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Improving Valorization of Waste Textiles: Assessing Separation Efficiency of Cotton–PET Blends via Alkaline and Enzymatic Hydrolysis

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Abstract

Recycling cotton–PET textile blends using alkaline solutions has gained increasing attention, with studies showing promising treatment pathways with diverse process setups. However, these separation processes use various input materials and focus on a small number of treatment parameter values which render the comparison of results over a large parameter range difficult. This study presents the feasibility of recovering cotton or PET at fabric level from cotton–PET blends across a wide range of temperatures (from −30 °C to 95 °C) and alkaline concentrations (from 0 to 40% (*w*/*w*)). The focus of this study is centered on the share of separation and recoverable fiber mass after hydrolyzing one component using alkaline hydrolysis or alkaline pre-treatment followed by enzymatic hydrolysis. A comparison of purity and material loss of the recovered polymers for all parameter sets is given. Experiments were performed on two distinct textiles while process parameters were selected in a straightforward manner, excluding catalysts, co-solvents and defibration. The results map temperature and alkaline concentration areas where these cotton–PET separation processes are feasible regarding recoverable fiber mass. Based on these results, separation efficiency could be optimized to design economic and environmentally friendlier process conditions.

Keywords: textile recycling; enzymatic hydrolysis; PET recovery; alkaline hydrolysis; cotton recovery

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1. Introduction

Global economy is facing newly arising and persistent difficulties, the two most urgent being criticality of resources and environmental impact of primary material consumption. The textile and fashion industry contributes over 1.7 billion tonnes of CO_2 annually, which account for up to 10% of global CO_2 emissions [1]. This can be attributed to the rise in textile production due to the reduction in the durability of textiles and apparel [2] and fast fashion trends [3]. Fiber production volume reached ~132 Mt in 2024 with polyester (77.7 Mt) and cotton (CO, 24.5 Mt) being the most dominant fiber types [4]. While CO fiber production has remained largely unchanged over the last twenty years, polyester fiber production has tripled [5]. Nowadays, many garments consist of fiber blends to improve comfort and functionality, typically combining polyester and CO [6,7]. However important these two materials may be for recycling, there are currently few industrially applicable methods for their recovery, as the separation of blended materials still poses

a considerable challenge [8,9]. Various approaches have been explored to recycle textile materials and these polymers in general [10–15]. Mechanical recycling, such as tearing and garneting, is widely used due to its simplicity and cost-effectiveness. It is, however, limited by fiber-length reduction and the ability to recycle textile blends effectively [10]. Thermomechanical recycling, for example melt spinning polyester, can overcome the limitation of fiber-length reduction, as the polymer is reprocessed into new fibers. Nevertheless, this approach requires well sorted input streams, since contaminants can significantly impair the process and product quality [14]. Chemical recycling offers a third main pathway by breaking down textile materials into their monomer or polymer building blocks. Thermolysis can be used to generate chemical raw materials but typically requires high energy input and does not allow fiber-to-fiber-recycling [10,16]. More selective approaches are based on solvolysis, in particular hydrolysis, enabling a targeted removal or conversion of individual components in blended materials. In this context, the number of studies on separation and recovery of polyester (in this case polyethylene terephthalate, PET) and cellulose (CO as well as manmade cellulosic fibers) from their blends has been increasing in recent years [7,17–20]. The general approach is to hydrolyze one component to recover the other, the key challenge, however, is to avoid degrading the target material. This is particularly relevant for cellulose, because once the average polymer chain length—the degree of polymerization (DP)—falls below a certain threshold, the material becomes unsuitable for subsequent spinning processes. For PET, however, this effect is less critical, because the molecular weight can be increased again through solid-state or liquid-state post-condensation [21–24]. When looking at the depolymerization of PET, research regarding packaging PET already exists [25,26] with an increasing number of publications addressing PET fibers as well [18,19,27–30]. Alongside relatively new approaches such as the use of enzymes for PET hydrolysis [31] or deep eutectic solvents [32], a commonly used method is separation using alkaline solutions which can be influenced using co-catalysts, co-solvents, or others. Catalysts are commonly employed to accelerate or enable chemical recycling processes; however, their use can be associated with increased costs, additional separation steps [33], as well as potential environmental impacts [34]. Compared to other solvolytic processes like alcoholysis or glucolysis, using aqueous alkaline solutions offers a comparatively simple process concept often without the need for catalysts, organic solvents, high pressure or high temperatures [35], thereby reducing process complexity. Furthermore, the reaction products of PET hydrolysis are its monomers terephthalic acid (TPA) and ethylene glycol (EG), used in PET production [29]. Palme et al. [18] demonstrated a viable approach hydrolyzing PET from defibrated CO-PET blends and recovering cellulose and terephthalic acid (TPA) by using sodium hydroxide solutions (NaOH) and a co-catalyst. However, this alkaline treatment affects not only the PET but also the DP of the cellulose. Over the last century, not only the effect of NaOH on PET but also its interaction with the cellulose–water system has been extensively researched [36,37]. Diluted NaOH solutions have remarkable swelling properties on cellulose, which can be noticed especially at low temperatures and concentrations [38]. Due to the properties of the sodium ion and its hydration shell in water, it is highly effective at disrupting the intermolecular hydrogen bonds within the cellulose polymer [39,40]. This results in swelling, which widens the space between the cellulose chains and makes it more accessible for further reactions and even allows smaller cellulose chains to be dispersed within the solution [41].

For a selective removal of cellulose from PET–cellulose blends, enzymatic hydrolysis is the most suitable pathway by converting cellulose into glucose using cellulases. Benefits of using enzymatic hydrolysis compared to other processes are the high selectivity of cellulases [42], mild process conditions [43] as well as their non-toxicity, environmental friendliness and lower energy consumption [44]. Vecchiato et al. [45] showed that

viscose—a regenerated cellulose fiber composed of short polymer chains—can be removed without any pre-treatment steps. However, for cellulose with a high DP and high crystallinity such as CO, a pre-treatment step is necessary for a complete hydrolysis [19,46]. Jeihanipour et al. [47] studied the conversion rate of CO fibers pre-treated with NaOH solutions into ethanol. Other publications (e.g., [19,48,49]) assessed either the glucose yield or the CO removal from CO-PET blend using different pre-treatment steps, predominantly NaOH solutions. At certain NaOH concentrations, mercerization of CO occurs, which describes the conversion from cellulose I to cellulose II. Stana-Kleinschek et al. [50] mention that NaOH concentrations of 12% are needed to start the mercerization process and when raising the NaOH concentrations between 15% and 20% NaOH at room temperature (RT), complete mercerization of CO occurs [51]. However, more recent publications also achieved a complete mercerization using only 1% NaOH below 0 °C [52].

Alkaline solutions appear to be a feasible approach either for the direct hydrolysis of PET or as a pre-treatment step for CO to enable a subsequent enzymatic hydrolysis, allowing recovery of both materials at the polymer or monomer level. Since both approaches use the same treatment principle, temperature and concentration of the alkaline solution have a decisive impact on separation and recoverability when using cellulose-PET blends. Existing studies in this field often concentrate on a small number of (pre-)treatment parameter values or narrow experimental conditions, with a primary focus on separation rather than quantifying recovery yields. In addition, many of these studies employ co-catalysts, co-solvents or energy-intensive processes such as steam explosion or pressurized systems [18,28,49,53]. This study therefore systematically investigated the influence of temperature and NaOH concentrations on the separation of cotton or PET from textile material blends. It examined (i) the separation of CO and the recovery yield of PET at fabric level using alkaline pre-treatment and enzymatic hydrolysis and (ii) the separation of PET and the recovery yield of CO at fabric level, using alkaline hydrolysis. Two different textile materials—60:40 cotton–PET pre-consumer fabric and 50:50 cotton–PET post-business fabric—were tested across a broad range of parameters. By deliberately maintain a simple and practical setup, these approaches facilitate a reduction on overall process complexity and material intensity. Avoiding co-catalysts and co-solvents limit the number of auxiliary chemicals required, which facilitates process handling and simplifies solvent recovery and recycling. In addition, these processes operate under relatively mild conditions which can lower the overall energy intensity compared to more complex recycling processes. The recovery and separation efficiency for the respective polymers were quantified for all parameter value sets.

