Synthesis of Ni-Co Layered Double Hydroxide

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INTRODUCTION

- Water splitting is becoming a more common method to convert energy due to its sustainability and limited negative impacts to the environment.
- The production of hydrogen and oxygen fuel through water splitting requires catalysts for the oxygen evolution reaction (OER) portion of the reaction as it is inefficient and slows the overall reaction down. (Song & Hu, 2014)
- Catalysts used currently are commonly composed of precious/noble metals. These are expensive and not abundant on earth. (Song & Hu, 2014)
- One application of layered double hydroxide (LDH) nanosheets are as a catalyst in these water splitting reactions.
- LDHs are ionic lamellar compounds made up of brucite-like layers with anions in between to balance charge. (Wang & O'Hare, 2012)
- These LDH compounds can be separated into nanosheets as it is the most functional way to use them.

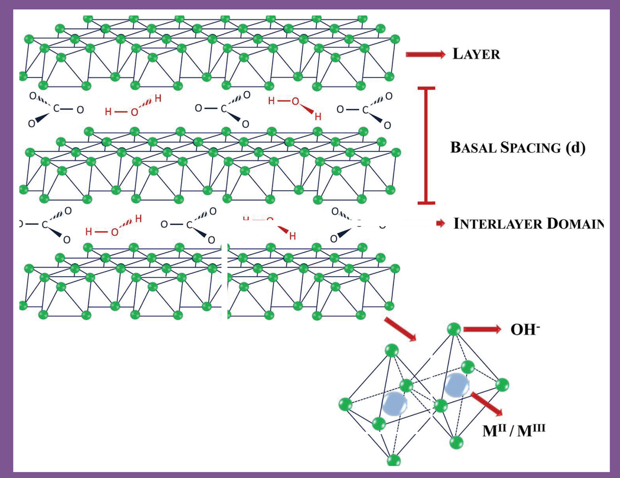


Figure 1: Layered double hydroxide structure (Benício et al, 2015)

OBJECTIVES

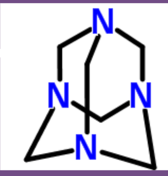
- To synthesize a layered double hydroxide catalyst using transition metals that is more efficient and cheaper than the catalysts used currently.
- To repeat an experiment and obtain similar results.
- Investigate the effect that changing the concentration of HMT has on the experiment.

METHODS

- The LDH was synthesized using the chemicals $CoCl_2 \cdot 6H_20$, $NiCl_2 \cdot 6H_20$ and hexamethylenetetramine (HMT) in deionized (DI) water.
- HMT was used in 2 different concentrations: 45mM (100%) and 4.5mM (10%).
- The solution was then purged with argon gas for ~3 hours before it was put under magnetic stirring and heating at ~90°C for 5 hours in an oil bath, still under an argon atmosphere.

METHODS

- The coprecipitation of these chemicals took place through HMT hydrolysis. (Fig. 2)



$$+ 6H_2O \rightarrow 6HCHO + 4NH_3$$

$$NH_3 + H_2O \rightarrow NH_4^+ + OH^-$$

Figure 2: HMT Hydrolysis Equation

- This hydrolysis of the water allows for OH^- to form and combine with the Co^{2+} and Ni^{2+} ions.
- After 5 hours, the precipitate was recovered through filtration using 0.45µm filter paper and rinsed with DI water and anhydrous ethanol.
- The precipitate was then air-dried at room temperature.
- After it was dried, we prepared samples for characterization tests.

