Mem4Bat

Sustainable membrane-based recovery of solvents for battery manufacturing

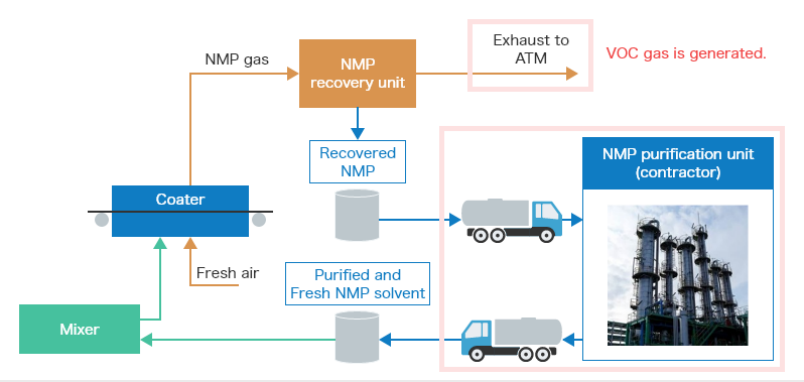
Mem4Bat aims to explore membrane filtration for purification of spent solvents reclaimed from Li-ion battery manufacturing plants

THE CONTEXT

Liquid-ion Battery (LIB) manufacturers are using large amounts of solvents. Especially N-Methyl-pyrrolidone (NMP) is an indispensable auxiliary material for manufacturing of cathodes. After slurry coating NMP is evaporated in a drying step, yielding a hydrous NMP solution for recovery. The amount of this waste NMP/water mixture is varying from approx. 1 000 tons to more than 10 000 tons/year, depending on the size of the LIBs production site.

Today in Europe there are recovery solutions for this NMP waste, but all battery producers must sell the recovered NMP on the market for other uses as the purity is not high enough for reuse in LIB manufacturing. However, since the market for other uses is limited, and the production capacity of LIBs keeps on growing, there is a need for more efficient purification processes.

Indaver has developed a process to purify NMP from LIB manufacturing waste till battery grade specification using dedicated distillation units. Due to the very stringent specifications, especially on metal contaminations (typically 10 ppb), transport of purified NMP from these dedicated units to the LIB factory poses an additional challenge, with a significant risk of metal (re-)contamination. Therefore, Indaver is looking for alternative downstream purification technologies, capable of removing metal contaminations arising during LIB manufacturing, as well as during solvent processing and transport.



Recycling of spent NMP in LIB manufacturing involving a contractor as Indaver

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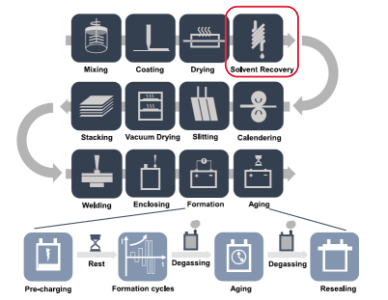
THE CHALLENGE

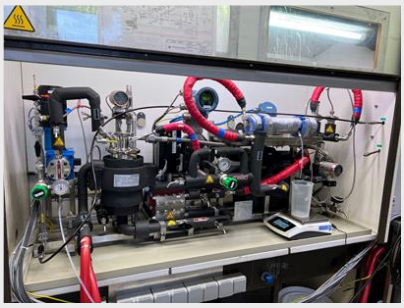
The Mem4Bat project will leverage on the successful developments and insights gained in the former EU project SOLVER where VITO together with several solvent re-refiners studied membranes for trace metal removal from electrochemical grade solvents, mainly alcohols. In this latter project, organic solvent nanofiltration (OSN) proved to be capable of removing 35 key trace metals till (sub-)ppb levels, which was demonstrated up to pilot scale.

However, purification of spent NMP poses significant challenges, as few membranes, even dedicated OSN membranes and modules, provide long-term stability in such aggressive polar aprotic solvents which are commonly used for membrane manufacturing as well. Only a small number of commercial membranes, including ceramic membranes, are robust enough without significant swelling, which is essential for selective removal of very small metal contaminations.

Moreover, the purity specifications are very low (ppb level), complicating characterisation and analysis of the NMP streams before and after purification. These stringent purity requirements also pose significant challenges for the membrane filtration installations and peripherals. Multi-step rinsing protocols will be essential here.



Schematic LIB manufacturing process



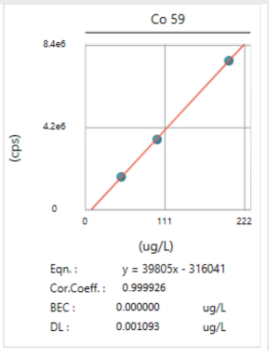
Membrane filtration installation at VITO

THE RESULTS

At first, a suitable external partner for ppb-level metal contaminant analysis in NMP was searched for. Various academic and commercial service labs were approached, in the end only one party was found to have the required capabilities and analytical instruments, showing the complexity of this analytical challenge. Even though significant progress was made in the method development, allowing for evaluation of the membrane separation trials, accurate analysis in the lower ppb range remains very challenging due to pronounced matrix effects.

First membrane screening tests were performed on an industrial spent NMP sample provided by Indaver. This stream proved to be particularly challenging due to its high contamination level. Results showed that membranes cannot cope well with this stream, with generally very low fluxes, while post-cleaning of equipment proved to be cumbersome. Therefore, a pre-treated NMP sample with only trace amounts of water and metals was sought by Indaver. On this sample, various membrane technologies, incl. OSN, but also pervaporation (PV) and membrane distillation (MD) are being tested, with first promising results regarding removal of metals and water.

Sample Id	Co 59 Ammonia DRC (ug/L)
Blank	
50	50.705
100	98.943
200	200.352
NMP vervuild 1-1000	372.4
NMP vervuild 1-3000	131.0
qc 50 ppb	49
qc 100 ppb	89
qc 200 ppb	178



ICP-MS analysis of typical crude NMP stream

CONCLUSION

The Mem4Bat project allows to draw the following promising conclusions:

- Analysis of metal traces at ppb levels proved to be very challenging, requiring more efforts and taking far more time than expected, however significant progress was made by an external lab, and a suitable method for evaluation of the membrane trials was developed.
- Direct processing of a highly contaminated crude spent NMP sample using UF/NF membranes was not successful.
- A clean pretreated NMP sample was sourced and various membrane technologies, incl. OSN, PV and MD have been tested on it, with encouraging results regarding removal of metals and water.

TECHNIQUES USED

In Mem4Bat the following services and capabilities of the INNOMEM OITB, all available at INNOMEM partner VITO, were used:

- Lab-scale testing of polymeric/ceramic membranes on real-life aggressive solvent streams under realistic conditions, requiring dedicated flexible crossflow installations.
- Proof-of-concept testing of integrated separation process using various membrane technologies and membrane types.
- First techno-economic analysis based on experimental result.

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