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Development of an efficient route for combined recycling of PET and cotton from mixed fabrics

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Abstract

Most textile waste is either incinerated or landfilled today, yet, the material could instead be recycled through chemical recycling to new high-quality textiles. A first important step is separation since chemical recycling of textiles requires pure streams. The focus of this paper is on the separation of cotton and PET (poly(ethylene terephthalate), polyester) from mixed textiles, so called polycotton. Polycotton is one of the most common materials in service textiles used in sheets and towels at hospitals and hotels. A straightforward process using 5–15 wt% NaOH in water and temperature in the range between 70 and 90 °C for the hydrolysis of PET was evaluated on the lab-scale. In the process, the PET was degraded to terephthalic acid (TPA) and ethylene glycol (EG). Three product streams were generated from the process. First is the cotton; second, the TPA; and, third, the filtrate containing EG and the process chemicals. The end products and the extent of PET degradation were characterized using light microscopy, UV-spectroscopy, and ATR FT-IR spectroscopy, as well as solution and solid-state NMR spectroscopy. Furthermore, the cotton cellulose degradation was evaluated by analyzing the intrinsic viscosity of the cotton cellulose. The findings show that with the addition of a phase transfer catalyst (benzyltributylammonium chloride (BTBAC)), PET hydrolysis in 10% NaOH solution at 90 °C can be completed within 40 min. Analysis of the degraded PET with NMR spectroscopy showed that no contaminants remained in the recovered TPA, and that the filtrate mainly contained EG and BTBAC (when added). The yield of the cotton cellulose was high, up to 97%, depending on how long the samples were treated. The findings also showed that the separation can be performed without the phase transfer catalyst; however, this requires longer treatment times, which results in more cellulose degradation.

Keywords: Polycotton, Textile recycling, Alkaline hydrolysis, Polycotton separation, Polyester recycling, Cotton recycling

Background

Today, there are well-established systems for the material recycling of glass, metals, and paper. However, to accomplish the material recycling of more complex materials, such as textiles, efficient methods for the separation of the components are needed. Since there are systems for the collection of textiles (Elander and Ljungkvist 2016), a major barrier to accomplish textile recycling is the large mix of materials, coatings, dyes, and non-textile objects (Wang 2006). This diversity is especially challenging in the chemical recycling of textiles. Due to greater awareness of the high environmental cost of textiles, the interest in textile recycling has increased very much in recent years. This creates a high demand for the development of recycling technology. Chemical recycling requires pure fractions, and thus, the development of separation processes is a central issue for the progress of textile recycling.

Chemical fiber-to-fiber recycling comprises different processes where changes on the molecular level are made to the textile fibers, through chemical processing, to form recycled fibers. Textiles may also be recycled mechanically, i.e., in processes where the textiles are

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mechanically defibrated to fibers, which may then be spun with or without the addition of virgin fibers into yarns for textile production. However, the mechanical recycling of most textile materials leads to fibers of inferior quality and may, thus, be regarded as downcycling (Morley et al. 2006). No commercial-scale chemical fiber-to-fiber recycling of textiles exists today; however, small-scale projects are ongoing, such as Eco Circle (Teijin), Worn Again, Evrnu, Re:newcell, and Ioncell (Asaadi et al. 2015; Elander and Ljungkvist 2016).

Polyester (poly(ethylene terephthalate)), PET or PES, and cotton are the most common synthetic and natural fibers, respectively, used in the world today (CIRFS European Man-Made Fibres Association 2016). The differences in their strengths and weaknesses make the two a perfect combination. Cotton contributes with softness and high water absorbency, and PET contributes with durability, strength, and a low price (Hatch 1993). This makes mixes of PET and cotton, in varying percentages, one of the most common textile materials, which are generally referred to as polycotton. Polycotton is used in the main part of all service textiles, such as sheets, towels, and workwear. Recycling of service textiles could be an important first step to achieving textile recycling since the service sector uses large quantities of textiles with similar quality.

In order to recycle polycotton chemically, cotton and PET must be separated. This may be done by depolymerizing or dissolving one of the components while maintaining the other. If the PET is to be maintained, it has been proposed that the cotton may either be degraded with acid (Ouchi et al. 2010) or with microbes (Bernd and Gerahard 1996). Dissolving cotton has also been proposed using different solvents, such as ionic liquids or *N*-methylmorpholine *N*-oxide (NMMO), and separating the two components with filtration (De Silva et al. 2014; Jeihanipour et al. 2010).

