

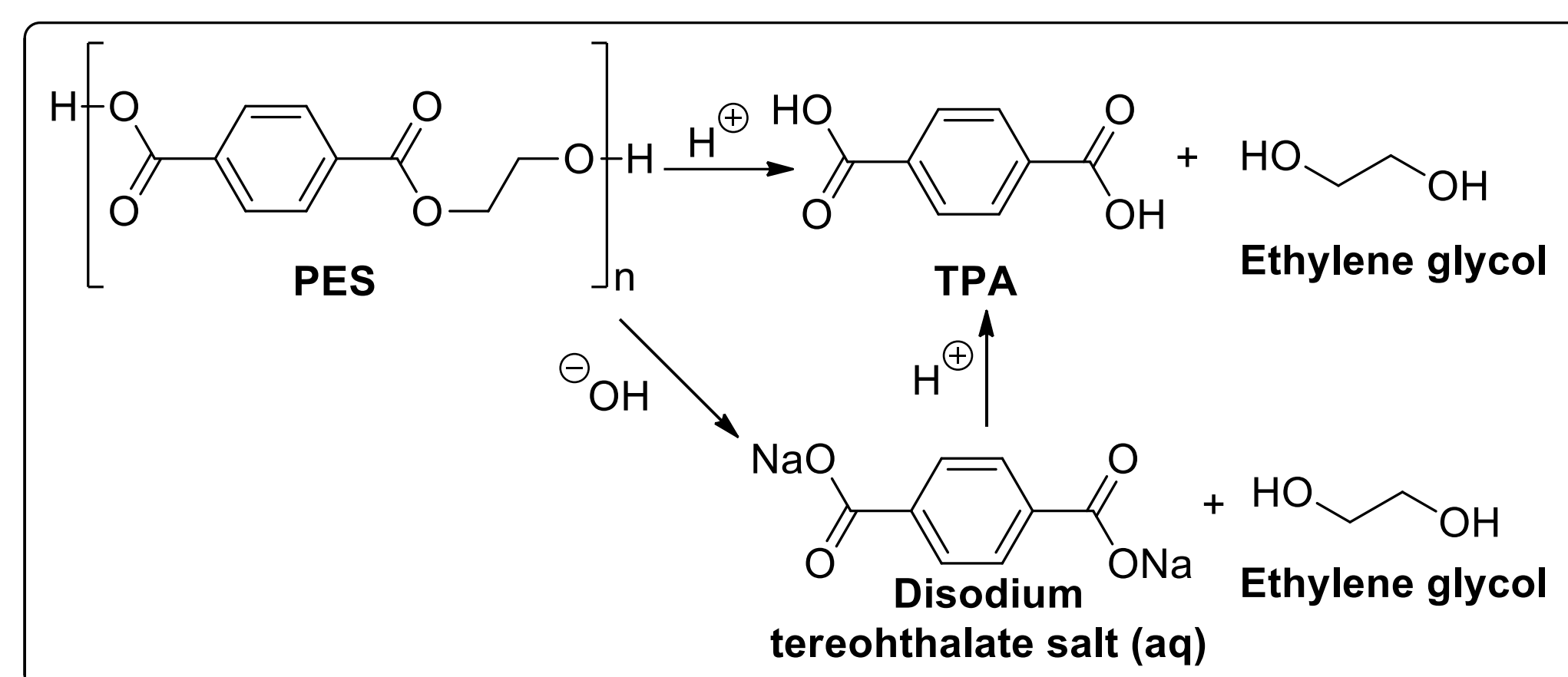
Recovery and recycling of cotton and cotton-containing garments

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Introduction

Textile waste represent an abundant source of synthetic and natural fibres (nearly 92 Mt year) that must be reused to mitigate the environmental impact of the petrochemical extraction, as well as an intensive use of chemicals and water requirements. In this work, cotton (CO) recovery was tested through selective removal of polyester (PES) from the mixture and subsequent terephthalic acid (TPA) isolation (Scheme 1), using post-consumer polycotton textile waste (Figure 1).



Scheme 1. Schematic representation of the acidic and alkaline hydrolysis of PES and respective products



Figure 1. Pictures of the original materials. From left to right: PCS (75% PES/23% CO/2% EL) and SQUASH POPLIN (84% CO/14% PES/2% EL)

Optimization of PES Hydrolysis

PES removal was optimized using different co-solvents for 24 hours, bath ratio of 1:50 and 50 °C (Figure 2). The best results were obtained when NaOH 2M/DMSO or NaOH 3M/isopropanol. This was confirmed by the disappearance of the C=O stretching signal at 1713 cm⁻¹ characteristic of PES fibre (Figure 2).

