

Supporting Information

Instantaneous hydrolysis of PET bottles: an efficient pathway to the chemical recycling of condensation polymers

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The present document displays a series of characterizations that complement the ones in the published version, as well as more detailed experimental issues related with the depolymerization reaction of the KMH system with PET bottles.

About Experimental details

It was verified that the reaction is not sensitive to small differences in PET flakes, either in shape or size; the KMH solution is able to depolymerize all range of shapes under 2 cm² with the same efficiency. The key factor is the thickness, which was kept constant. As a matter of fact, a significant difference was observed when the depolymerization was carried out on flakes and on pellets (0.1 mm vs 4 mm thickness). This demonstrates that in spite of the high shrinking layer of the PET-KMH system, the reaction is governed by diffusion limitations of the reactant species into the PET matrix.

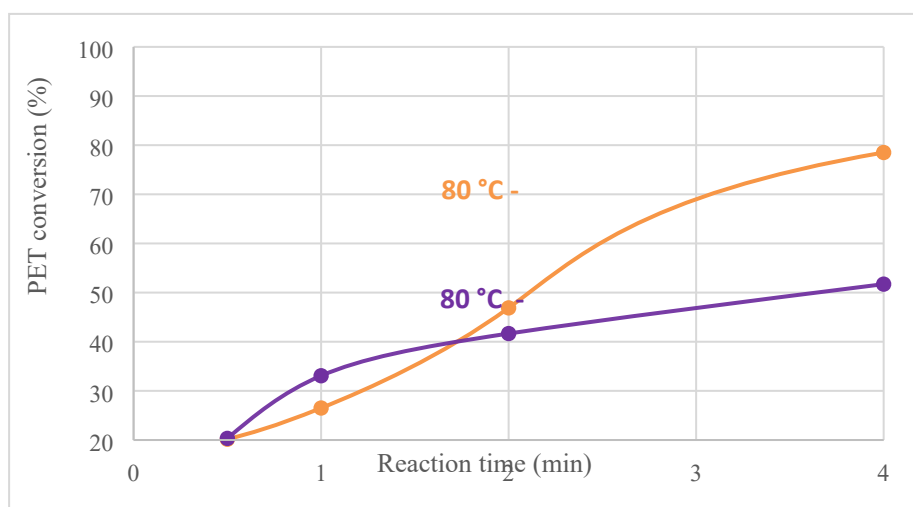
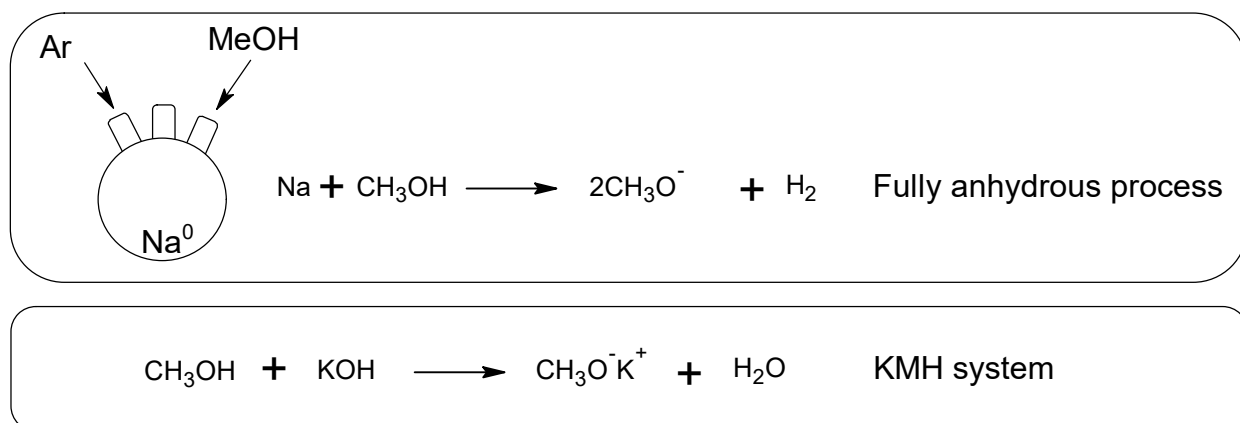


Figure S1 Comparison between depolymerization of PET bottle flakes and PET pellets under microwaves assisted heating using KMH solution.

