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The effect of temperature on degradation of PET under hydrothermal carbonization

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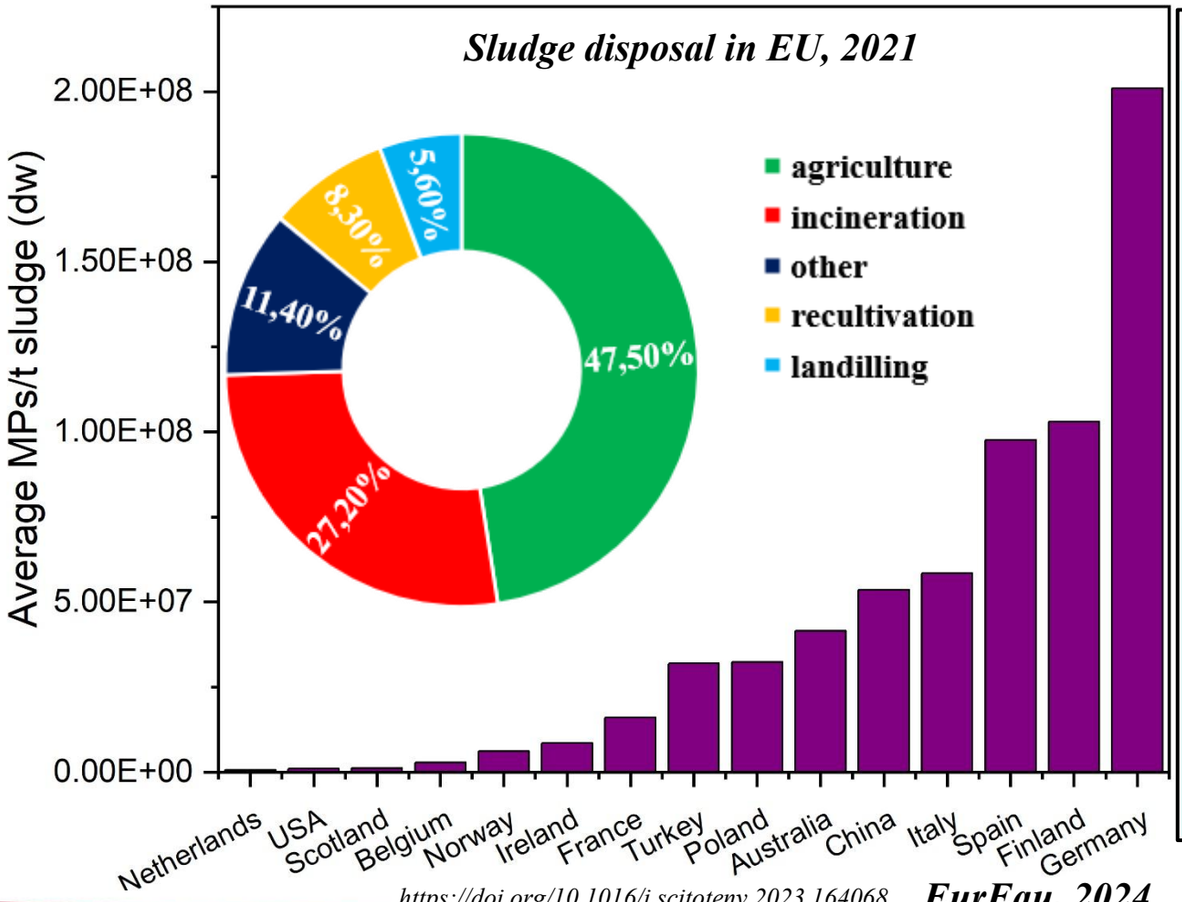
Outline

- Introduction
  - Microplastics in sewage sludge
  - Main representative of microplastics - PET
  - Hydrothermal carbonization (HTC)
- Experimental set-up
- Research methodology:
  - the effect of HTC temperature on PET degradation
  - Identification of PET
- Results
- Conclusion