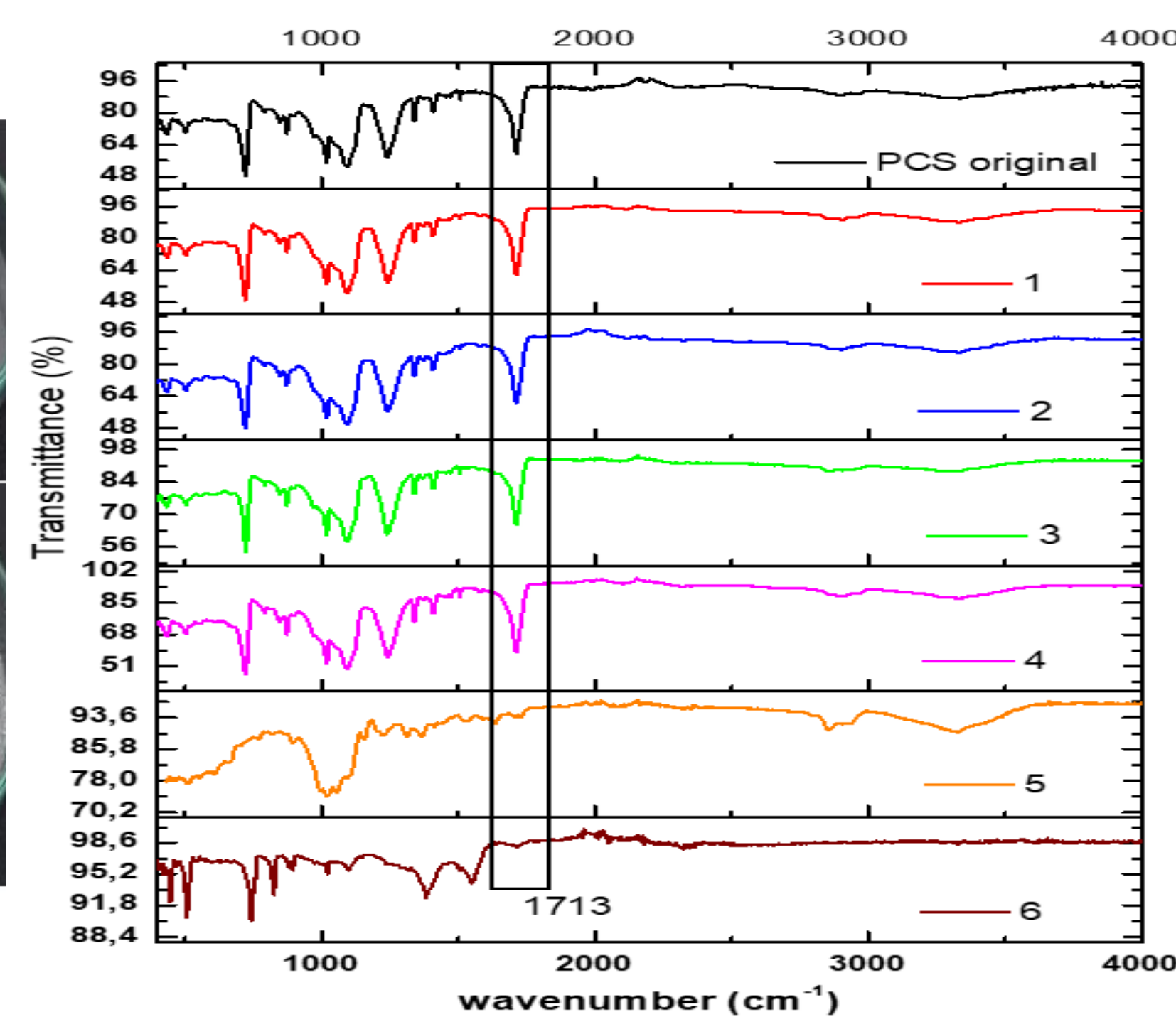