The activity of water was followed by forming sodium methoxide *in-situ* without producing any water inside the reaction system, which led to approximately the same results as for the KMH solution at high temperature. The KMH solution was chosen because of its use of a cheap reagent (KOH), while the *in-situ* production of sodium methoxide involves the reaction of metallic sodium under dry, inert (in our case Argon) atmosphere. There were no observable differences among the TPA produced from the fully anhydrous and the KMH open system as demonstrated by FTIR. Further, we demonstrated that at low temperatures and through a higher control of the water inside the system, the formation of DMT can be observed as reported in the published document.



Scheme S1 Differences between the fully anhydrous process and the KMH system. While fully anhydrous requires the use of metallic sodium and inert atmosphere, the KMH system only uses potassium hydroxide and methanol.

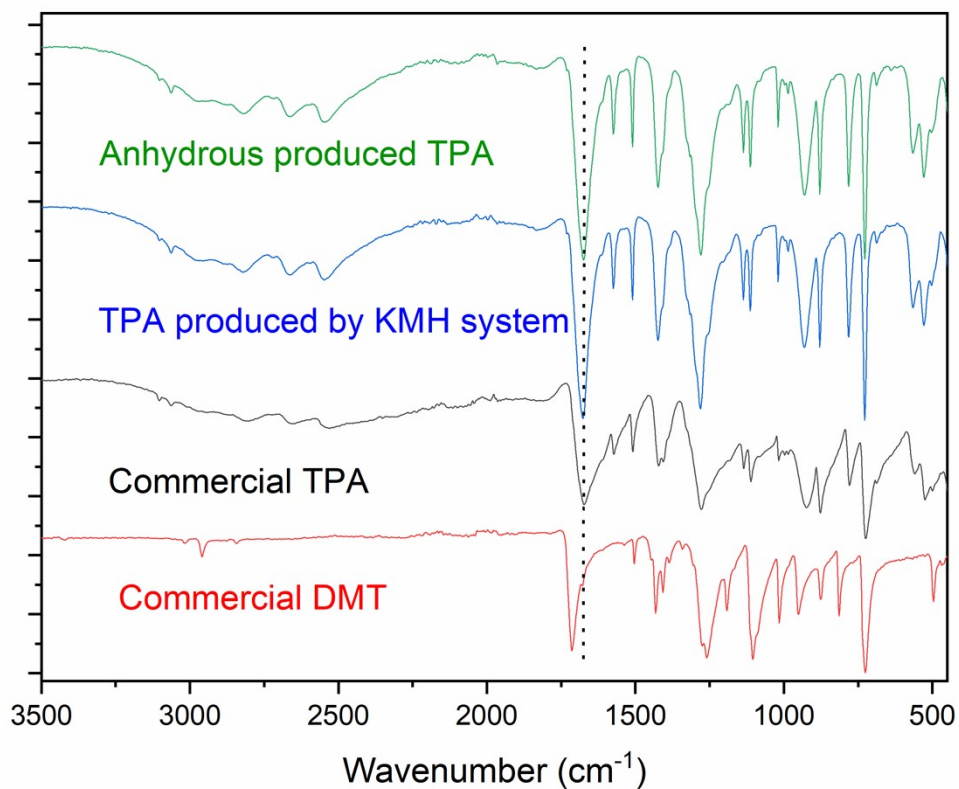


Figure S2 FTIR spectra of the TPA produced by the anhydrous system and produced by open atmosphere KMH system.