2. Results and Discussion

This section is structured to systematically present the experimental findings according to the investigated materials and process conditions. Section 2.1 describes the tests on pre-consumer textiles, where screening was performed over a large temperature and alkaline concentration range. Sections 2.1.1 and 2.1.2 elaborate on the results of CO separation and PET loss which are compared directly in Section 2.1.3; Section 2.1.4 shows the results from the microscope images. Section 2.2 shows the tests on post-business textiles which were systematically conducted for both the recovery of CO and PET on a selected parameter range. Results for CO removal are described in Section 2.2.1 and for PET removal in Section 2.2.2.

2.1. Part 1: Tests on Pre-Consumer Textiles

For this first part of the study, alkaline pre-treatment and subsequent enzymatic hydrolysis was studied on pre-consumer CO–PET T-shirts. The composition stated on the label (60% CO, 40% PET) was initially checked and verified according to DIN ISO 1833-11:2017 [54] and AATCC Test Method 20-1977 [55]. The actual polymer mass fractions $w_{0,PET}$ and $w_{0,CO}$ were calculated after weighing the mass of the remaining fabric and relating it to the initial sample mass. Tables S2 and S3 in the Supplementary Materials contain the exact mass percentages as well as all the masses necessary for the calculations. The analysis focused on two key topics: the separation efficiency for CO and the quantification of recoverable mass and quality of PET.

2.1.1. Cotton Removal

Figure 1 shows the results for the separation efficiency of CO after alkaline pre-treatment and subsequent enzymatic hydrolysis. Using concentrations and temperatures starting from 15% NaOH and RT, a high to almost complete removal of CO from the fabric after a 6 h pre-treatment was observed. Similar results were achieved by Piribauer et al. [19] and Gritsch et al. [49] for defibrated samples using 20% NaOH at RT after 1 h and 3–6 h, respectively. As described in Section 1, at these conditions mercerization of CO is coming into effect. According to Haga et al. [56], mercerized cellulose is more accessible to enzymatic hydrolysis than cellulose I, which in turn can partially be attributed to the reduction in crystallinity [50]. However, raising NaOH concentration to 40%, a decrease in mass reduction was found when using temperatures at and below RT (see Figure 1). This can be explained due to the increase in viscosity of the solution [57] and the reduction in the hydration shell of sodium at higher concentrations. Subsequently, the accessibility for water molecules to enter the cellulose polymer is restricted and the cellulose does not undergo sufficient swelling for the cellulases to reach inside the polymer chain. When raising the temperature here to 53 °C, neither CO nor PET remained in an intact fabric structure. In contrast to the other concentrations used, performing alkaline pre-treatment with 7.8% NaOH, the lowest separation yield for CO was achieved at RT (see Figure 1). While a greater CO mass reduction was noted when raising the temperature to 95 °C (32% of the initial CO mass remained), which can trivially be explained with higher reactivity and chain mobility at higher temperatures, a similar reduction was detected using temperatures below RT. At 5 °C and −10 °C, only 39% and 29% of the initial CO mass remained in the respective recovered fabric. As described by Sobue et al. [38], this can be explained by the increase in the swelling properties of cellulose using these conditions. This effect is advantageous for cellulase accessibility and could potentially be a beneficial operating window for CO separation processes on an industrial scale since those processing conditions might be achieved with lower resource consumption (energy, operating chemicals).

Color impression of the treated textiles was affected by the separation process. Figure 2 shows the recovered fabrics when using 7.8% and 20% NaOH at 5 °C, RT and 53 °C. Since only the CO fibers were dyed (black hue), the extent of CO separation at different parameter value settings could be visually tracked in the washed textiles by the varying degrees of decoloration of the fabric after treatment. The textile became lighter in color impression the more CO was separated, which is clearly a consequence of the quantitative removal of dye (see Figures 1 and 2, below RT). When using 20% NaOH (see lower row in Figure 2), only minor cotton residues were observed already below RT, while above RT removal was almost complete, which is reflected in the lighter hues for the samples treated at 20% NaOH below RT compared to the samples treated at 7.8% NaOH below RT.

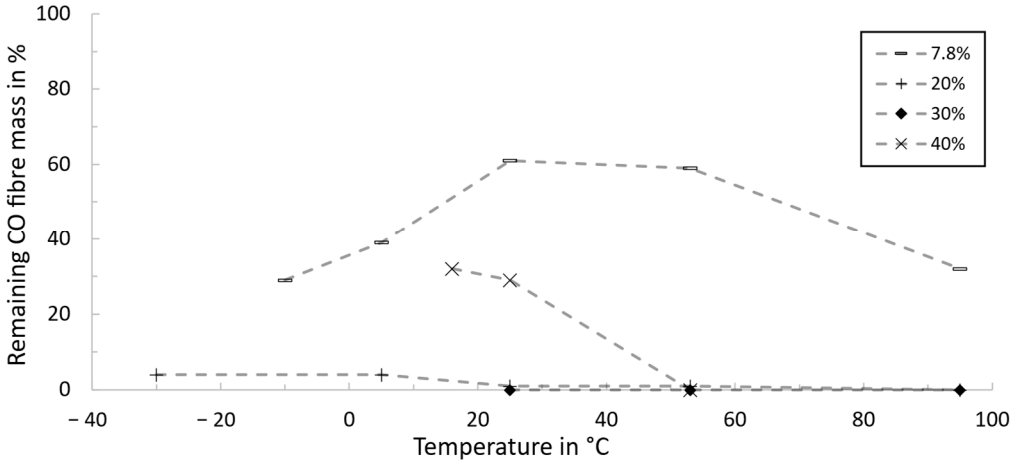


Figure 1. Remaining mass of CO fibers relative to initial CO fiber mass ($X_{m,\,CO}$) after a six-hour pre-treatment and enzymatic hydrolysis from CO–PET T-shirts using different temperatures and NaOH concentrations. For better visualization, four selected NaOH concentrations were used in this figure, the complete results can be found in Table S1 in the Supplementary Materials.

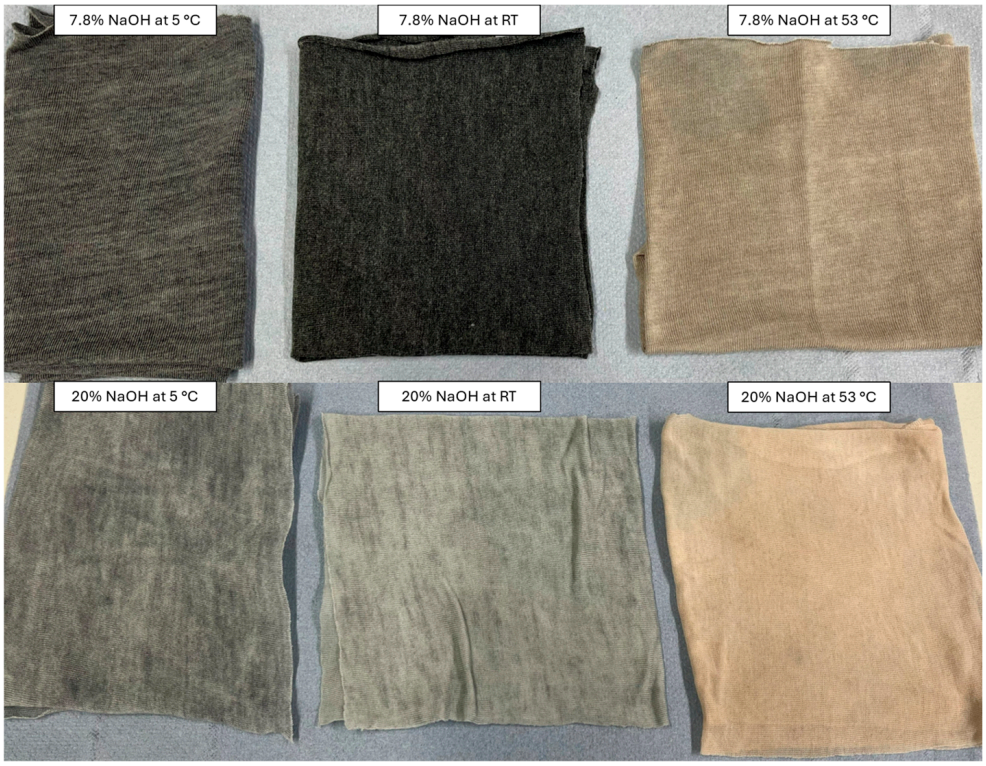


Figure 2. Varying degrees of decoloration of the fabric in accordance with CO mass reduction reflecting a selection of chosen treatment parameter values.

