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Solvolytic Depolymerization of PA6 from Marine Waste Using Structurally Diverse Deep Eutectic Solvents

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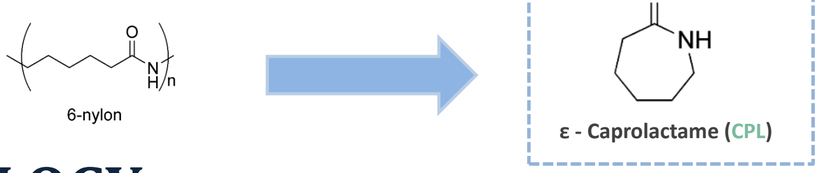
INTRODUCTION & AIM

The accumulation of **discarded fishing nets** in coastal zones significantly contributes to marine pollution, with **polyamide 6** (PA6) being the predominant polymer in these residues.

Chemical recycling through depolymerization offers a promising strategy to transform this waste into high-value building blocks, particularly **ε-caprolactam**, supporting circular economy initiatives while reducing ocean contamination.

In this study, **deep eutectic solvents** (**DESs**) were evaluated as green media for depolymerizing PA6 recovered from fishing nets.



Project

REDES4VALUE - Valorization of fishing nets for the production of high added value productS (INNEST/2024/313)

METHODOLOGY

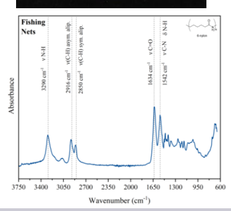
WP1. Definition of requirements and technical specifications.

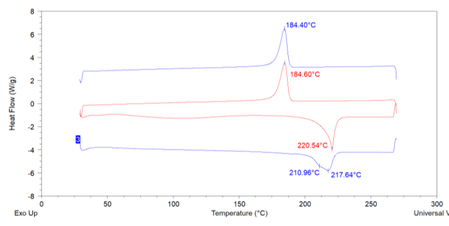
WP2.Mechanical recycling of waste and study of modification and properties modulation.

Discarded fishing nets collected in Ghana.

SEA2SEE

Initial physicochemical characterization





Melting point and typical FTIR spectra of polyamide 6, with some traces and impurities.

WP3. Chemical recycling and repolymerisation of the products.

Materials

Fishing nets: Pretreatment by AIMPLAS Institute of Technology.
Catalyst: 4-(dimethylamino)pyridine (DMAP)
DES: Mixture of MEA (C_2H_7NO) 99% , DEA ($C_4H_{11}NO_2$) 99% and ChCl ($C_5H_{14}ClNO$) ≥ 98%. Different molar ratios.