Main source of microplastics – sewage sludge

90% of MPs flowing into WWTP are accumulated in sewage sludge

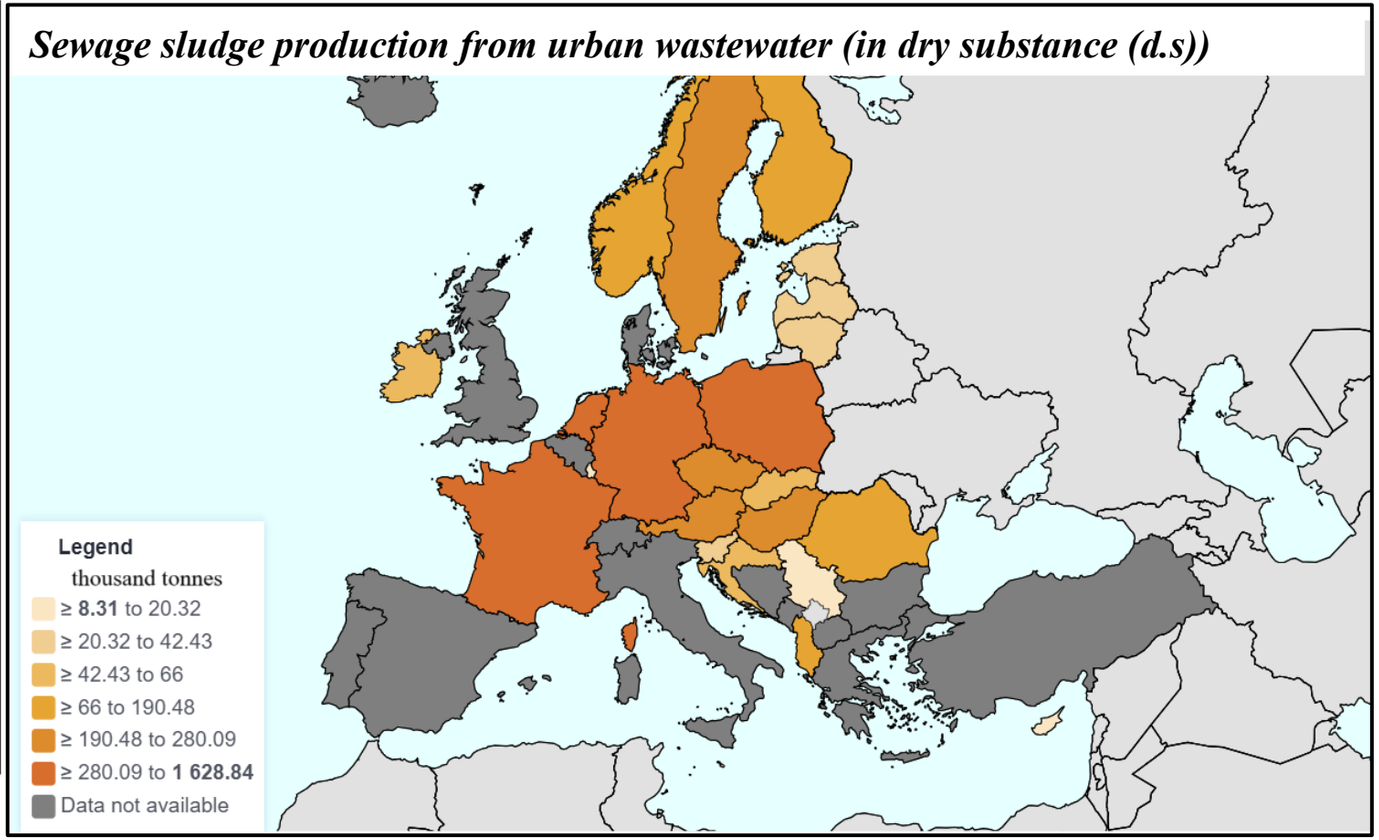
Sludge disposal in EU, 2021



https://doi.org/10.1016/j.scitotenv.2023.164068

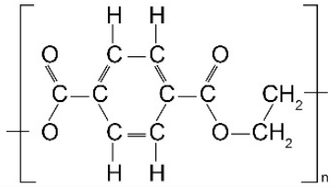
EurEau, 2024

Sewage sludge production from urban wastewater (in dry substance (d.s))



Eurostat, 2025

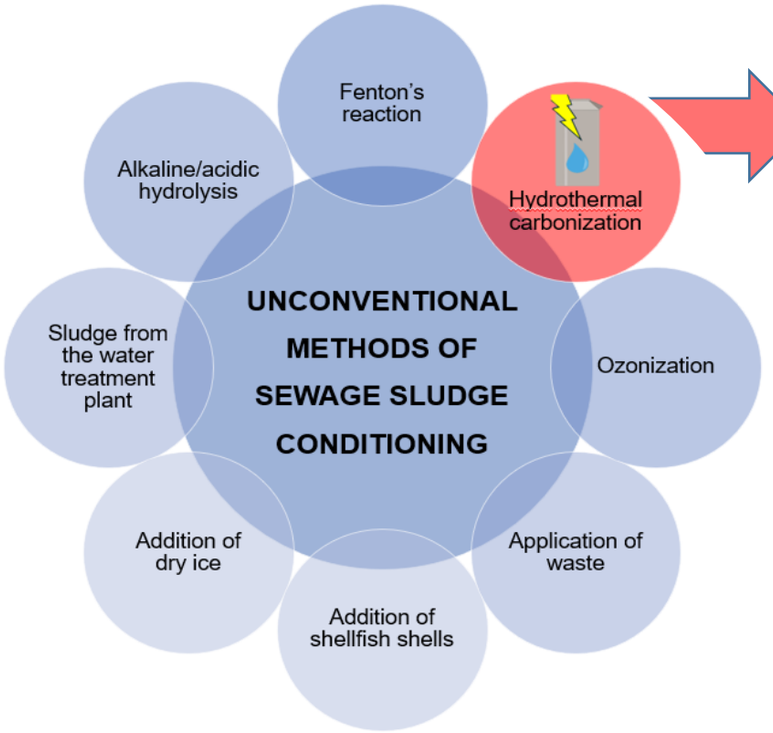
Common polymers found in sewage sludge

Name of compound	Polyethylene terephthalate (PET)
	