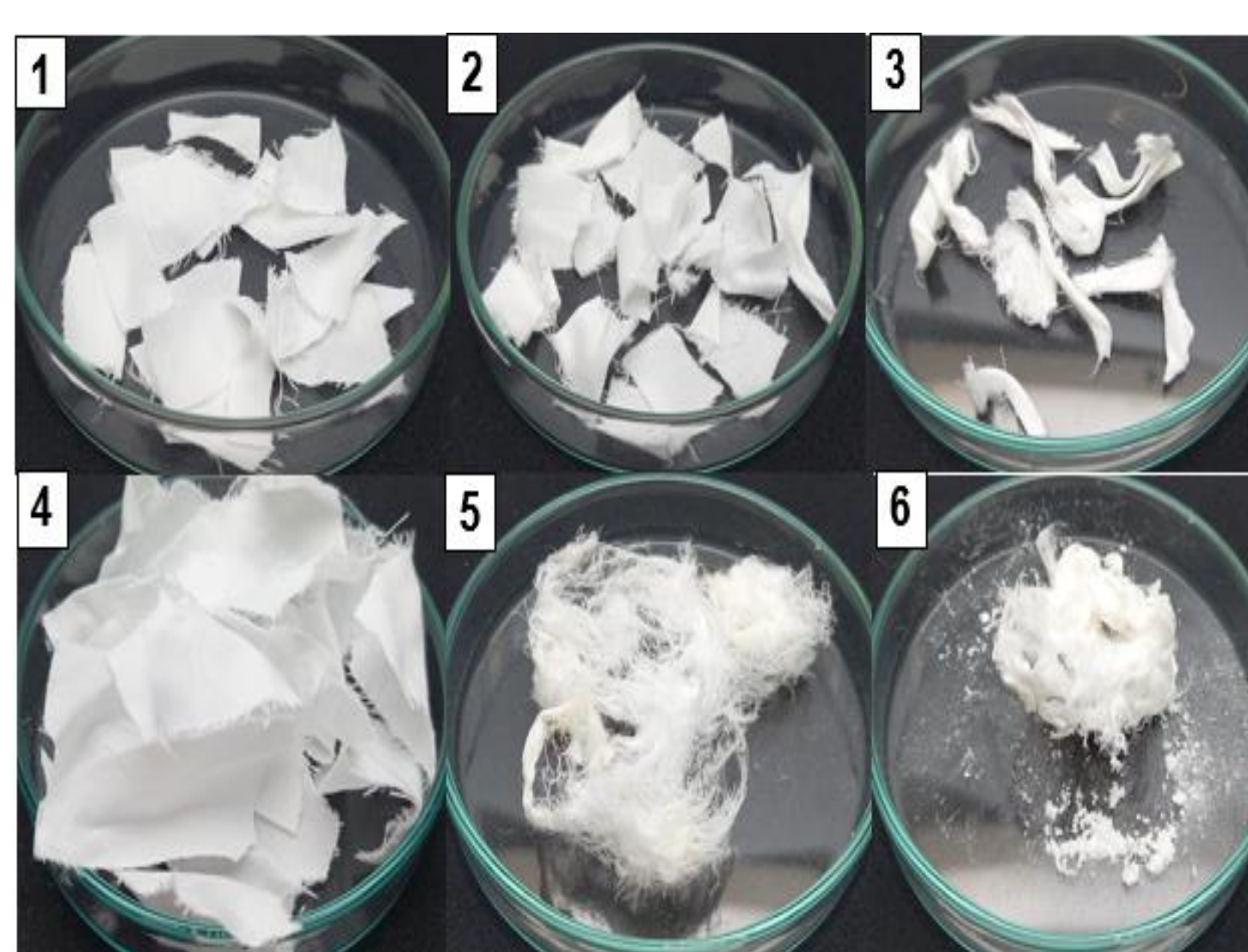