Design of experiments

Temperature
160, 170 and 180 ºC

Time
30, 60, 120 and 240 min

DESs
- ChCl:Urea [1:2]
- ChCl:DEA [1:6]
- ChCl:MEA [1:6]
- ChCl:Glycerol

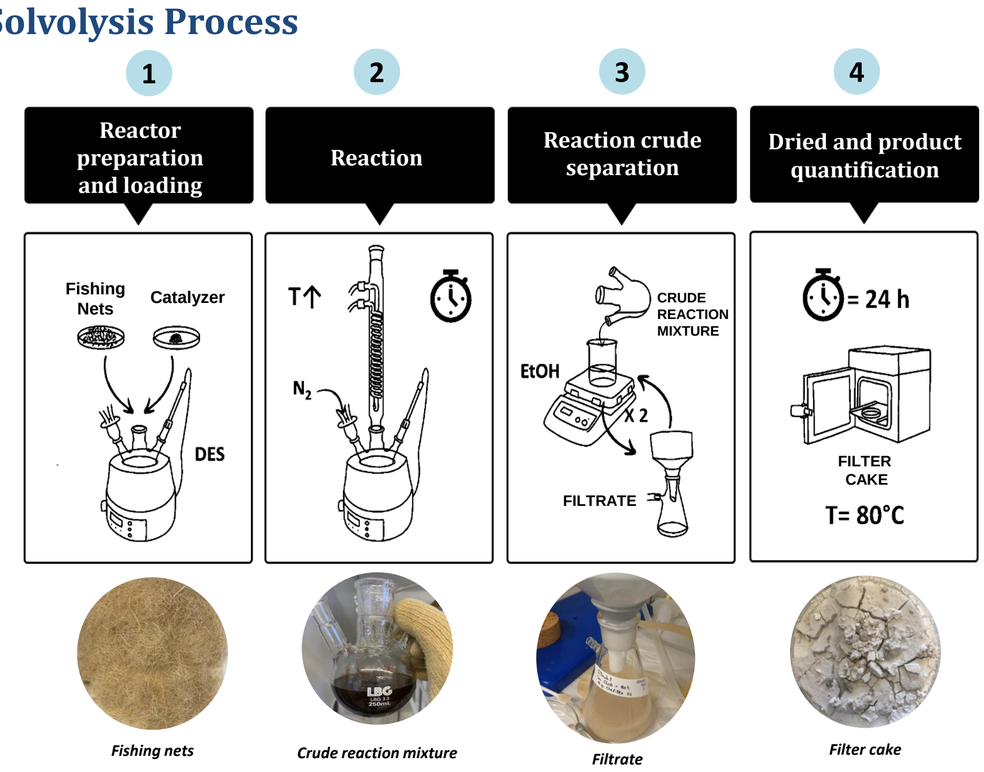
Characterization

Gravimetry

Fourier infrared Spectroscopy (FTIR)
Agilent Technologies Cary 630, 600-4000 cm^{-1}, 4 cm^{-1}, 32 scans

Differential Scanning Calorimetry (DSC)
Setaram Setline+, 40-300 ºC, 10 ºC·min^{-1}, N_2 atmosphere

Solvolysis Process

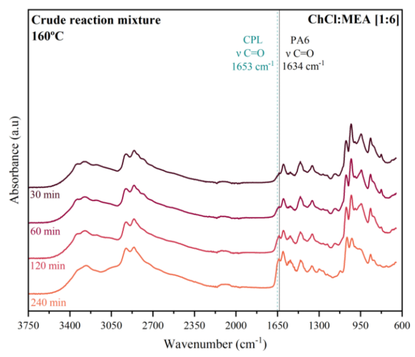


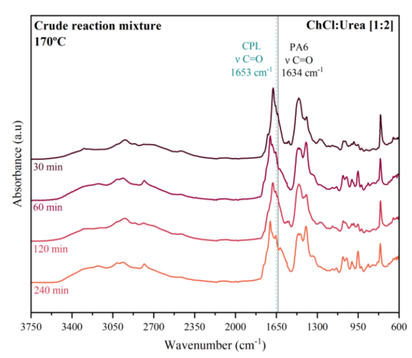
Fishing nets

Crude reaction mixture

Filtrate

Filter cake





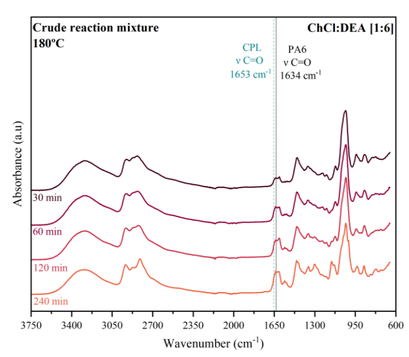


Table 1. Conversion percentage as a function of sample condition (presence/absence of fishing net after filtration)

Nº	DESs	Temperature [ºC]	Time [min]	Conversion [%]
1.	$ZnCl_2$: Urea [1:3.5]	170	30	Partial solubilization
2.	ChCl : Urea [1:2]	170	30	48.2
3.	ChCl : Glycerol [1:3]	160	60	65.1
4.	ChCl : MDEA [1:6]	170	60	4.1
5.	ChCl : DEA [1:6]	180	120	92.4
6.	ChCl : MEA [1:6]	160	120	94.8

The intensity of the **carbonyl band of CPL** (C=O, 1653 cm^{-1}) increases with longer reaction time, while the carbonyl band associated with polyamide 6 (C=O, 1634 cm^{-1}) decreases, indicating solvolysis of fishing nets.

CONCLUSION & FUTURE WORK

- Solvolysis was promoted in amine-based systems, which enabled solubilization of the fishing net at lower temperatures and shorter reaction times.
- The highest conversion was achieved when using deep eutectic solvents (DES) containing **amine-based compounds**, indicating that the presence of amino groups enhances the depolymerization efficiency of the system.
- Development of recycled materials for FDM additive manufacturing, to be carried out in **Work Package 4.**
- Process optimization and validation of the obtained products and demonstrators, to be carried out in **Work Package 5.**

References

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2. G. Marco-Velasco, A. Gálvez-Subiela, R. Jiménez-Robles, M. Izquierdo, A. Cháfer, and J. D. Badia, "A Review on the Application of Deep Eutectic Solvents in Polymer-Based Membrane Preparation for Environmental Separation Technologies," Polymers (Basel), vol. 16, no. 18, p. 2604, Sep. 2024, doi: 10.3390/polym16182604

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