thus, this study demonstrates the value of combining structural and microstructural characterization techniques for the identification of poorly documented crystalline coordination phases. It also highlights the suitability of the Size-Strain Plot and Halder-Wagner models for evaluating the microstructural parameters of metal carboxylate-based coordination polymers, particularly when the contributions of coherent crystallite size and crystal defects need to be distinguished.

Author Contributions

Ferland Ngoro-Elenga: conceptualisation, data curation, methodology, writing-original draft and writing-reviews and editing; Hubert Makomo: Methodology, writing-reviews and editing; Nicaise Narcisse Obaya: methodology, writing-review and editing; Grâce Jeanci Viboudoulou: methodology; Ibrahima Elhadji Thiam: supervision and validation; Timothée Nsongo: supervision and validation. All authors have read and approved the final version of the manuscript.

Data Availability Statement

The authors confirm that all data supporting the finding of this study are included in the article.

Conflict of Interest

The authors declare that they have no conflict of interest.

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