Fourier Transform Infrared spectroscopy of unreacted residues and produced TPA under microwave irradiation

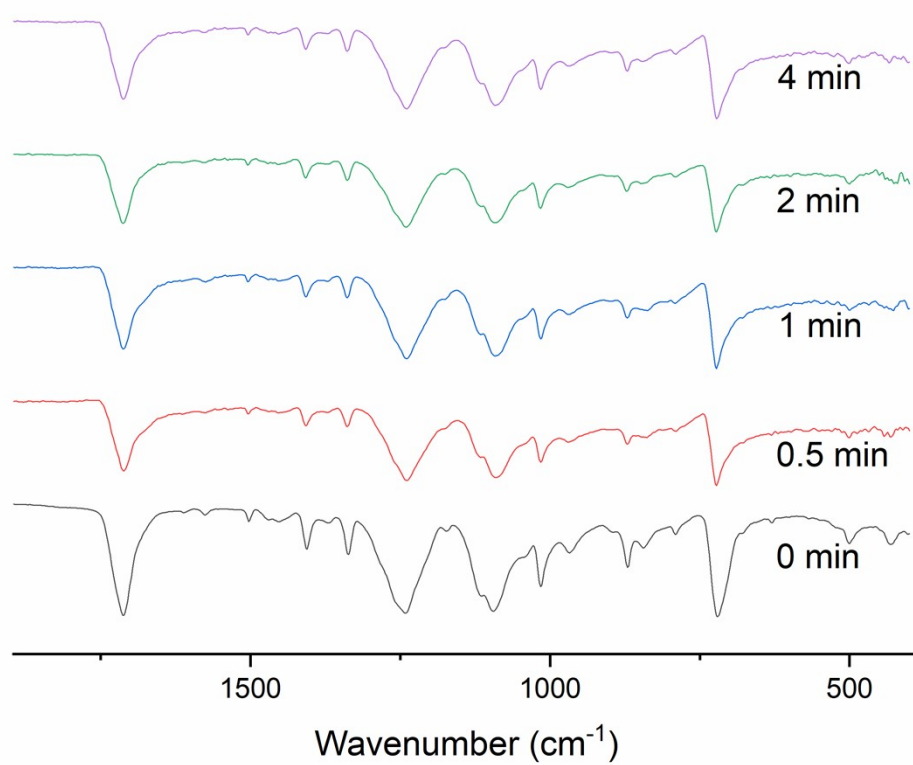


Figure S3. FTIR spectra of unreacted PET at different times after treatment with KMH solution 1.25 M at 90 °C

