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The efficacy of alkaline hydrolysis on selective degradation and recovery of high-purity terephthalic acid and cotton cellulose from postconsumer polyester/cotton waste

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A R T I C L E I N F O

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A B S T R A C T

Textile waste is one of the biggest global waste problems requiring innovative and sustainable solutions. This study focused on developing a potential sustainable solution to textile waste by recycling of blended polyester/cotton fabric waste. The blends were pretreated prior to selective alkaline degradation and then vacuum filtered into constituent materials. Terephthalic acid (TPA), ethylene glycol (EG), and cotton were the constituent materials. The effects of the processing conditions and determination of optimum conditions were ascertained by analysis of the constituent materials' properties. Electron microscopy (SEM), Ultraviolet spectroscopy (UV–VIS), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), thermogravimetric analyses (TGA-DSC) and viscometer were utilized in characterizing the recovered materials. The optimum processing conditions were found to be 15 % (w/v) NaOH in the presence of 1 mol BTBAC: 1 mol per repeating unit of polyester phase transfer catalyst and processing temperature and time of 80 °C and 270 mins, respectively. These conditions resulted in complete hydrolysis of the polyester in polyester/cotton blended fabrics waste achieving a TPA recovery rate of 80 % and recovery rate of 97 % for cotton fibres. The recovered cellulose had viscosities between 340 and 520 ml/g which is within the range of pulp utilized in the production of regenerated fibres. The successful separation of TPA and cotton fibres implies that the recycling of blended textiles can be accomplished contributing to sustainable textile waste management and circular economy. The research indicates significant potential for scalable textile waste solution to confront the increasing crisis of textile waste globally.

1. Introduction

There is an ever-growing demand and use of textile materials resulting in high volumes of textile waste being disposed of to the environment. The quantity of discarded textiles rises annually raising concerns on the management of textile waste. Therefore, the need for innovative sustainable management of end-of-life textiles and textile waste cannot be overemphasized. Recycling end-of-life textiles and textile waste is one of the techniques that could be effectively applied to deal with textile materials disposed of to the environment. However, one of the biggest obstacles in the realm of textile recycling is the prevalence of multi-material textiles, which refers to textile materials consisting of two or more distinct types of fibrous polymers. Some of the available recycling methods such as mechanical recycling of multi-material textiles lead to the production of recycled materials with inferior properties. This makes recycled materials unfavourable for some applications or to be used in the production of materials for low-end or inferior applications only. This implies that such recycled materials would quickly find their way back into the environment or landfills or get incinerated. Therefore, this study focused on a more lasting and sustainable solution for recovering high-purity terephthalic acid and cotton cellulose from post-consumer polyester/cotton fabric waste while preserving their intrinsic properties for enhanced applications.

The utilisation of a blend of polyester (PET) and cotton fibres is

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prevalent in the textile industry. This is primarily due to the advantageous properties of both materials. Polyester, known for its durability and cost-effectiveness, is combined with cellulose fibres from cotton, which possess excellent water absorbency. As a result, this combination enhances the fabric's comfort, making it highly desirable. Together, these two fibres make up over 75 % of global fibre production and hence comprise the majority of textile waste [1]. Separation of the two components is essential for the recycling of these materials to enable further valorisation [2]. Several approaches for cellulose/PET separation have been studied, including degradation, depolymerization, or dissolution of at least one of the components [2–8].

Studies in which the polyester was maintained required the degradation of cotton by acids or by microbes or dissolution in selective cellulose solvents [9–11]. Depolymerization of cellulose into glucose facilitates the creation of specialty chemicals or biofuels [12,13]. Valorisation of cotton-based textile waste has been demonstrated through a process of pretreatment followed by enzymatic hydrolysis [14–16]. However, cotton cannot be repolymerised as it is a natural polymer and this limits the use of the residue if it is severely degraded. Dissolution of cotton components has also been proposed using different solvents, including N-methyl morpholine N-oxide(NMMO) or ionic solvents [17,18]. This approach maintains the polyester intact, however, the molecular weight of PET is diminished in the postconsumer fabric when contrasted with a new fabric due to laundering and regular wear. Most laundering is performed using alkali detergents which attack the ester bonds of polyester, leading to significant degradation after many laundering cycles [2]. Additionally, most textile polyester is produced from recycled PET from waste bottles and hence the processing of remelting and recycling in the first stage leads to a significant decrease in the molecular weight [19]. The reduced molecular weight of PET could potentially lead to recycled PET fibers exhibiting substandard mechanical characteristics, or even render the melt-spinning procedure unfeasible.

Conversely, depolymerizing PET results in the formation of PET monomers, which can be utilized in the manufacturing of virgin PET or other value-added products following appropriate purification processes. There are three main chemical degradation methods for polyester, that is, hydrolysis [2,20,21], alcoholysis [1,22] and glycolysis [22,23] and these have been extensively researched in the chemical recycling of plastic bottle PET [19,24]. However, hydrolysis is preferred in this study because it could degrade polyester back into the original monomeric terephthalic acid and ethylene glycol, used in producing virgin polyester [3]. In addition, cellulose can be recovered in its polymeric form, suitable for recycling into regenerated fibres or microcrystalline cellulose. Alkali hydrolysis is known to reduce the degree of polymerisation of cotton fibres [12] to the degree of polymerisation range of dissolving pulp, making it suitable as the raw material for conventional fibre-to-fibre recycling.

Alkaline hydrolysis has been previously shown to be a successful method for both depolymerizing PET and recovering cotton cellulose simultaneously [3,25–27]. The nucleophilic attack of hydroxide ions facilitates the cleavage of ester bonds present in PET, yielding disodium terephthalate salt and ethylene glycol, both of which are soluble in the aqueous phase. Upon completion of the reaction, the aqueous phase is acidified to a pH of approximately 2–3, leading to the formation and precipitation of TPA. The downside of this method is that severe conditions such as long reaction time, high temperature and pressure are utilized. This is because the rate of hydrolysis is comparatively slow due to the presence of hydrophilic hydroxide ions in the aqueous phase whilst polyester is a hydrophobic solid state. To improve hydrolysis, phase transfer catalysts which facilitate the hydroxyl ions onto the polyester surface to facilitate the reaction are used [28–30]. Quaternary ammonium or phosphonium salts with lipophilic side chains are widely recognized as the prevailing forms of phase transfer catalysts [27,31]. The utilization of sodium hydroxide (NaOH) solutions along with a phase transfer catalyst has proven to be highly efficient in depolymerizing PET at moderate temperatures (70–95 °C) and alkaline levels between 5 % and 15 % NaOH [2,3]. Recently, Palme et al. (2017) reported that the addition of benzyltributylammonium chloride (BTBAC), PET hydrolysis was completed at 90 °C with 10 % NaOH within 40 min [3]. However, there is a possibility of using milder temperatures when dealing with waste polyester, considering their work was reported on new polyester-based fabrics.

This current study focuses on the efficient recovery of terephthalic acid and cotton cellulose from alkali hydrolysis of polyester/cotton waste denim. Previous research focused on depolymerizing new fabrics or fabrics which were never dyed to any colour. The effect of a pretreatment to remove dyes and other impurities is thus not reported though it represents a more possible approach considering that most polyester/cotton textile waste is dyed. This work aimed to selectively depolymerise polyester in cotton/polyester postconsumer waste denim which was pretreated according to the method described by Baloyi et al., [32] to remove dyes and other impurities which might affect the purity of the final products. Alkaline hydrolysis of a mixture of cotton/PET blend was performed, and the cellulose and TPA were evaluated with respect to their purity, yield, molecular weight, fibre morphology and crystallinity. Process parameters that maximize PET degradation, that is, NaOH concentration, time and temperature were used as these would make the process feasible for separating the two components.

