Preparation of nickel-coated gas diffusion electrodes for (bio)electrochemical ammonium recovery

Adapted from Hou et al. 2018[1]

Materials:

- Hydrophobic polypropylene (PP) membrane (0.22-μm, Tisch Scientific)
- Polyvinylidene fluoride (PVDF)
- Multiwall carbon nanotubes (MWCNTs, outer diameter: 8−15 nm, length: 10−50 μm, CheapTubes)
- Dimethylacetamide (DMAc)
- Nickel foam
- 150 mM NiSO4·7H2O, 25 mM NiCl2·6H2O, and 500 mM H3BO3 (at pH 2.00 ± 0.05)
- Titanium wire

Protocol:

1) Dissolve 0.5 g of PVDF and 1.0 g of MWCNT in 100 g of dimethylacetamide (DMAc) and stir with a magnetic stirrer for 24 h
2) Ultrasonicate the dispersions for 2 h
3) Cut a 12cm x 12 cm piece of the PP membrane (always use gloves when touching the membrane)
4) Spray the PVDF−MWCNT dispersion uniformly over the middle section of the PP membrane support leaving 2 cm of non-coated membrane to the sides with an airbrush
5) Dry overnight in an oven at 50 °C
6) Electrodeposition of nickel:
   a) Prepare a bath containing 150 mM $NiSO_4 \cdot 7H_2O$, 25 mM $NiCl_2 \cdot 6H_2O$, and 500 mM H_3BO_3 (at pH 2.00 ± 0.05)
   b) Connect an equal size nickel foam piece to a current collector and place it in the bath. This is used as the anode.
   c) Connect a current collector to the coated membrane and place it in the bath. This will work as the cathode. Place the electrodes close to each other, but make sure that they are not touching.
   d) Apply a constant DC current of 0.2 A for 24 h
   e) Rinse the prepared membrane electrode with deionized water to remove Ni salts and unbound Ni residues before drying overnight at 50 °C

[1] Hou, D., Iddya, A., Chen, X., Wang, M., Zhang, W., Ding, Y., ... & Ren, Z. J. (2018). Nickel-based membrane electrodes enable high-rate electrochemical ammonia recovery. Environmental science & technology, 52(15), 8930-8938.