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# STEPPING COLORATION OF Co $(OH)_2/Ni(OH)_2$ ELECTROCHROMIC FILM PREPARED BY A SIMPLE CHEMICAL BATH DEPOSITION METHOD

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Article History
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**Abstract**

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Electrochromic smart windows enable dynamic regulation of solar irradiation, thereby contributing to improved energy efficiency in building environments. In this work, $Co(OH)_2/Ni(OH)_2$ composite films were successfully deposited onto ITO-coated glass substrates via a facile chemical bath deposition method. Two configurations were explored: a homogeneously mixed composite and a double-layer architecture. The structural, morphological, and optical properties of the films were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), and UV–Vis spectroscopy. Both film types exhibited voltage-dependent, stepwise color modulation under applied potentials, confirming their electrochromic behavior. Notably, the mixed $Co(OH)_2/Ni(OH)_2$ film demonstrated enhanced optical modulation, with a transmittance change (ΔT) of 11.10% and an optical density change (ΔOD) of 0.09 at 0.15 V, increasing to 50.14% and 0.91 at 0.75 V. In contrast, the bilayer configuration showed significantly lower ΔT and ΔOD values of 1.36% and 0.13 at 0.15 V, and 9.64% and 0.31 at 0.75 V. These results highlight the synergistic role of $Co(OH)_2$ in tuning the optical response of $Ni(OH)_2$-based electrochromic films and suggest that compositional mixing is more effective than stratified layering for optimizing optical contrast in electrochromic devices.

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**Keywords:** Electrochromic, composite, $Co(OH)_2$, $Ni(OH)_2$, chemical bath

## 1. Introduction

Solar radiation serves as a fundamental energy source, providing both illumination and thermal energy to sustain life on Earth. Notably, near-infrared (NIR) radiation accounts for approximately 50% of the total solar energy and is primarily responsible for heat generation upon absorption (Khandelwal *et al.*, 2017; Renaud *et al.*, 2017). In the context of built environments, this absorbed heat significantly contributes to elevated indoor temperatures. Presently, energy demands associated with indoor climate regulation—including lighting and temperature control—constitute nearly 30–50% of global energy consumption, with projections indicating a continued upward trend in the coming decades (Davy *et al.*, 2017; Khandelwal *et al.*, 2017; Renaud *et al.*, 2017; Zhang *et al.*, 2018). These growing energy requirements have stimulated intensive research into the development of smart window technologies capable of modulating both solar light and heat transmission (Wang *et al.*, 2019).

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Electrochromic smart windows typically consist of a multilayer architecture comprising a transparent electrolyte layer sandwiched between two transparent conductive electrodes, along with an electrochromic film and a complementary ion storage (accumulation) layer. Electrochromic materials are capable of reversibly modulating their optical transmittance in response to an applied electric potential, transitioning between transparent (bleached) and colored states (Shchegolkov *et al.*, 2021). This tunable optical behavior is driven by redox reactions that alter the material's electronic structure. Classical electrochromic materials include transition metal oxides such as $WO_3$, $IrO_2$, $MoO_3$, and $V_2O_5$, which have been extensively studied due to their high coloration efficiency and cycling stability (Zhang *et al.*, 2021). However, their widespread application is limited by the high cost and complexity of their synthesis and device integration processes.

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Among alternative electrochromic materials, nickel hydroxide ($Ni(OH)_2$) has emerged as a promising candidate for smart window applications due to its rapid coloration–bleaching kinetics, high optical contrast, and excellent cycling stability (Kotok *et al.*, 2021). The performance improvement of $Ni(OH)_2$ can be done by adding $Co(OH)_2$ to $Ni(OH)_2$. Recent studies have demonstrated that the electrochemical and optical performance of $Ni(OH)_2$ can be further enhanced through the incorporation of cobalt hydroxide ($Co(OH)_2$). As reported by Bushra et al. (2021), the addition of $Co(OH)_2$ into the $Ni(OH)_2$ matrix improves the material's electrical conductivity and lowers its redox potential, thereby facilitating faster charge transfer during electrochromic switching (Safdar *et al.*, 2021). Furthermore, the formation of a bilayer $Co(OH)_2/Ni(OH)_2$ structure via electrodeposition has been shown to induce rapid and distinct multicolor transitions, expanding its functionality for dynamic tinting applications (Lee *et al.*, 2020).