The opposite approach is also possible, in other words, maintaining the cotton and depolymerizing or dissolving the PET fibers. This approach was used in this study for two main reasons. First, if the PET is to be maintained and remelted into new fibers, then the PET should not be degraded during laundering and use. However, since laundering most commonly is performed under alkaline conditions, and the ester bonds in the PET fibers are sensitive to alkali, it is probable that significant degradation will have occurred after many laundering cycles (International Fabricare Institute 1995). This degradation may cause problems during remelting since PET, which has a molecular mass that is very low, may form fibers of inferior quality. This can, however, be avoided if the PET is degraded to its monomers and repolymerized. Second, cotton is a natural polymer, and it can, thus, not be repolymerized, which limits the use of the cotton
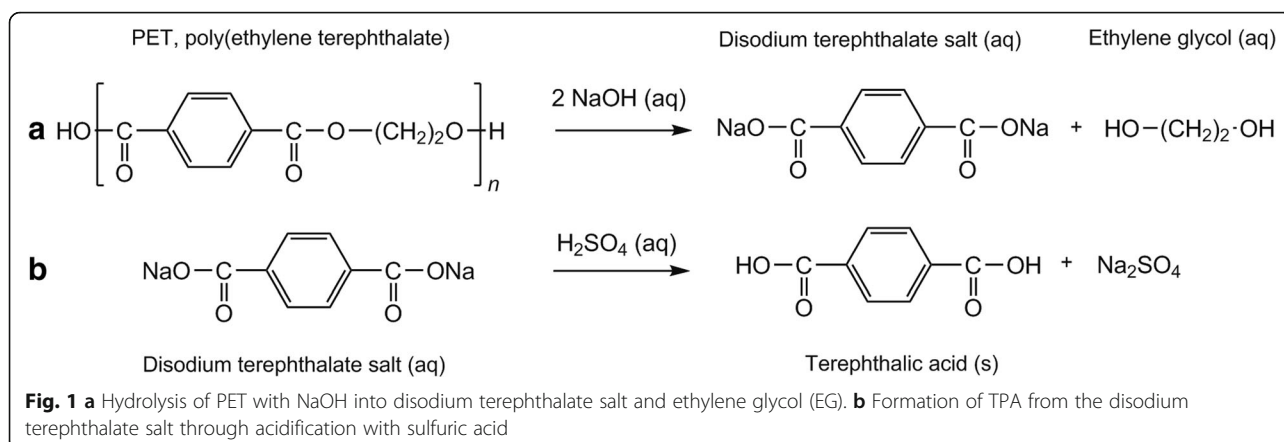
residue if it is severely degraded. An approach to maintaining the cotton and depolymerizing the PET has been proposed earlier, using both hydrolysis (Negulescu et al. 1998) and alcoholysis (Oakley et al. 1993) of the PET.

Recycling of PET through depolymerization has been extensively studied and reviewed due to the large volumes of PET bottles, which are creating a fast-growing waste problem (Al-Sabagh et al. 2016; Chen et al. 2011; Dutt and Soni 2013; George and Kurian 2014; Geyer et al. 2016; Paliwal and Mungray 2013; Sinha et al. 2010). There are three main chemical degradation methods for PET; hydrolysis (acid, neutral, or alkaline), alcoholysis (Oakley et al. 1993), and glycolysis (Viana et al. 2011). However, only hydrolysis degrades the PET back to terephthalic acid (TPA) and ethylene glycol (EG), which are the monomers mainly used in PET production today (Gupta et al. 1997).

Alkaline hydrolysis in sodium hydroxide (NaOH) solutions with the addition of a phase transfer catalyst has been shown to be very effective at depolymerizing PET at moderate temperatures (70–95 °C) and alkalinity in the range of 5–15% NaOH (Das et al. 2007; Kosmidis et al. 2001; López-Fonseca et al. 2009; Polk et al. 1999). The hydrolysis of PET is shown in Fig. 1. Ester bonds in the PET are cleaved by the nucleophilic attack of hydroxide ions under the formation of the disodium terephthalate salt and ethylene glycol, which are both soluble in the aqueous phase. When the reaction is finished, the aqueous phase is acidified to a pH around 2.5–3, which causes the formation and precipitation of TPA.

Without a phase transfer catalyst, the hydrolysis is relatively slow since the hydrophilic hydroxide ions are dissolved in the aqueous phase and the PET is a hydrophobic, solid material. The effect of the phase transfer catalyst is, thus, to carry the hydroxide ions to the surface of the PET to facilitate the reaction (Naik and Doraiswamy 1998). Therefore, the phase transfer catalyst should be cationic and have sufficient organic character to be lipophilic, but remain small enough to avoid steric hindrance (López-Fonseca et al. 2009; Polk et al. 1999). The most common types of phase transfer catalysts are quaternary ammonium or phosphonium salts with lipophilic side chains (Naik and Doraiswamy 1998). López-Fonseca et al. tested the difference between these two types and found only minor differences between phase transfer catalysts based on ammonium or phosphonium ions (López-Fonseca et al. 2009). It is, thus, the side chains that are the most important for the effect of the catalyst. Some attention, should, however, be paid to the counter ion to ensure that this does not create any problems further along the process.

No research papers dealing with alkaline hydrolysis as a method for degrading PET in polycotton textiles as part of a recycling process have been found in the literature. In this study, alkaline hydrolysis of polycotton, with

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Fig. 1 a Hydrolysis of PET with NaOH into disodium terephthalate salt and ethylene glycol (EG). **b** Formation of TPA from the disodium terephthalate salt through acidification with sulfuric acid

the addition of a phase transfer catalyst, was evaluated for the separation of cotton and PET from a blended textile. The aim was to generate end products that could be recycled into textiles. The isolated end products from the process were TPA, EG, and cotton. The separation and purification of EG were not considered in this study. The TPA may be used to produce new PET after repolymerization with ethylene glycol (recycled or virgin) and the cotton residue may, depending on its quality, possibly be recycled into regenerated cellulose fibers, such as viscose or lyocell.

