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Synthesis, characterization and environmental applications of Zn-Al and Co-Al mixed oxides derived from layered-like precursors

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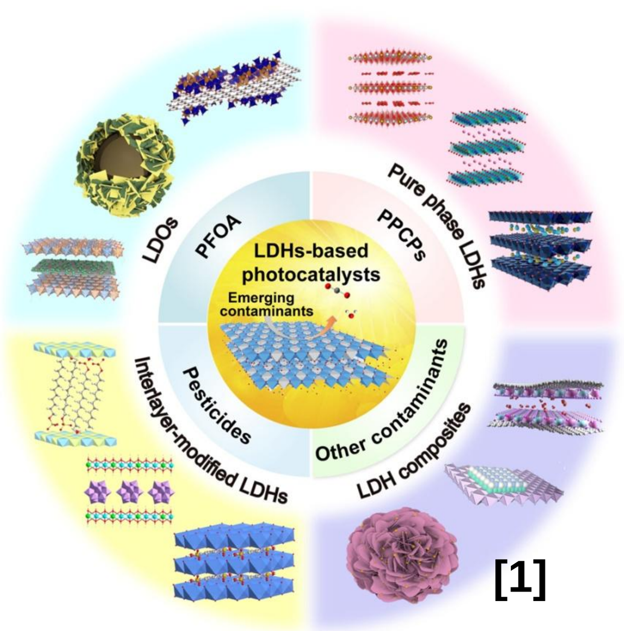
INTRODUCTION & AIM

Development of cost-effective advanced nanomaterials with tailored surfaces and bulk properties play a fundamental role in enhancing the efficiency for environmental and energy-related applications.



Layered double hydroxides (LDHs)

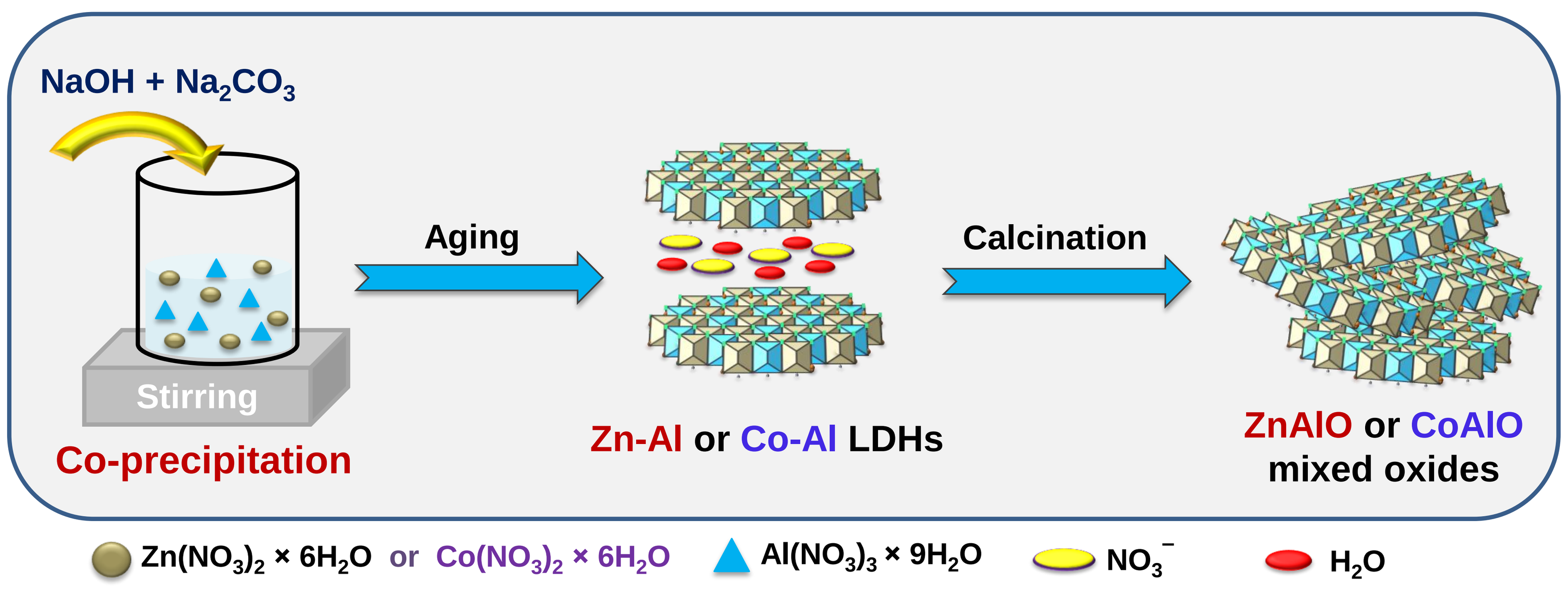
- ✓ a family of **two-dimensional materials;**
- ✓ **$[M^{2+}_{1-x}M^{3+}_x(OH)_2]^{x+}(A^{n-})_{x/n}\cdot mH_2O$** formula, where **$M^{2+}$, M^{3+}** are di-, and trivalent cations, **A^{n-}** is an anion.
- ✓ **outstanding physical and chemical properties**[1,2] being widely **used in various fields** (e.g., drug delivery, clean energy, catalysis).