Ni(OH)-based electrochromic films can be synthesized using a variety of techniques, including hydrothermal synthesis (Yang *et al.*, 2014), electrochemical deposition (Lee *et al.*, 2020), solvothermal (Liu *et al.*, 2018), and chemical bath deposition (CBD) (Lokhande and Chavan, 2018). Among these, the CBD method is particularly attractive due to its simplicity, low-temperature processing, scalability for large-area deposition, and cost-effectiveness (Bidier *et al.*, 2017). In the present study, $Co(OH)_2/Ni(OH)_2$ films with mixed and bilayer architectures were fabricated via CBD to explore their electrochromic behavior, with a focus on achieving multistep coloration suitable for advanced smart window applications.

## 2. Methodology

### 2.1 Materials

Indium tin oxide glass substrate (ITO, <10 ohm/sq, Welljoin, China), ammonium hydroxide ($NH_4OH$,
14 30%, Merck, Germany), nickel sulfate hexahydrate ($NiSO_4.6H_2O$, 98%, Merck, Germany), cobalt sulfate
heptahydrate ($CoSO_4.7H_2O$, 98%, Merck, Germany) acetone, isopropyl alcohol (IPA), deionized water (DI), sulfuric acid 98% ($H_2SO_4$), hydrogen peroxide 30% ($H_2O_2$), KOH.

### 2.2 Instrumentation

Characterization was performed using an X-Ray Diffractometer (SHIMADZU XRD-7000), a Scanning Electron Microscope (JEOL JSM-6510), a UV-Vis Spectrophotometer (Thermo Scientific Genesys 150), and a Rigol power supply.

### 2.3 Experimental Procedure

26 ITO-coated glass substrates were successively washed with acetone, IPA, and deionized water to ensure
removal of surface contaminants. To enhance surface wettability, the cleaned substrates were treated
25 with a freshly prepared piranha solution consisting of concentrated $H_2SO_4$ (98%) and $H_2O_2$ (30%) in a
3:1 v/v ratio for 10 minutes.

39 For the synthesis of the mixed $Co(OH)_2/Ni(OH)_2$ films, the activated substrates were immersed in a 0.05
M aqueous solution containing both $NiSO_4{\cdot}6H_2O$ and $CoSO_4{\cdot}7H_2O$, complexed in ammonium hydroxide
2 ($NH_4OH$) to facilitate controlled hydroxide precipitation. The $Co(OH)_2/Ni(OH)_2$ double-layer films were
29 obtained through sequential immersion of the substrate in 0.05 M $CoSO_4{\cdot}7H_2O$ and $NiSO_4{\cdot}6H_2O$
precursor solutions. All deposition processes were carried out at 70 °C, with immersion durations varied systematically at 10, 15, 20, 25, and 30 minutes. Following deposition, the films were rinsed, dried, and stored prior to electrochromic evaluation.

30 To assess the coloration performance, external voltages of 0, 0.15, 0.30, 0.45, 0.60, and 0.75 V were
7 applied in a two-electrode configuration employing 1 M KOH aqueous solution as the electrolyte. The
36 $Co(OH)_2/Ni(OH)_2$-coated glass served as the anode, and a carbon electrode was used as the cathode.
The stepwise change in optical transmittance under different applied voltages was monitored using a lux meter application to estimate the relative transparency.