On the other hand, above RT, both the 7.8% and 20% NaOH treatments seem to have caused a brightening in color, even though the achieved CO contents after treatment are each similar (~60% for 7.8% NaOH at RT and 53 °C; almost complete removal for 20% NaOH at RT and 53 °C). This can only be explained by a direct decoloration on the cotton itself (increased color stripping to the solvent) or a chemically induced color-shift effect (chemical alteration of the dye), as otherwise a significantly darker shade would be expected for the sample treated at 7.8% NaOH/53 °C.

2.1.2. PET Loss

The mass of the recovered PET fabric after alkaline pre-treatment and enzymatic hydrolysis was calculated from Equation (4) and the loss of mass is given in Figure 3. Using RT and below, an almost complete recovery of the PET fabric was achieved. Gritsch et al. [49] describe mass losses of PET for their experiments using NaOH concentrations of 4% and below and stated similar results. The loss of PET, however, increases with higher NaOH concentrations. While there was no PET loss at RT and 7.8% NaOH, it increased to ~20% when using 30% and 35% NaOH. Using NaOH concentrations above 35%, the loss of PET slightly decreases again which can be attributed to increased viscosity of the solution (see Section 2.1.1). Our analyses suggest, however, that temperature poses a stronger influence than NaOH concentration. Raising the temperature, the amount of recovered PET drastically decreases which makes the process inefficient. In this context, acceleration of the alkaline hydrolysis of PET at higher temperatures has been described in the literature [58,59]. At 20% NaOH and RT, a PET mass loss of ~10% was observed, while at 53 °C this loss already reached 65%. Raising the temperature to 95 °C, no PET could be recovered across the entire NaOH concentration window. In contrast to this, Peterson et al. [30], report no significant removal of PET from a CO–PET fabric after a 6 h pre-treatment using only slightly milder conditions, namely 90 °C and 5% NaOH. Čorak et al. [60] describe that at 4% NaOH, 100 °C and a duration of one hour, hydrolysis started to take place but still 78% of the initial PET content remained. Assuming a comparable textile and PET yarn structure, this might be attributable to the lower NaOH concentrations the authors used. However, as can be seen from results given in Section 2.2, a significant PET mass reduction was already observed at those concentrations. This could be attributed to an already lowered quality of the PET in the post-business material. As those results do not provide a final understanding, we recommend further research on this topic.

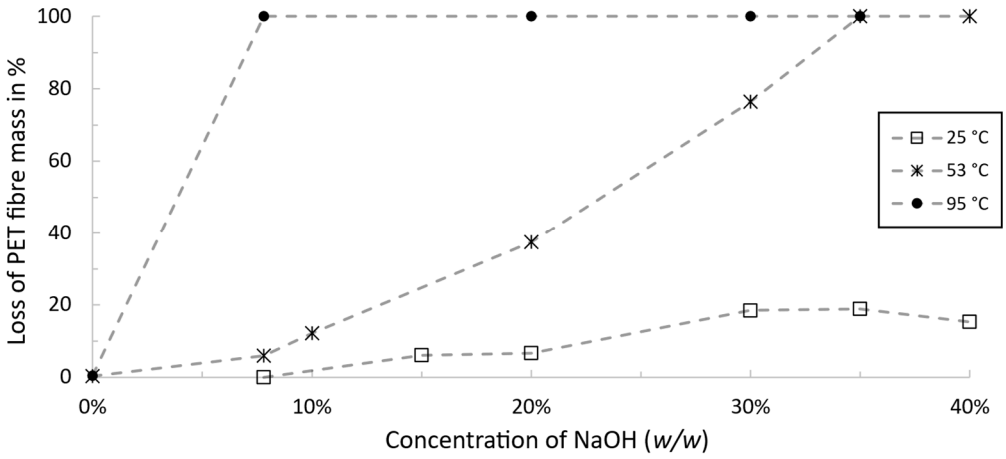


Figure 3. Loss of PET fibers relative to initial PET fiber mass ($X_{m,PET,loss}$) after a six-hour pre-treatment and enzymatic hydrolysis. Data points below RT were not included in this graph to better visualize the results as no significant reduction in mass was detected. The complete results can be found in Table S1 in the Supplementary Materials.

2.1.3. Comparing Cotton Removal and PET Loss

Figure 4 summarizes the remaining CO and PET for the T-shirts after exposure to the enzymatic treatment by overlaying the results from Figures 1 and 3. The numbers given for each data point reflect the remaining CO and PET masses relative to either of their initial masses in percent (left and right of the slash, respectively) within the recovered fabric. This enables a comparison of the separation yield of CO with the correlated mass loss of PET for each processing condition. From the results, it is apparent that a concentration

above 10% NaOH is required to achieve an almost complete removal of CO from this fabric. A more vigorous mechanical treatment during enzymatic hydrolysis or a second hydrolysis step might enable the removal of any remaining residual amounts of CO. Cavaco-Paulo [61] and Cortez et al. [62] describe a positive effect on the separation yield of CO using mechanical agitation during enzymatic hydrolysis. Figure 4 also shows that the pre-treatment temperature had almost no effect on the separation of CO when using concentrations between 20% and 35% NaOH. Using lower (7.8% NaOH) or higher (40% NaOH) concentrations, the influence of the temperature is clearly visible. As discussed earlier, for the recovery of PET, low temperatures are preferable, as a significant loss of PET from the fabric is already detectable when treated above RT. If temperature and NaOH concentration are too high, neither CO nor PET remain intact on fabric level.

