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STUDY OF MORPHOLOGY AND STRUCTURAL FEATURES OF METAL HYDROXIDES OF THE IRON TRIAD

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ABSTRACT

Hydroxides of the iron triad represent an important class of inorganic materials whose functional behaviour is governed not only by composition but also by the structural pathway followed during phase formation. In the present work, $Fe(OH)_3$, $Co(OH)_2$, and $Ni(OH)_2$ were synthesised under identical precipitation conditions to provide a direct and physically meaningful comparison of morphology and structural organisation. The materials were examined using scanning electron microscopy (SEM) and X-ray diffraction (XRD). The comparative analysis demonstrates that each hydroxide develops a distinct structural scenario. Iron hydroxide forms highly dispersed amorphous aggregates characterised by a loose porous architecture, explaining its strong affinity toward adsorption processes. Cobalt hydroxide evolves into thin plate-like units consistent with low-crystalline layered structures. Nickel hydroxide exhibits the highest structural ordering, forming nanosheet and flower-like assemblies associated with α/β-phase organisation. The obtained results reveal a clear relationship between cation chemistry, hydrolysis kinetics, and morphology development. The study provides a unified framework explaining how synthesis parameters govern structural evolution within iron-triad hydroxides and offers practical guidance for designing inorganic materials with tailored properties for environmental, catalytic, and electrochemical technologies.

Keywords: Iron-Triad Hydroxides, $Fe(OH)_3$, $Co(OH)_2$, $Ni(OH)_2$, Morphology, SEM, XRD, SDG-9.

INTRODUCTION

The behaviour of metal hydroxides in functional applications is strongly dependent on how their structure develops during synthesis rather than solely on chemical composition. Features such as particle geometry, crystallite dimensions, aggregation pathways, and pore distribution determine the accessibility of active surface sites and directly influence transport phenomena, interfacial reactions, and charge exchange processes.[1–5] Consequently, morphology becomes a key factor controlling catalytic efficiency, sorption capacity, and electrochemical performance. Layered or low-dimensional architectures formed by transition-metal hydroxides are known to demonstrate superior functional characteristics compared with bulk materials. In particular, ultrathin $Co(OH)_2$ and $Ni(OH)_2$ structures often display enhanced electrochemical activity due to shortened diffusion paths and increased exposure of reactive surface planes. Variations in phase composition-including transitions between α- and β-modifications — together with ionic modification or doping can substantially affect conductivity, stability, and long-term operation.[6–10] Despite extensive research, a unified understanding of how synthesis parameters control the structural evolution of Fe-, Co-, and Ni-based hydroxides has not yet been achieved. Many investigations address isolated systems and vary experimental parameters independently, which makes direct comparison difficult and prevents the identification of general formation principles. Published data show that studies comparing $Fe(OH)_3$, $Co(OH)_2$, and $Ni(OH)_2$ under strictly identical conditions remain limited. Most works focus on individual compounds, while comparative approaches employing unified pH conditions, ionic environments, and neutralisation procedures are relatively rare.[11–13] This lack of consistency complicates the development of predictive relationships linking synthesis conditions with

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resulting morphology. Another important issue concerns hydrolysis and nucleation kinetics. For example, Fe(III) species are known to undergo rapid hydrolysis, often leading to amorphous intermediates, whereas slower precipitation processes can stabilise more ordered phases. The composition of the reaction medium and pH strongly influence nucleation pathways and subsequent growth mechanisms.[14–16] From a methodological standpoint, differences in characterisation strategies also limit cross-comparison. SEM and XRD data are frequently interpreted separately and using non-unified approaches, which leads to inconsistencies when comparing structural ordering among Fe-, Co-, and Ni-based systems.[17–19] Equally relevant is the role of precursor concentration and neutralisation pathways in controlling phase stability and morphology. The use of ionic additives, dopants, and variations in alkalization strategy may influence stabilisation of specific polymorphs, including α- and β-$Ni(OH)_2$, and promote formation of hierarchical architectures.[20,21] Recent literature dedicated to nickel hydroxide mainly focuses on structural polymorphism, hydration state, and defect chemistry due to their strong influence on electrochemical energy-storage performance.[22] At the same time, morphology engineering through ionic modification remains a major research direction.[23] Similar observations are reported for $Co(OH)_2$ and related cobalt oxyhydroxides, where morphology design largely determines pseudocapacitive and catalytic properties.[24] In contrast, $Fe(OH)_3$ and related Fe(III) oxyhydroxides are more commonly investigated from an environmental perspective, particularly in adsorption and immobilisation systems.[25–28] Over the past decade, advances in synthesis strategies-including hydrothermal approaches, two-dimensional growth control, and defect engineering-have significantly broadened the application potential of hydroxides based on the iron triad.[29,30] Nevertheless, a unified comparison of $Fe(OH)_3$, $Co(OH)_2$, and $Ni(OH)_2$synthesised under equivalent conditions remains missing, and systematic links between precipitation kinetics and structural evolution are still insufficiently established. The present study addresses these gaps by combining controlled synthesis with comparative SEM and XRD analysis in order to identify the key physicochemical factors governing morphology formation.[31,32] This work aims to establish a direct comparative understanding of the morphology and structure of iron-triad hydroxides and to correlate synthesis parameters with structural development and functional behaviour. This aim is consistent with SDG-9: Industry, innovation and infrastructure.