3 The optical properties of the electrochromic films were characterized using UV–Vis spectrophotometry.
Films exhibiting the most pronounced transmittance modulation, one from the mixed and one from the bilayer system, were further analyzed using scanning electron microscopy (SEM) to evaluate surface
16 morphology. Crystallographic analysis was performed using X-ray diffraction (XRD) to confirm phase
formation and structural characteristics of the deposited films. The experimental procedure is illustrated in Figure 1.

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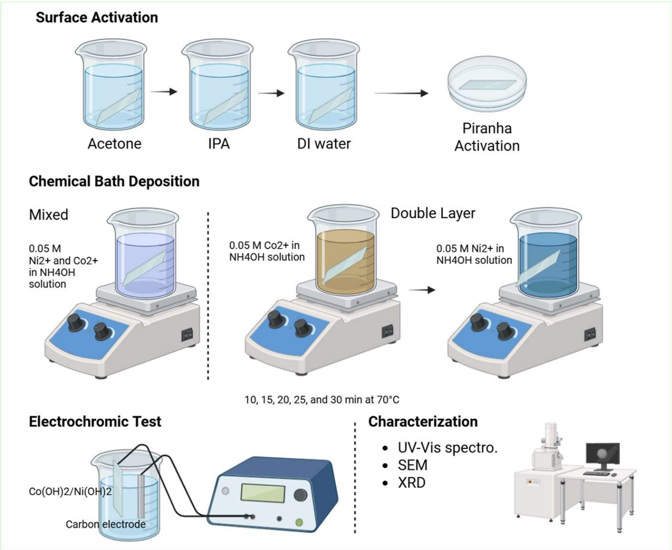



**Figure 1**. Schematic illustration of experimental procedure

## 3. Result and Discussion

During chemical bath deposition, the nucleation and growth of $Co(OH)_2$ and $Ni(OH)_2$ crystallites proceed from a supersaturated solution, facilitated by the gradual evaporation of ammonia. This evaporation shifts the equilibrium of complexation and hydrolysis reactions, leading to the controlled precipitation of metal hydroxide species from their respective $Co^{2+}$ and $Ni^{2+}$ ions [68]. The underlying mechanism of film formation can be described by the following reactions (Park *et al.*, 2020):

Upon dissolution of $NiSO_4 \cdot 6H_2O$ and $CoSO_4 \cdot 7H_2O$ in $NH_4OH$, complex ions $[Ni(NH_3)_6]^{2+}$ and $[Co(NH_3)_6]^{2+}$ are formed, typically appearing as purplish-blue and brownish-red solutions, respectively, as described by Equation (2). Upon heating, the liberation of $NH_3$ shifts the equilibrium, resulting in the release of free $Ni^{2+}$ and $Co^{2+}$ ions, which subsequently react with hydroxide ions to yield green $Ni(OH)_2$ and pink $Co(OH)_2$ precipitates, according to Equation (3).

To study the effect of deposition duration on film growth, time intervals of 10, 15, 20, 25, and 30 minutes were employed. Deposition time is known to strongly influence film thickness and morphology in CBD-derived films (Jin *et al.*, 2020). As shown in Figure 2, an increase in deposition time correlates with an increase in film mass per unit area, indicating greater nucleation and accumulation of hydroxide crystallites on the substrate surface. Specifically, the mass loadings for the mixed $Co(OH)_2/Ni(OH)_2$ films at 10, 15, 20, 25, and 30 minutes were 6.08 x $10^{-4}$; 6.00 x $10^{-4}$; 6.72 x $10^{-4}$; 7.04 x $10^{-4}$; and 7.20 x $10^{-4}$ g/cm$^2$, respectively. For the bilayer $Co(OH)_2/Ni(OH)_2$ films, the respective masses were 5.86 x $10^{-4}$; 6.13 x $10^{-4}$; 7.6 x $10^{-4}$; 1.04 x $10^{-3}$; and 1.63 x 10-3 g/cm$^2$.