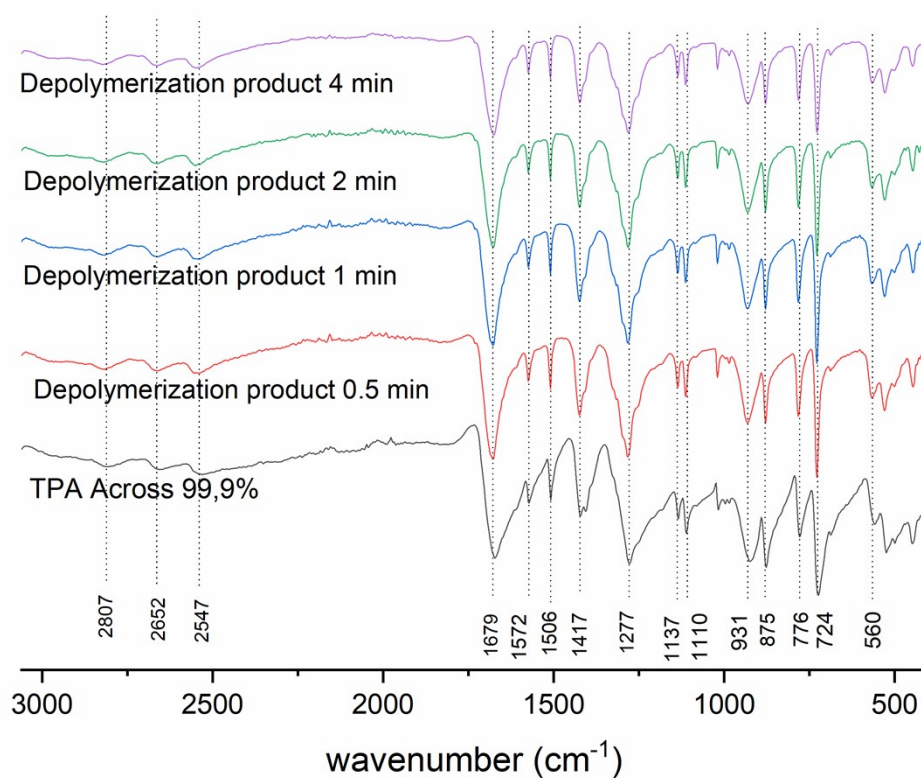


Figure S4. FTIR of depolymerization products obtained after treatment of PET flakes with KMH 1.25 M at 90 °C. The wavenumbers of each peak are shown and compared to commercial TPA (99.9%) from Across.

Table S1 FTIR peaks of the obtained TPA from PET waste and the corresponding vibrations according to literature ^{1,2}

Wavenumber (cm-1)	Vibration
875	C=C bending
927	O-H bending
1110	C-O stretching
1137	C-O stretching
1277	C-O stretching
1417	O-H bending
1679	C=O stretching
2807	C-H stretching

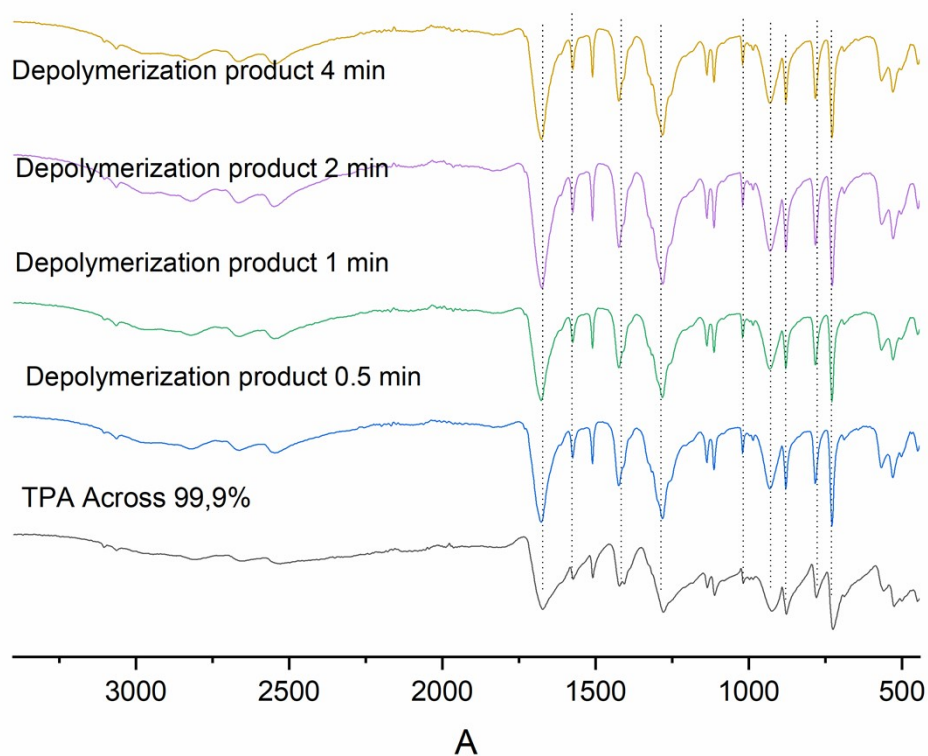


Figure S5 FTIR of depolymerization products obtained after treatment of PET flakes with KMH 1.25 M at 80 °C compared to TPA 99.9% from Across.