Figure 2. Pictures of a selection of the recovered material after optimization of the reaction conditions, within a fixed time of 24 hours, bath ratio of 1:50 and 50 °C (left), and superimposition of their respective FTIR ATR spectra (1) NaOH 2M; (2) NaOH 2M, DMSO 10% (v/v); (3) NaOH 2M/acetone (1:1); (4) NaOH 2M/HFIP (1:1); (5) NaOH 2M/DMSO (1:1); (6) NaOH 3M/isopropanol (1:1)

TPA recovery

For TPA recovery, the laundering mixture was acidified to pH= 2 and stirred at 50 °C for 2 hours, then cooled to 0 °C and filtered under vacuum. TPA recovery denoted moderate to high yields, with high degree of purity as confirmed by ¹H NMR (Figure 4).

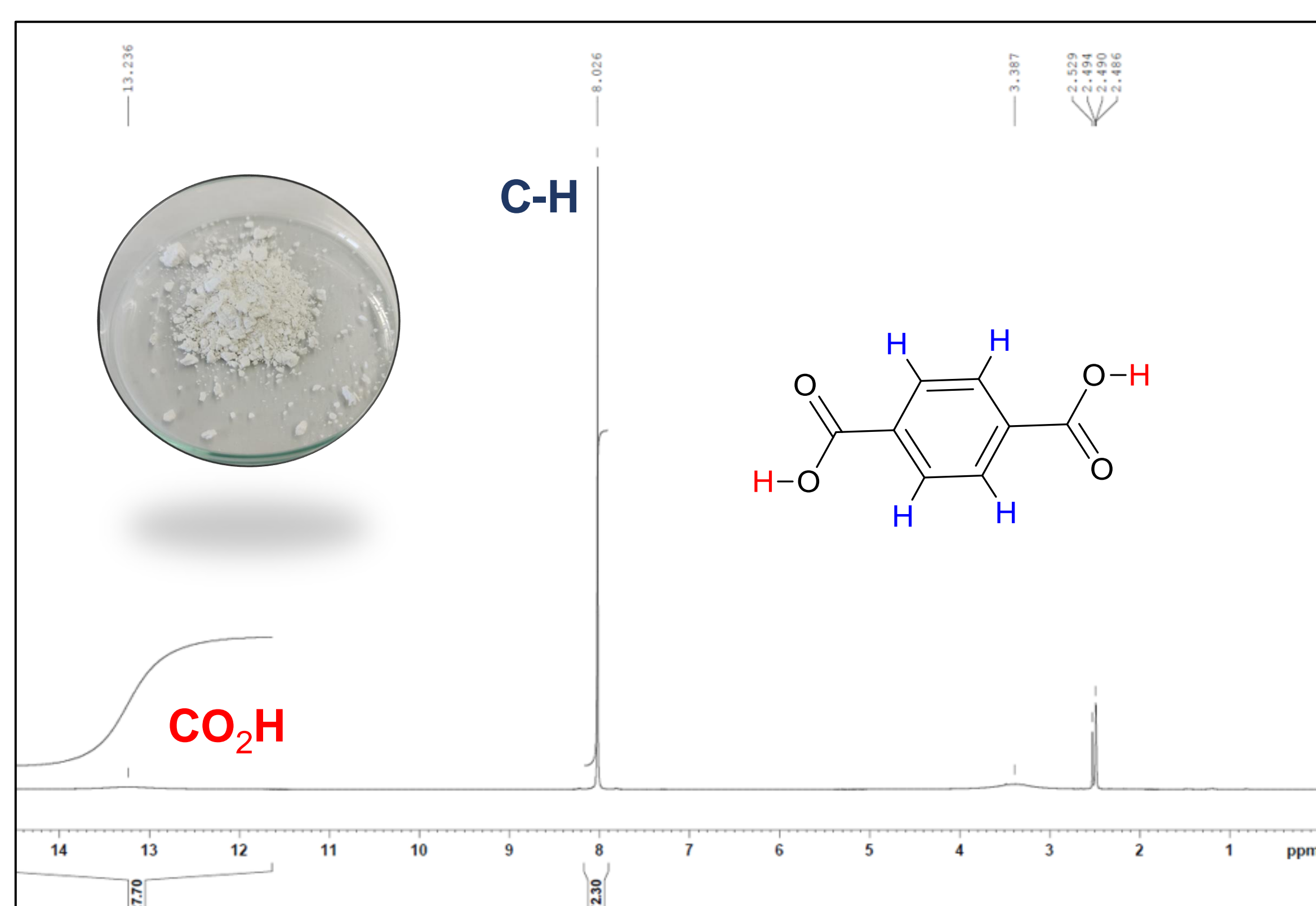


Figure 4. Example of recovered TPA from the hydrolysis of PET using NaOH 2M/DMSO 1:1, 50 °C, Bath ratio of 1:50, for 24h (top); and its respective ¹H NMR spectrum (400 MHz, DMSO-d₆; bottom)

Aknowledgments

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PES removal cycles

The best results were obtained using NaOH 2M with DMSO or iso-propanol. Hydrolysis cycles were performed to evaluate the mixtures' capacity for PES removal in successive uses, displaying a maximum yield of 3 cycles, with nearly 100% of CO recovery (Figure 3A and B). The removal of PES was followed by FTIR ATR spectroscopy (figures 3C and D), targeting the absence of the 1713 cm⁻¹ signal.