Molecular structure	
Molecular monomer weight, g/mol	192.17
Polymer type	Thermoplastic, condensation
Glass transition temperature, °C	70-80
Melting point, °C	250-260
Degradation mechanisms	Hydrolysis, oxidation
Main degradation products	Terephthalic acid, ethylene glycol, benzoic acid

- **Major polymer**
- **Production ~ 56 Mt/year ~ 10.8% of plastic waste**
- **10–30% found in sewage sludge**
- **Low collection rates: 50% globally, 33% USA, 64% EU**

Chemical Recycling. Certain types of plastics, such as PET, can be disposed of through chemical recycling. *https://www.businesswaste.co.uk/news/plastic-waste-disposal-guide/*

HTC of sewage sludge contaminated by MPs



Chem Eng J 450 (2022) 138163

HTC for 0.5 h; MPs: PC, PMMA, PS, PU, PVDF, PA, PE, PET, PO, PP, PVC

- at 170°C: PC, PMMA, PS, PU, PVDF completely degraded
- at 220°C**:** (PA, PE, PET, PO, PP, PVC) reduced by ~50.21%
- **87.35% total reduction at 220°C**

Chemosphere 297 (2022) 134203

HTC for 3 h; MPs: PU, PS, PET, PA, PP

- at 180°C: PU & PS totally removed
- at 220°C: PET & PA totally removed
- at 260°C: PP (79.34% reduction) and PE (55.93% reduction)
- **79% total reduction at 260°C**

Degradation mechanisms excluding organic matter

ACS Sustain Chem Eng (2024)14187−14199

HTC for 6h; MPs: PS, PP, PET

- **~90% total reduction at 200°C**

J Environ Chem Eng 13 (2025) 118262

HTC for 3 h; MP: PE

- **~71% total reduction at 220°C**

HTC feedstock sludge: 80-90% water

Objective

- **Is to study the effect of HTC temperature on degradation PET-MPs during a residence time of 2 hours**
- **Is to find out the adequate HTC temperature for PET degradation which in the same time is suitable for sewage sludge pretreatment**

