

1 Experiment

1.1 Purpose of experiment

To make a sample of **2.5%NiO-MgO** precursor, following the details from MM Millet, adapted to the Optimax

Millet, Marie-Mathilde, et al. "Ni single atom catalysts for CO2 activation." Journal of the American Chemical Society 141.6 (2019): 2451-2461.

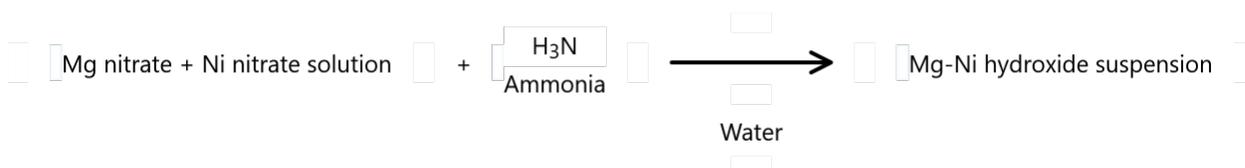
1.2 Pre-conditions

None

1.3 Conclusions

A white-pale green suspension is obtained, which is then filtered and centrifuged, giving a blue solution (not avoidable, formation of Ni(NH₃)₆ complex)

2 Chemistry



Chemical	Type	Mol Weight	Stoic. Coeff	Actual Moles	Actual Amount	Conc.	Comment
Mg nitrate + Ni nitrate solution	Reagent	NaN g/mol	1	NaN	300.18 g	100 %	
Ammonia	Reagent	17.031 g/mol	1	17.627	300.2 g	100 %	
Water	Solvent	18.02 g/mol	-	11.099	200.34 ml	100 %	
Mg-Ni hydroxide suspension	Product	NaN g/mol	1	NaN	0 g	100 %	

Results of Reactions	Value
Actual Yield Reaction 1	n/a
Total Est. Actual Weight	700 g
Total Est. Actual Volume	860.85 ml

3 Setup

OptiMax 1001	Description
Device	OptiMax 1001 (Serial #: C051747718 / Firmware: 6.1.2.2)
Reactor	1000 ml
Stirrer	Overhead (Pitched-blade down, C22, Ø 45mm / Standard shaft, C22, Length 400mm)
Other	Tr Sensor

UCB	Description
Sensors	pH input 1 : pH prob2, Trend Name = pH1 (pH)
Dosing Controller	Item name : Dosing1 Output port : 1 A : Prominent pump Balance port : COM 1 Temperature : constant at 60°C Dosed amount is added to Mr.
Dosing Controller	Item name : Dosing2 Output port : 2 B : Prominent pump Balance port : COM 2 Temperature : constant at 60°C Dosed amount is added to Mr.