2. Materials and methods

2.1. Materials

Unused dyed 100 % cotton, 100 % polyester and different blends of polyester/cotton waste acquired from a local landfill in Durban, South Africa, NaOH pellets, cupri-ethylenediamine (CED) solution, BTBAC, 98 % sulphuric acid terephthalic acid and ethylene glycol purchased from United Scientific (Durban, South Africa) and deionized water.

2.2. Methods

2.2.1. Material pretreatment

The fabric waste was first cut into 1 cm^2 pieces and then defibrillated for 10 mins in a 220 Voltage blender with six stainless steel blades, powered by 2200 W and at a speed of 26,000 rpm. This process was responsible for defibrillation in the case of 100 % cotton or polyester fabric waste and both defibrillation and homogenous mixing in the case of the blended waste material. The materials were then decolourised using a method reported by Baloyi et al. [32]. The 100 % samples were used as control specimens whilst the waste blends were used in the alkali hydrolysis process to test the effectiveness of the separation technique on decolourised blended textile waste. The cotton/polyester ratio of the sourced blends and the mixed waste blend was determined according to ASTM D63 – Standard Test Methods for Quantitative Analysis of Textiles. 75 % sulphuric acid was mixed with the material at room temperature for 1 h after which the undissolved material was filtered off using vacuum filtration. Fibre composition was then expressed on the oven-dry mass of the original sample or the oven-dry mass of the clean fibre after the removal of non-cotton material. The cotton/polyester ratio of the blends was Blend 1 (76.30/23.70), Blend 2 (82.58/17.42) and Blend 3 (60.67/39.53).

2.2.2. Alkali hydrolysis

To determine the optimum conditions for achieving complete degradation of PET in blended textiles, a series of experiments utilising 100 % waste polyester fabric were conducted. These helped in understanding the parameters necessary for complete degradation. In test tubes, approximately 0.1 g of 100 % decolourised polyester fabric was mixed with different concentrations of NaOH at a temperature ranging between 50 and 90 °C and the hydrolysis was carried out for 60–300 min s with or without the addition of a phase transfer catalyst as shown in

Table 1.
Alkali hydrolysis experiments.

Parameter	Temperature (°C)	Time (minutes)	Concentration (%)	Catalyst ratio	
				No catalyst	Catalyst *
15 % NaOH, 240 min	50–90	constant	constant	0	1:1
80 °C,240 min	constant	constant	5–25	0	1:1
15 % NaOH, 1:1 catalyst	70–90	60–300	constant	@ 90°C	constant

* Catalyst ratio is based on 1 mol of BTBAC: 1 mol of repeating unit of PET.

Table 1. A material-to-liquor ratio of 1:50 was employed in this study and the solution was stirred with a glass rod every 15 mins. Prior to the alkali hydrolysis experiments, the polyester was dried in an oven at 105 °C for 1 h to remove any moisture in the sample.

After the required hydrolysis time, the test tubes were quenched in cold water to stop the reaction. The contents were then filtered through a 45 µm nylon filter paper under vacuum. The unreacted polyester fibres were washed thoroughly and dried in the oven for 12 h. The weight of the remaining polyester fibres was then taken to calculate the PET conversion. The filtrate containing sodium terephthalic was precipitated with concentrated sulphuric acid to a pH of 2 and then it was filtered off under vacuum on 45 µm filter paper to collect the TPA and the ethylene glycol in the filtrate. TPA was then dried in a vacuum oven at 105 °C for 12 h before characterization. Eqs. (1) and (2) were used to calculate the PET conversion and the TPA yield, respectively. The calculation of the TPA theoretical yield was based on the observed mass loss at specified time intervals, on the premise that PET is produced via the esterification

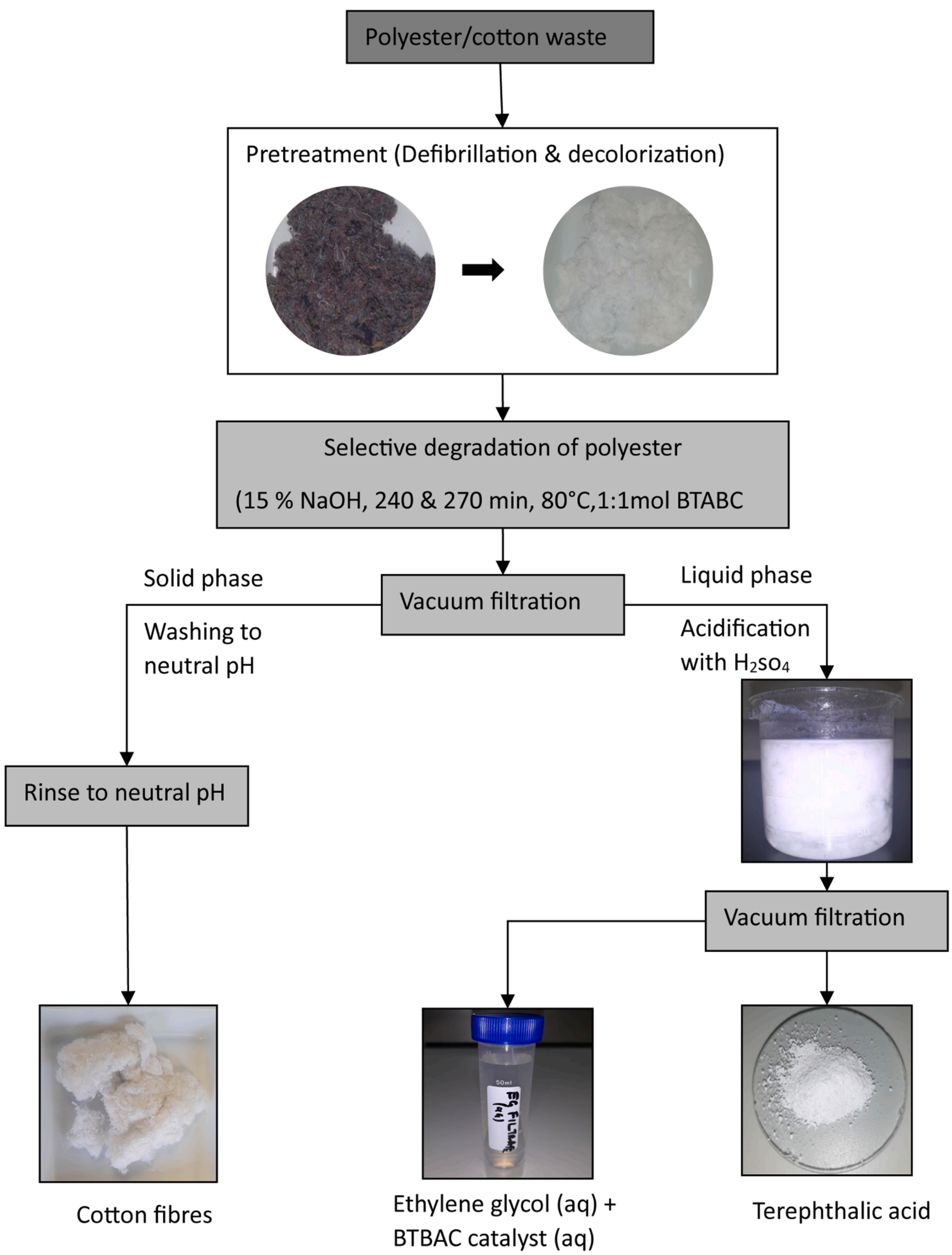

Fig. 1. Overview of the hydrolysis process of polyester/cotton blended fabrics.