Controlled thermal decomposition of the **LDHs** precursors → **mixed oxide catalysts with semiconducting properties** and **desirable features** (e.g. high specific surface area, thermal stability, homogeneity of metals).

This study describes a facile route to prepare Zn-Al and Co-Al mixed oxides from LDH precursors, their characterization and the evaluation as photocatalysts for the degradation of phenol.

METHOD

Both **Zn-Al** and **Co-Al LDH precursors** were prepared by a **co-precipitation** route at constant pH. The corresponding metal nitrates (molar ratio M^{2+}/M^{3+} = 3) were mixed together with a precipitating solution (1M NaOH & 0.2M Na_2CO_3) and kept under continuous stirring. After complete precipitation, the slurry was **aged at 75 °C** for 12 h; then washed and dried at 110 °C overnight prior to the **calcination at 650 °C** for 6 h, in air atmosphere **(see Scheme 1)**.



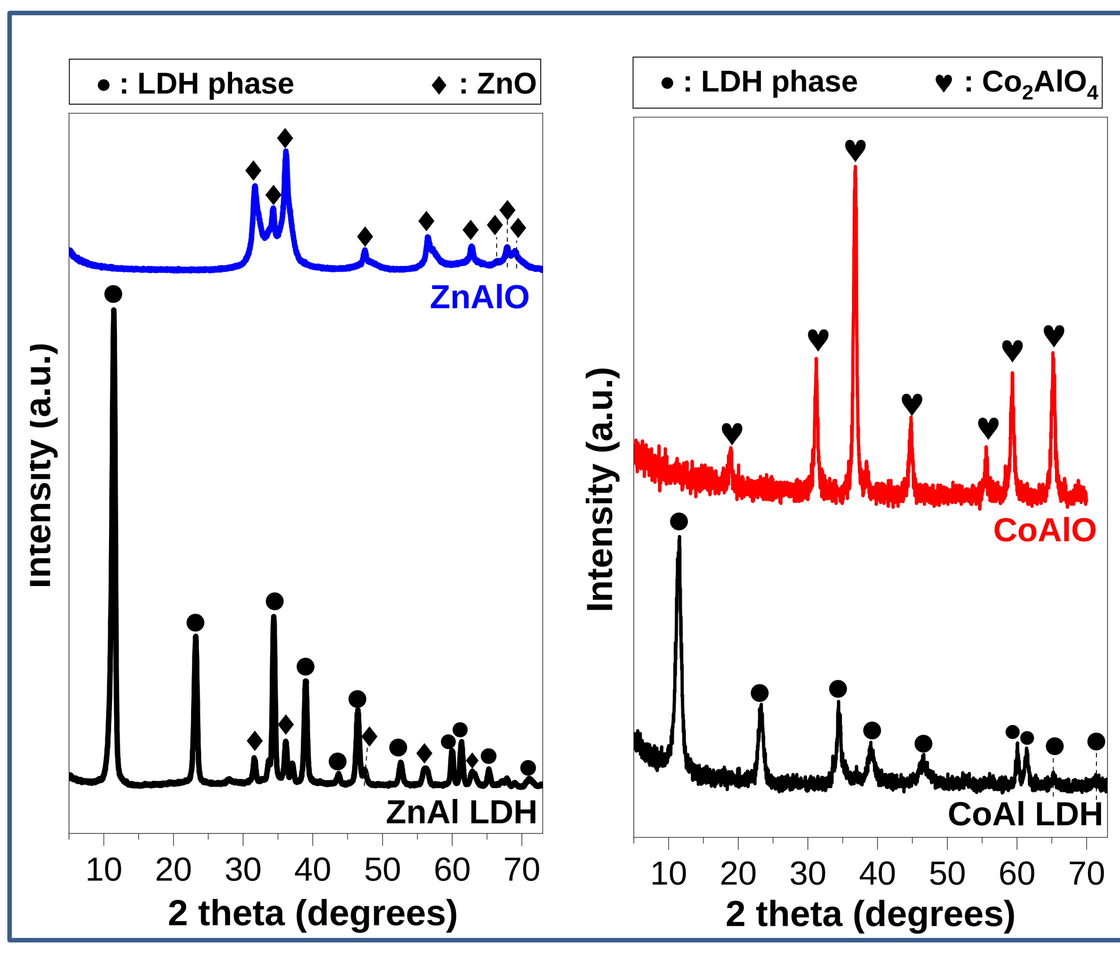
Scheme 1. Synthesis steps for the fabrication of Zn(Co)AlO mixed oxides derived from LDH precursors