EXPERIMENTAL

Materials

$FeCl_3 \cdot 6H_2O$, $CoCl_2 \cdot 6H_2O$, and $Ni(NO_3)_2 \cdot 6H_2O$ (analytical grade) were used without additional purification. Alkaline precipitation was carried out using a 1 M NaOH solution or a $Ca(OH)_2$ suspension.

Synthesis Conditions

Hydroxides were obtained under controlled precipitation conditions summarised in Table-1[33], including pH range, temperature, and OH^- addition rate.

Table-1: Conditions for the Formation of Iron Triad Hydroxides

Parameter	$Fe(OH)_3$	$Co(OH)_3$	$Ni(OH)_3$
The initial ion	Fe^{3+}	Co^{2+}	Ni^{2+}
Optimal deposition pH	6.0–8.5	>9	>10–11
Temperature	20–25°C	20–60°C	20–80°C
The speed of adding OH^-	High	Average	Average
Crystallinity	Amorphous	Low	Average
Type of morphology	Floccules	Nanoplates	Nanoflakes

Electron Microscopy

SEM micrographs were recorded at accelerating voltages of 10–20 kV with spatial resolution up to 20 nm.

X-Ray Diffraction

Diffraction patterns were collected using a MAXima XRD-7000 instrument with $Cu_{K\alpha}$ radiation (λ = 1.5406 Å), scanning within 10–80° (2θ).

RESULTS AND DISCUSSION

Figure-1 shows SEM images of the obtained hydroxides. SEM analysis revealed clear differences in structural organisation among the synthesised hydroxides. Iron hydroxide forms loose, weakly ordered aggregates composed of nanoscale particles (approximately 10–50 nm) with pronounced porosity. Such architecture indicates rapid nucleation and explains the high adsorption efficiency typically observed for $Fe(OH)_3$ -based sorbents.[25,26] Cobalt hydroxide exhibits a markedly different morphology. The material consists of thin plate-like structures with characteristic thicknesses of approximately 5–10 nm[15,16,34], indicating partial layer ordering consistent with brucite-type structural motifs. Nickel hydroxide shows the highest morphological organisation. SEM images demonstrate nanosheet assemblies combined into hierarchical flower-like structures[35], resulting in increased surface accessibility and potentially favourable electrochemical behaviour. X-ray diffraction analysis[36] confirmed differences observed by microscopy. Figure-2 shows X-ray diffraction patterns of the synthesised hydroxides.

