Efficient recovery of benzoic acid from the condensed phase of textile residues obtained by slow pyrolysis

L. S. Fressato¹, A. Salimbeni¹, L. Bettucci¹, G. Lotti¹

¹Renewable Energy Consortium for Research and Demonstration (RE-CORD), Viale Kennedy, 182, 50038 Scarperia e San Piero, Italy

Keywords: Benzoic Acid recovery, Slow pyrolysis, Byproducts Presenting author email: larissa.fressato@re-cord.org

Introduction

The textile industry, driven by relentless consumerism and the rapid turnover of fast fashion, stands as one of the largest contributors to global waste, generating a staggering 92 million tons of textile waste annually (Fan et al., 2024). Alarmingly, 85% of this waste is improperly managed, leading to immense resource loss and posing significant environmental challenges. Addressing this crisis requires innovative solutions, and one promising approach is pyrolysis—a thermal process capable of converting textile waste into valuable byproducts. Pyrolysis not only produces biochar, a material with applications such as activated carbon (Rubi et al., 2023) and energy generation (Park et al., 2024), but also yields organic and aqueous phases rich in benzoic acid. This versatile compound finds widespread use as a preservative in the foods, cosmetics and pharmaceutical industries (del Olmo et al., 2017). By unlocking the potential of these byproducts, pyrolysis offers a pathway to integrate textile waste into a circular economy, transforming a pressing environmental burden into valuable resources and opportunities.

This study focuses on efficiently recovering benzoic acid from pyrolysis byproducts using simple physical and chemical methods, enhancing the sustainability and economic viability of slow pyrolysis while adding value through its industrial applications.

Methodology

Textile waste materials were ground and pelletized before being subjected to slow pyrolysis in an inert nitrogen atmosphere. The process was conducted at 500°C in a rotary drum reactor with a maximum capacity of 100 kg/h, located at the industrial plant facilities of RE-CORD (Renewable Energy Consortium for Research and Demonstration). Additionally, the generated pyrogas was condensed, resulting in two separate phases: an organic phase and an aqueous phase. The process flows with the global mass balance is depicted in Figure 1. Analysis of the organic and aqueous phases via GC-MS (Gas Chromatography-Mass Spectrometry) was performed.

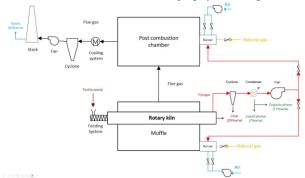


Figure 1: Mass balance for slow pyrolysis process.

To recover benzoic acid, 10 grams of the organic phase were mixed with distilled water at 90°C under agitation at 400 rpm for 1 hour, employing a solid-to-liquid ratio of 1:10. After this period, the hot solution was filtered and allowed to cool to room temperature. During cooling, crystal formation occurred, and the crystals were separated by simple filtration and dried in an oven at 70°C. These crystals were labeled as Solid A. The liquid remaining after filtration was designated as Liquid A. The residual organic solid was subjected to a second solid-liquid extraction under identical conditions, yielding another batch of crystals (Solid B) and a liquid phase (Liquid B). The remaining solid fraction after both extractions was named Solid C.

The liquid solutions obtained from the extraction processes (Liquid A and Liquid B), along with the aqueous phase from pyrolysis, underwent a freezing process at -5°C. Afterward, these samples were thawed and filtered sequentially, resulting in additional solid and liquid products. All products were analyzed using GC-MS for characterization.

Results and Discussion

The organic phase obtained from the pyrolysis process was analyzed and quantified, revealing benzoic acid (23% w/w) as the major component, followed by phenol (5% w/w), methyl benzoic acid (5% w/w), and naphthalene (2% w/w).

The solid-liquid extraction process was effective, with the first and second extractions achieving efficiencies of 43% and 28%, respectively, resulting in a total recovery of 71% of the initial benzoic acid in the organic phase. These results demonstrate the method's simplicity and efficiency.

The liquid phases were homogeneous and yellowish when hot, forming crystals upon cooling (Figure 2), primarily composed of benzoic acid. Analysis of Solid A showed that 93% of the benzoic acid from the first extraction was recovered. This aligns with the solubility of benzoic acid, which is 6.8 g/L at 90°C and decreases to 3.4 g/L at room temperature. The residual benzoic acid in Liquid A matched its solubility limit, and Solid A had a purity of 60%. The second extraction followed a similar trend, with Liquid B containing residual benzoic acid at 3.4 g/L, while the excess precipitated in Solid B. However, only 38% of the benzoic acid from this step was recovered, likely due to the water used. Reducing the solid-to-liquid ratio could improve recovery efficiency in future extractions.



Figure 2: Liquid A before and after the cooling process.

Freezing experiments with Liquids A and B improved recovery, forming solids primarily composed of benzoic acid. The solid from Liquid A showed a 64% extraction efficiency, while that from Liquid B achieved 45%. The residual liquid in both cases contained approximately 1.4 g/L of benzoic acid, indicating efficient separation. The aqueous phase from the pyrolysis process was also frozen, yielding a 65% recovery efficiency. However, the resulting solid had a low purity of 15%, highlighting the need for further optimization. These results demonstrate the potential of recovering valuable compounds from pyrolysis byproducts, supporting circular economy practices.

Conclusion

The experiment demonstrated the effectiveness of recovering benzoic acid from products of slow pyrolysis. The two-stage solid-liquid extraction of the organic phase achieved a 71% recovery of the total benzoic acid, with 60% recovered as solid with over 60% purity. The freezing method applied to the aqueous solutions also recovered about 60% of the benzoic acid.

Although the purity of the recovered solid can be improved, this will be addressed in future studies. These results highlight the potential of simple extraction and freezing processes for recovering high-demand industrial compounds, supporting circular economy principles by adding value to textile waste thermal treatment and offering an efficient waste valorization pathway.

Notes

The authors request that the results presented in this project not be disclosed, as this content is confidential.

References

- del Olmo, A., Calzada, J., Nuñez, M., 2017. Benzoic acid and its derivatives as naturally occurring compounds in foods and as additives: Uses, exposure, and controversy. Crit. Rev. Food Sci. Nutr. 57, 3084–3103. https://doi.org/10.1080/10408398.2015.1087964
- Fan, W., Wang, Y., Liu, R., Zou, J., Yu, X., Liu, Y., Zhi, C., Meng, J., 2024. Textile production by additive manufacturing and textile waste recycling: a review. Environ. Chem. Lett. 22, 1929–1987. https://doi.org/10.1007/s10311-024-01726-2
- Park, J., Tsang, Y.F., Lee, D., Cho, S.-H., Kwon, E.E., 2024. Syngas Production from Textile Dyeing Sludge via Carbon Dioxide-Assisted Pyrolysis. J. Anal. Appl. Pyrolysis 106916. https://doi.org/10.1016/j.jaap.2024.106916
- Rubi, R.V.C., Allayban, J.P.O., Deduque, D.A.B., Mena, J.A.B., Robles, N.M.D., Rubio, B.D., Roque, E.C., Joy Janaban, P., Olay, J.G., 2023. Production and characterization of activated carbon from pyrolysis biochar of cellulosic cotton-based textile wastes. Mater. Today Proc. https://doi.org/10.1016/j.matpr.2023.05.443