

Quantitative comparison of conventional and microwave-assisted heating in the ZnO-catalyzed glycolysis of virgin PET.

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Introduction

Polyethylene terephthalate (PET) is the most widely used synthetic polyester in textiles and packaging (El Darai et al., 2024), due to its low cost, processability, and reliable performance. However, its growing production, combined with its non-biodegradability, and low fibre-to-fibre recycling rate (<1%) (Ellen MacArthur Foundation, 2017) constitute a risk to environmental integrity and a matter of considerable economic concern. Therefore, it is important to underscore the need for more effective valorisation routes based on selective depolymerization.

Among the available chemical recycling options, glycolysis stands out as the most practical and industrially mature route for PET depolymerization due to its simplicity, low energy demand, and operation at temperatures from 180 °C to 240 °C (Umdagas et al., 2025). It selectively produces bis(2-hydroxyethyl) terephthalate (BHET), a high-purity monomer that can be repolymerized into PET with properties comparable to virgin material and a reduced carbon footprint. However, reaction rate and yield remain critical points. This work highlights microwave-assisted glycolysis as an approach to overcome these limitations. Microwaves convert electromagnetic energy into heat directly within the reaction medium, without contact. Microwave-assisted heating is faster and more selective than conventional methods, with lower energy losses (Sun et al., 2016). By coupling selective microwave heating with an efficient ZnO catalyst, the process achieves rapid energy transfer, enhanced kinetics, and high conversion (Selvam et al., 2023). This study provides a quantitative comparison between conventional and microwave-assisted glycolysis, focusing on reaction efficiency, operating severity, and catalyst and solvent requirements, to elucidate the potential of microwave heating as an intensified and more sustainable depolymerization route.

Materials and methods

The materials used in this study include virgin PET (vPET) pellets (approximately 4x3x2 mm) and ZnO nanoparticles (particle size 18 nm), both supplied by AITEX (Alcoy, Spain). Ethylene glycol (analytical grade), employed as the depolymerization agent and solvent, was purchased from VWR Chemicals. Conventional glycolysis was performed in a 500 mL two-neck round-bottom flask equipped with a reflux condenser and a thermocouple for accurate temperature control. Microwave-assisted glycolysis was carried out in a MW-assisted reactor (Milestone SynthWAVE), which can operate at a maximum microwave power of 1500 W. Pressurized 100 mL vials were employed to maintain the reaction medium in the liquid phase, while continuous agitation ensured homogeneous conditions.

For the glycolysis experiments, vPET, ZnO and EG were mixed in the aforementioned reactors in different proportions. Standard operating procedures, such as maintaining stirring rate and external nitrogen pressure, in the MW-assisted procedure, were applied to run the reactor under controlled conditions to guarantee stable and comparable reaction environments.

For BHET isolation and purification, a defined volume of water was added to the reaction mixture after each experiment. The suspension was heated to 80 °C and stirred for 15 minutes to selectively dissolve BHET in the aqueous phase while leaving unreacted PET insoluble. The hot mixture was then vacuum-filtered, and the solid vPET was separated from the filtrate containing dissolved BHET and suspended catalyst. The vPET residue was finally dried, and the filtrate was cooled at 5 °C overnight to promote BHET crystallisation. The resulting BHET was recovered by filtration and dried at 80 °C overnight.

Glycolysis efficiency was evaluated by quantifying PET conversion from the mass of residual PET and determining BHET yield from the mass of BHET recovered relative to the theoretical monomer amount obtainable from the initial PET.

Results and discussion

Figure 1 summarizes the influence of the key operational parameters in conventionally heated glycolysis and as it can be extracted, the optimal conditions were identified as 190 °C, 8 h reaction time, 0.5 % w/w ZnO, and an EG:PET mass ratio of 3.5.