of equal molar amounts of TPA and EG. The optimum conditions were then deduced from these results and used in the alkali hydrolysis experiments with the blended waste. To assess the effect of the optimized conditions on the cotton cellulose, 100 % waste cotton fabric which had been stripped of its dye was treated with the same conditions and the weight of the remaining cotton residue was weighed using a Kern ADB 100–4 analytical balance and calculated using Eq. (3). Recovery of cellulose and viscosity were used to check any effect of degradation on the cotton cellulose.

$$PET\ conversion\ (\%) = \frac{Initial\ weight - final\ weight}{Initial\ weight} x\ 100 \tag{1}$$

$$TPA\ yield\ (\%) = \frac{Practical\ yield\ (g)}{Theoretical\ yield\ (g)} x\ 100 \tag{2}$$

$$Recovered\ cotton\ fibre = \frac{Cotton\ residue\ weight}{Initial\ cotton} x\ 100 \tag{3}$$

2.2.3. Alkali hydrolysis of blended textile waste using optimized conditions

5 g of decolourised mixed denim waste was mixed with 250 g of 15 % NaOH in a volumetric flask mixed with 1 mol ratio of BTBAC catalyst to 1 mol for every repeating unit of polyester. The solution was left in the oil bath set to 80 °C and the hydrolysis was left to run for 4 h with stirring at 150 rpm. The flask was placed in cold water to quench the reaction. These conditions were chosen as the optimum conditions as they gave maximum degradation of polyester and minimum degradation of cotton fibres as it is necessary for complete separation of polyester in mixed blends to occur. However, after 240 min, some polyester fibres were still visible in these experiments hence the time was increased to 270 mins to make sure no polyester fibres remained in the residue. The remaining cellulose was then filtered off using a vacuum filter and washed thoroughly before drying. The filtrate was precipitated following the same stages explained in Section 2.2. Fig. 1 shows the overview of the hydrolysis process.

2.3. Characterisation

2.3.1. UV–VIS

The concentration of ethylene glycol in the filtrate was estimated using a Thermo Scientific GENESYS 50 UV–Vis Spectrophotometer. Absorbance at 200 nm was collected with the solution of the catalyst and water as the background. Firstly, standard solutions of EG in water of concentration 2 to 17 mol/L were prepared and their absorbance obtained. A graph of concentration against absorbance was plotted and the molar absorptivity (ε) of EG calculated using linear regression. The calculated absorptivity was then used to calculate the concentration of ethylene glycol in the filtrate using the Beer-Lambert law as shown in Eq. (4).

$$C = \frac{A}{\varepsilon b} \tag{4}$$

Where C is the concentration, A is absorbance, $\mathcal{E} = 0.0045$ L/mol^{-1} cm^{-1} which is the molar absorptivity of ethylene glycol, which was calculated using standard EG concentration and b is the length of light path.

2.3.2. Scanning electron microscopy

Surface morphologies of the fibres during the hydrolysis and the remaining cellulose after hydrolysis were done using a scanning electron microscope Phenom Pharos (Thermofisher Scientific, USA) at a voltage of 15 kV. The specimens were mounted onto a metal stub with the use of carbon tape and subsequently gold coated using a Q150RES sputter coater (Quorum, UK) to make the specimens conductive.

2.3.3. FTIR

The functional groups of the recovered cellulose and TPA were characterised using a PerkinElmer (USA) FT-IR spectrophotometer with a universal attenuated total reflectance (ATR) accessory utilized to record the spectra of recovered cellulose and the TPA. The recording spanned from 4000 to 600 cm^{-1}, with a total of 4 scans resolution set at 4 cm^{-1}. FTIR was also used to monitor the change in functional groups on the recovered 100 % cotton during alkali hydrolysis.

2.3.4. Thermal analyses

A simultaneous Thermal Analyser (Perkin Elmer STA 600, USA) was used to perform both the thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) tests. TGA focused on analysing the thermal decomposition behavior of the recovered cellulose and matched it with that of pure unused cotton. DSC analyses focused on the melting point and thermal behaviour of the recovered TPA. The test samples were heated in a nitrogen environment with a flow rate of 20 mL/min. The temperature was raised from 30 °C to 600 °C at a heating rate of 10 °C/min. TG and DTG data were collected using STA Pyris Series software (version 11), and OriginPro 2024 software was used for graphing.

2.3.6. Intrinsic viscosity

The cellulose samples' intrinsic viscosity was determined using a glass capillary viscometer. The recovered cellulose and also the 100 % cotton was dissolved in CED (copper (II) ethylenediamine) and measured the time it took for each sample to pass through a narrow capillary. The sample solvent mixture was first mechanically shaken in a closed plastic bottle with copper beads. The intrinsic viscosity was then calculated using Eq. (4). This measurement method was according to ISO 5351:2010 standard titled "Pulps—Determination of limiting viscosity number in cupri-ethylenediamine (CED) solution."

$$V = Ctd \tag{5}$$

Where V is the viscosity of the cellulose solution in CED (ml/g), C is the viscosity constant obtained by calibration, t is the average efflux time in seconds and d is the density of the pulp solution g/ml^3

3. Results and discussion

3.1. Effects of hydrolysis conditions on pet conversion

Fig. 2 shows the effects of the parameters on the conversion of PET under different conditions. Fig. 2a shows that the alkaline hydrolysis at 15 % (w/v) NaOH concentration without the addition of a catalyst resulted in a PET conversion of 97 % at a temperature of 90 °C after 240 mins.

When hydrolysis was performed at the same conditions but with the addition BTBAC catalyst, only a temperature of 80 °C was required to completely degrade PET. The BTBAC catalyst, therefore, lowered the conditions required to degrade the PET completely. Similar effects of the catalyst were observed when different concentration of NaOH was used. Fig. 2b shows the effects of concentration and phase transfer catalyst on PET conversion. The rate of degradation of PET increases with increasing NaOH concentration. At 15 % concentration, the addition of catalyst resulted in total degradation. However, a concentration of 25 % was required to degrade the PET completely within the time of 240 mins. Hydrolysis without BTBAC was much slower compared to when there was a catalyst.

In Fig. 2c, the time dependence of hydrolysis at 70, 80 and 90 °C and 15 % NaOH with BTBAC is compared to hydrolysis at 90 °C and 15 % NaOH without any addition of catalyst. Results show that the conversion rate increased with increasing temperature. A higher temperature without BTBAC could result in complete PET degradation, however, the rate will still be slower compared to when a lower temperature (80 °C) is coupled with a catalyst. A temperature of 70 °C was not adequate to degrade the PET completely even with the addition of a BTBAC catalyst.