The obtained Zn(Co)AlO mixed oxides were characterized by several techniques revealing their physical and chemical properties.

CONCLUSION

- ➢ **Zn(Co)Al mixed oxides** issued from **LDH precursors** were successfully prepared by the co-precipitation route**;**
- ➢ **After the calcination** of the **ZnAl LDH, pure ZnO phase** was obtained, whereas **Co-based LDH** lead to **Co_2AlO_4 spinel** phase**;**
- ➢ Both **Zn(Co)AlO solids** maintained a **platelet-like morphology** issued from the original LDH precursors**;**
- ➢ **CoAlO** mixed oxide present **a lower band gap** than that of **ZnAlO** solid;
- ➢ The **CoAlO** catalyst displayed higher photocatalytic performance for the phenol degradation compared to ZnAlO material. This behavior is due to its lower band gap, allowing for better light absorption and improved charge separation.

RESULTS & DISCUSSION

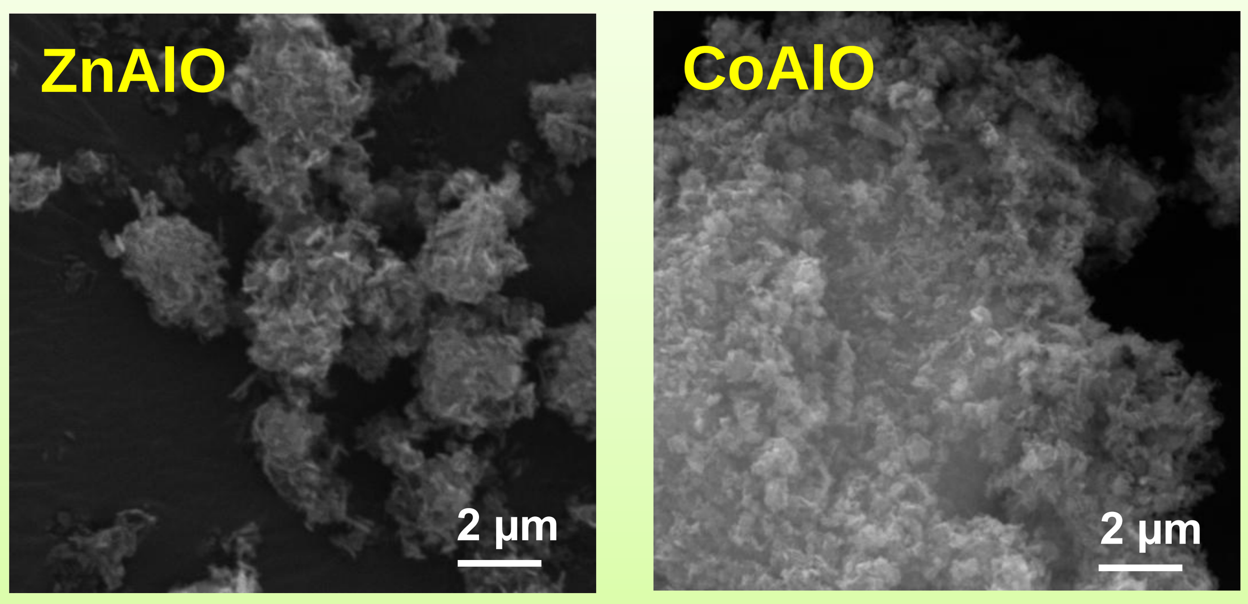


XRD pattern showed:

- **ZnAl LDH sample** - characteristic lines of layered double hydroxide (LDH) phase and a segregated ZnO;
- **CoAl LDH sample** - pure LDH phase;

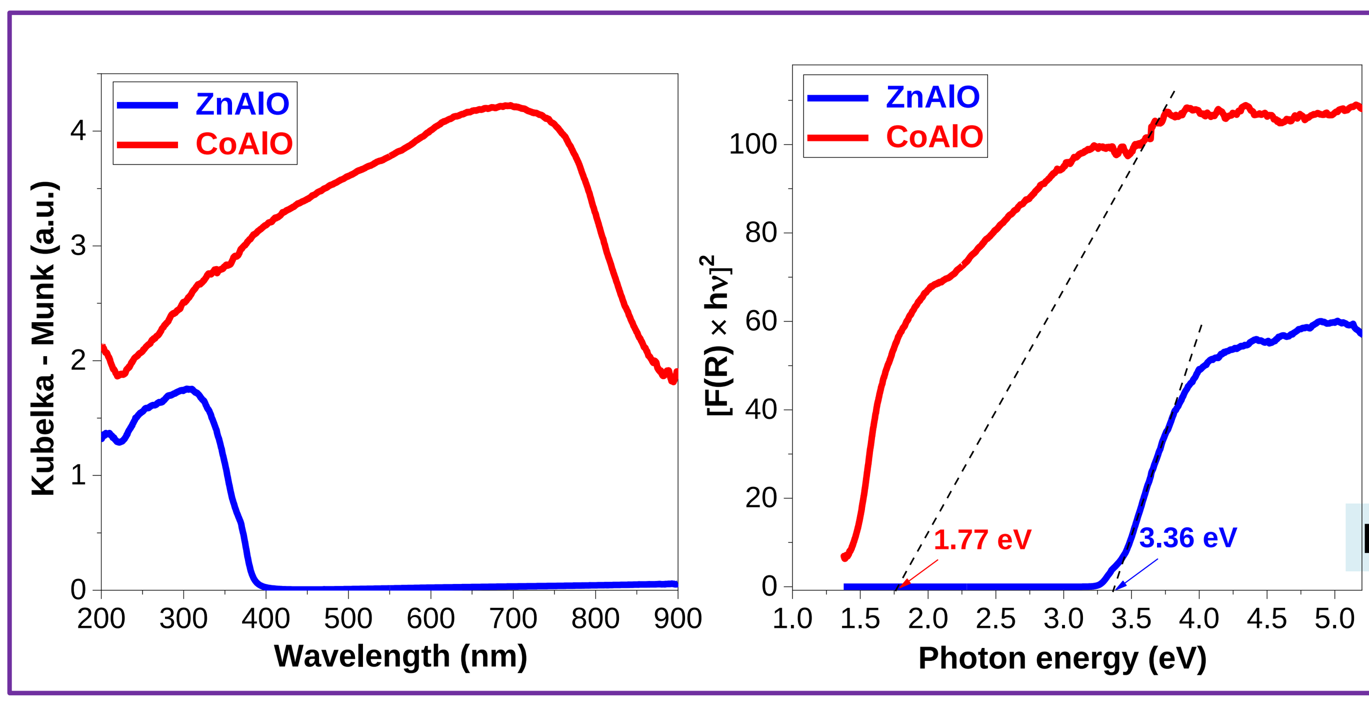
➢ **After LDHs calcination at 650 °C, for 5 h, in air atm.:**

- **ZnAlO sample** – obtaining of ZnO crystalline phase
- **CoAlO sample** - producing of Co_2AlO_4 spinel phase.