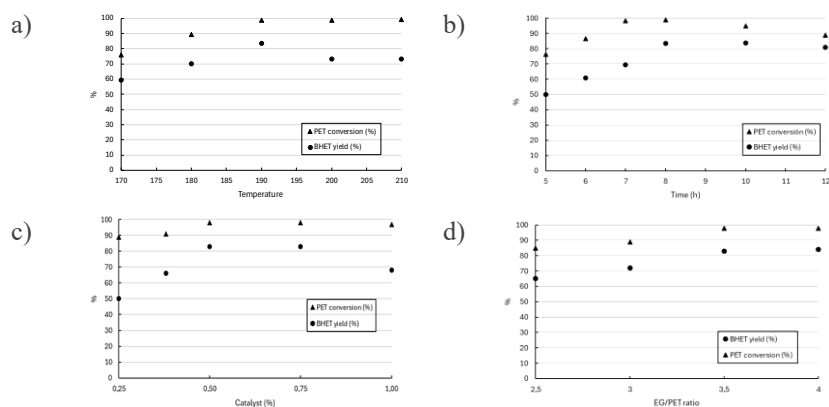


Figure 1: Effect of conditions on glycolysis under conventional heating efficiency: a) effect of temperature; b) effect of reaction time; c) effect of catalyst concentration (% w/w) and d) effect of PET:EG mass ratio.

In contrast, under microwave-assisted heating (Figure 2), the process reaches optimal performance at higher temperature but significantly reduced severity, namely 210 °C, 1 h reaction time, 0.139 % w/w ZnO, and an EG:PET mass ratio of 0.20. These results clearly illustrate the efficiency gains achieved through microwave irradiation.

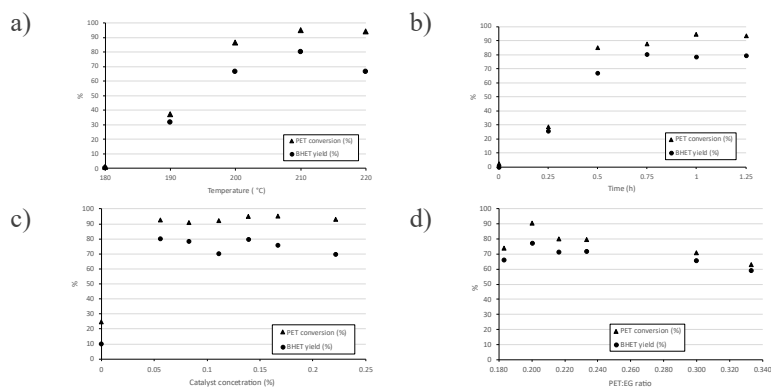


Figure 2: Effect of conditions on microwave-assisted glycolysis efficiency: a) effect of temperature; b) effect of reaction time; c) effect of catalyst concentration (% w/w) and d) effect of PET:EG mass ratio.

Conclusions

Microwave-assisted heating reduces the reaction time by up to seven hours, even when the reactant loading is reduced relative to the PET feedstock. Both under conventional heating and microwave-assisted glycolysis, similar performance was achieved, with PET conversion of approximately 95% and BHET yield exceeding 80%. These results show that microwave-assisted glycolysis delivers robust performance while reducing reaction time and reagent consumption.

Future research should extend this methodology to post-consumer PET and assess the activity of alternative catalyst systems. In addition, further work is required to evaluate the scalability of microwave-assisted glycolysis and the long-term reusability of ZnO over multiple reaction cycles.

El Darai, T., Ter-Halle, A., Blanzat, M., Despras, G., Sartor, V., Bordeau, G., Lattes, A., Franceschi, S., Cassel, S., Chouini-Lalanne, N., Perez, E., Déjugnat, C., & Garrigues, J.-C. (2024). Chemical recycling of polyester textile wastes: Shifting towards sustainability. *Green Chemistry*, 26(12), 6857–6885. <https://doi.org/10.1039/d4gc00911h>

Ellen MacArthur Foundation. (2017). A New Textiles Economy: Redesigning fashion's future. <https://www.ellenmacarthurfoundation.org/a-new-textiles-economy>

Selvam, E., Luo, Y., Ierapetritou, M., Lobo, R. F., & Vlachos, D. G. (2023). Microwave-assisted depolymerization of PET over heterogeneous catalysts. *Catalysis Today*, 418, 114124. <https://doi.org/10.1016/j.cattod.2023.114124>

Umdagas, L., Orozco, R., Heeley, K., Thom, W., & Al-Duri, B. (2025). Advances in chemical recycling of polyethylene terephthalate (PET) via hydrolysis: A comprehensive review. *Polymer Degradation and Stability*, 234, 111246. <https://doi.org/10.1016/j.polymdegradstab.2025.111246>

Sun, J., Wang, W., & Yue, Q. (2016). Review on Microwave-Matter Interaction Fundamentals and Efficient Microwave-Associated Heating Strategies. *Materials*, 9(4), 231. <https://doi.org/10.3390/ma9040231>