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Chemical recycling of PA6 from discarded fishing nets using choline chloride-ethanolamine (ChCl:MEA) as Deep Eutectic Solvent (DES)

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INTRODUCTION & AIM

The extensive use of synthetic polymers, especially in the fishing industry, has led to the accumulation of **non-biodegradable plastic waste** in marine ecosystems.

Around 10% of total marine plastic waste comes from abandoned polymeric fishing gear, mainly made of **polyamide 6** (PA6).

Highest proportion of macro- and mega-plastics (>50 cm) floating on the ocean's Surface.

PA6 is a versatile engineering polymer often used in fishing gear applications due to its good mechanical resistance, thermal stability, chemical durability and low costs. It is produced via ring-opening polymerization from the monomer **ε-caprolactam**

Recycling and recovery → Mechanical recycling / **Chemical recycling** → Solvolysis using Deep Eutectic Solvents (DES): Depolymerization using solvents to disrupt the polymer chains.

PA6 → Cleavage of amide bonds → Intramolecular cyclization → Formation of **ε-caprolactam**

DES → Hydrogen bond aceptor: Choline chloride (**ChCl**)
Hydrogen bond donor: Monoethanolamine (**MEA**)

Low-melting solvent

METHOD

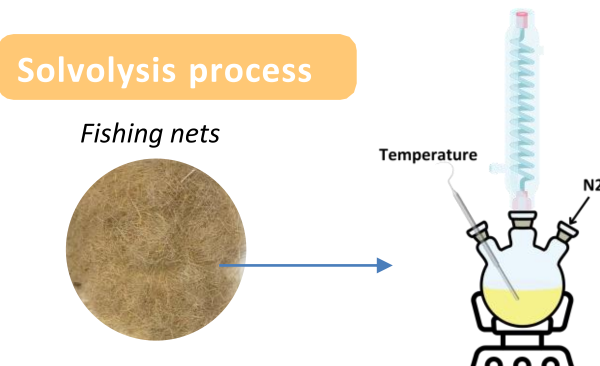
Materials

Fishing nets: Pretreatment by AIMPLAS Institute of Technology.

DES: Mixture of MEA (C_2H_7NO) 99% and ChCl ($C_5H_{14}ClNO$) ≥98%. Different molar ratios stirred at 80 °C for 1h.

Catalyst: 4-(dimethylamino)pyridine (DMAP)

Solvolysis process



Batch reactor under N_2 atmosphere, with EG-cooled reflux

Post-Solvolysis - Conversion

i. Dilution with 96% Ethanol 1:1 (PA6 precipitation).
ii. Filtration → PA6 dissolution efficiency.
iii. Washed with EtOH until no solvent remained in the filter cake.

Design of experiments (DoE) → **Central Composite Design (CCD)**

3 Factors and 2 replicates:

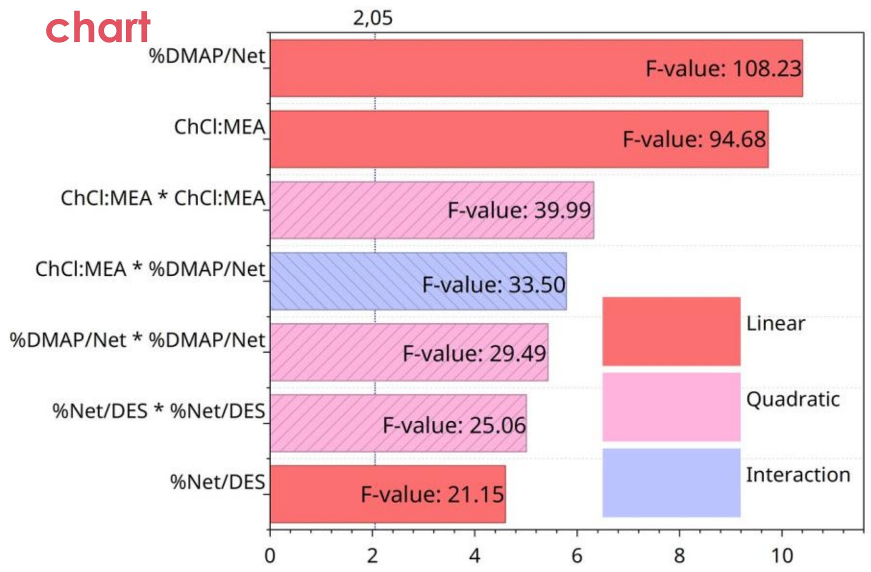
- Ratio of net mass to DES (%Net/DES):: 6.64 - 13.36 %
- Proportion of catalyst relative to net mass (%DMAP/Net): 0.95 - 11.05 %
- Molar ratio of ChCl to MEA in the DES (ChCl:MEA): 1:2.64 - 1:9.36