Methods

Materials

New cotton/PET bed sheets supplied by a large supplier of Swedish service textiles were used in the study. The sheets were cut into smaller pieces and defibrated in a Wiley mill before being chemically treated. Benzyltributylammonium chloride (BTBAC, purity ≥98%), sodium hydroxide (NaOH, reagent grade), and sulfuric acid (H_2SO_4, reagent grade, 95–97%) were obtained from Sigma-Aldrich. The purchased chemicals were used without any further purification.

Small-scale experiments

Small-scale experiments were performed in 50-ml falcon tubes. The NaOH concentration was 10 or 15 wt%, and the BTBAC concentration was varied between 0 and 1 mol BTBAC/mol repeating unit in PET, where 1 mol/mol corresponds to 52 mmol BTBAC/kg hydrolysis solution. The hydrolysis experiments were begun by mixing NaOH and BTBAC in water to the selected concentrations and followed by heating the solution to the selected temperature (80 or 90 °C). The reaction was started by adding a 1.0 g ovendry (o.d.) polycotton sample to the reaction tube, and the hydrolysis was performed for the selected time period (15–240 min). The reaction was quenched by immersing the reaction tube into an ice bath. The remaining solid phase was separated using a glass

microfiber filter and rinsed with water. The liquid phase was acidified to pH 2–3 by the addition of H_2SO_4, which caused the terephthalic acid (TPA) to precipitate. The TPA was separated using a glass microfiber filter, rinsed with water, and after drying in an oven at 105 °C, the weight of the isolated TPA was determined. The yield of TPA was calculated as the percentage of the theoretical yield of TPA that can be obtained from PET assuming that the PET is formed by the esterification of equimolar amounts of TPA and EG.

Large-scale experiments

Large-scale experiments were performed in a glass reactor containing 500 g of hydrolysis solution. The NaOH concentration in the hydrolysis solution was 5, 7.5, or 10 wt%, and the temperature was 70, 80, or 90 °C. The BTBAC concentration was 52 mmol/kg hydrolysis solution. The hydrolysis was begun by mixing NaOH and BTBAC to the selected concentrations and heating the solution to the selected temperature. Then, a 5.0 g o.d. polycotton sample was added, and the hydrolysis was started. During the hydrolysis, samples of the hydrolysis solution, 1.5 g at a time, were taken out regularly to follow the course of the reaction.

After the selected reaction period (240–420 min), the reaction was stopped by filtering off the solid (cotton) residue through a glass microfiber filter. The cotton residue was then rinsed with water, dried, and weighed. The liquid phase was acidified and isolated as described in the small-scale experiments.

Microscopy

Micrographs were obtained to follow the hydrolysis of polycotton fibers over the course of 1 h using time-lapse mode with a Zeiss SteREO Discovery.V12, equipped with an Axio Cam IC1 and a Linkam PE120 heating stage, at 80 °C. The polycotton fibers were treated with an alkaline solution containing 10 wt% NaOH and 0.1 M BTBAC.

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Solid-state ^{13}C NMR spectroscopy

Solid-state NMR experiments were performed using a Varian Inova-600 instrument operating at 14.7 T and equipped with a 3.2-mm solid-state probe. All measurements were conducted at 298 K at a MAS spinning rate of 15 kHz with a cross-polarization magic angle spinning (CP/MAS) pulse sequence. The number of acquisitions for each spectrum was 16,384. All NMR spectra were processed with MestReNova 8.1 software. First-order polynomial baseline correction was used in all spectra.

Solution-state NMR spectroscopy

Solution-state NMR experiments were performed on a Bruker Advance III HD operating at 18.8 T and equipped with a 5-mm TXO cryoprobe and a sample-changing robot. Measurements were performed at 298 K. For ^{1}H spectra, 64 acquisitions were recorded. For ^{13}C spectra, 1024 acquisitions were recorded.

Before analyzing the filtrate sample, water was evaporated prior to the addition of DMSO-d_6 to the solid residue. The isolated TPA was directly dissolved in DMSO-d_6.

Analysis of TPA content using UV-absorption spectroscopy

Absorbance at 242 nm was collected using a UV-vis spectrophotometer (Specord 205, Analytic jena) with the initial hydrolysis solution as the background. The concentration calculations for the large-scale experiments were adjusted for the removal of solvent and TPA.

Attenuated total reflectance Fourier transform infrared spectroscopy (ATR FT-IR)

The spectra were recorded at room temperature in air from 4000 to 400 cm^{-1} with 20 scans using a PerkinElmer FT-IR spectrophotometer with a diamond ATR attachment GladiATR from Pike Technologies. The resolution was 2 cm^{-1}, and the interval scanning was 0.5 cm^{-1}.

Intrinsic viscosity

The intrinsic viscosity of the cellulose samples was measured after dissolution in CED (copper(II) ethylenediamine) by measuring the time required for each sample to run through a thin capillary, according to the ISO 5351:2010 "Pulps—Determination of limiting viscosity number in cupri-ethylenediamine (CED) solution standard."