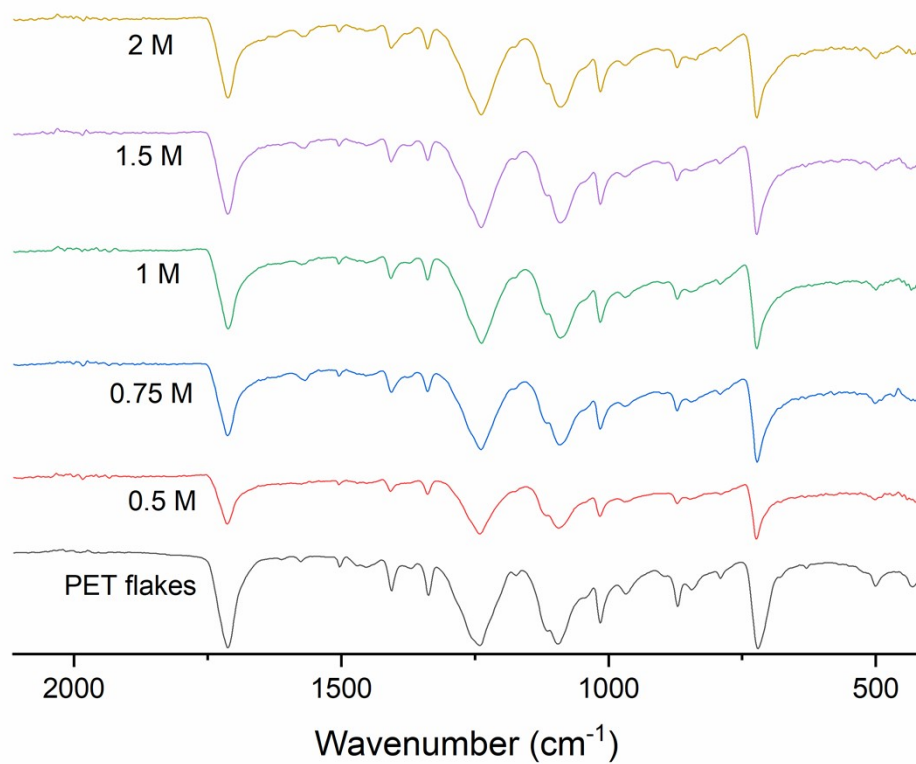


Figure S6 FTIR of unreacted PET after treatment with KMH solution 1.25 M for 3 min at 80 °C