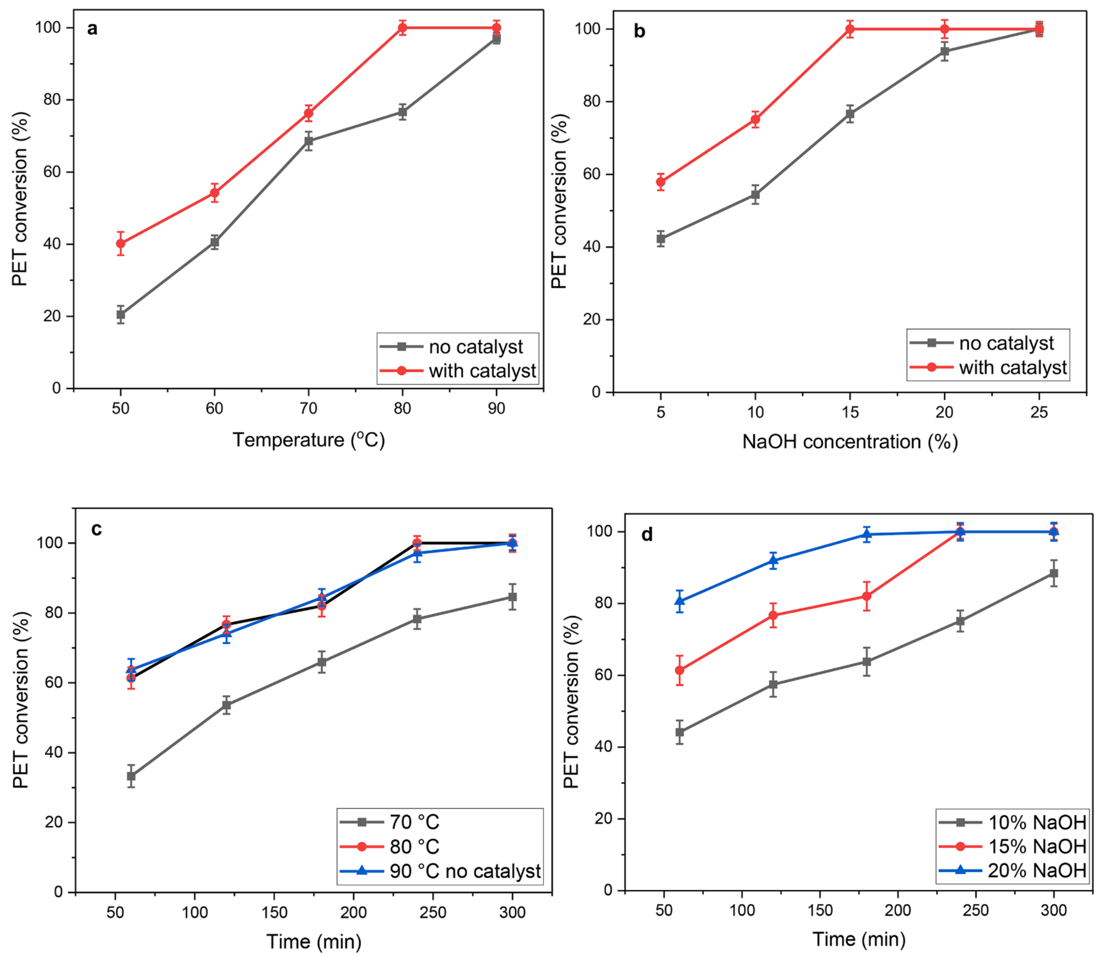

Fig. 2. PET conversion from 100 % white polyester fabric at different hydrolysis conditions.

In Fig. 2d, hydrolysis at 80 °C with 10, 15 and 20 % NaOH were compared. With increasing NaOH concentration, the conversion rate increases. The result with 20 % NaOH showed complete conversion could be achieved at 180 mins. The elevated concentration led to diminished yields of the monomer TPA, prompting the selection of a lower concentration of 15 % as the optimal level. A temperature of 80 °C and a hydrolysis duration of 240 mins were sufficient to achieve complete degradation of PET. In experiments involving blended waste, an additional 30 mins of hydrolysis time was incorporated to guarantee complete conversion, given the complexity introduced by the mixture. Table 2 shows the average TPA yield and recovered cellulose from the optimum conditions of 80 °C, 15 % NaOH and 240 mins.

In determining the theoretical output of cotton and TPA, the fibre ratio was considered an essential element in the calculations. A cotton fibre recovery of 97.48 % was obtained as a cotton cellulose residue. The reduction in cotton weight following the treatment can be attributed to the oxidation reactions occurring within the cellulose chain when exposed to strong alkalis in oxidative environments. The hydrolysis was carried out in an environment with oxygen leading to an oxidative reaction. According to the literature, oxidative reaction results in surface peeling of the cotton fibre outer layer [33].

Table 2.
Effect of optimum conditions on the pure waste samples at optimum conditions.

Sample ID	PET conversion (%)	TPA yield (%)	Recovered cotton fibre (%)	EG concentration (mol/L)
100 % Cotton	–	–	97	–
100 % PET	99	79	–	12

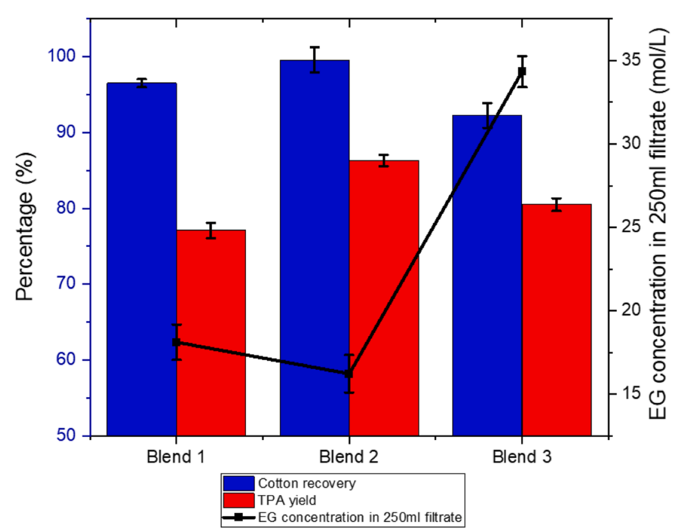

Fig. 3. Yield of PET monomers and recovered cotton cellulose from the blend waste samples at 80 °C, 1:1 mol BTBAC/mol PET, 15 % NaOH for 270 min.

3.2. TPA yield and cotton recovered from blended post-consumer waste

Fig. 3 shows the percentage yield and cotton fibre recovered from the blended textile waste after hydrolysis at optimum conditions and the remaining ethylene glycol concentration in the filtrate.

In Fig. 3, it can be seen that the maximum yield of TPA was recorded as 77.11, 86.32 and 80.51 % for Blends 1, 2 and 3, respectively. The resulting theoretical yield is subsequently compared to the experimental yield of TPA, which is measured using gravimetric techniques. Blend 2 had the least amount of PET (17.42 %) compared to the other blends; however, it had the highest yield of TPA which was surprising. This phenomenon can be explained by the ratio of PET to the available NaOH, which contributed to a more efficient hydrolysis and degradation of the PET material. A further explanation may lie in the differing molecular weights of the PET, which can significantly impact its reactivity. The multiple laundering cycles that the waste has undergone could result in varying molecular weights among the three samples. It is anticipated that PET with a lower molecular weight will hydrolyse more readily than those with higher molecular weights, consequently facilitating a greater yield of TPA monomer. The yields obtained in this work are in agreement with the 88.51 % yields of TPA obtained by Li et al., [34] from the effective method combining hydrolysis, reactive processing and decolouration of PET fabrics. A significant weight loss was observed in the recovered cellulose leading to a lower cellulose yield. This is due to the longer treatment times. Extended exposure to alkali can lead to significant breakdown of cellulose fibres resulting in a decrease in the degree of polymerization, reducing the strength and integrity of the fibres. As expected, blend 3 had the lowest yield of 92.26 %. Pre-treatment method of blending and mixing the sample, exposed more surface area, enhancing the effectiveness of alkali hydrolysis and subsequent degradation of the chain ends.

The concentration of residual ethylene glycol was estimated using UV analysis. A higher concentration was observed in Blend 3 which had a higher portion of PET followed by Blend 1 and then Blend 2. The concentration of the EG was proportional to the amount of PET which was in the waste fabrics. However, further analysis on EG was not included in this paper as they were not part of this study.