Results and discussion

This study evaluated a process to separate cotton and PET from mixed textiles. Figure 2 shows an overview of the process. The process started with the degradation of PET into disodium terephthalate and ethylene glycol (EG). When the degradation was complete, the cotton that remained could be filtered off. The filtrate contained two

Fig. 2 Overview of the separation process of polycotton textiles presented in the current paper
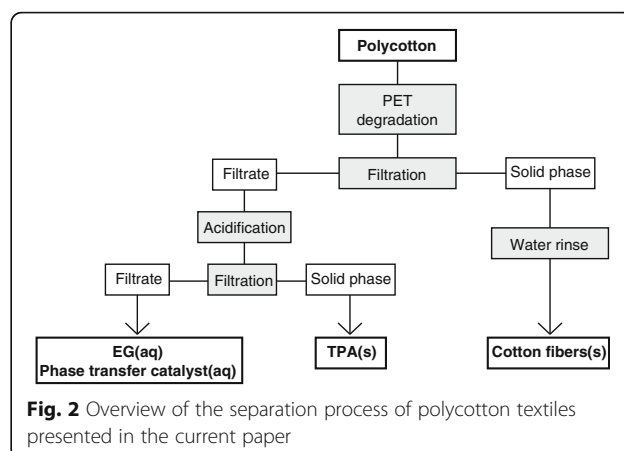
PET components (TPA in sodium form and EG). After acidification, the TPA was present in its protonated form, as a precipitate, which could also be filtered off. Thus, three product streams resulted from the process: cotton, TPA, and a filtrate containing EG and process chemicals.

Characterization of sheets

Before separation, the polycotton bed sheets were characterized using solid-state ^{13}C NMR spectroscopy and the method described in the European standard "Mixtures of cellulose and polyester fibres (method using sulfuric acid)" (ISO 1833-11:2006). Figure 3 shows the solid-state NMR spectra of (a) the polycotton mix, (b) a pure PET sample, and (c) a pure cotton sample. The figure shows that signals from both materials (PET and cellulose) are present in the polycotton sheets. No signs of other materials were observed in the spectra. Analysis according to ISO 1833-11:2006 showed that the PET content of the sheets was 52%.

Hydrolysis and separation of TPA

The selective hydrolysis of PET can be followed on the micrographs in Fig. 4, which were performed at 80 °C. The hydrolysis solution contained 10% NaOH and 0.1 mol BTBAC per kg solution. The micrographs illustrate that during hydrolysis, PET was degraded while the cotton fibers only swelled. Throughout the course of the PET degradation, the micrographs revealed the formation of a new phase surrounding the areas where the PET fibers were located, seen as liquid bubbles in the micrographs. When PET fibers are hydrolyzed, sodium terephthalate is released. This new phase can, therefore, tentatively be explained by the high local concentrations of sodium terephthalate.

Effect of concentration of NaOH and phase transfer catalyst on hydrolysis

In the small-scale experiments, the samples were treated for 100 min, and then, the reaction was quenched and

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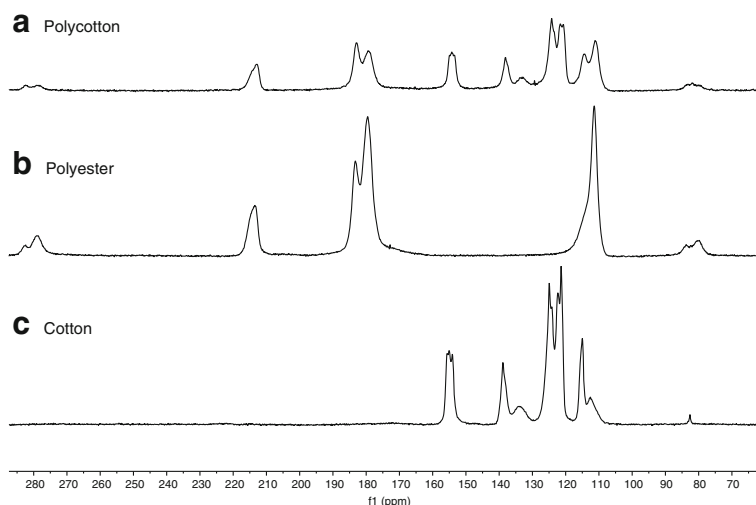
Fig. 3 Solid-state NMR spectra of **a** the polycotton sheets used in this study, **b** pure polyester (PET), and **c** pure cotton

the products were collected, as described in the "Methods" section. The impact of the NaOH and BTBAC concentration and temperature on PET degradation was investigated using this set-up. The results in Fig. 5a show that the alkaline hydrolysis of PET without the addition of BTBAC gave a TPA yield of 97% after 100 min with 15% NaOH in the solution. At NaOH concentrations ≤5%, virtually no TPA was released without the addition of BTBAC. When hydrolysis was performed at the same conditions, 90 °C and 100 min, but with the addition of 0.1 mol BTBAC per mol repeating unit in PET, only 10% NaOH was needed to almost complete PET hydrolysis. In addition, when the BTBAC was added, substantial PET degradation was observed as early as at 5% NaOH.

Figure 5b shows the effects of temperature and the addition of the BTBAC on the extent of hydrolysis after 100 min. At the lower temperature tested, 80 °C, a clear effect of an increase in the addition of BTBAC on the extent of hydrolysis can be seen in the figure. It should be noted that to reach complete PET hydrolysis after

100 min in 80 °C, an addition in the range of 0.7–1 mol BTBAC per mol repeating unit in PET is required. When the temperature is increased to 90 °C, the hydrolysis rate increases, and in order to reach the same degree of hydrolysis in 100 min reaction, the addition of only 0.05 mol BTBAC per mol repeating unit is needed.