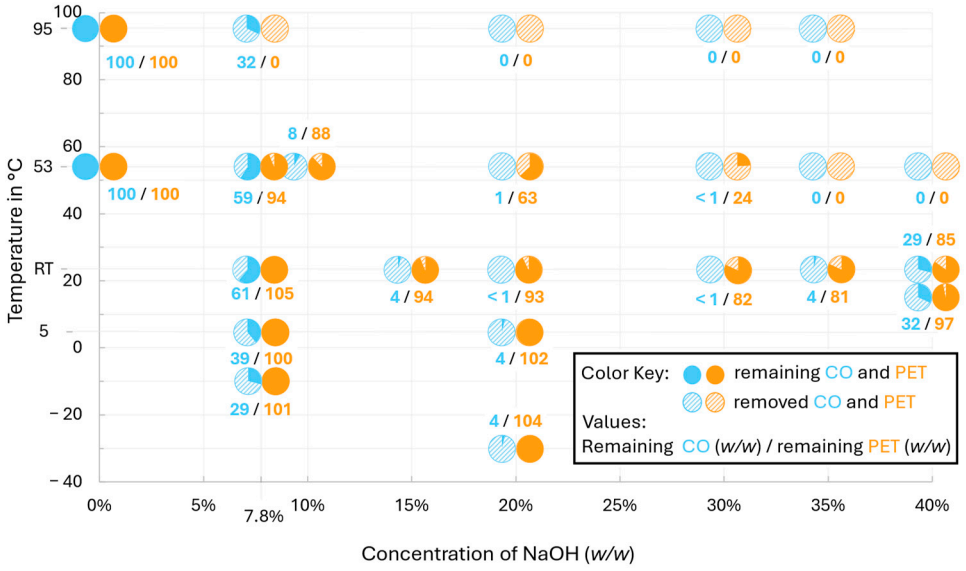


Figure 4. Summary of the remaining CO and PET (initial textile composition CO:PET = 60:40) after pre-treatment and enzymatic hydrolysis. The numbers to the left of the slash of each data point indicate the percentage by mass of the remaining CO in the recovered textile. This shows the separation efficiency of CO from pre-consumer textiles. Directly alongside this, the loss of PET for each data point is shown to the right of the slash, also as percentage by mass. Each data point therefore indicates how much CO and PET remain in the recovered textile relative to either of their respective initial masses. Data point colors visualize the presence of CO or PET at the respective parameters (blue = CO, orange = PET; solid colors indicate the remaining fraction, hatched areas correspond to the removed fraction). Values above 100% result from small weighing errors or local deviations from the initial composition. The complete results can be found in Table S1 in the Supplementary Materials.

2.1.4. Optical Microscopy

As discussed in Section 2.1.1, a distinct change in sample color (dark to beige) was evident when treatment temperatures were increased from RT to 53 °C. While we largely linked this to either a physical or chemical effect on the dye and dyed fiber surface, we also recognized a competing effect, namely a roughening of the surface of PET fibers and a resulting superimposed color effect (Figure 5, lower magnification). Because the disrupted surface produces a random scattering pattern for the incoming light, this renders the fibers less translucent and causes an opaque color impression. While pure PET should appear white in polychromatic light, chemical alterations of the polymer during treatment or small amounts of chromophore substances adhering to the roughened PET fiber surface could have created the strong beige impression in the affected samples.

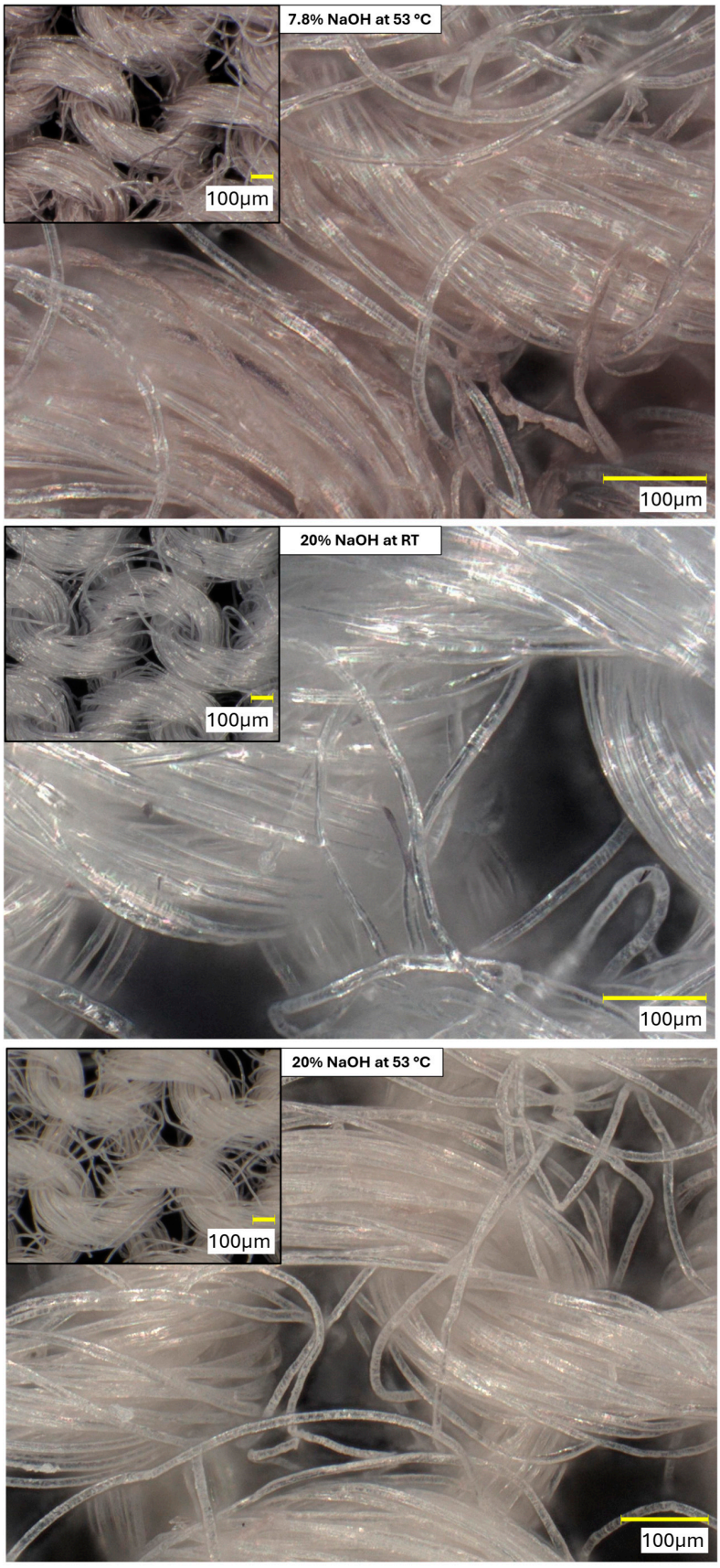


Figure 5. Light-microscope images of sample sections (2 magnifications each) showing the influence of treatment parameter values on color impression and surface morphology of PET fiber. Note that for 7.8% NaOH treatment a considerable amount of CO fibers is still present.

Figure 5 (higher magnification) clearly shows the effect on fiber surface. For a treatment in 20% NaOH at 53 °C fibers exhibit a characteristic scarification (indentions/protrusions at intervals of a few µm), while for a treatment at the same alkaline concentration but at RT the surface remains largely undamaged. A similar roughening is observed for a 7.8% NaOH treatment at 53 °C, albeit much less pronounced and harder to discern, since CO fibers still co-exist. We attribute the effect to an increased chemical attack on the fiber sheath at rising temperatures, which weakens the PET fibers and causes a loss in surface tension, leading to the observed structural change and possibly a flaking reaction. This is consistent with observations regarding the mass loss of PET (Figure 3), where the concentration-dependent mass loss increases more as the chosen treatment temperature rises.

Even at reduced mass loss, disrupting the PET fiber surface inevitably leads to reduced mechanical stability. Since recovered PET fibers do not have to be used directly to produce new yarns [13], this is a minor problem if no adverse effect on polymer integrity is detected. Yet, special attention should be paid to the discussed effects during processing at elevated temperatures, and lower treatment temperatures should be considered overall preferable for preventing PET loss. Furthermore, the recovered PET must fulfill certain quality requirements and cannot be damaged gravely by the separation process. Data from the literature show that alkaline pre-treatment under moderate conditions does not result in a significant reduction in polymer quality (see Supplementary Materials).

2.2. Part 2: Tests on Post-Business Textiles

In the second part of the study, experiments were extended to post-business bed sheets. According to the methods described in Sections 2.1 and 3.4, the composition stated on the label (50% CO, 50% PET) was checked. This resulted in a CO:PET ratio of 44:56 which deviates slightly from the labeled values and can be attributed to the wear and tear of the fabric particularly in frequent industrial washing (see Section 3.3.1). A parameter range was selected to systematically analyze the recovery and separation of both polymers. Tables S4–S6 in the Supplementary Materials contain the exact mass percentages as well as all the masses necessary for the calculations.