DSC of produced terephthalic acid

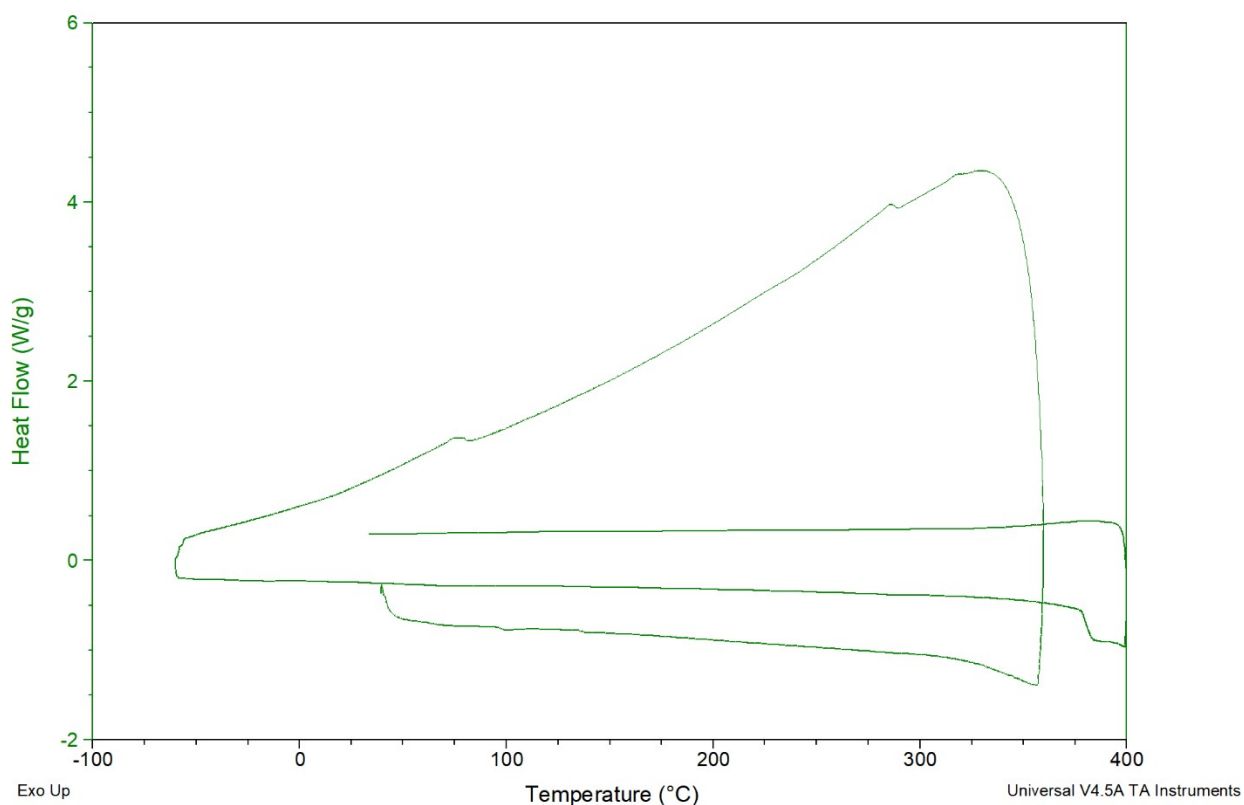


Figure S7 Typical DSC result obtained from produced terephthalic acid. All the heating-cooling cycles are presented with no evidence of melting transition

DSC results of produced terephthalic acid had as main feature the absence of any melting transition, which is expected since, normally, its degradation occurs first, explaining why there are no observable melting peaks in DSC. TGA on the other hand, confirmed this via the cDTA accessory, which shows only one large endothermic peak corresponding to TPA degradation above 400 °C.