Hydrolysis at different times and alkalinities

In the large-scale experiments, samples were removed continuously during the experiment and analyzed for TPA using UV-vis spectroscopy. This made it possible to follow the extent of PET hydrolysis as it proceeded. The residual cotton was isolated after the experiments were completed, and the yield and intrinsic viscosity were determined. In Fig. 6a, hydrolyses at 70 °C with 5, 7.5, and 10% NaOH are compared. The results show that with increasing NaOH concentration, the rate of hydrolysis increases. The experiment with 5% NaOH at 70 °C was stopped after 120 min, and thus, only a 66% TPA yield was reached.
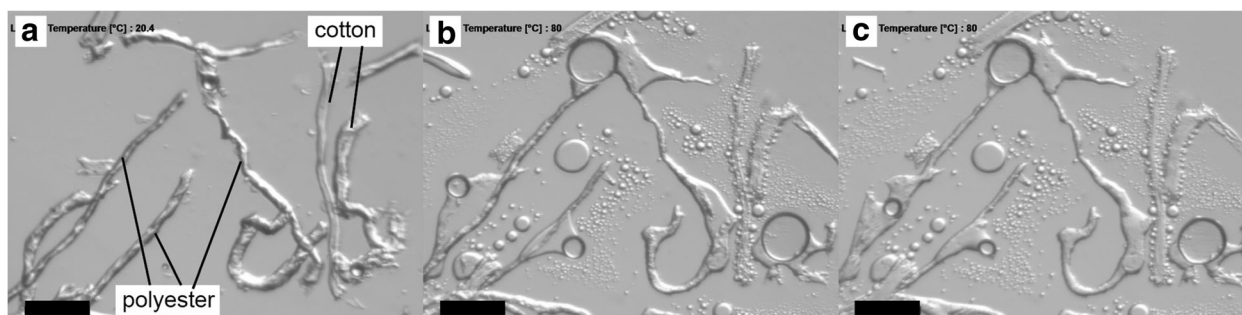
Fig. 4 Micrographs of selective hydrolysis of PET in 10% NaOH, 0.1 mol BTBAC/kg solution at 80 °C, **a** at start, 23 °C, **b** after 37 min, 80 °C, and **c** after 1 h, 80 °C. The *black scale bar* indicates 0.1 mm

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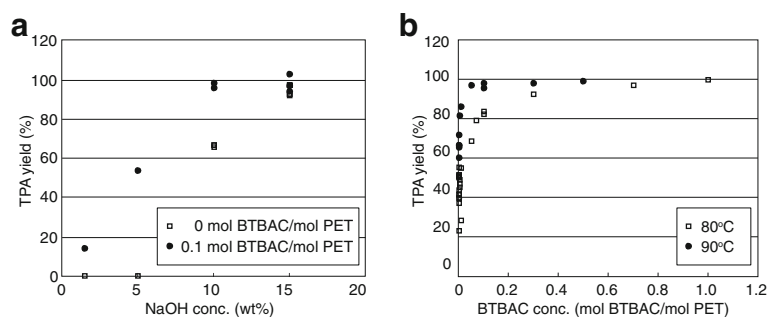
Fig. 5 a Yield of TPA against the NaOH concentration, with and without the addition of BTBAC at 90 °C. **b** Yield of TPA against different BTBAC concentrations at 80 and 90 °C using 10% NaOH concentration. Both figures are based on data from the small-scale experiments, and the reaction time was 100 min

In Fig. 6b, the time dependence of hydrolysis at 70, 80, and 90 °C and 10% NaOH with BTBAC is compared to hydrolysis at 90 °C and 10% NaOH without any addition of BTBAC. Results in the figure show that the reaction rate increased with temperature. Hydrolysis without BTBAC was much slower than with this additive. However, after approximately 150 min, the PET had been completely degraded. This shows that the additive only impacts the rate of the degradation and that complete degradation can be accomplished without any additive.

Purity of degradation products from PET
The purity of the resulting materials was tested after hydrolysis. As described in the "Methods" section and as shown in Fig. 2, the TPA was precipitated by lowering the pH (with the addition of H_2SO_4) and was separated from the hydrolysis solution through filtration. Solution-state ^1H and ^{13}C NMR spectra of the precipitated fraction (dissolved in DMSO-d_6) are shown in Fig. 7. The only signals that could be observed were from the TPA, showing that the purity was very high.

In order to analyze the filtrate from the large-scale experiments, the water was evaporated from the filtrate, and the solid residue was dissolved in DMSO-d_6.