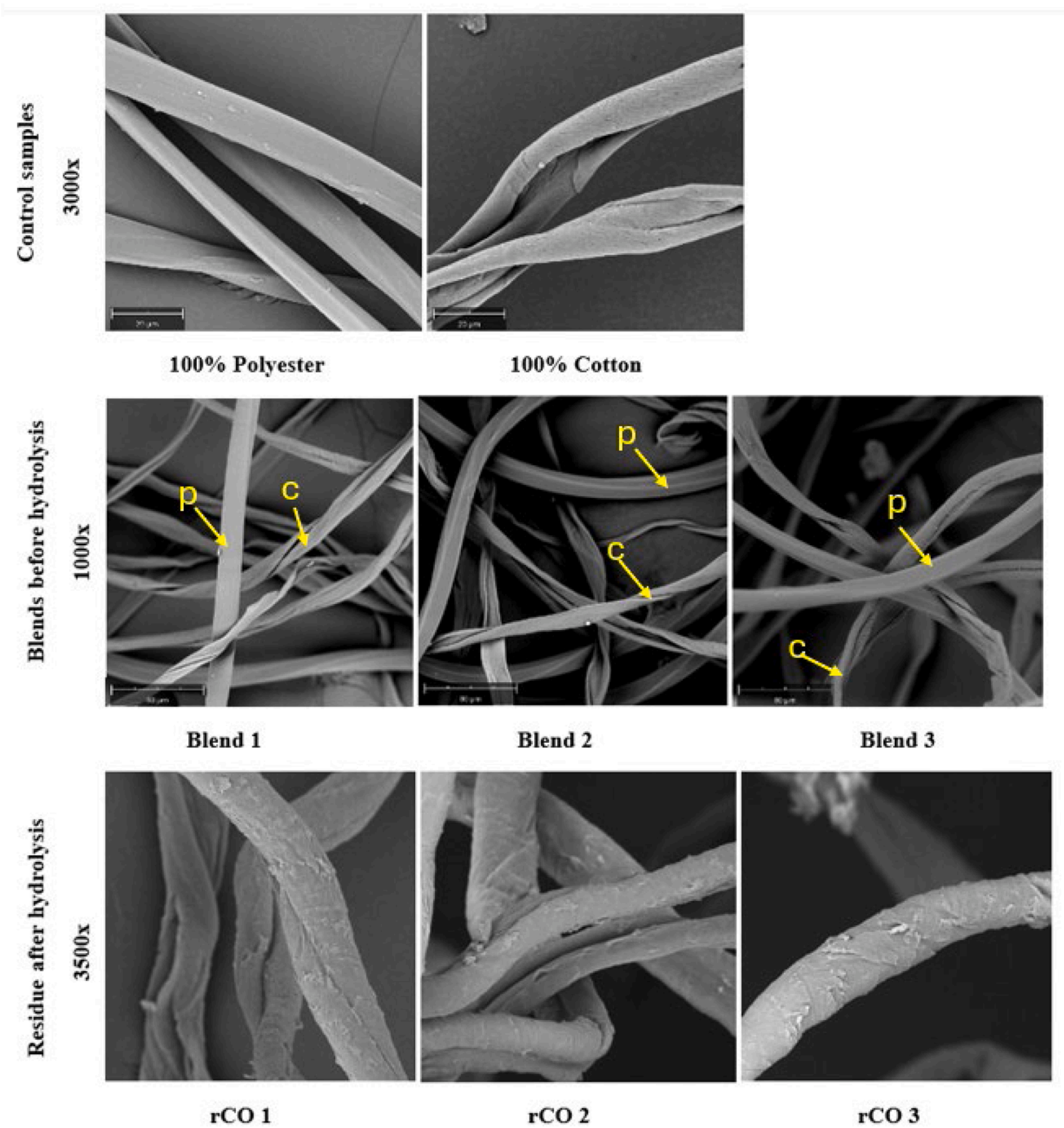

Fig. 4. Micrographs of the control samples and the blend before and after alkali hydrolysis.

3.3. Microscopy

Fig. 4 shows the SEM images of the polyester fibre control sample, cotton fibre control sample, the decolourised cotton/polyester blended fabrics, and the recovered cotton fibre hereby labelled rCO 1, rCO 2 and rCO 3 which is recovered cotton from blend 1, blend 2 and blend 3, respectively. Labels c and p in Fig. 4 refer to fibres resembling cotton fibre and polyester fibre, respectively. All the blended materials showed the presence of polyester fibre and cotton fibres which resemble the control polyester and cotton fibres, The convoluted fibres in the blends with typical morphological features such as the ribbon shape, rolled in a helicoidal manner around the axis are cotton fibres. This is in agreement with other published literature which states that the cross-sectional structure of cotton fibre has convolutions [35,36]. The synthetic fibre with the scalloped oval shape is identified as the polyester fibre [37,38]. The micrographs of the recovered residue indicate the changes in the morphology of cotton fibres after alkali hydrolysis. There is increased surface roughness resulting from many grooves on the fibre surface. This occurrence can be explained by the hydrolysis effect induced by sodium hydroxide (NaOH), which is known to partially degrade the hydrogen-bonded network on cellulose surface when exposed to oxygen for prolonged durations [39]. However, the fibres still presented a spiral structure, which seemed to show that the cotton fibre recovered its rigidity and natural convolution.

Fig. 5a shows that after 1 h of selective degradative treatment, the polyester fibres started showing signs of degradation as shown by the traces of degradation on the polyester fibre surface. After 2 h (Fig. 5b), degradation was observed to have increased and more visible microscopically. After 3 h (Fig. 5c), excessive degradation was observed. In Fig. 5d, after 4 h and 30 mins, the absence of polyester fibres in the material suggests that the degradation of PET was complete, leaving only cotton as the remaining component. This also indicates that the selective degradative treatment did not destroy the cotton fibre. Comparable degradation trends indicated by mass loss were noted by Bengtsson et al., [29], revealing that after a reaction time of 1440 mins, the polyester present in textile waste exhibited a complete mass loss of 100 %.

3.4. FTIR

The FTIR spectra for the recovered TPA (hereby labelled as rTPA 1 denoting recovered TPA from blend 1, rTPA 2 from blend 2 and rTPA 3 from blend 3) and cellulose are shown in Fig. 6. The analysis of the change in functional groups during the alkali hydrolysis is discussed as well as a comparison of the recovered products to pure commercial samples.

In Fig. 6a, the primary alterations observed from PET/CO blend 3 compared to the recovered cotton rCO showed a reduction in intensities of peaks corresponding to the wavenumbers of PET, specifically at 1714 cm^{-1} for C = O, 1239 cm^{-1} for C–O, 1043 cm^{-1} for –O–(CH_2)–O–, and 723 cm^{-1} for C–O. This is in agreement with Guclu et al. (2003) [40]. With increasing hydrolysis time, the disappearance of the 1714 cm^{-1}C=*O* peak was observed. However, after running the hydrolysis at the optimised time of 240 mins, small peaks at 1714 and 1239 cm^{-1} were still visible showing that there was still traces of polyester in the recovered cellulose. The discrepancy could stem from the use of pure PET waste in the optimization experiments. Nevertheless, in the case of a blend, it is feasible that some traces of polyester continue to be adsorbed on cotton surfaces. As a result, an additional 30 mins was added to the experimental time. When compared to the 100 % cotton control, the rCO after 270 mins showed similar characteristic bands.