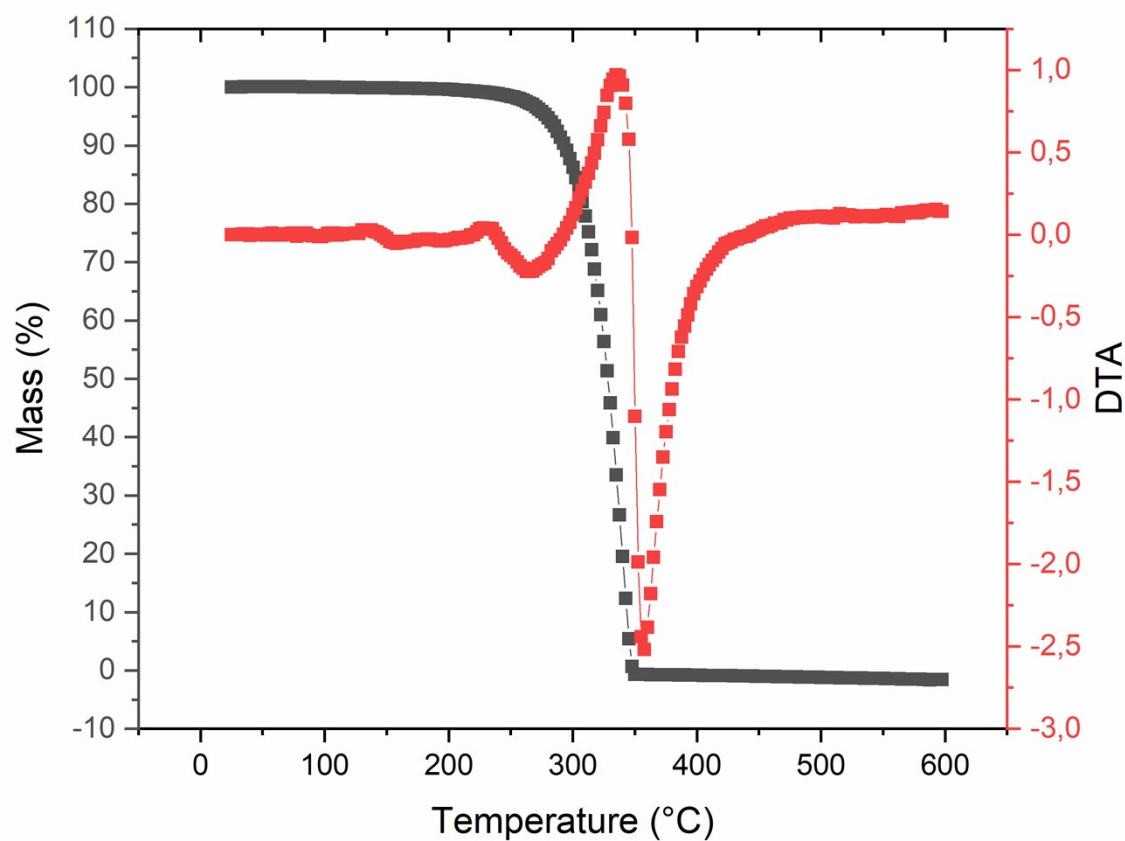


Figure S8 Typical TGA and DTA for obtained TPA

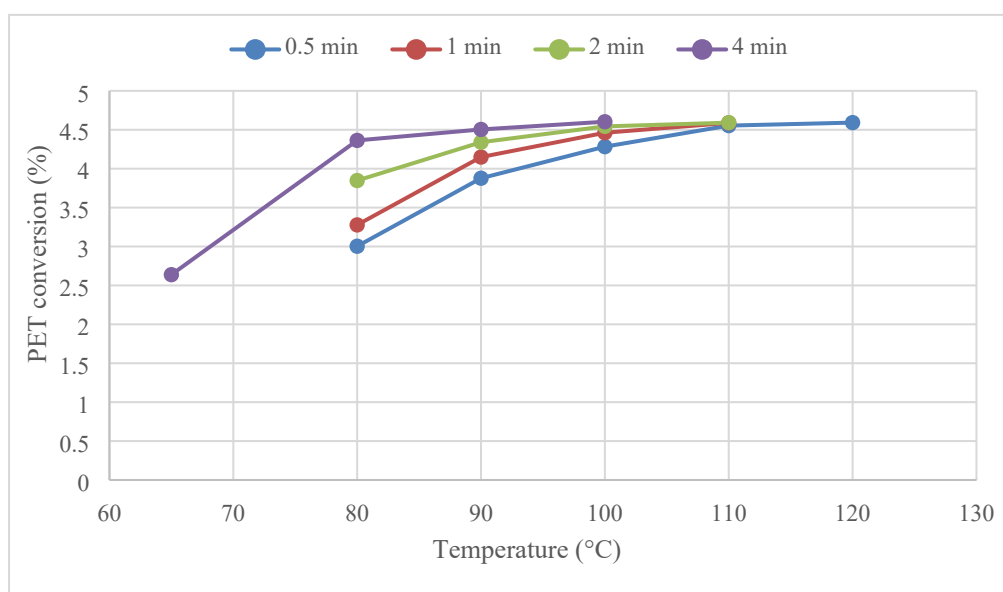


Figure S9. PET conversion as a function of temperature for different reaction times

References

- (1) Karthikeyan, N.; Joseph Prince, J.; Ramalingam, S.; Periandy, S. Electronic [UV-Visible] and Vibrational [FT-IR, FT-Raman] Investigation and NMR-Mass Spectroscopic Analysis of Terephthalic Acid Using Quantum Gaussian Calculations. *Spectrochim. Acta - Part A Mol. Biomol. Spectrosc.* **2015**, *139*, 229–242. <https://doi.org/10.1016/j.saa.2014.11.112>.
- (2) Deshmukh, S.; Kandasamy, G.; Upadhyay, R. K.; Bhattacharya, G.; Banerjee, D.; Maity, D.; Deshusses, M. A.; Roy, S. S. Terephthalic Acid Capped Iron Oxide Nanoparticles for Sensitive Electrochemical Detection of Heavy Metal Ions in Water. *J. Electroanal. Chem.* **2017**, *788*, 91–98. <https://doi.org/10.1016/j.jelechem.2017.01.064>.