2.2.1. Cotton Removal

Figure 6A shows the results for the separation efficiency of CO from post-business textiles. It displays results similar in nature to those obtained for the experiments in Section 2.1, while accounting for material-specific treatment conditions. Higher separation rates for CO fibers were expected here due to the high wear and tear at use and cleaning compared to pre-consumer textiles. However, this hydrolysis-enhancing precondition is in competition with the denser structure of the woven bed sheets, which rather hinders cellulases (sterically) to enter the fabric. Vasconcelos et al. [63] concluded that knitted textiles seem to be more accessible to hydrolysis due to the greater exposed fiber surface. Reducing the pre-treatment time had no negative effect on the separation yield of CO which was assessed in preliminary tests carried out on a selected parameter set (results can be derived from Tables S7 and S8 in the Supplementary Materials). Evaluating the results using 10% NaOH at different temperatures, the same trends for CO mass reduction can be identified for this post-business material blend compared to the pre-consumer knitted textile (see Figure 1). This again is in line with the parameter range defined by Sobue et al. [38] for increased swelling properties of CO at low temperatures. At 5 °C, more than 80% of the initial CO mass was removed. This value decreases to 40% between RT and 65 °C and increases again to 70% at 85 °C. The results using 5% NaOH did not show the same trend, as this concentration lies just outside of the parameter range described by Sobue et al. [38]. When using 15% NaOH and above, an almost complete removal of

CO from the blend was achieved. These results stayed consistent over the entire analyzed temperature range.

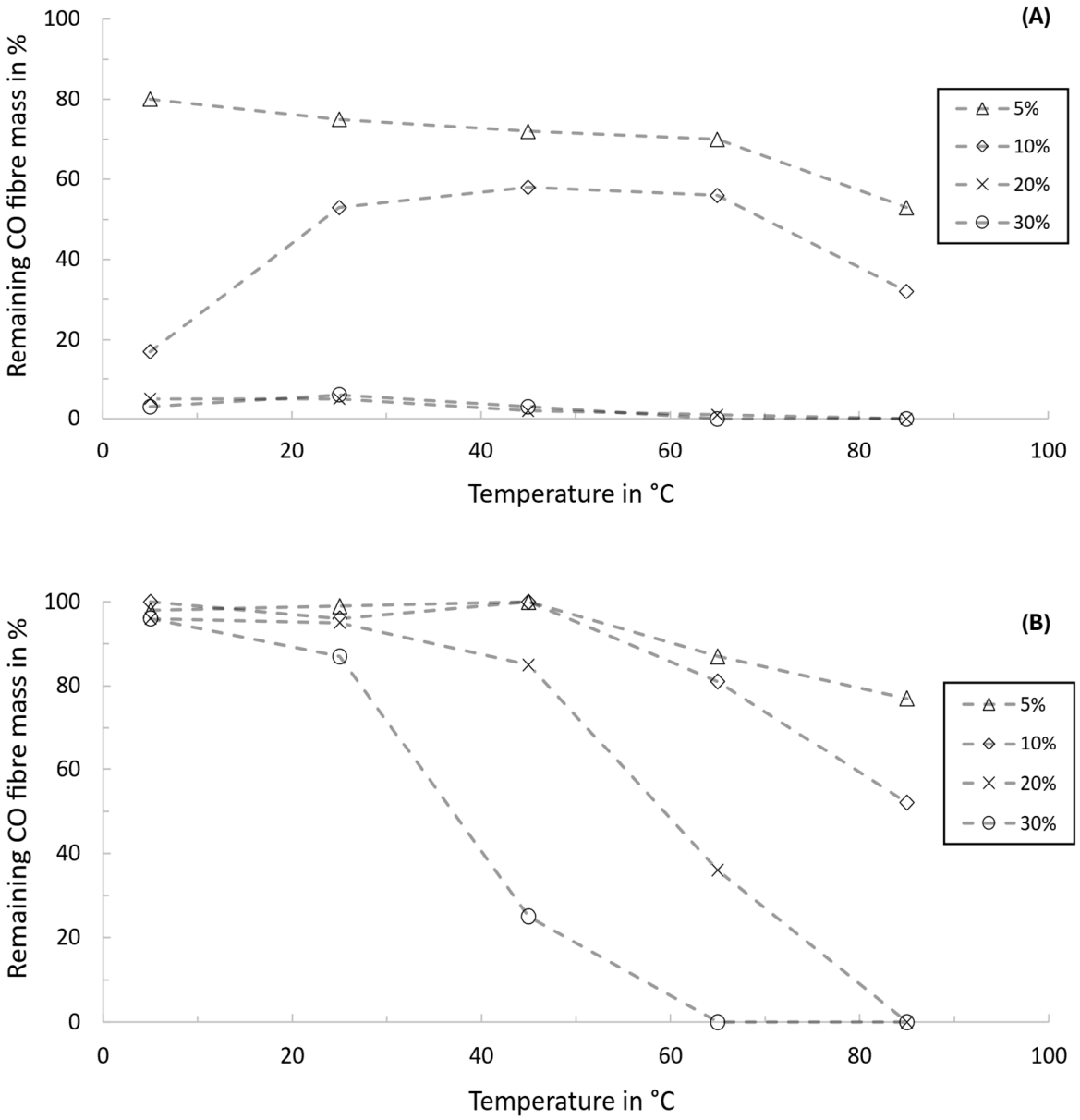


Figure 6. (**A**) Reduction in the initial mass of CO fibers after a two-hour pre-treatment and enzymatic hydrolysis. The figure shows four of the six analyzed NaOH concentrations for better visualization and comparability to the results from Section 2.1. (**B**) Reduction in the initial mass of PET fibers after a two-hour alkaline hydrolysis at different temperatures and NaOH concentration for a treatment time of 2 h. A complete list of all results may be obtained from Table S4 in the Supplementary Materials.

2.2.2. PET Removal

Complementary to the removal of CO by alkaline pre-treatment and subsequent enzymatic hydrolysis, the separation of PET from the blend was investigated with the intention to recover CO on fabric level, using alkaline hydrolysis of the PET as the core step. This in turn allows a parallel assessment of the loss of CO during alkaline hydrolysis. Results provided in Figure 6B (see also Table S3 in the Supplementary Materials) again show a pattern similar to the results for the pre-consumer textiles in Figure 3, within the same parameter range but using shorter treatment times. At RT and below, less than 20% of the initial PET mass was removed regardless of the NaOH concentration. Complete

removal at fabric level could be achieved at 65 °C using 25% NaOH and when raising the temperature to 85 °C, 20% NaOH was sufficient. Following that trend, and in line with the effects encountered in Section 2.1, using higher temperatures would reduce the NaOH concentration needed for complete separation even further. In addition, longer treatment times have a positive effect on PET separation as was observed during the preliminary tests (see Table S6 in the Supplementary Materials). This was also stated by Bengtsson et al. [64] for viscose–PET textiles, especially when using low NaOH concentrations. For a comparison of the difference between the untreated fabric and the recovered CO using 20% NaOH at 85 °C by light-microscope imaging, see Figure S1 in the Supplementary Materials.

Using elevated temperatures, on the other hand, not only increases the separation yield for PET but also decreases the mass of recovered CO (calculated using Equation (2), see also Table S3 in the Supplementary Materials). While there were almost no mass losses between 5 °C and 65 °C, up to 15% of the initial CO mass was lost when raising the temperature to 85 °C. These results were observed regardless of the NaOH concentrations used. Concurrently, alkaline treatment also triggers a decrease in the DP of cellulose, in particular when using higher NaOH concentrations, temperatures and treatment durations, as is described in the literature. A short overview on recent publications regarding the DP and fiber integrity of recovered CO can be found in the Supplementary Materials.