The FTIR spectra of recovered cellulose are shown in Fig. 6b. Related characteristic bands associated with cotton celluloses are observed at 1019 cm^{-1} assigned to C—O stretching vibrations from the ether linkages in the cellulose structure and out-of-plane bending C—H vibrations from the methylene groups in cellulose backbone at 897 cm^{-1}. This was in agreement with literature [41,42]. The absence of distinct peaks associated with polyester in Fig. 6b at the wavelengths of 1714, 1239, 1040, and 723 cm^{-1} suggests that the polyester had undergone complete hydrolysis. This is supported by the observations of Liu et al. (2020) [43]. Surprisingly, no distinct features associated with the treatment of cotton by NaOH were visible in the spectra as expected. The C—O stretch at around 1050–1150 cm^{-1} that show shifts or intensity changes because of the disruption of crystalline cellulose during treatment with NaOH, leading to more amorphous regions, or formation of new bands at around 1500 cm^{-1} which can be associated with the formation of new structures such as sodium cellulose complexes or altered functional groups. This could be because these changes were already made in the pure cotton waste that was used in the control as most fabrics go through the process of mercerisation during their manufacture. Fig. 6c shows the resemblance of functional groups of the recovered TPA from blends and the commercial TPA. Even the fingerprint area is also the same indicating that they were identical materials. The stretching vibrational absorption in the region of 2500–3000 cm^{-1} represents the stretching vibration of the –OH group in the carboxylic acid groups. Due to the conjugation between the carboxyl groups and the benzene rings, the C = O stretching vibration of the carboxylic acid groups is detected at 1683 cm^{-1}. The peak observed at 1505 cm^{-1} is attributed to the skeletal vibrations of the benzene ring. Meanwhile, the peak at 1279 cm^{-1} is indicative of the characteristic frequency associated with the C–O bond.

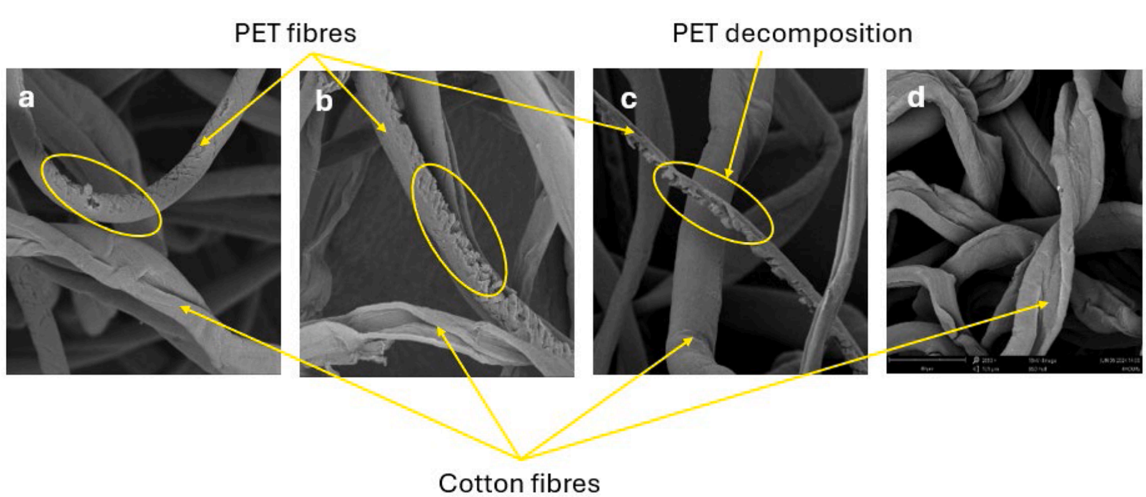

Fig. 5. PET degradation progression period after a) 1 h, b) 2 h, c) 3 h and d) 4 ½ h.

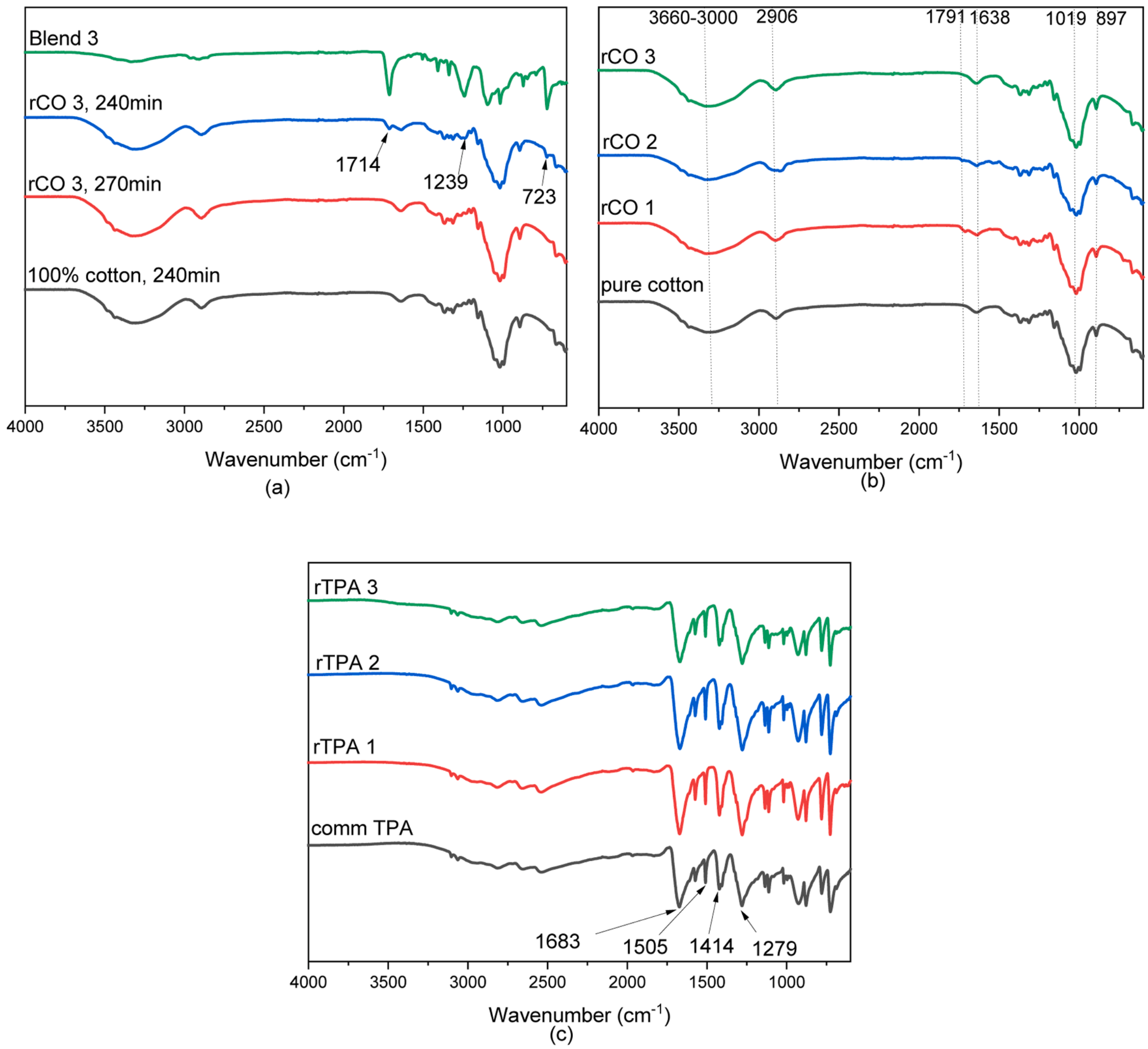

Fig. 6. FTIR of (a) comparison of the textile waste blend 3 and its obtained cellulose residue after 240 and 270 mins, (b) recovered cotton cellulose as compared to pure cotton, and (c) recovered TPA from textile blends as compared to commercially sourced TPA.

Furthermore, the absorption peaks corresponding to the flexural vibrations of the O–H groups are located at 1426 and 929 cm^{-1}. The observed peaks for the recovered TPA are consistent with the commercial product and also results reported literature [7,44].

3.5. DSC

The DSC thermal profiles for the blended textile waste are shown in Fig. 7.