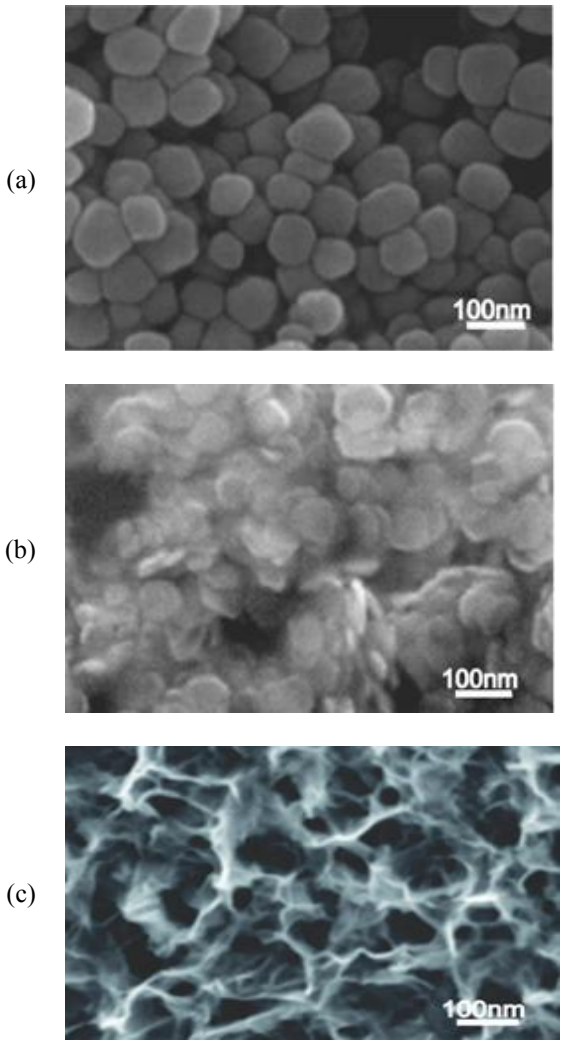

Fig.-1: SEM Images of Hydroxides of (a) Iron; (b) Cobalt, (c) Nickel

$Fe(OH)_3$ displays a broad diffuse halo typical of amorphous or poorly ordered phases. $Co(OH)_2$ presents reflections corresponding to layered brucite-like structures, whereas $Ni(OH)_2$ exhibits more defined diffraction peaks associated with α- and/or β-phase organisation. Table-2 shows the comparative morphological characteristics of metal hydroxides of the iron triad and possible areas of their application.

The comparative characteristics summarised in Table-2 highlight a clear trend: increasing structural ordering from Fe → Co → Ni correlates with decreasing hydrolysis rate and differences in cation chemistry.

Thus, morphology formation is controlled not only by synthesis conditions but also by intrinsic properties of the metal ion.

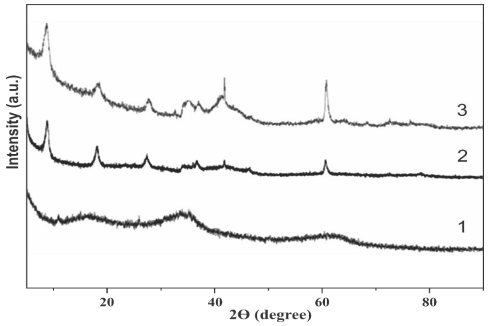

Fig.-2: X-ray Diffraction Spectrum of Hydroxides: 1 - iron (III), 2 -cobalt (II) and 3 -nickel (II)

Table-2: Comparative Morphological Characteristics of Fe, Co, and Ni Hydroxides

Property	$Fe(OH)_3$	$Co(OH)_2$	$Ni(OH)_2$
Crystallinity	Amorphous[13]	Low[16,29]	Average[21]
Basic structure	Floccules[23]	Nanoplates[16]	Nanoflakes[35]
Particle size	10 -40 nm[23]	10-30 nm[16,31]	10-25 nm[18]
Porosity	Very high[30]	Average[31]	Medium/High [19,22]
Application areas	Water purification[26,32,33]	Catalysis, batteries[34,36]	Sensorics, energy[37,38]

CONCLUSION

The comparative SEM and XRD study demonstrates that hydroxides of the iron triad follow distinct formation pathways even under equivalent synthesis conditions. $Fe(OH)_3$ forms highly dispersed amorphous aggregates generated by rapid hydrolysis, resulting in a porous structure favourable for adsorption processes. $Co(OH)_2$ develops layered plate-like architectures with moderate crystallinity, providing structural stability suitable for catalytic and electrochemical applications. $Ni(OH)_2$ exhibits the highest degree of structural ordering through the formation of nanosheet-based hierarchical assemblies, making it attractive for energy-storage and sensing technologies. The results confirm that morphology and crystallinity are governed by predictable relationships between cation chemistry, hydrolysis kinetics, and precipitation parameters. Understanding these relationships enables rational design of hydroxide-based materials with targeted functional properties and provides a foundation for their further application in environmental and electrochemical technologies. In this context, the study directly contributes to the implementation of Sustainable Development Goal SDG-9 (Industry, Innovation and Infrastructure) by enabling the development of advanced, structurally optimised inorganic materials suitable for industrial deployment in energy technologies, environmental engineering, and catalytic manufacturing processes.

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The work used elements of artificial intelligence, ChatGPT 5.1, to create a list of references and check grammar.

CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to*SDG-9: Industry, Innovation and Infrastructure*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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