The electrochromic performance, particularly ion intercalation/extraction behavior, is closely related to film thickness (Patil *et al.*, 2006). Thicker films provide more active sites and increased ion storage capacity, thereby enhancing optical modulation.

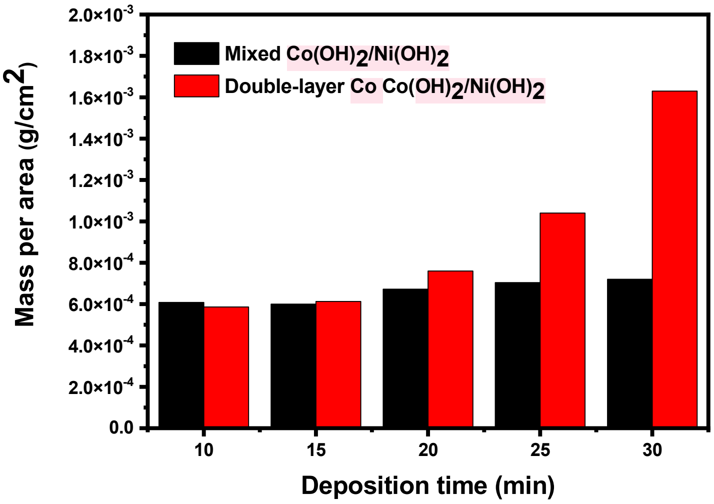


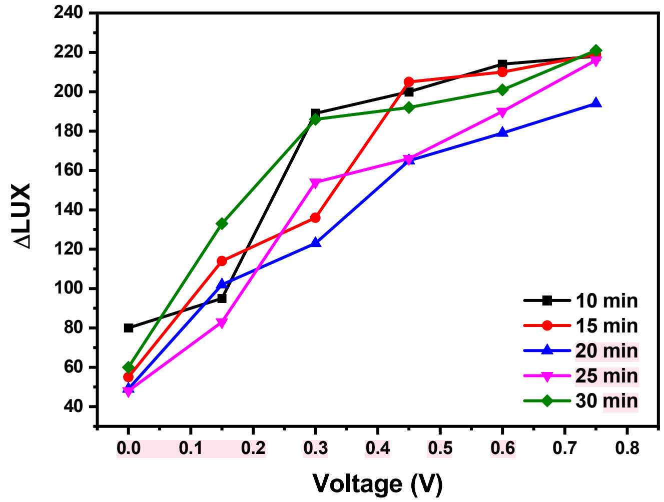

**Figure 2.** Mass per area mixed dan double-layer $Co(OH)_2/Ni(OH)_2$

The light transmittance modulation of the electrochromic films was evaluated by measuring the difference in illuminance (ΔLUX) between the unblocked light source and the light intensity transmitted through the $Co(OH)_2/Ni(OH)_2$ coated substrate. As illustrated in Figures 3 (a) and (b), both mixed and bilayer configurations exhibited a clear trend: ΔLUX increased with increasing applied voltage, indicating a corresponding decrease in optical transmittance. This behavior reflects a stronger light-blocking capability at higher redox states, which is attributed to the progressive oxidation of $Ni^{2+}$ to $Ni^{3+}$ and $Co^{2+}$ to $Co^{3+}$. Such redox transitions intensify coloration and confirm the increased electrochromic activity of the films.

Deposition time significantly influences ΔLUX by affecting the film's thickness and density. Longer deposition durations result in thicker films, which not only host a greater concentration of redox-active species but also exhibit more pronounced color changes during electrochemical switching. This enhancement in optical modulation correlates with increased mass loading, as discussed earlier.