Recovered TPA has a melting point of around 353 °C for all 3 samples, which is 5.3 % lower than that of the commercial TPA. However, the DSC profile reveals a similar endotherm pattern to that of the commercial variant and reported literature, confirming the purity of the TPA [7,40]. The minor reduction in melting point can be ascribed to the existence of byproducts from hydrolysis which could have interfered with the integrity of the crystal lattice manifesting as impurities leading to diminished melting point. This is in agreement with Cosimbescu et al. (2021) who also reported the interference of the hydrolysis byproducts on the integrity of the crystal lattice resulting in reduced melting point [45]. Furthermore, given that TPA is being extracted from textile waste, the occurrence of chain scission within the polymer chains is unavoidable. Laundering and the pretreatment of samples play a significant role

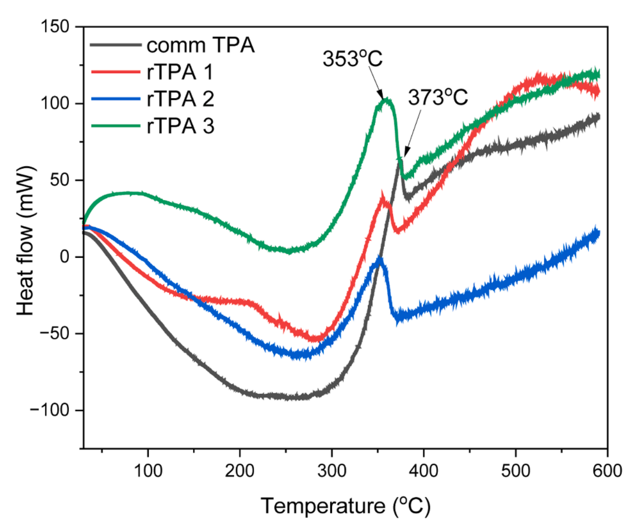

Fig. 7.. DSC curves of recovered TPA from blended waste Blends 1, 2 and 3 in comparison with the commercial TPA.

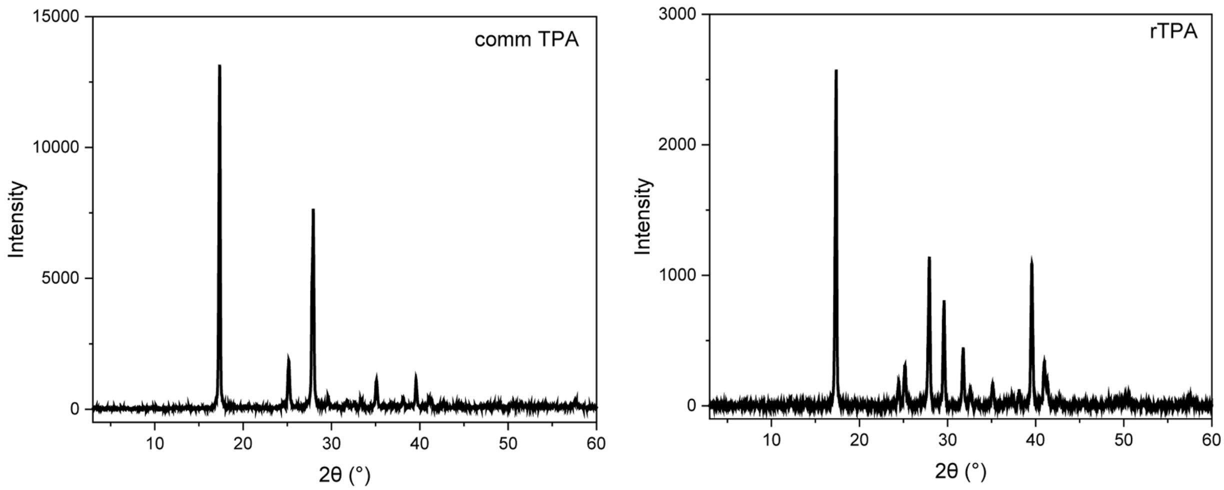

Fig. 8. . XRD patterns of recovered TPA and commercial TPA.

in the breakdown of polymer chains, producing shorter molecular chains or oligomers [46,47]. This phenomenon results in a decreased crystallinity level which subsequently leads to a lower melting point. The X-ray diffraction (XRD) results in Fig. 8 show a reduction in the crystallinity level of the recovered TPA which further substantiates the postulations that laundering and pretreatment reduce the crystallinity level of the textile materials.

3.6. XRD

Fig. 8 shows the XRD pattern of the recovered TPA and the commercial TPA.

Characteristic diffraction peaks were identified at 2θ values of 17.34°, 27.84°, 29.49°, 35.03° and 39.68° conforming to typical diffraction patterns of TPA crystal structure [48]. These peaks and patterns align well with XRD observations reported by Ling et al. (2019) on related work [7]. Additionally, the sharpness and narrowness of these diffraction peaks were comparable to those observed in commercial TPA, suggesting that the recycled TPA exhibits a high crystallinity index. The recycled TPA exhibited an average crystallinity of 70.2 %. There was no significant difference in the crystallinities compared to that of the pure commercial TPA of 71.1 %. The slightly lower average crystallinity can be attributed to the effects of laundering chemicals, weathering (UV causing photodegradation, acid rain) & use of heat in ironing clothes which cause polymer chain scission [33]. Additional reasons could be due to the TPA precipitation rate from the disodium terephthalate phase. Fast precipitation caused by vigorous stirring, high temperatures in the media or the concentration of the acid used can lead to rapid precipitation [49] which leads to the formation of smaller crystals and finally lower crystallinity of TPA [50]. Additionally, the presence of impurities or additives such as surfactants which could have been left from the decolourisation process might inhibit the formation of well-ordered crystals, leading to lower crystallinity of TPA [50].

3.7. TGA

The thermogravimetric properties of the recovered cotton cellulose are illustrated in Fig. 9 and a summary of their thermal degradation temperatures are listed in Table 3. Figs. 9a and 9 show that the recovered cellulose is characterised by a single-stage thermal decomposition as evidenced by a single peak for all the recovered cellulose and pure cellulose. Generally, the peak decomposition temperature of the recovered cotton was higher than that of pure cotton and this is attributed to the mercerisation effect which increases the molecular alignment within the cellulose chains. The decomposition below 100 °C is for the loss of moisture and volatile organic compounds.

Table 3 shows the thermal degradation temperatures and the mass loss of the recovered cotton due to thermal degradation.

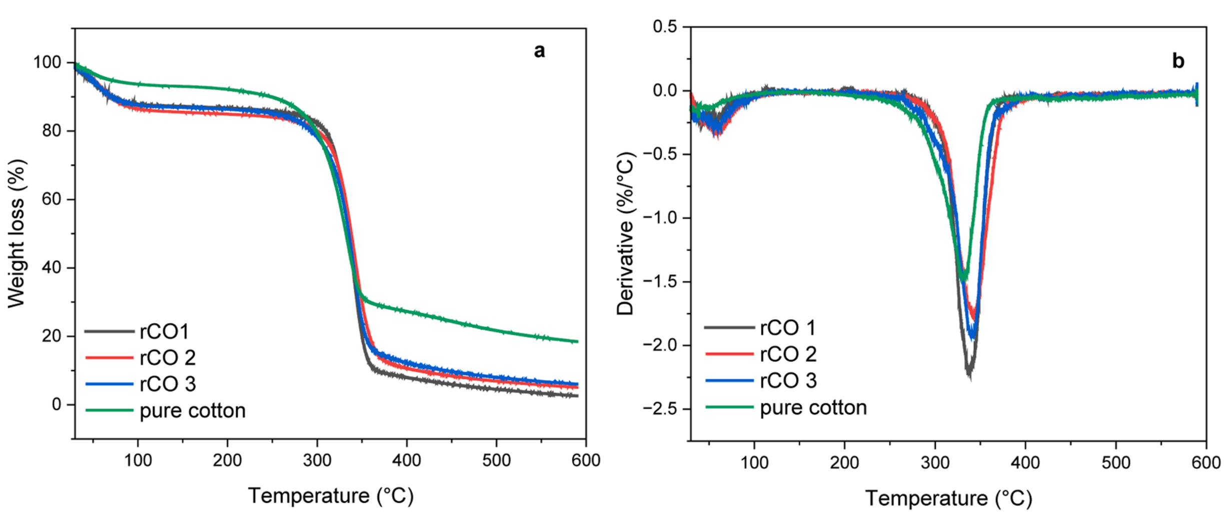

Fig. 9. TGA (a) and DTG (b) curves of cotton recovered from the blended waste in comparison with the 100 % cotton curve.