Experimental set-up

HTC reactor

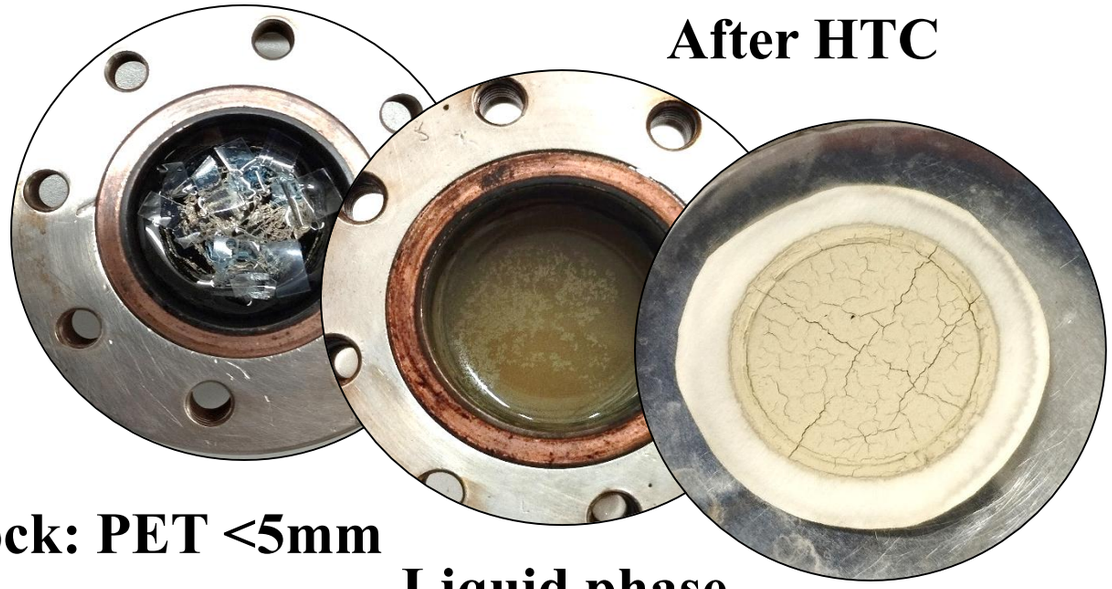
50 mL autoclave
University of Trento

Temperature: **220, 240 and 260°C**

Residence time: **2 h**

Plastic-to-water ratio: **~0.007**

Feedstock: PET <5mm



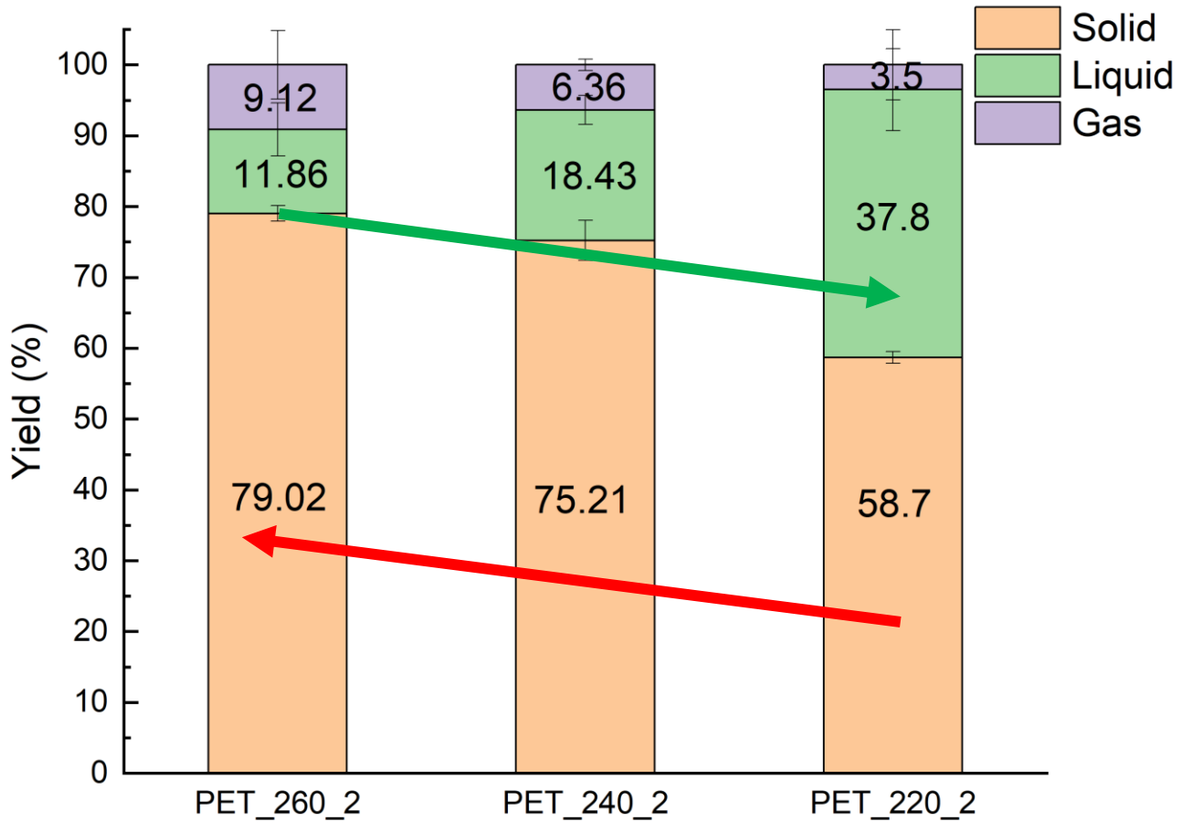
Hydrolysis mechanism

$$[\text{–OC–C}_6\text{H}_4\text{–COO–CH}_2\text{CH}_2\text{O–}]_n + nH_2O \xrightarrow{\text{HTC}} n\text{HOCH}_2\text{CH}_2\text{OH (EG)} + n\text{HOOC–C}_6\text{H}_4\text{–COOH (TPA)}$$