2.3. Limitations and Recommendations

The composition of all samples and recovered fabrics was assessed as described in Section 3.4. Therefore, a small equivalent of the respective textile was analyzed. However, it is not guaranteed that the composition is homogenous across the entire textile. Especially for post-business bed sheets, it is possible that some areas have sustained more abrasion than others which may lead to local variations in composition. This can change the resulting values slightly, but the overall trend regarding separation yield and mass loss remains the same. Furthermore, it cannot be guaranteed that the tests described in Section 3.4 did not influence the results as well. In preliminary trials on pure CO and PET textiles, we verified that the fabric to be recovered does not depolymerize. However, particularly during the washing step, fibers from the recovered fabric can detach, resulting in a slightly lower mass. To quantify possible mass losses from washing, an analysis of the washing water should be included in future tests.

In this study, the separation of CO and PET from blends was analyzed on a broad (pre-)treatment parameter range, focusing on separation efficiency and recoverable mass. Each parameter set was investigated as a single experiment run, enabling the evaluation of a large number of parameters and the identification of general trends across the explored range. While this approach limits the statistical robustness of the individual data points, it allows comprehensive screening of process behavior. Verifying suitability of these materials for recycling processes is the next crucial step for closing the loop. Therefore, after identifying optimized operating conditions, conducting analyses on morphology and material properties like DP and intrinsic viscosity as well as replicated experiments should be addressed in future work.

For our experiments, we used textiles with a simple construction (e.g., no multi-layer items), and without any disruptors (buttons, zippers, prints, third fiber type, etc.) or sewing yarn to guarantee high validity of the results from the screened parameter matrix. For extended analyses, an assessment of the influence of these disruptors on the overall recycling processes is advisable. But although they render recycling processes—particularly downstream processing—more complex or may impede the recovery of pure materials, the polymer separation principle holds. Apart from adverse effects on mixing behavior and a reduction in accessibility of the textile substrate imposed by certain disruptors appearing

in large amounts, we do not expect those items to impede the investigated hydrolysis steps themselves to an intolerable extent. The general challenge, however, could be circumvented by implementing a sophisticated design for recycling for textiles. Limiting the number of different fiber polymers in a textile and avoiding fibers present in small percentages only (e.g., PET sewing yarns in "100" CO textiles or an elastane content < 2%) can eliminate the need for additional stepwise fiber separation and make pure material streams more feasible. Furthermore, reducing the number of additives and dyes used in textile production as well as implementing easily removable disruptors can also greatly improve recyclability. Still, recycling not only requires a well-prepared material input stream, but it also calls for an economically and ecologically sensible process design. Catalysts can accelerate or enable reactions, yet selectivity of core processing steps and durability (input material quality vs. quality of the recycled polymers) seem to be the bigger challenges in the recycling of post-consumer textiles [65] when compared to batch time. In addition, the production of certain catalysts can have considerable environmental impacts, as described for urea production [34]. Catalysts must therefore be kept within the process, otherwise the loss of these often costly substances must be compensated for and elaborate separation alongside (by-)products is necessary to prevent leakage to the environment and causing negative health effects by contaminated garments produced from recycled fibers. The same applies for all other chemicals used to ensure a resource-efficient process. Peterson et al. [30] also discussed this in respect to purification and reusability of NaOH after PET hydrolysis and Gritsch et al. [49] demonstrated that it is possible to recover and reuse the cellulases used for CO hydrolysis.

3. Materials and Methods

To enable a comprehensive overview of the results, the work presented here is structured in two parts. The schematic visualization of the conducted test series can be seen in Figure 7. The processes were kept simple, without the need to add auxiliary substances to, or separate them from, the reaction solution other than the primary reagents. Furthermore, we avoided the implementation of process steps like defibration as keeping the structural integrity would not only allow easier handling of the material but could also lower the energy input for recycling processes compared to fiber production starting at monomer level [66,67]. Regarding PET separation, Bengtsson et al. [64] also described no improvement by mechanical pre-shredding. The investigation of the recovery of either CO or PET was therefore conducted within an intact fabric structure, while the other component was removed. It is essential to assess the feasibility of these processes regarding the parameters at which significant quantities of each respective polymer can be recovered. Furthermore, many studies focused their research on one input material or fabric type. Production, weaving, intended usage, washing (industrial or household) and wear and tear can greatly influence textile properties and recovery yield. Therefore, we used two different textile types representative of those criteria (see Figure 7), which will be described in detail in Sections 3.2.1 and 3.3.1.

In the first part, focus was put on pre-consumer textiles, where screening was performed over a large temperature and alkaline concentration range with selected parameter values to assess the recovery performance for PET after enzymatic hydrolysis. The second part of tests was then carried out on post-business textiles, and a selected parameter range was analyzed systematically for both the recovery of PET after enzymatic hydrolysis and CO after alkaline hydrolysis. All parameter values are listed in Table S8 in the Supplementary Materials.

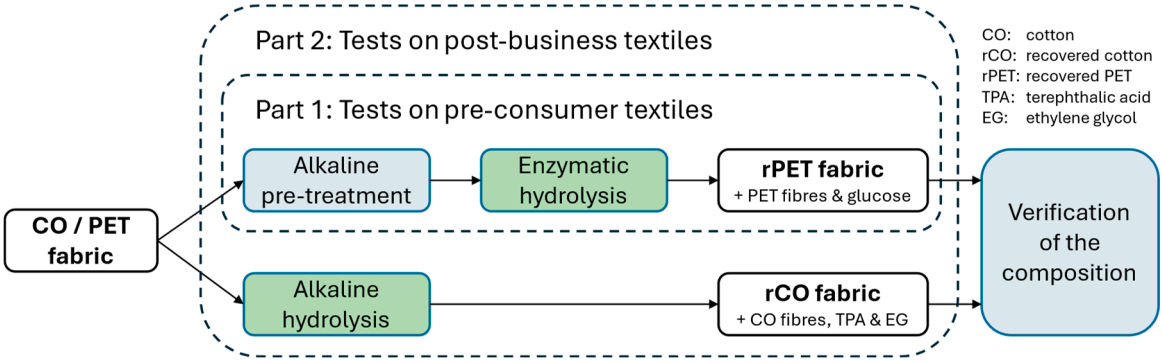


Figure 7. Schematic illustration of the conducted test series. Part 1 shows the test procedure for the pre-consumer textiles with the focus on CO separation and PET recovery after alkaline pre-treatment and enzymatic hydrolysis. Part 2 shows the procedure using post-business textiles analyzing both CO separation/PET recovery after alkaline pre-treatment and enzymatic hydrolysis and the PET separation/CO recovery after alkaline hydrolysis. The recovered PET fabric is referred to here as rPET, the recovered CO as rCO.

3.1. Chemicals

Citric acid monohydrate (purity ≥ 99%, CAS: 5949-29-1), sodium hydroxide (NaOH, ≥99%, CAS: 1310-73-2), m-cresol (purity ≥ 99%, CAS: 108-39-4) and sulfuric acid (H_2SO_4, reagent grade 95–97%, CAS: 7664-93-9) were obtained from Carl Roth GmbH + Co. KG, Karlsruhe, Germany. If not specified otherwise, deionized water taken from a Sartorius Arium® Pro UV ultrapure water system, Sartorius AG, Göttingen, Germany (0.055 µS/cm) was used for all purposes. Two aqueous cellulase preparations, NS 59116 (used in finishing/biopolishing) and NS 59143 (used in biorefineries), containing endoglucanases, exoglucanases and β-glucosidases in varying shares were used for enzymatic hydrolyses. To maintain functionality, those preparations were stored at 4–8 °C and were provided for research purposes by Novozymes Switzerland AG, Aesch, Switzerland. The above-mentioned chemicals were used without further purification.