RESULTS

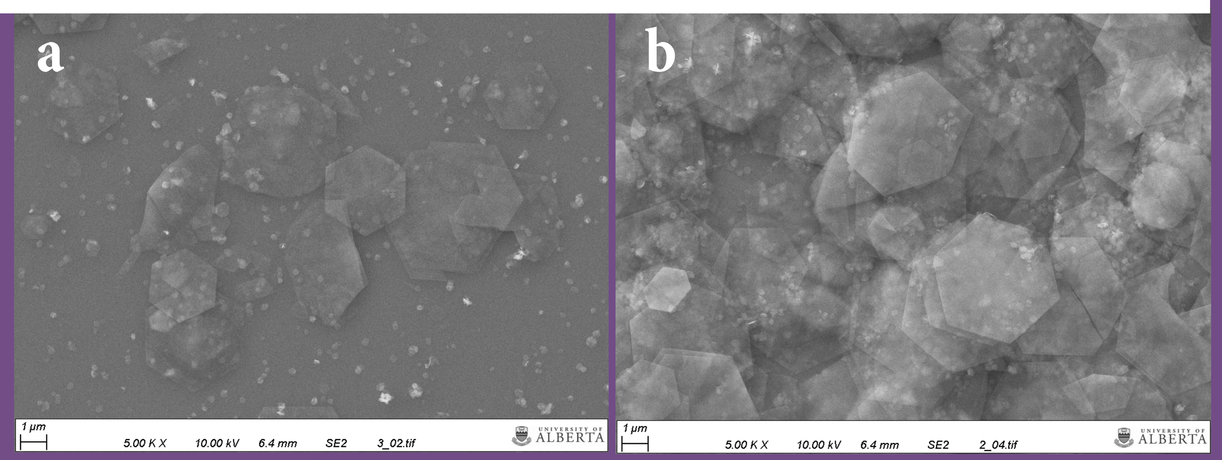


Figure 3: (a, b) SEM images of the samples with 100% HMT

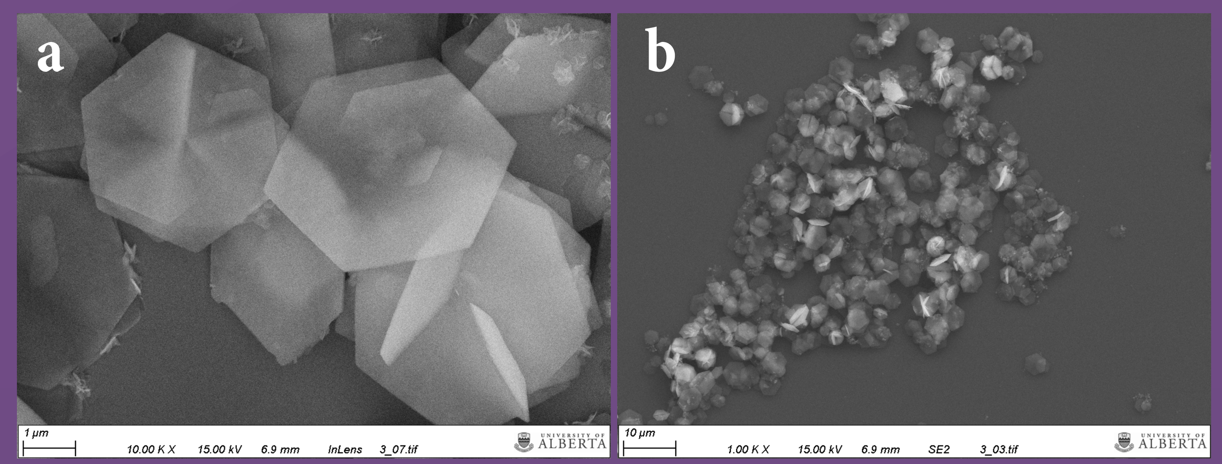
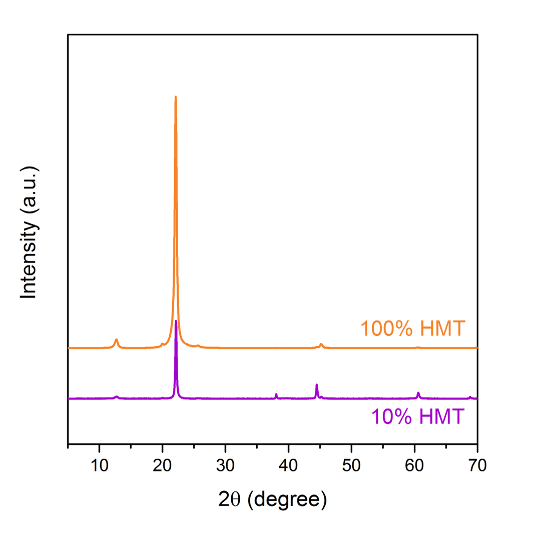


Figure 4: (a, b) SEM images of the samples with 10% HMT

- Some modifications had to be made to the experiment including: purging the solution with gas for a few hours before beginning the heating and stirring. This is important to expel the air out of the flask and obtain the hexagon shape for the samples.
- The scanning electron microscope (SEM) images show that the Co-Ni hydroxide samples have formed thin hexagonal platelets.
- XRD graph shows the structure of the sample. EDS graph shows what elements are in the samples.
- In Figure 5, high sharp peaks mean that the sample is highly crystallized.

RESULTS

Figure 5: X-ray diffraction (XRD) patterns of 100% (45mM) and 10% (4.5mM) HMT



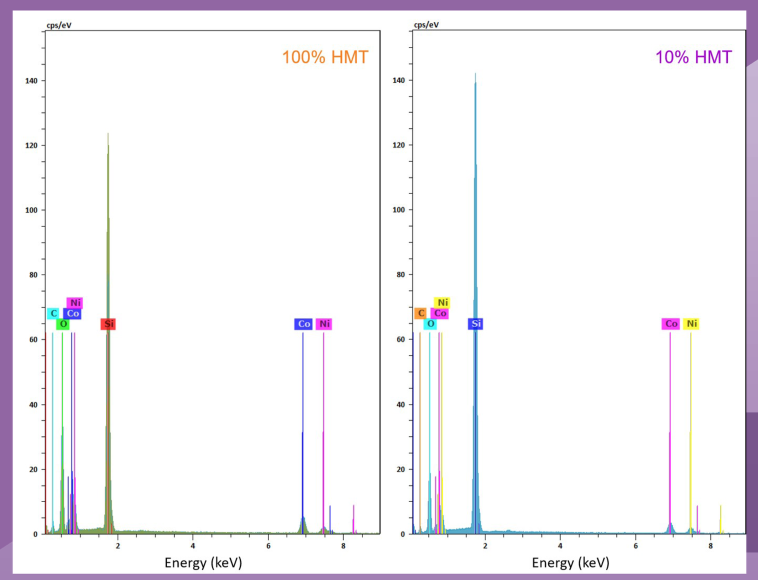


Figure 6: Electron diffraction spectroscopy (EDS) graph of 100% and 10% HMT.

CONCLUSIONS

- These results show that we could successfully synthesize Ni and Co and obtain samples that are highly crystalized and have uniformly shaped hexagonal platelets.
- We discovered that changing the concentration of HMT influenced the samples. Using 100% HMT yielded thinner platelets with more small particles. Using 10% HMT yielded thicker platelets with a more uniform shape.

LITERATURE CITED

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