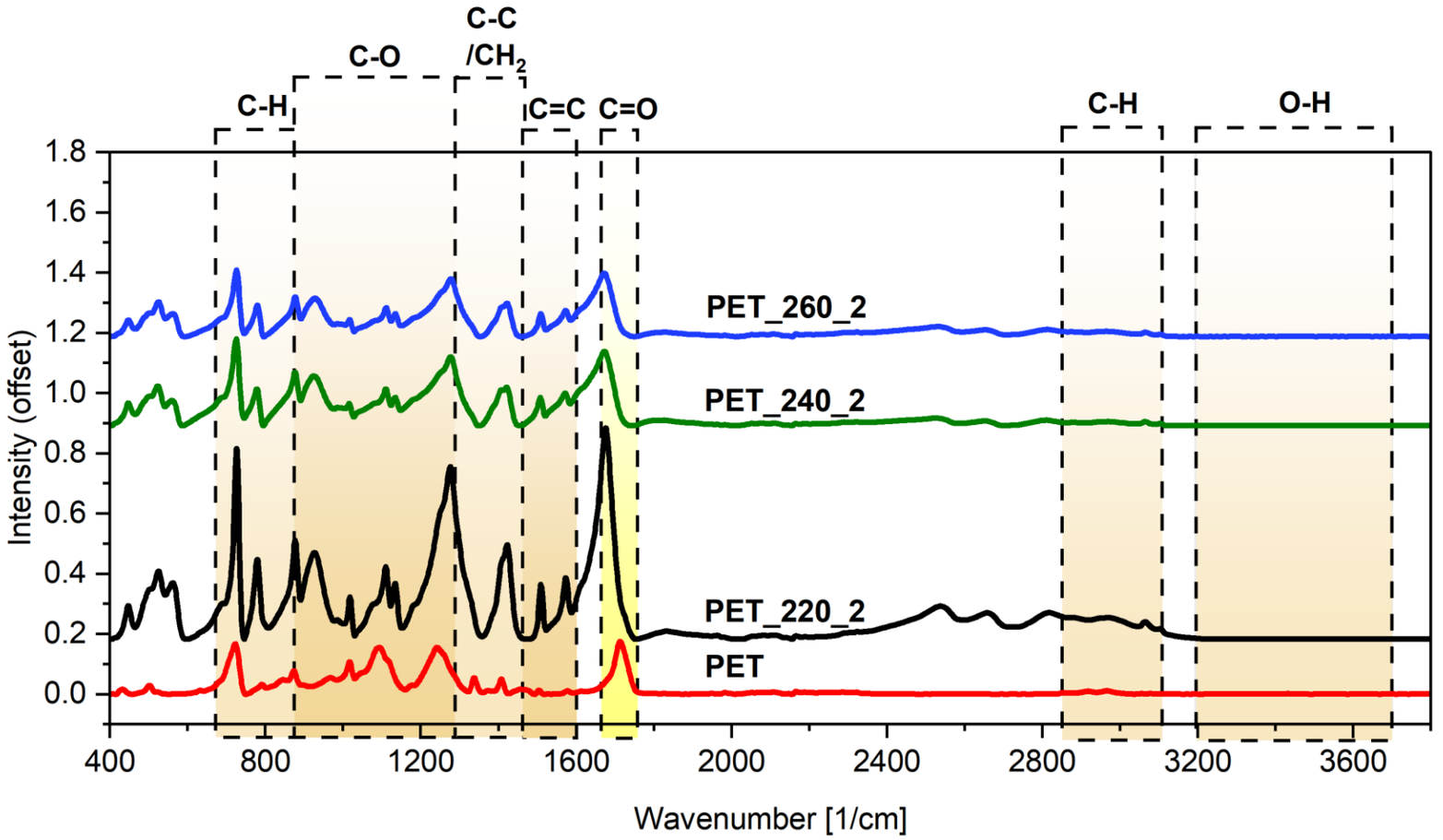
Results

HTC yields

- **Yields determined from two HTC runs**
- **Liquid fraction calculated by difference**
- **Solid yield increased with increasing temperature**
- **Liquid fraction decreased with increasing temperature**
- **Gas < 10% increased with increasing temperature**
- **220 °C – hydrolysis dominant**
- **≥ 240 °C – condensation and solid formation (secondary char)**