Filtrates from experiments both with and without BTBAC were included and analyzed with solution-state ^1H NMR and ^{13}C NMR spectroscopy, see Fig. 8.
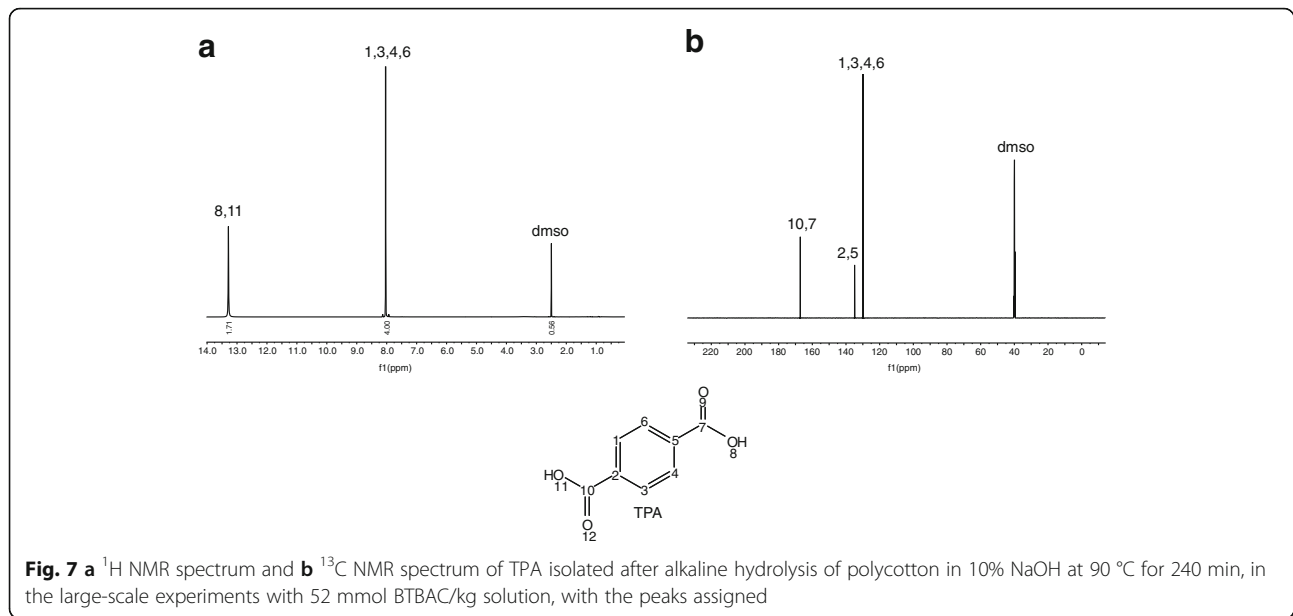
Analysis of the NMR spectra of the filtrate without BTBAC, in Fig. 8a, c, showed that the EG was the dominant peak, and no other components could be found in the filtrate. In the filtrate with BTBAC, in Fig. 8b, d, only BTBAC and EG were found. It was expected that at least signs of some cellulose degradation products, i.e., hydroxy acids, would be present, since cellulose is known to generate such degradation products when treated under alkaline conditions, cf. e.g., (Sjöström 1993). However, more in-depth analysis would probably reveal the presence of some cellulose degradation products.

Cellulose characterization
Figure 9a shows ATR FT-IR spectra of samples treated in 5% NaOH at 70 °C for 120 min and in 10% NaOH at 90 °C for 240 min. The sample treated in 5% NaOH still contained some unhydrolyzed PET, cf. Fig. 6a, and the peaks seen around 1240 and 1720 cm^{-1} are most likely related to ester linkages in the PET that remained in the sample (Peterson 2015). However, these peaks are not
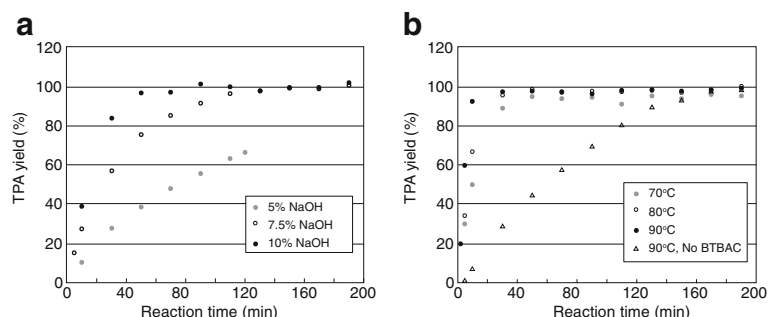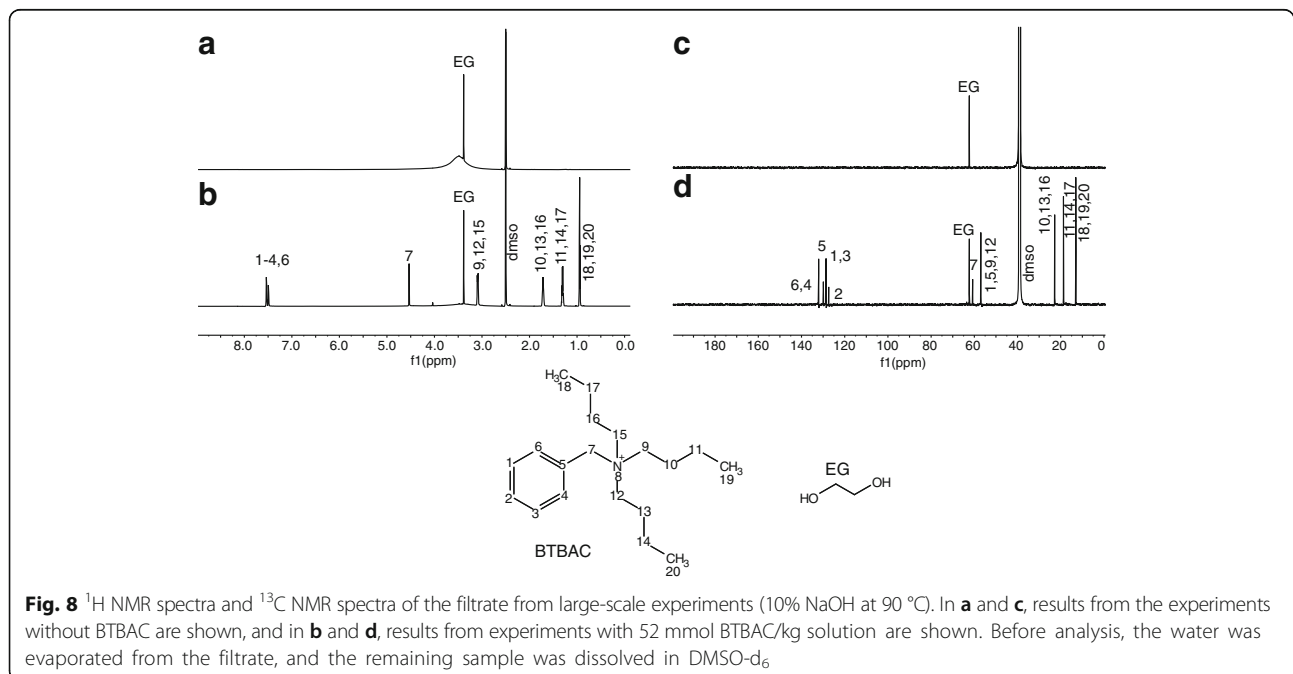