Both the mixed and double-layer $Co(OH)_2/Ni(OH)_2$ films demonstrated multistep coloration behavior, attributed to the presence of $Co(OH)_2$, which introduces an additional redox potential distinct from that of $Ni(OH)_2$. Notably, $Co(OH)_2$ has a more negative oxidation potential (−0.22 V) compared to $Ni(OH)_2$ (+0.52 V), making it more readily oxidized under an applied bias. The coloration process is governed by the oxidation of $Co^{2+}$ and $Ni^{2+}$ to their respective trivalent states ($Co^{3+}$ and $Ni^{3+}$), which leads to a decrease in transmittance due to the formation of CoOOH and NiOOH, as described in the following redox reactions:

(a)

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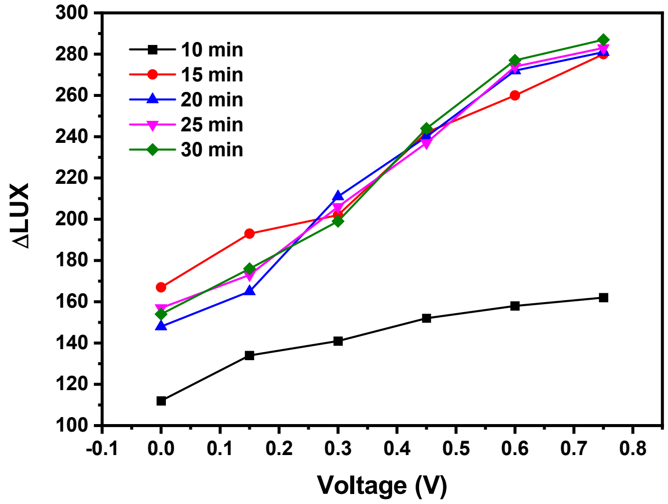


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1 **Figure 3.** Multistep coloration of mixed (a) dan double-layer (b) $Co(OH)_2/Ni(OH)_2$

23 The optical properties of the mixed and double-layer $Co(OH)_2/Ni(OH)_2$ electrochromic films were investigated using UV–Vis spectrophotometry, as presented in Figure 4. Optical modulation was assessed by measuring the change in transmittance (ΔT) at 0.15 V and 0.75 V, which characterizes the
21 film's ability to regulate light transmission through stepwise coloration. As the applied voltage increased from 0.15 V to 0.75 V, ΔT correspondingly increased, confirming the multistep electrochromic response of both film architectures.

The mixed $Co(OH)_2/Ni(OH)_2$ film exhibited superior optical modulation, with ΔT values of 11.10% and 50.14% at 0.15 V and 0.75 V, respectively. In contrast, the double-layer film demonstrated significantly lower modulation, with ΔT values of only 1.36% and 9.64% under the same voltage conditions. The improved performance of the mixed film can be attributed to its more uniform composition and optimized thickness, which facilitate better ion intercalation and redox activity. In the double-layer configuration, the greater overall film thickness may hinder ion diffusion, leading to reduced transmittance contrast and less efficient switching.