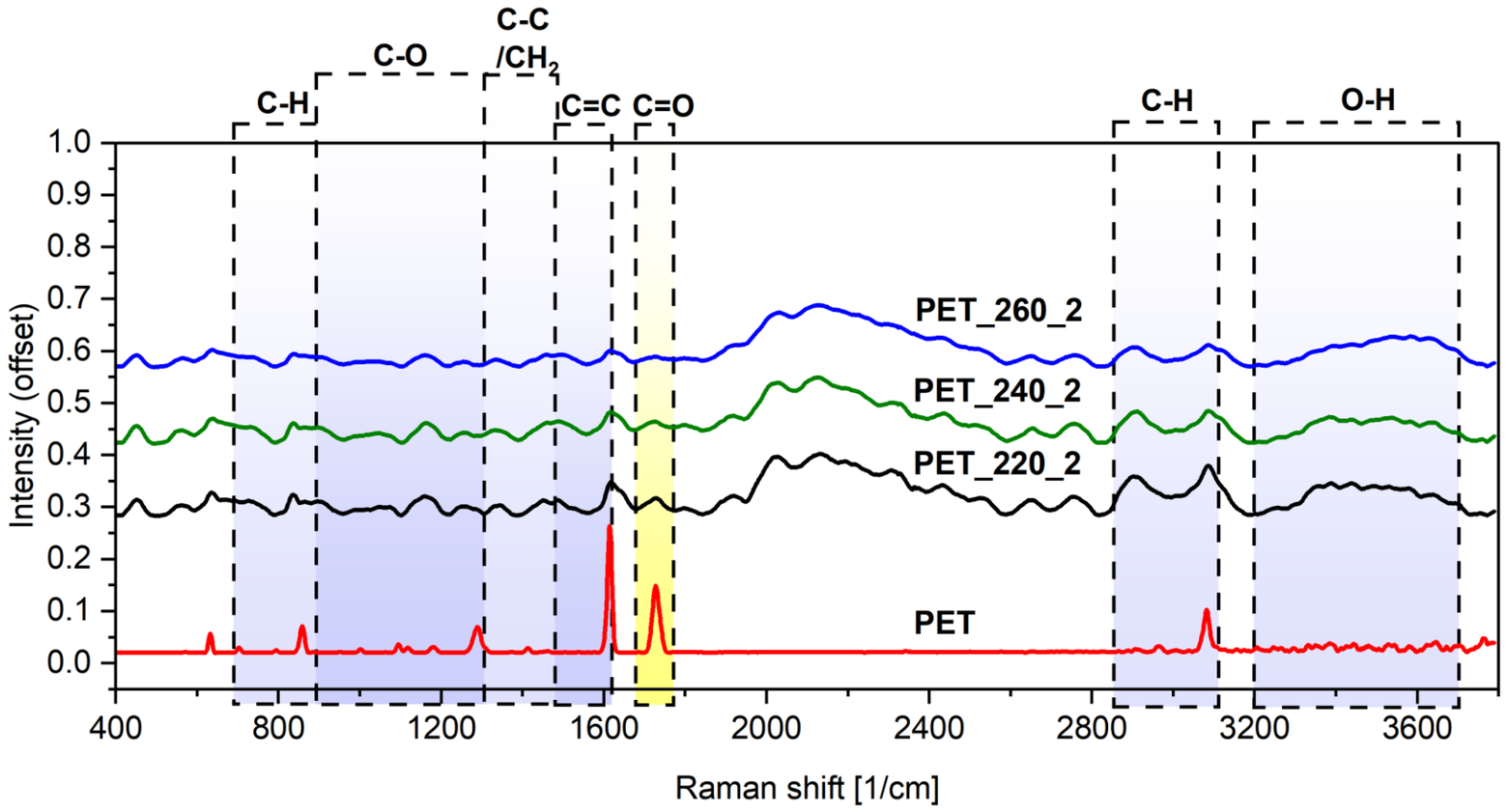


FTIR



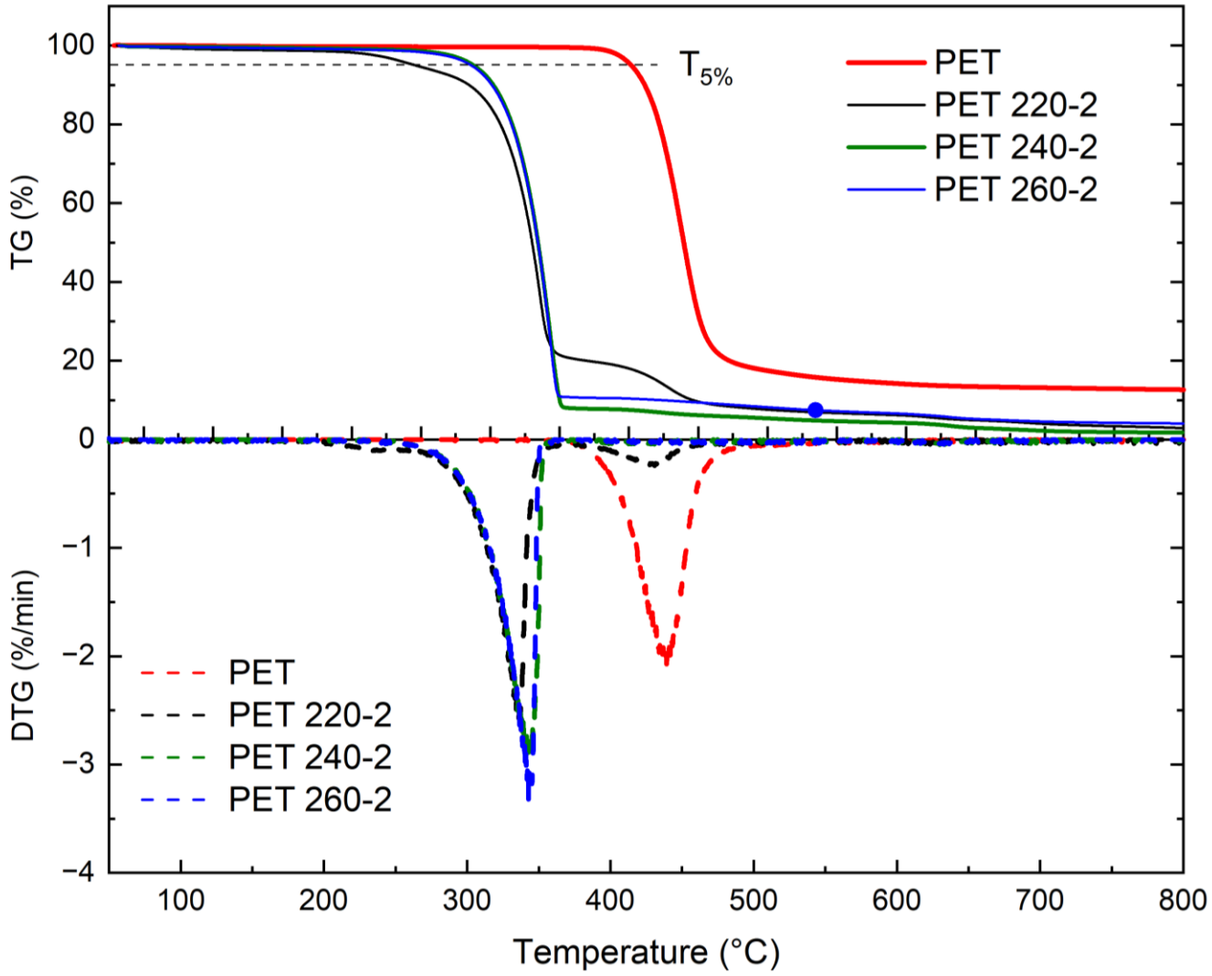
- **Pretreated sample peaks: 520, 565 cm^{-1} (condensed structures), 1135 cm^{-1} (chain scission)**
- **Aliphatic bending shift 1410 (PET) → 1420 cm^{-1} (treated PET); O–H/C–H broad/shifted band > 2400 cm^{-1} (H-bonding)**
- **PET_240_2 and PET_260_2: spectra flatter, lower intensity; loss of aromatic bands and H-bonding → amorphous, stable char**