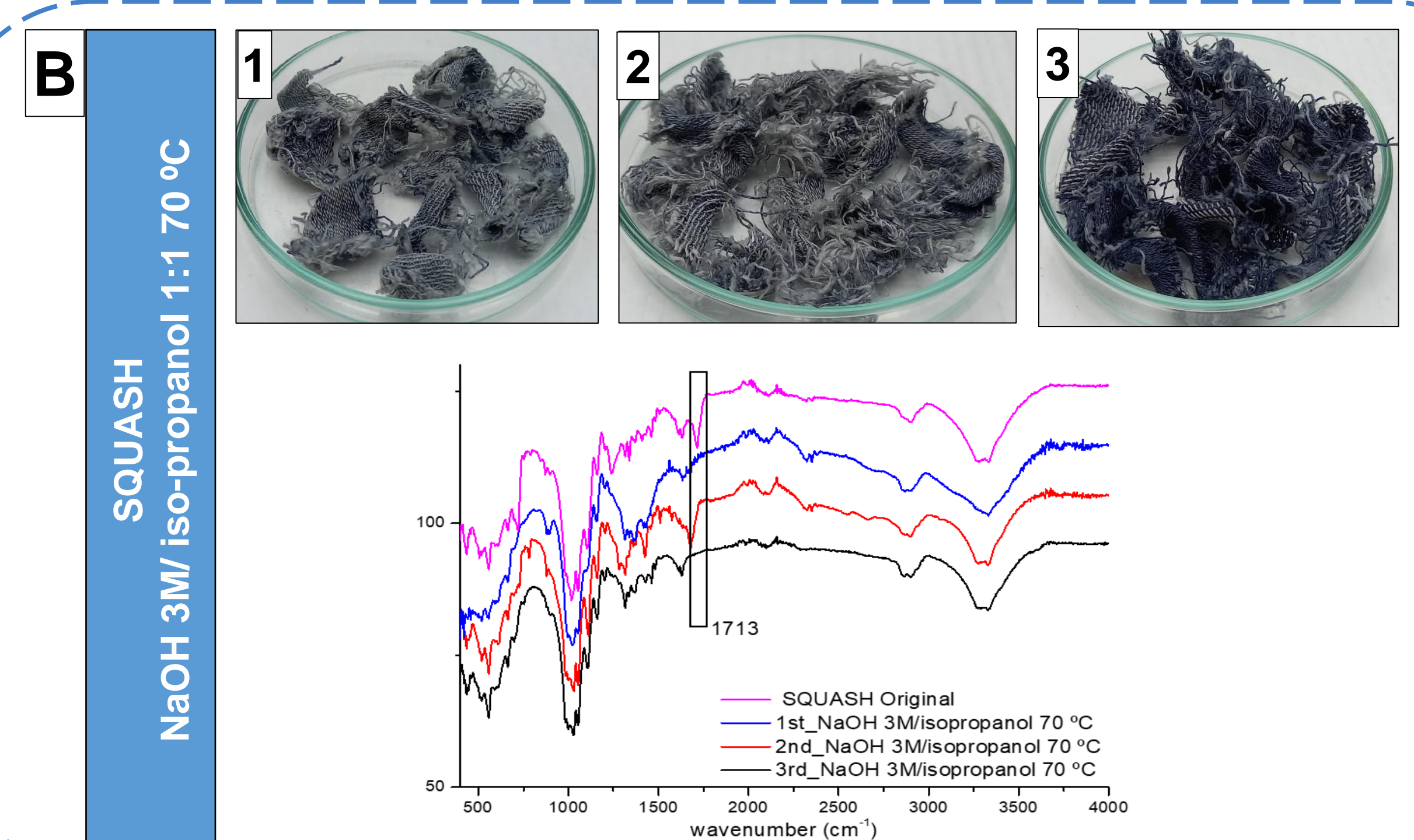
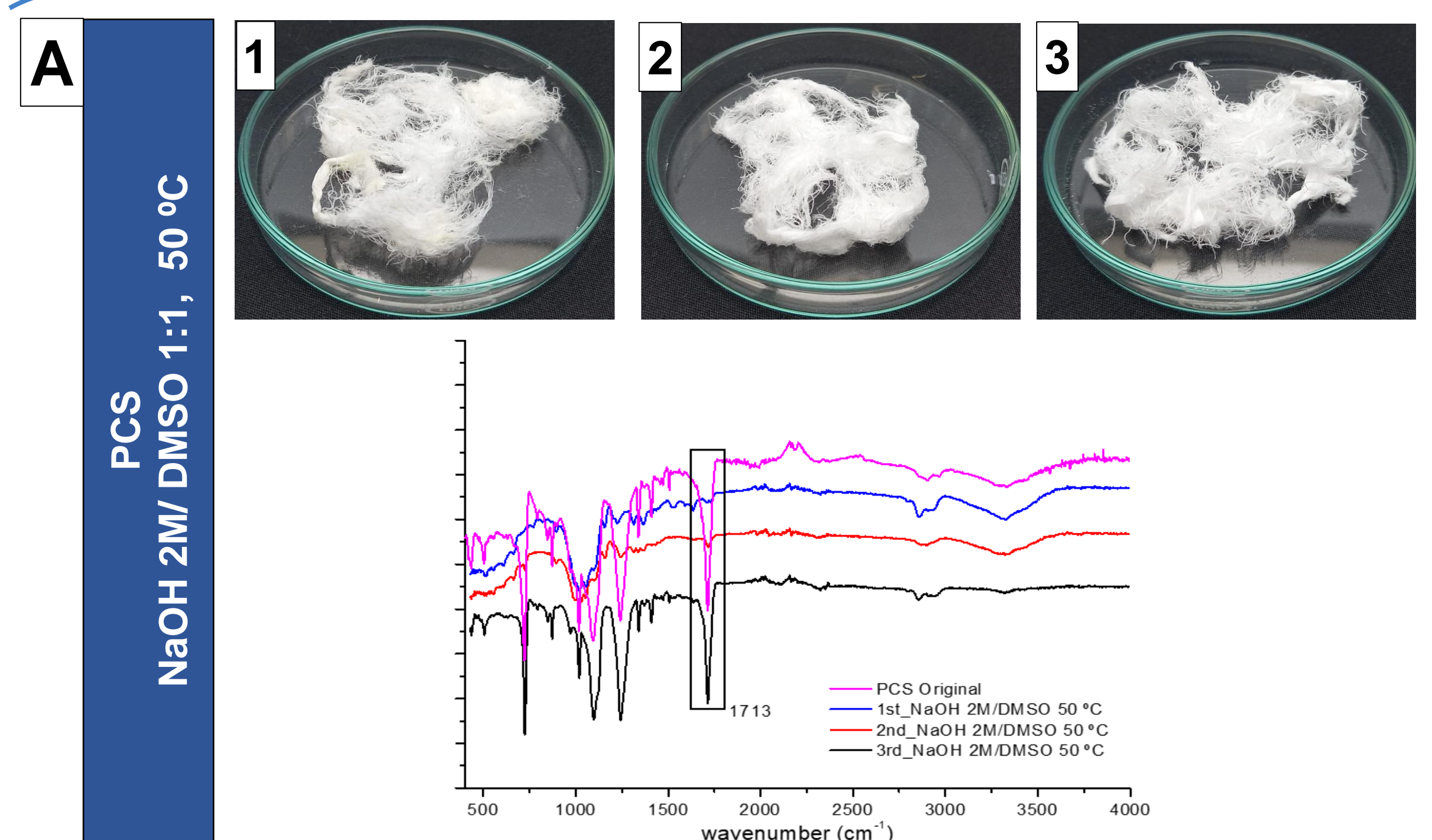


Figure 3. Pictures of a selection of the recovered material after successive hydrolysis cycles, from 1st to 3rd cycle, and respective FTIR ATR spectra; (A) PCS; (B) SQUASH

Conclusions

- ✓ A new and effective method for the recovery of CO and removal of PES was achieved in high yields regarding both cotton recovery and polyester degradation.
- ✓ The optimized reaction conditions were successfully optimized for usage in tandem hydrolysis cycles up to 3 cycles.
- ✓ Both the recovered CO and TPA demonstrated good properties that allow a reincorporation into the industrial process.
- ✓ Further studies are being completed to evaluate the mechanical and physical properties of the recovered CO.