SEM images:

Revealed that **Zn(Co)AlO mixed oxides** displayed a platelet-like morphology preserved from the parental LDH precursors.

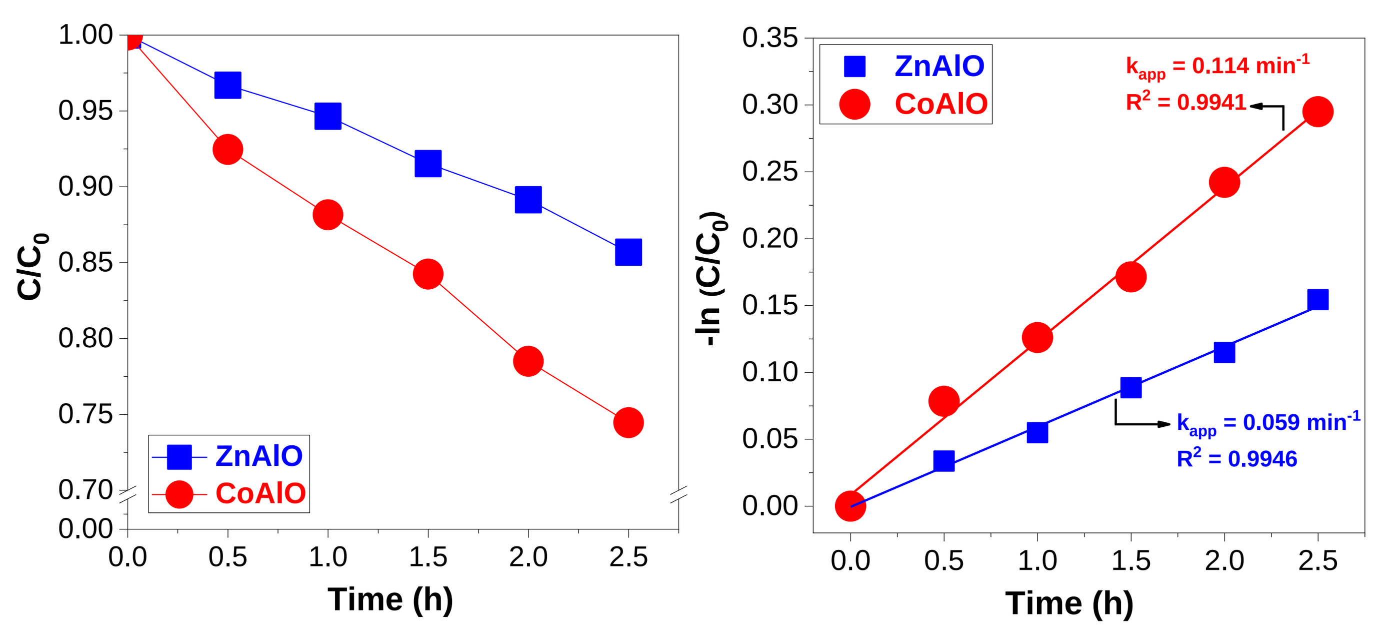


UV-Vis spectra:

Absorption edges associated with electron transfer from the VB to the CB

E_g CoAlO < E_g ZnAlO

Photocatalytic degradation of phenol under simulated solar irradiation



Experimental:

- $m_{catalyst}$ = 0.050 g
- C_{Phenol} = 25 mg/L
- T = 20 °C
- V sol = 110 mL

- **Higher photocatalytic activity** for phenol degradation over **CoAlO** solid compared to **ZnAlO** material;
- **Pseudo-first order kinetic model** with an apparent rate constant (K_{app}) **greater** for **CoAlO** than the **ZnAlO** catalyst.

FUTURE WORK / REFERENCES

- ✓ Variation of the phenol concentration, light intensity, and mass of photocatalysts;
- ✓ Stability runs and TOC measurements need to be completed**.**

[1]. L. Luo et al. *Catalysts* (2024), *14(4)*, 252. https://doi.org/10.3390/catal14040252.
[2]. J. Prince. *Appl. Catal. B. (2015),* 163, 352-360. https://doi.org/10.1016/j.apcatb.2014.08.019.