Confocal Raman microscope CRM



- **Pretreated PET spectra nearly identical; major changes already at 220 °C**
- **Aromatic bands (1620, 1730 cm^{-1}) diminished and broadened → depolymerisation**
- **New broad D-band (1350 cm^{-1}) and G-band (1580–1620 cm^{-1}) → disordered/graphitic carbon**
- **O–H/C–H broad/shifted band >2400 cm^{-1} (H-bonding)**

Thermogravimetric Analysis (TGA)



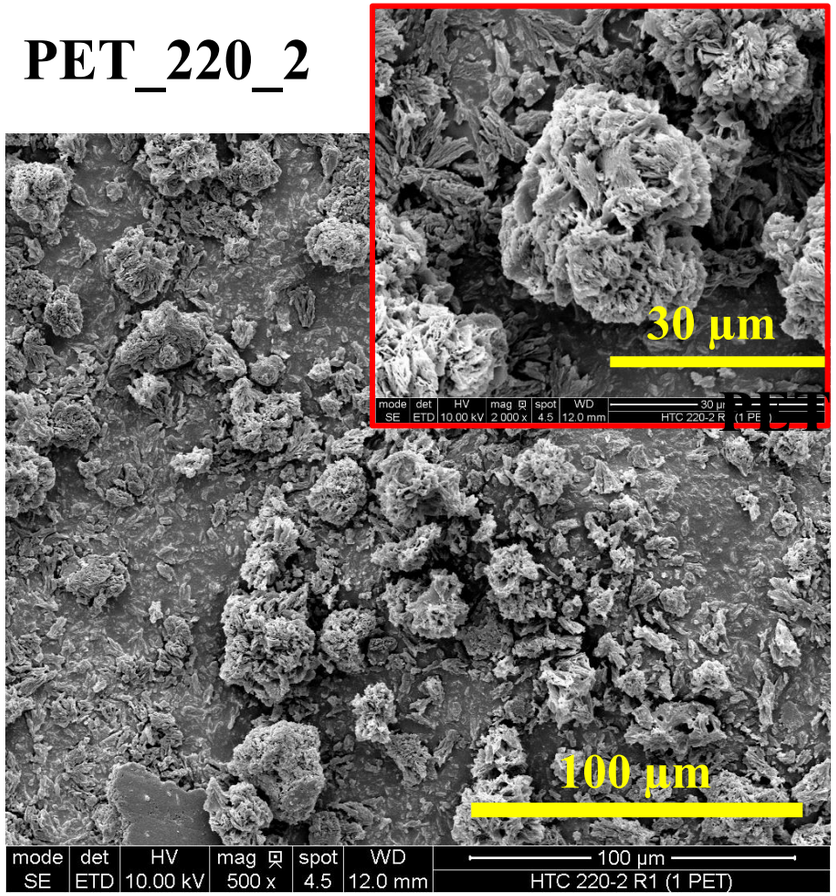
- **Single-stage degradation: PET, PET_240_2, PET_260_2**
- **Multi-step decomposition: PET_220_2**
- **PET: $T_5\%$ ~ 410 °C, T_{max} ~ 450 °C, >85% mass loss, ~10% residue**
- **After HTC: lower stability**