Fixed parameters: 158 °C, 2 h

Response variable: PA6 conversion

RESULTS & DISCUSSION

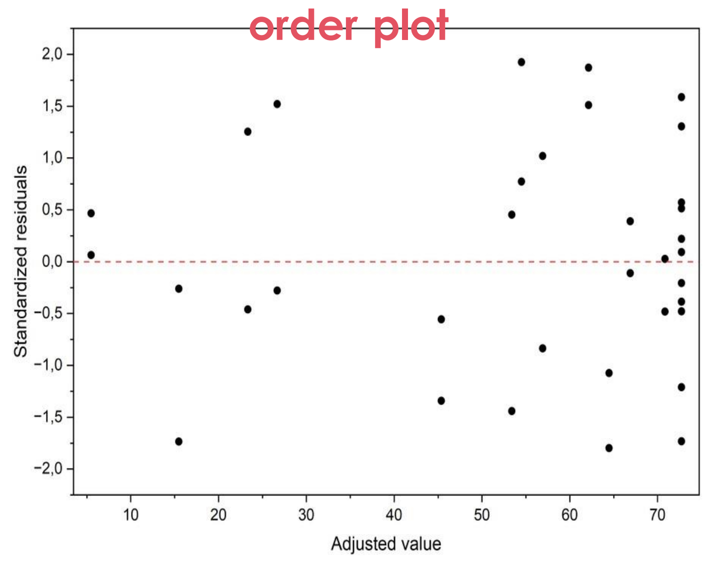
Pareto chart



The model obtained from the CCD is *statistically significant*: *p-value* = 0.00 (< 0.05)

- Main factors: **%DMAP/Net** and **ChCl:MEA ratio**
  Highest F-value ⟶ Most significant variables
- Significant interactions: ChCl:MEA × %DMAP/Net

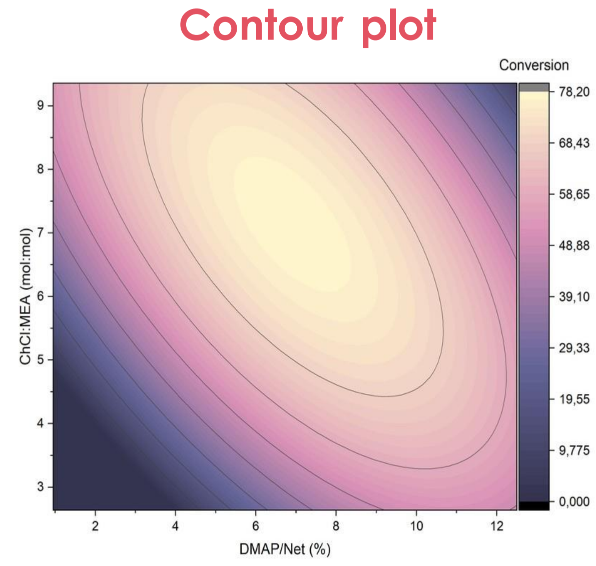
The residuals vs. observation order plot



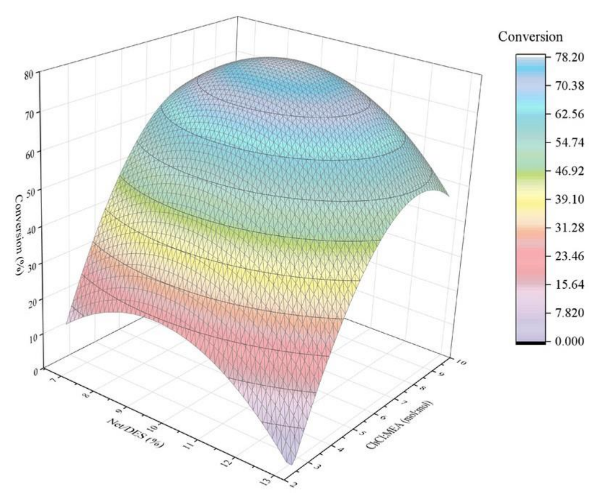
Random dispersion around zero. No systematic error or bias was present.

- Significant quadratic effects: $(ChCl:MEA)^2$, $(\%DMAP/Net)^2$
- Regression F-value = 48.47 ⟶Strong relationship between the factors and X(PA6)

Contour plot



Response surface plot



Statistically significant quadratic terms: Evidence of curvature, the optimum not achieved at the extremes.

Maximum PA6 conversion was achieved at intermediate levels of %Net/DES and ChCl:MEA ratio.

Optimized conditions:
9.29% (w/v) Net/DES
6.97% (w/w) Net/DMAP
1:7.12 ChCl:MEA

CONCLUSION

- ChCl:MEA-based solvolysis effectively enabled the chemical recycling of PA6 fishing nets under mild conditions.
- The second-order model was statistically significant and accurately predicted the depolymerization behavior.
- Catalyst concentration and DES molar ratio were the most influential factors.
- The optimized conditions yielded up to ~80% PA6 conversion.

FUTURE WORK / REFERENCES

- Conduct separation of DES and ε-caprolactam and purification for re-synthesis.
- Comparation of ionic liquids for PA6 depolymerization.

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