3.2. Part 1: Tests on Pre-Consumer Textiles

3.2.1. Materials

Textile consumer goods are a heterogenic material stream with a frequently complex material composition and presence of accessories (e.g., buttons, zippers, prints, etc.) which makes them challenging to recycle. Since fibers in pre-consumer textiles experienced no wear and tear through usage and washing; they retain higher quality properties than post-consumer or post-business textiles. Especially repeated washing can damage the fabric not only through particle shedding or microfiber production but also due to the decrease in DP, impacting the tensile strength [68,69]. To avoid any influence by the above-mentioned disruptors to recycling, and to test the efficacy of our treatments on cellulosic material with high DP, we used knitted T-shirts (200 g/m^2) purchased at retail. Those items comprised dark colored CO and uncolored PET with a mass ratio of 60:40 according to the label (see Figure S2 in the Supplementary Materials).

3.2.2. Alkaline Pre-Treatment

For each experiment, 10 g of textile material (sample size of ~22 × 22 cm) were prepared and treated in 300 mL NaOH at different temperatures and alkaline concentrations. Alkaline pre-treatment was performed in glass beakers placed on temperature-controlled heating plates. To promote homogenous heat transfer and temperature stability, the reaction beakers were positioned inside larger beakers serving as water-baths. Textile samples were fully immersed in the alkaline solution throughout the treatment and agitation was

provided by periodic manual stirring to ensure uniform contact between the material and the solution. The parameters ranged from −30 °C to 95 °C and from 0 to 40% (*w*/*w*) NaOH (see Table S8 in the Supplementary Materials). Parameters were partly derived from the literature data with the intention of realizing the most straightforward technical approach. RT (~23 °C), 53 °C and 95 °C, were chosen because at RT no additional heating or cooling is required, 53 °C is the temperature suitable for enzymatic hydrolysis (see Section 3.2.3) and 95 °C is still below the boiling point of water, which means that no pressurized system is required. Parameters of 5 °C and −10 °C using 7.8% NaOH were selected because these parameter values lie within the range defined by Sobue et al. [38] at which cellulose undergoes strong swelling. Furthermore, 20% NaOH was selected, as this concentration is used to mercerize CO [70] and the parameter sets 20% NaOH at −30 °C and 40% NaOH at 16 °C were used, as they are close to the liquidus curve of the binary NaOH–water system [36]. Taking into consideration the properties of the material described in Section 3.2.1 and the treatment times used by other studies, ranging from 1 h [19] to 3–6 h [49] on defibrated material, the treatment time in the alkaline solution was fixed to six hours to ensure sufficient pre-treatment. Afterwards, the samples were washed until reaching neutral pH and immediately transferred to the enzyme–buffer solution without drying.

3.2.3. Enzymatic Hydrolysis

An enzyme–buffer solution containing a 3:1 mixture of NS 59116 and NS 59143 was prepared. This ratio was based on previous research regarding hydrolysis of viscose fibers present in protective textiles [71] and the effectiveness was verified in preliminary trials on CO–PET textiles [72]. The optimal working conditions for cellulases in this context are a pH value between 4.8 and 5 and ~53 °C in accordance with industry practices [73] and research [45,74]. To provide stable conditions, a 50 mmol/L citric acid buffer was prepared from citric acid monohydrate, NaOH and deionized water and adjusted to the desired pH value. For each experiment, 1 L of this buffer solution was transferred into a one-liter glass bottle alongside 10 g of enzyme mixture and the pre-treated material. The dispersion was then heated to and maintained at 53 °C in a closed, temperature-controlled water bath connected to an external thermostat with the textile samples fully submerged in the solution. Each fabric was treated for 24 h, and agitation was achieved by periodical manual mixing to ensure adequate contact between the fabric and the solution. Afterwards, the samples were washed, dried at RT, analyzed gravimetrically and the remaining CO content was checked (see Section 3.4). Li et al. [46] discovered that the glucose yield after enzymatic hydrolysis decreases if the substrate loading (textile mass per volume of solution) exceeds 3%. For this reason, we fixed our substrate loading to ≤1% to avoid inhibitory effects and to ensure good mixing behavior. According to established practice in lignocellulosic biorefinery [73], an enzyme dosage of 30 g per 1000 g substrate, corresponding to an enzyme loading of 0.5–0.6% (enzyme mass per volume of solution, at a liquor ratio of ~1:5) is typical to achieve ~80% conversion after five days. Since those values are not practical in textile recycling, the enzyme dosage could not be matched to the lower acceptable substrate loading for sufficient conversion. On this assumption and because in aqueous systems reaction progress is largely governed by reactant concentration, we therefore targeted at least the values typical for lignocellulosic biorefineries and fixed an enzyme loading of 1%. This also lies within the range reported in other studies [19,45]. For reference, the activity of the same enzymes was previously reported as a filter paper unit of 1850 U/g in a study conducted under comparable conditions within our research group and is described in Piribauer et al. [74]. Since near-quantitative cellulose conversion is crucial, we integrated a

parallel enzymatic treatment of a reference material used by Ipsmiller et al. [71] to verify maintained catalytic activity.

3.3. Part 2: Tests on Post-Business Textiles

3.3.1. Materials

In this study, we use the term post-business textiles to encompass flat linen and ward linen textiles provided by textile rental companies in large quantities for hotels/restaurants and the healthcare sector [75]. The composition of those materials is typically simple, either consisting of CO only or a CO–PET blend and furthermore, the presence of recycling disruptors is limited. Hence, they are a well-suited material stream for recycling. On the other hand, post-business textiles are usually exposed to industrial washing conditions, resulting in higher wear and tear especially of CO fibers [76]. While this may result in fibers with lower fiber integrity [76], which is disadvantageous for recovery, it may have a beneficial impact on separation due to a possibly higher accessibility to hydrolysis processes. As a representation of such post-business textiles, white bed sheets with a 50:50 mass ratio of CO and PET were provided (165 g/m^2; see Figure S2 in the Supplementary Materials).

3.3.2. Alkaline Hydrolysis/Pre-Treatment

For each experiment, 10 g of textile material (sample size of ~24 × 24 cm) were prepared in duplicate, where one sample was used for alkaline hydrolysis and one for alkaline pre-treatment. All samples were treated in NaOH at different temperatures and concentrations. The same experimental setup was used as described in Section 3.2.2. To systematically assess the separation/recovery yield, the parameter values were narrowed down to temperatures from 5 to 85 °C and concentrations from 5 to 30% (*w*/*w*) NaOH (see Table S8 in the Supplementary Materials). An incubation time of 2 h was fixed for both alkaline pre-treatment of CO and alkaline hydrolysis of PET to take the lower expected fiber integrity into account. Consequences implied by this choice are discussed in Section 2.2. Shorter treatment times also have a positive effect on industrial processes from an economic and ecological standpoint, as less energy would be required under similar conditions. After each alkaline hydrolysis test, the sample was washed with water until reaching neutral pH, dried at ambient conditions until reaching constant mass and the remaining PET content was checked (see Section 3.4). The post-business samples pre-treated for enzymatic hydrolysis were washed until reaching neutral pH and immediately transferred to the enzyme–buffer solution.

3.3.3. Enzymatic Hydrolysis

The enzymatic hydrolysis for assessing the removal of CO was carried out applying the same procedure as described in Section 3.2.3.

3.4. Identification of the Material Composition

After verification of initial component share (non-treated material), separation efficiency (composition after treatments) was assessed. A sample equivalent was cut from each textile and analyzed for polymer content according to the following procedures: (1) The reduction in mass of CO by enzymatic hydrolysis was checked according to DIN ISO 1833-11:2017 [54] by treating the textile in 75% (*w*/*w*) H_2SO_4 at 50 °C for 1 h; (2) the reduction in mass of PET by alkaline hydrolysis was verified via the AATCC Test Method 20-1977 [55] by treating the textile in pure m-cresol at 140 °C for 5 min. After washing, drying and weighing, the reduction in mass was determined and the effective shares of each polymer were calculated using Equations (1)–(4).