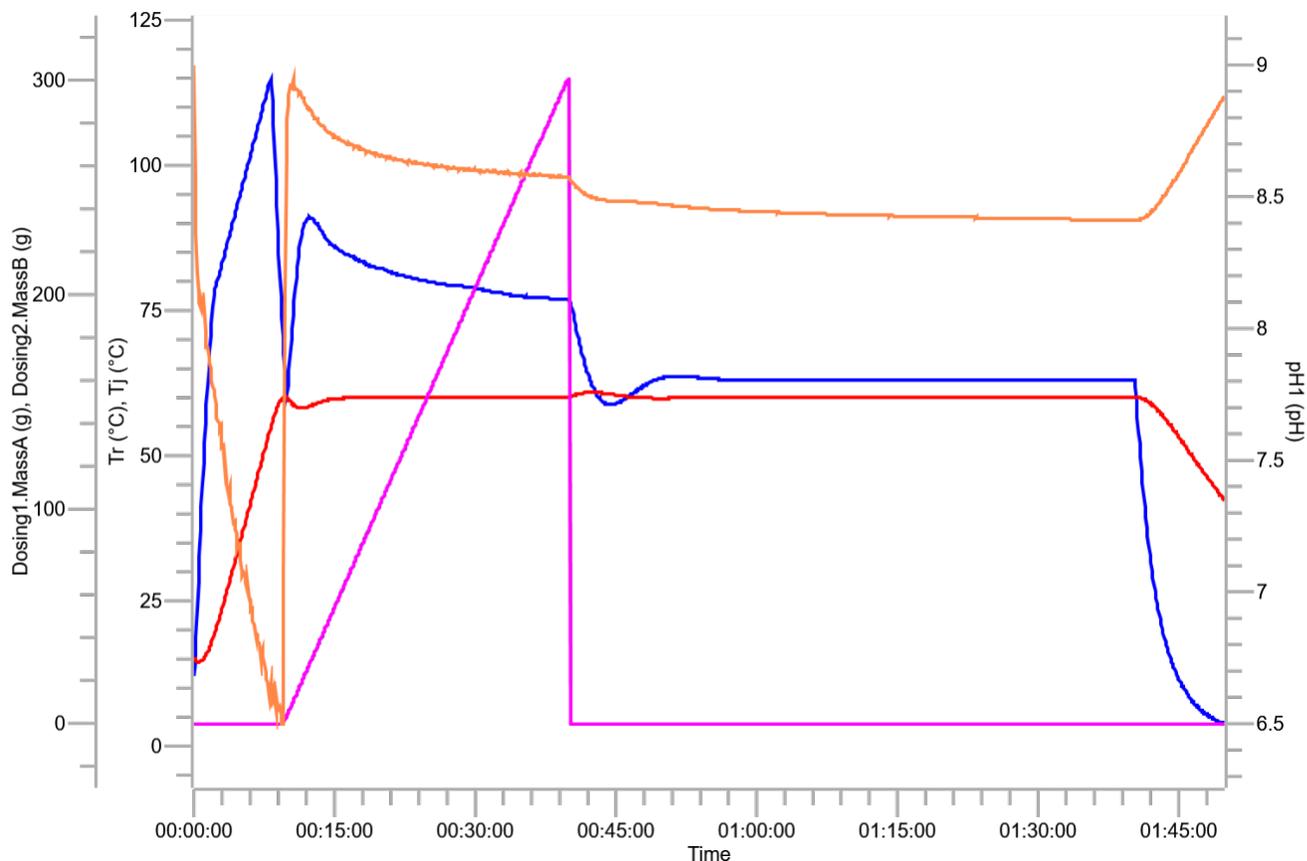
4 Recipe

#	Action / Note / Sample	Start Time	End Time
1	Start of experiment on 09.02.2022 at 11:15:15 with thermostat off and stirrer off Details: Initially the operation started as 'First Fill and Safety Limits '	00:00:00	00:00:05
2	Add 200.34 ml of Water at once	00:00:05	00:00:21
3	Ramp stirrer speed to 300 rpm over 10 sec	00:00:06	00:00:17
4	Heat Tr to 60 °C as fast as	00:00:06	00:09:09

	possible		
	Phase 2: Dosing phase Start condition: Tr ≥ 60 °C Details: After -00:00:25, Waiting for start condition before starting phase.	00:09:35	00:40:08
5	Dose 300 g of Ammonia at 10 g/min using Dosing2	00:09:35	00:40:08
6	Dose 300 g of Mg nitrate + Ni nitrate solution at 10 g/min using Dosing1	00:09:35	00:40:08
	Phase 3: Aging phase	00:40:08	01:40:08
7	Wait 60 min	00:40:08	01:40:08
	Phase 4: Cooling	01:40:08	01:49:53
8	Cool Tr to 25 °C as fast as possible Details: After 00:09:43, Operation was aborted as the Experiment was aborted.	01:40:08	01:49:52
9	Ramp stirrer speed to 200 rpm over 10 sec	01:40:08	01:40:21
10	End of experiment on 03.08.2022 at 13:39:57 with Tj set to 25 °C and R set to 100 rpm Details: Initially the operation started as 'End Experiment Tj set to 25 °C and R set to 100 rpm'	01:49:52	01:49:52

5 Trend Graphs

Trends



Trend	Color	Units
Tr	—	°C
Tj	—	°C
Dosing1.MassA	—	g
Dosing2.MassB	—	g
pH1	—	pH

#	Action / Note / Sample
	Phase 2: Dosing phase Start condition: Tr ≥ 60 °C Details: After -00:00:25, Waiting for start condition before starting phase.
	Phase 3: Aging phase
	Phase 4: Cooling

6 Work up procedure

Sample is recovered and filtered with 15A, mother liquor is collected and conductivity is measured. After that, sample is put in 350 mL mqH₂O to stir for 15 min, then filtered, but most solid passed through the filter, even though it was 15A. Then, sample was put in centrifuge, 5000 rpm, 15 min, in 3 big beakers, each with 150-200 mL mqH₂O. Sample is then washed 2 more times, directly in the beaker (3 in total). Conductivity is measured after every step, and the final solid is put to dry in static air cabinet at 80 °C.

	Conductivity
Mother liquor	56,39 mS/cm
1st washing	1391 µS/cm
2nd washing	97,48 µS/cm
3rd washing	60,22 µS/cm