Table 3
. Degradation and mass loss of recovered cotton fibres.

Sample ID	Onset degradation temperature (°C)	Peak degradation temperature (°C)	End degradation temperature (°C)	Mass loss at End degradation temperature (%)	Mass loss at 600 °C (%)
100 % Cotton	232	329	362	75	82
rCO 1	264	338	380	86	98
rCO 2	267	342	392	81	95
rCO 3	266	341	370	82	94

Table 3 shows the recovered cotton fibres had a higher onset thermal decomposition temperature indicating improved thermal stability. The average improvement in thermal stability was 14.51 %. Likewise, the peak decomposition temperature and the end decomposition temperature of the recovered cotton fibres increased also signalling that the recovered fibres had better thermal stability than the commercial cotton fibre. The average improvement of the peak thermal decomposition and end thermal decomposition are 3.45 and 5.15 % respectively. Mass losses observed between 320 and 370 °C are attributed to the breakdown of cellulose into smaller oligosaccharides and monosaccharides through depolymerization and finally decomposing into char [7,51,52]. As observed by the decomposition peaks in Fig. 9b, the recovered cotton cellulose is slightly more thermally stable compared to the pure cotton. This is a result of the mercerisation of fibres during alkali hydrolysis which enhances their integrity and molecular alignment, reducing the likelihood of structural breakdown when exposed to heat. A comparable finding was reported by Yue et al. (2013) who indicated that the treatment of cotton fibres with sodium hydroxide concentrations exceeding 10 % led to enhanced thermal stability due to the conversion of cellulose I to cellulose II [53]. Decomposition continues at temperatures greater than 370 °C, resulting in the creation of char. The recovered cotton reveals a more purified form of cellulose as evidenced by the minimal char residue observed after heating to 600 °C, in contrast to that of waste cotton.

3.8. *Viscosity*

In Fig. 10, the intrinsic viscosity and the recovery of cellulose are shown as a function of time. The results in the graphs show that there was a major effect on the cellulose degradation in terms of both the recovery yield and the intrinsic viscosity. Under the optimised conditions of hydrolysis, it was observed that the intrinsic viscosity significantly drops in the first 60 mins of hydrolysis and gradually continues to decrease within the second hour of hydrolysis. However, after 120 mins, there was no significant change in the viscosity of the recovered cellulose. The decrease in intrinsic viscosity is primarily linked to the breakdown of the cellulose polymer into shorter fragments. Alkali hydrolysis does not depolymerise cellulose, however, the lower viscosity is a result of endwise peeling at the reducing end of a cellulosic chain changing the degree of polymerisation of the cellulose chains [12,54]. Shorter polymer chains result in lower intrinsic viscosity and consequently lower cellulose recovery. Complete PET degradation was achieved without much severe degradation of the cellulose yield. After treatment for 300 mins, cellulose recovery was 93.5 %, however, a yield of > 95 % could be achieved at a reaction time of 270 mins which was then used for the blended waste.

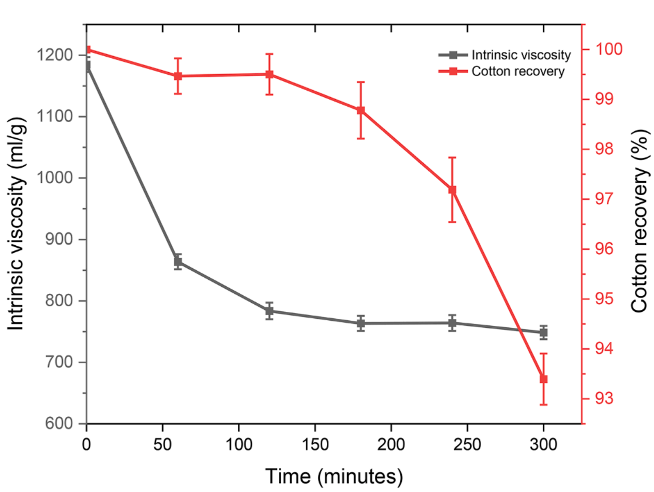

Fig. 10. Viscosity of 100 % cotton as a function of time.

The mean intrinsic viscosity for the recovered cellulose was 521.8 ml/g, 402.5 ml/g and 346,8 ml/g for rCO 1, rCO 2 and rCO 3, respectively. It is not feasible to ascertain the viscosity value before the alkali hydrolysis, primarily because cellulose is mixed with polyester. This complicates the ability to reach conclusive insights about the reduction in the degree of polymerization. However, one can compare this value to the intrinsic viscosity measurements of 100 % cotton fibres, which are detailed in Fig. 10. The mean intrinsic viscosity of the cellulose residue from textile waste demonstrates that blend 1 and 2 cellulose could be considered a potential feedstock for the manufacturing of viscose staple fibres. According to Sixta [55], the intrinsic viscosity deemed appropriate for dissolving pulps used in the production of standard viscose fibres is between 450 and 500 ml/g. Blend 3, however, fell short of the desired viscosity range. It is advisable to enhance this cellulose by incorporating cellulose pulp with a higher viscosity, particularly if it is intended for use in textile fibre production.

4. Conclusions

In this study, alkaline hydrolysis technique was successfully employed in achieving selective degradation and recovery of high-purity terephthalic acid and cotton cellulose from postconsumer polyester/cotton blended fabrics waste. The optimum conditions for the hydrolysis process were found to be 15 % (w/v) NaOH in the presence of 1 mol BTBAC: 1 mol per repeating unit of polyester phase transfer catalyst and processing temperature and time of 80 °C and 270 mins, respectively. These conditions resulted in complete hydrolysis of the polyester in polyester/cotton blended fabrics waste. The conversion rate of PET was found to increase with increase in temperature up to 90 °C which was the highest temperature used in this work. PET conversion rate also increased with increase in NaOH concentration up to 15 % (m/v) NaOH which was the highest NaOH used in this work. Addition of catalyst also improved the conversion rate of PET. Even though, non-optimum processing conditions, that is, processing times beyond 270, temperatures exceeding 80 °C in the absence of a catalyst can lead to the complete hydrolysis of PET, such conditions have some adverse effects to the process and recovered materials. Some of the adverse effects non-optimum conditions include increased energy consumption while simultaneously causing a significant reduction in the degree of polymerization of the cotton when using high temperatures and high NaOH concentrations negatively affects yield of TPA and also causes a decrease in the viscosity of the recovered cotton. Recovered TPA can reused in the production of polyester fibres through repolymerization with either virgin or recycled ethylene glycol while the recovered cotton fibres may be used in the production of regenerated fibres through a dissolution process. The successful separation of TPA and cotton fibres implies that the recycling of blended textiles can be accomplished and scaling up the processes would result in commercialisation of the recycling technique.

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CRediT authorship contribution statement

Rivalani Baloyi Baloyi: Writing – original draft, Methodology, Investigation, Conceptualization. **Bruce Bishop Sithole:** Writing – review & editing, Supervision, Funding acquisition. **Mufaro Moyo:** Writing – review & editing, Supervision, Formal analysis.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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