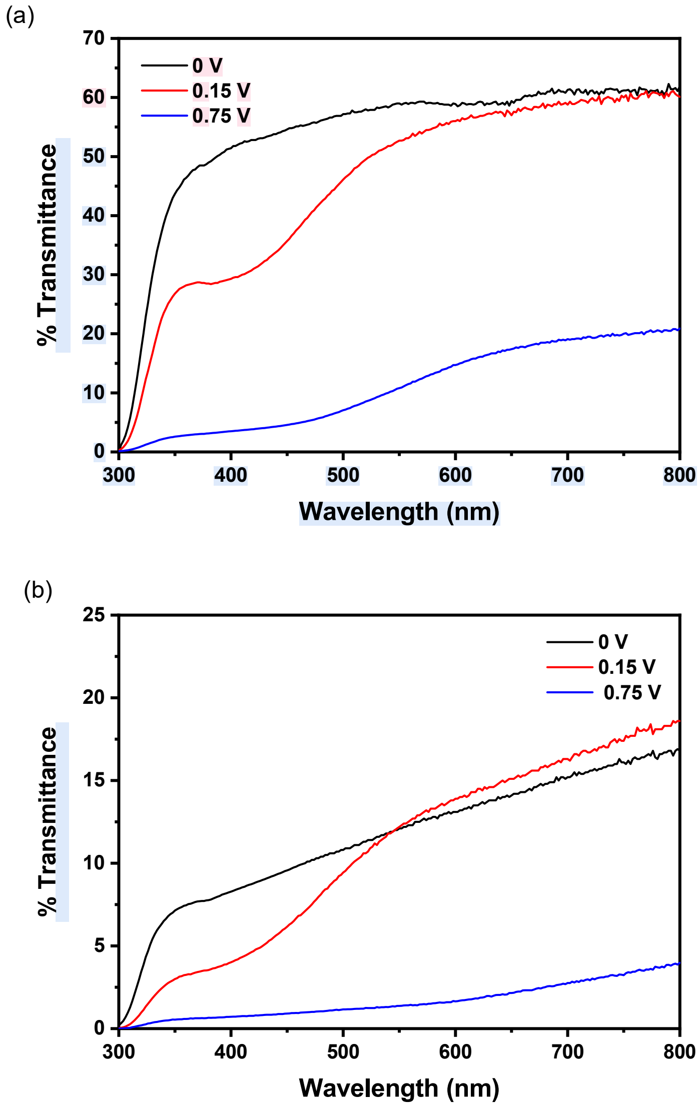


**Figure 4.** Transimttance spectrum of mixed (a) dan double-layer (b) $Co(OH)_2/Ni(OH)_2$

The surface morphology of the mixed and double-layer $Ni(OH)_2/Co(OH)_2$ films was examined by scanning electron microscopy (SEM), as presented in Figure 5. The mixed film exhibited a nanowall-like morphology, whereas the double-layer film displayed a nanoplate-like structure. These nanoscale architectures are advantageous for electrochromic performance, as they provide open channels and high surface area for efficient electrolyte penetration and ion exchange, thereby facilitating rapid and enhanced color switching (Wu and Yang, 2007). The observed morphological features are consistent with previous studies by Xia et al., who reported that electrochromic films synthesized via chemical bath deposition (CBD) exhibit superior ion intercalation pathways and more pronounced electrochromic behavior compared to those prepared by alternative methods. This highlights the efficacy of CBD in producing porous, electrochemically accessible microstructures. (Xia *et al.*, 2008).