Fig. 6 Time dependence of degradation with **a** increasing NaOH concentrations (5, 7.5, and 10% NaOH at 70 °C) and **b** increasing temperature (10% NaOH and 70, 80, and 90 °C at 10% NaOH, with and without BTBAC (52 mmol/kg solution)). Both figures are based on data from the large-scale experiments

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Fig. 7 a ^{1}H NMR spectrum and **b** ^{13}C NMR spectrum of TPA isolated after alkaline hydrolysis of polycotton in 10% NaOH at 90 °C for 240 min, in the large-scale experiments with 52 mmol BTBAC/kg solution, with the peaks assigned
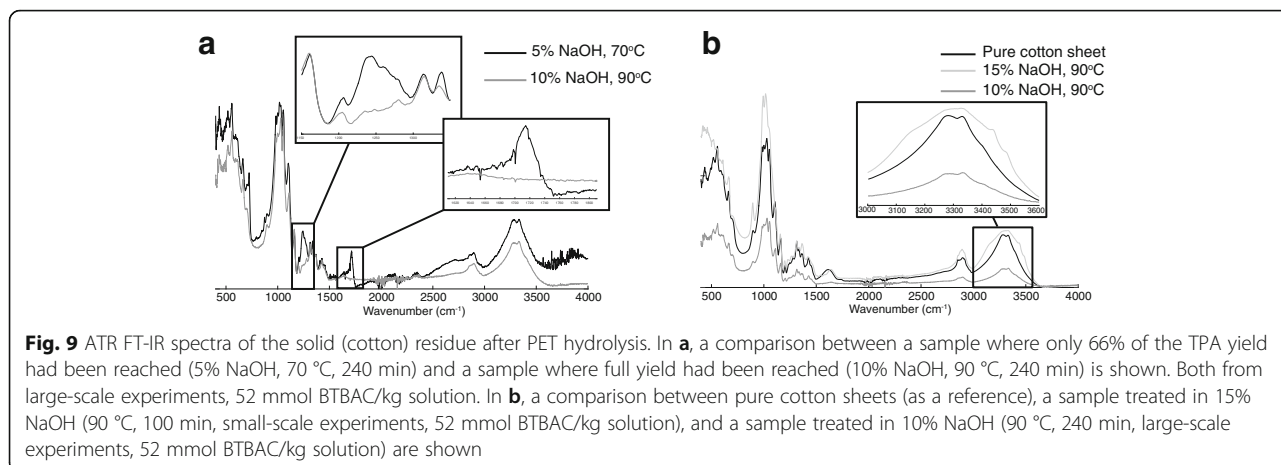
visible in the sample treated in 10% NaOH at 90 °C, which indicates complete PET removal.

Treatment of cellulose with alkaline solutions is known to cause the conversion of cellulose I into cellulose II (Sjöström 1993). The cotton samples were analyzed with ATR FT-IR to obtain a first indication if this conversion had occurred during hydrolysis. In Fig. 9b, the ATR FT-IR spectrum of a pure cotton reference sample is compared with spectra of samples treated in 10 and 15% NaOH at 90 °C. The reference sample was analyzed without any treatment. In the sample treated with 15% NaOH, the cellulose I seems to, at least partly, have been shifted to cellulose II. This was concluded because the two shoulders around 3447 and 3488 cm^{-1}, which are specific for intramolecular OH-stretching in cellulose II (Carrillo et al. 2004) are present in the sample treated in 15% NaOH, but not in the other two samples. The sample treated with 10% NaOH, on the other hand, seems to mainly consist of cellulose I since its spectrum is very similar to the spectrum of the pure