The remaining mass of PET, $X_{m,PET}$ (%), can be seen in Equation (1) with m being the mass of the textile after treatment, m_0 the initial mass of the textile, $w_{0,PET}$ the mass fraction

of PET in the original textile, m_{cresol} the mass of the textile equivalent after identification of material composition and $m_{0,cresol}$ the mass of the textile equivalent before identification. Equation (2) shows the unintended loss of CO, $X_{m,CO,loss}$ (%), during the treatment with $w_{0,CO}$ being the proportion of CO in the original textile.

$$X_{m,PET}\ (\%) = \frac{m \cdot \left(1 - \frac{m_{cresol}}{m_{0,cresol}}\right)}{m_0 \cdot w_{0,PET}} \cdot 100 \quad (1)$$

$$X_{m,CO,loss}\ (\%) = \left(1 - \frac{m \cdot \frac{m_{cresol}}{m_{0,cresol}}}{m_0 \cdot w_{0,CO}}\right) \cdot 100 \quad (2)$$

Equation (3) shows the calculation for the remaining mass of CO, $X_{m,CO}$ (%), with $m_{H_2SO_4}$ being the mass of the textile equivalent after identification of material composition and $m_{H_2SO_4}$ the mass of the textile equivalent before identification. Equation (4) shows the unintended loss of PET, $X_{m,PET,loss}$ (%), during the treatment.

$$X_{m,CO}\ (\%) = \frac{m \cdot \left(1 - \frac{m_{H_2SO_4}}{m_{0,H_2SO_4}}\right)}{m_0 \cdot w_{0,CO}} \cdot 100 \quad (3)$$

$$X_{m,PET,loss}\ (\%) = \left(1 - \frac{m \cdot \frac{m_{H_2SO_4}}{m_{0,H_2SO_4}}}{m_0 \cdot w_{0,PET}}\right) \cdot 100 \quad (4)$$

As a complementary method, light-microscopic images (VHX-6000 digital light microscope, Keyence, Osaka, Japan) were recorded in reflected-light mode to visually verify separation efficiency.

4. Conclusions

To the best of our knowledge, this is the first work that investigates the feasibility of recovering CO (via the alkaline hydrolysis of PET) or PET (via an alkaline pre-treatment and enzymatic hydrolysis of CO) from textile blends across a wide range of processing parameter sets and that compares material separation efficiency and material loss of the desired polymer at fabric level for each data point.

The recovery trends for CO and PET from pre-consumer (T-shirts) and post-business textiles (bed sheets) were found to be largely consistent. A NaOH pre-treatment concentration of 15% was required to achieve near-complete CO separation via enzymatic hydrolysis under the tested conditions. At temperatures below 45 °C, 90% of PET was recoverable in fabric form. Remaining CO residues on the recovered PET fabric could potentially be removed via more vigorous mechanical agitation or an additional hydrolysis step, both of which warrant future investigation. PET removal and recovery of CO via alkaline hydrolysis necessitate elevated temperatures and NaOH concentrations. At 65 °C, NaOH concentration of 25% was required to completely remove PET from the fabric. Higher temperatures reduced the required NaOH concentration: at 95 °C, 7.8% NaOH was sufficient for complete PET removal. However, using higher temperatures also increases the loss of recovered CO, resulting in mass losses of up to 18% of the initial CO mass. Careful parameter selection is critical not only for maximizing polymer recovery but also for preserving fiber integrity. A reduction in the DP of CO, as described in the literature, must be considered—but it may be advantageous or even desirable for the production of man-made cellulosic fibers from the recovered CO fraction. In case DP values are too low for these applications, alternative valorization pathways must be considered. One possible route could be the use of recovered CO as raw material for paper production, where DP

requirements are generally less stringent than for fiber production. In addition, production of microcrystalline cellulose represents another option, as this can be used as an additive in paper manufacturing. Beyond material-based uses, recovered cotton with low DP may also be suitable for (bio-)chemical conversion routes, including transformation into platform chemicals or fermentable sugars for bioethanol production.

The study also demonstrates that CO and PET can be recovered with minimal mass loss from both pre-consumer and post-business textiles under moderate treatment conditions—without the need for pressurized systems, catalysts or defibration. This further indicates that the processes used here offer comparatively environmentally friendly approaches for textile recycling, particularly when compared to more complex processes. The actual economic and environmental impact of these processes on an industrial scale, however, needs to be evaluated using life cycle analysis. Although this depends on site-specific conditions, evaluating the simplest possible processes and establishing a more in-depth understanding for parameter choice can serve as a useful basis for recyclers when implementing these processes. Further research is still needed to close the loop for textiles. Depending on the intended application and blend composition, alternative separation processes should also be explored. Moreover, the impact of recycling disrupters on overall process performance must be assessed for post-consumer textiles, which represent the most complex material stream.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/recycling11060100/s1, Table S1: Results of Part 1: Tests on pre-consumer textiles. The table shows the percentages of the remaining mass of cotton (CO) and the calculated remaining mass of PET after alkaline pre-treatment and enzymatic hydrolysis using pre-consumer T-shirts. Values above 100% result from small weighing errors or local deviations from the initial material composition; Table S2: Data produced from Part 1: Tests on pre-consumer textiles. This data is used in Equations (1)–(4) for calculating the percentage of separated cotton during the process. The composition was checked using H_2SO_4 (described in Section 3.4) except for the data marked with an * which was tested using m-cresol; Table S3: Results of Part 2: Tests on post-business textiles. The table shows the percentages of the remaining mass of CO after alkaline pre-treatment and enzymatic hydrolysis, the remaining mass of PET after alkaline hydrolysis and the calculated remaining mass of CO after alkaline hydrolysis using post-business bed sheets. Values above 100% result from small weighing errors or local deviations from the initial composition; Table S4: Data produced from Part 2: Tests on post-business textiles. This data is used in Equations (3) and (4) for calculating the percentage of separated cotton during the process. The composition was checked using H_2SO_4 (described in Section 3.4); Table S5: Data produced from Part 2: Tests on post-business textiles. This data is used in Equations (1) and (2) for calculating the percentage of separated PET and cotton loss during the process. The composition was checked using m-cresol (described in Section 3.4); Table S6: Results of the preliminary tests for Part 2: Tests on post-business textiles regarding the variation in treatment time. All experiments here were carried out as described in Sections 3.2.2 and 3.2.3 using 20% NaOH at RT. The table shows the percentages of the remaining mass of CO and the calculated remaining mass of PET after alkaline pre-treatment and enzymatic hydrolysis using post-business bed sheets; Table S7: Data produced from the preliminary tests for Part 2: Tests on post-business textiles regarding the variation in treatment time. All experiments here were carried out using 20% NaOH and 25 °C. This data was used in Equations (3) and (4) for calculating the percentage of separated cotton; Table S8: Overview of the temperature and sodium hydroxide (NaOH) concentrations used for the experiments of Part 1: Tests on pre-consumer textiles and Part 2: Tests on post-business textiles; Figure S1: Light-microscope images of untreated textile (left) and textile after alkaline hydrolysis (right). This not only visualizes a general reduction in mass by larger voids between the individual yarn strands, but by the morphology of the surviving fibers, which is typical of cotton, also provides evidence that no PET fibers remained; Figure S2: The pictures on the left side show the untreated T-shirt used for "Part 1: Tests in pre-consumer textiles"

at different magnifications and the pictures on the right side show the untreated bed linen used for "Part 2: Tests in post-business textiles" at different magnifications. References [77–79] are cited in the Supplementary Materials.

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Data Availability Statement: The original contributions presented in this study are included in the article and Supplementary Material. Further inquiries can be directed to the corresponding author.

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Abbreviations

The following abbreviations are used in this manuscript:

CO	Cotton
DP	Degree of polymerization
EG	Ethylene glycol
H_2SO_4	Sulfuric acid
NaOH	Sodium hydroxide
PET	Polyethylene terephthalate
rCO	Recovered cotton
rPET	Recovered PET
RT	Room temperature
TPA	Terephthalic acid

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