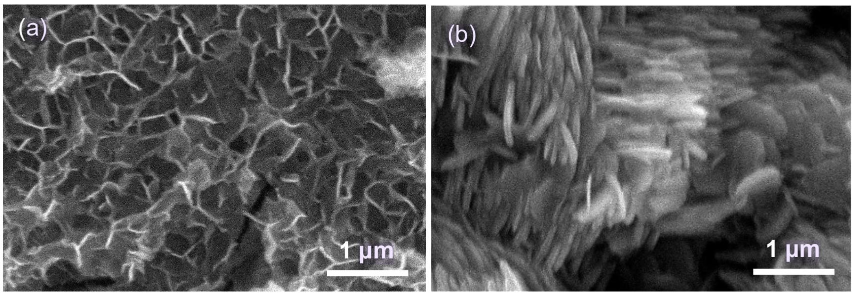


**Figure 5.** Surface morphology of mixed (a) dan double-layer (b) $Co(OH)_2/Ni(OH)_2$

The crystallographic properties of the mixed and double-layer $Co(OH)_2/Ni(OH)_2$ films were analyzed using X-ray diffraction (XRD), as shown in Figure 6. The XRD pattern of the double-layer $Co(OH)_2/Ni(OH)_2$ film exhibits well-defined diffraction peaks corresponding to both $Co(OH)_2$ and $Ni(OH)_2$ phases. Characteristic reflections of $Ni(OH)_2$ were observed at $2\theta \approx 38.46°$ and 51.90°, corresponding to the (011) and (012) planes, respectively, and are consistent with the JCPDS reference card PDF# 01-074-2075 (Ni *et al.*, 2013). In parallel, peaks attributable to $Co(OH)_2$ were identified at $2\theta \approx 19.52°$, 38.45°, and 51.95°, indexed to the (001), (011), and (012) planes, in agreement with PDF# 01-074-1057(Wang *et al.*, 2016). The absence of peaks associated with the ITO substrate (PDF# 00-039-1058) indicates that the conductive layer was fully covered by the hydroxide film, effectively suppressing the substrate signal.

These results confirm the successful formation of crystalline β-phase $Co(OH)_2$ and $Ni(OH)_2$ in the bilayer configuration. The presence of overlapping peaks near 38.45° and 51.90° from both $Co(OH)_2$ and $Ni(OH)_2$ suggests a degree of structural similarity between the two hydroxide phases, which may contribute to the uniformity of the electrochromic layer.

In contrast, the mixed $Co(OH)_2/Ni(OH)_2$ film exhibited no discernible diffraction peaks corresponding to either $Co(OH)_2$ or $Ni(OH)_2$, indicating a largely amorphous structure or an extremely thin film with insufficient crystallinity to yield detectable reflections. This result is consistent with previous observations that thinner films deposited via the CBD method may not exhibit prominent XRD features due to limited scattering volume or poor long-range order.

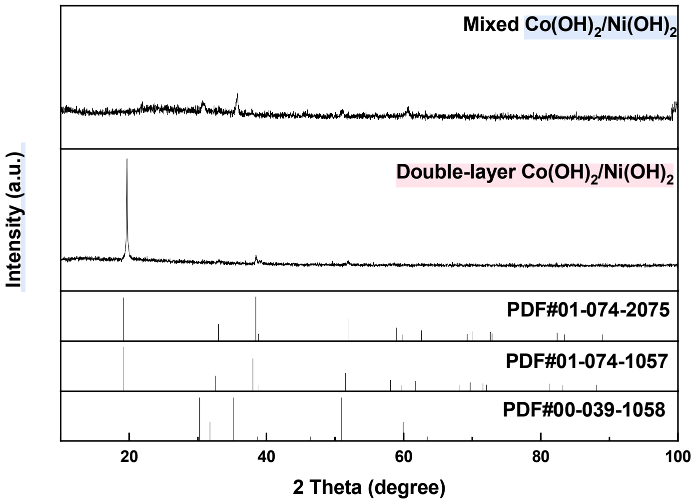


**Figure 6.** Diffraction patterns of mixed dan double-layer $Co(OH)_2$ and $Ni(OH)_2$

## 4. Conclusion

$Co(OH)_2$ and $Ni(OH)_2$ electrochromic films were successfully synthesized via chemical bath deposition. The deposition duration was found to significantly influence film thickness and, consequently, electrochromic performance. Two structural configurations, mixed and double-layer, were fabricated and evaluated for their optical and structural characteristics. Both exhibited multistep coloration behavior under applied voltage, enabling dynamic control of light transmittance. The mixed $Co(OH)_2$ and $Ni(OH)_2$ films demonstrated superior optical modulation compared to their double-layer counterparts, primarily due to their more favorable thickness, which facilitated greater contrast between bleached and colored states. SEM analysis revealed distinct nanostructures, nanowalls in the mixed films and nanoplates in the bilayer films, both of which promote ion diffusion and redox activity. XRD analysis confirmed the formation of crystalline β-phase $Co(OH)_2$ and $Ni(OH)_2$ in the double-layer films, while the mixed films exhibited limited crystallinity, likely due to reduced film thickness. These findings highlight the potential of mixed $Co(OH)_2/Ni(OH)_2$ films as tunable, cost-effective electrochromic materials for smart window applications.

## Acknowledgement

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