Fig. 8 ^{1}H NMR spectra and ^{13}C NMR spectra of the filtrate from large-scale experiments (10% NaOH at 90 °C). In **a** and **c**, results from the experiments without BTBAC are shown, and in **b** and **d**, results from experiments with 52 mmol BTBAC/kg solution are shown. Before analysis, the water was evaporated from the filtrate, and the remaining sample was dissolved in DMSO-d$_6$

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Fig. 9 ATR FT-IR spectra of the solid (cotton) residue after PET hydrolysis. In **a**, a comparison between a sample where only 66% of the TPA yield had been reached (5% NaOH, 70 °C, 240 min) and a sample where full yield had been reached (10% NaOH, 90 °C, 240 min) is shown. Both from large-scale experiments, 52 mmol BTBAC/kg solution. In **b**, a comparison between pure cotton sheets (as a reference), a sample treated in 15% NaOH (90 °C, 100 min, small-scale experiments, 52 mmol BTBAC/kg solution), and a sample treated in 10% NaOH (90 °C, 240 min, large-scale experiments, 52 mmol BTBAC/kg solution) are shown

cotton sheet. Based on the ATR FT-IR analysis, however, it cannot be excluded that a small amount of cellulose I had been converted to cellulose II.

Unwanted cellulose degradation reactions may also take place in the cellulose part of a polycotton sample during the strongly alkaline conditions applied in the PET-removing treatment. Such reactions lead to the cleavage of cellulose chains, which results in a lower degree of polymerization (DP) of the cellulose. Furthermore, the chain cleavage will give rise to new end-groups in the cellulose, which are sensitive to alkaline-induced reactions and lead to the formation of low-molecular degradation products (Sjöström 1993). In order to investigate the extent of loss of cotton and the decrease in the degree of polymerization, the cotton recovered after PET hydrolysis treatments was isolated and weighed. The weight-based yield and the intrinsic viscosity of cellulose could then be calculated.
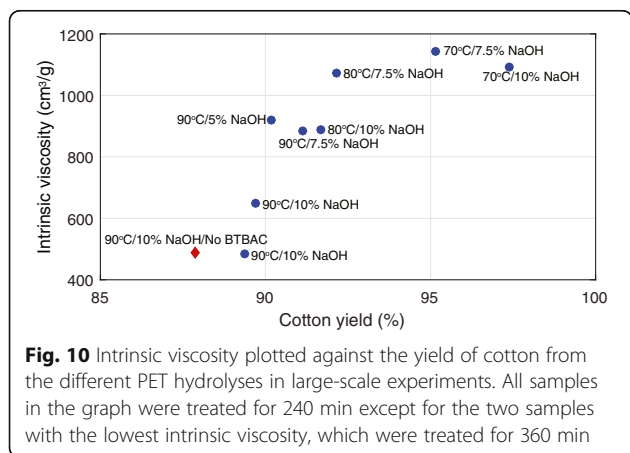
In Fig. 10, the intrinsic viscosity of the cotton cellulose is shown plotted against the yield of cotton. The intrinsic viscosity of cellulose may be correlated to the degree of polymerization (DP), and the results in the graph indicate that temperature had a major effect on the extent of

Fig. 10 Intrinsic viscosity plotted against the yield of cotton from the different PET hydrolyses in large-scale experiments. All samples in the graph were treated for 240 min except for the two samples with the lowest intrinsic viscosity, which were treated for 360 min

cellulose degradation, both on yield loss on and the decrease in intrinsic viscosity. However, results from the milder treatments show that complete PET removal can be achieved without any severe degradation of the cellulose. The two samples that had the lowest yield and intrinsic viscosity had been treated for 360 min, instead of 240 min, which shows impact of time on degradation. If this process was to be implemented, discarded sheets would be used, and they would probably have a lower intrinsic viscosity since laundering is well known to decrease the intrinsic viscosity of cotton (Palme et al. 2014).

Conclusions

This article presented a method to separate polycotton sheets into the PET monomers TPA and EG and to preserve cotton residue. The findings showed that:

- PET can be completely hydrolyzed within 40 min in 10% NaOH at 90 °C with the addition of 52 mmol BTBAC/kg hydrolysis solution, and it can be recovered as pure TPA.
- Increasing the temperature, NaOH concentration, and BTBAC concentration increase the reaction rate.
- Hydrolysis without BTBAC also yields pure streams; however, longer reaction times are required, and thus, the cotton yield will be lower.
- The isolation of cotton and TPA as pure streams implies that recycling may be accomplished through repolymerization of the TPA with ethylene glycol (recycled or virgin) and that the cotton fibers could possibly be used in the production of regenerated cellulosic fibers.

Abbreviations
(M)EG: (Mono)ethylene glycol; ATR FT-IR: Attenuated total reflectance Fourier transform infrared; BTBAC: Benzyltributylammonium chloride; CED: Copper (II) ethylenediamine; CP/MAS ^{13}C-NMR: Cross-polarization magic angle spinning carbon-13 nuclear magnetic resonance; NaOH: Sodium hydroxide; NMR: Nuclear magnetic resonance; o.d.: Oven dry; PET: Poly(ethylene terephthalate); TPA: Terephthalic acid

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Authors' contributions
A.Pa, HB, and HdlM designed the study. A.Pa, A.Pe, and HdlM performed the experimental work. All authors interpreted the results and wrote the manuscript. All authors have read and approved the final manuscript.

Competing interests
The authors declare that they have no competing interests.

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