  PET_220_2 $T_5\%$ ~ 250 °C

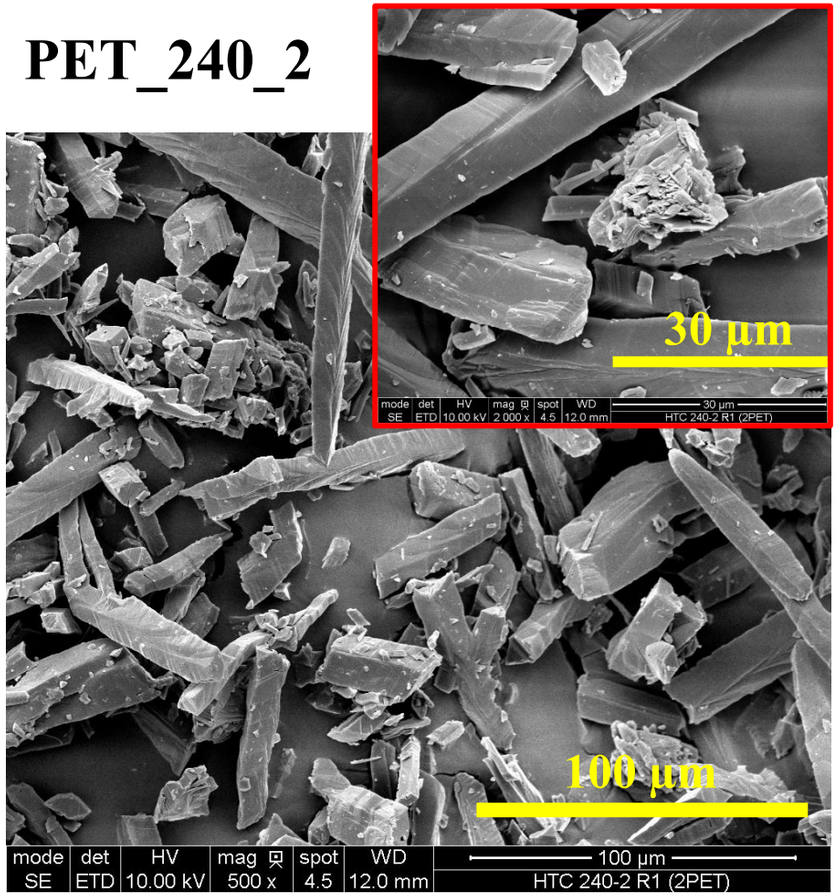
  PET_240_2 & PET_260_2 $T_5\%$ ~ 380 °C

Scanning Electron Microscopy (SEM)

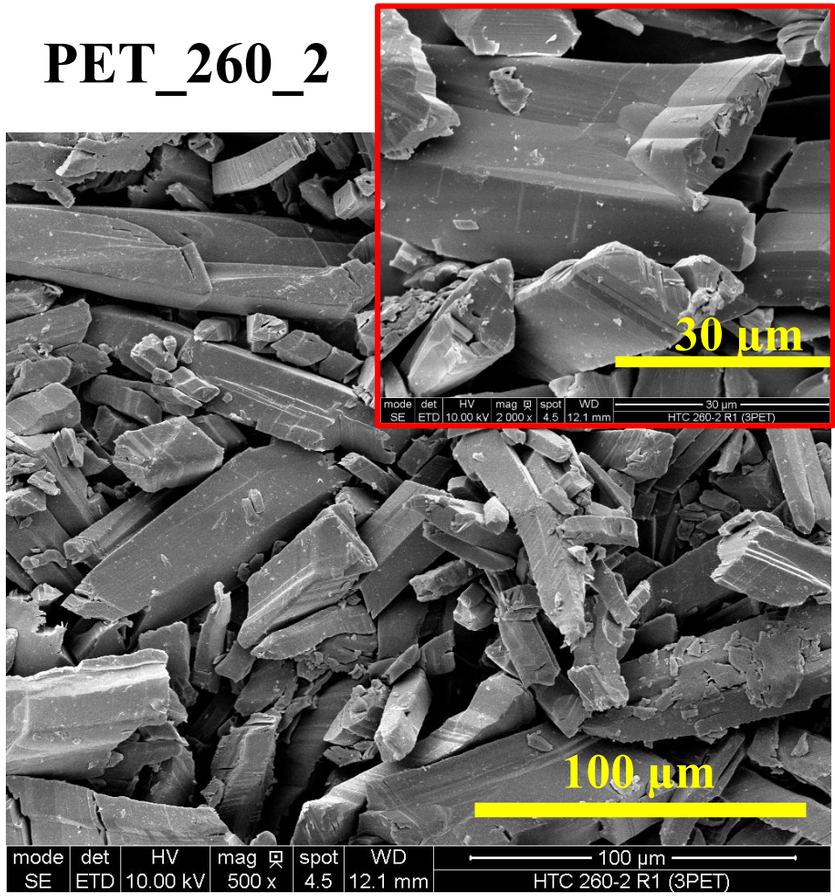
PET_220_2



PET_240_2



PET_260_2



Porous and powdery surface at 220 °C

Crystalline and stable structures at 240–260 °C

Conclusions

- **Yields results indicate the formation of secondary hydrochar with increasing HTC temperature**
- **Pretreated PET has the lowest thermal stability at 220 °C**
- **Above >220 °C PET lost its aromatic and hydrogen-bonding signals with crystalline structure, indicating the formation of more durable, amorphous, more carbonized solids**
- **New broad D- and G-bands in Raman spectra confirm carbonization and the development of graphitic domains**
- **HTC at 220 °C for 2 h is sufficient to degrade and remove PET-MPs from a highly water medium**

Acknowledgements

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Thank you for your attention

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