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Mild valorisation process of polycotton waste using a recyclable ionic liquid for lactic acid production, combined with polyester recovery

H. Poy [a], M. Capilla [a], E. Lladosa [a], B. Taroncher [b], P. San-Valero [a], S. Loras [a], C. Silvestre [a], P. Ferrero [b], J. Álvarez-Hornos [a,*]

[a] Department of Chemical Engineering, Universitat de València, Av. De la Universitat S/N, Burjassot 46100, Spain
[b] AIMPLAS, Technological Institute of Plastics, València Parc Tecnològic C. Gustave Eiffel 4, Valencia, Paterna 46980, Spain

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ABSTRACT

Textile waste, commonly found in blends, is a potential raw material that needs recycling due to the rise of the fast-fashion model. In this study, a method to valorise cotton-polyester textile waste (average composition 60 % cotton, 40 % polyester) into a value-added chemical, such as lactic acid was investigated. As pretreatment, the most effective ionic liquid was the 1-ethyl-3-methylimidazolium acetate, [Emim][OAc]. This one at a relatively low temperature (60 °C) and a short duration (15 min) drastically increased the digestibility of treated cotton textiles to levels exceeding 95 %. Furthermore, the proposed method enabled the recovery of the polyester fraction with a high similarity (96 %) to commercial polyester. The recovery and reuse of the [Emim][OAc] were assessed, with up to four recycling cycles conducted, showing minimal differences between cycles, with recovery values ranging from 81.8 % to 89.2 %. The pretreated textile waste was hydrolysed, and lactic acid was successfully produced with the same glucose uptake and lactic acid fermentation profiles regardless of the cycle of [Emim][OAc] utilisation. The lactic acid yields were excellent, ranging from 0.92 to 0.97 g/g and an overall yield of ~0.5 kg of lactic acid per kg of textile waste was achieved. The lack of differences in enzymatic hydrolysis and fermentation showed that there was no adverse effect on the reutilisation of [Emim][OAc] in the overall process; therefore, this approach may enhance the circularity of polycotton waste into high-value bioproducts.

1. Introduction

The textile industry is currently facing significant challenges related to resource management and environmental impact. Driven by the fast-fashion business model, the increasing global population, and rising per capita purchasing power, global fibre production has nearly doubled over the past two decades to 116 million tonnes in 2020 and is projected to reach 160 million tonnes by 2030 [1]. This fast-fashion model results in low-quality garments with limited reuse and recycling potential, leading to a significant increase in textile waste generation in recent years [2]. Worldwide, approximately 80 % of textile waste is landfilled or incinerated [3], resulting in significant environmental issues and a substantial loss of valuable resources. Indeed, the European textile industry ranks fifth in raw materials use and contributes approximately 10 % of greenhouse gas emissions [4]. This situation has prompted a paradigm shift towards the efficient recycling of textiles, facilitating the transition of the textile industry to a circular and low-carbon business model. For this purpose, different technologies have been proposed based on mechanical, chemical or biochemical textile recycling [5].

Most textiles are produced from fabric blends due to their adaptable properties, which typically involve mixing natural fibres with various proportions of synthetic fibres [6]. The global fibre market is primarily dominated by interwoven blends of cotton (mainly composed of 88–96 % w/w cellulose [7]) and polyester (mainly poly(ethylene terephthalate), PET), also known as polycotton blends [8]. The heterogeneous nature of polycotton blends constitutes a substantial obstacle to textile waste separation and recycling, often necessitating the use of harmful solvents and/or energy-intensive processes in conventional mechanical and chemical recycling methods [9].

Alternatively, the use of enzymes in biochemical recycling can provide an environmentally friendly and low-energy-demand pathway for selectively depolymerising a specific polymer in blended fabrics into its monomeric building blocks, while preserving the other components [5,10,11]. Specifically, cellulase enzymes are recognised as an essential

* Correspondence to: Research Group GI2AM, Department of Chemical Engineering, Universitat de València, Spain
E-mail address: Francisco.J.Alvarez@uv.es (J. Álvarez-Hornos).

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type of biocatalyst for the processing of polycotton, which in turn allows the concomitant solubilization and hydrolysis of cellulose into its glucose units [8,12]. The produced glucose can be used as a substrate in microbial fermentation to produce value-added chemicals, while the high-purity recovered PET can be recycled in a closed-loop system [6, 13]. Within the realm of the fashion industry, the production of optically pure lactic acid (LA) from glucose is of particular interest, as LA is used in the biosynthesis of polylactic acid (PLA), which is recognised as an eco-friendly bio-based polyester and a promising substitute for petroleum-based synthetic PET [14,15]. Therefore, producing LA from polycotton could help fill the gap in the textile industry for efficient recycling of blended fabrics while promoting sustainability.

A significant challenge in the valorisation of cotton-based waste lies in the recalcitrant structure of cellulose, which limits the accessibility of enzymes for glucose production and hinders the dissolution of fibres in water and common organic solvents [16,17]. Hence, the dissolution of cotton-based waste has emerged as a key pretreatment step to overcome the recalcitrance of cellulose before its valorisation by different processes. Recently, Cho et al. [18] have valorised cotton-based textile waste into different bio-based chemicals by an integrated biorefinery process. The proposed approach was based on conventional pretreatment of textile waste with 15 % NaOH to facilitate subsequent fermentation into lactic acid, yielding 0.84 g/g. However, in recent years, ionic liquids (ILs) have emerged as a promising alternative to traditional cellulose-dissolving solvents due to their greener nature and better performance under milder conditions [19]. ILs are organic salts with melting points below 100 °C, which are considered “green designer solvents” due to their low volatility and the possibility of tuning their physicochemical properties according to the cations and anions used in their synthesis [20]. Particularly, ILs based on 1-alkyl-3-methylimidazolium as a cation are among the most promising for processing cellulose [8], which has promoted their use as pretreatment reagents in the saccharification of lignocellulosic biomass [21] as well as in the chemical recycling of cotton-based waste for different purposes [22,23]. Moreover, the use of ILs for processing polycotton enables the selective dissolution of cellulose without interfering with the PET fraction [24, 25]. Nevertheless, the economic feasibility of employing ILs can be limited by the cost and recyclability of these materials themselves. Thus, researchers are keen to recover and reuse the ILs to reduce the overall cost and increase the environmental friendliness of the process [26].

In the present work, a mild recycling method involving the dissolution of polycotton with ILs followed by enzymatic hydrolysis was assessed for the valorisation of the cotton fraction into LA as a valuable precursor in the fashion industry, while recovering the PET fraction. For this purpose, a screening of the cellulose dissolution ability of several ILs was conducted, followed by the selection of the optimal operating temperature. Afterwards, the suitability of the process was evaluated by the enzymatic hydrolysis of the pretreated samples, followed by microbial fermentation into LA without further detoxification. Since recyclability is a key aspect influencing economic and environmental performance, the reuse of the best IL was evaluated.

2. Materials and methods

2.1. Materials, reagents and microorganisms

The textiles used in this study were discarded black-dyed and white T-shirts consisting of a 40/60 polyester/cotton blend, obtained from a household waste collection in Valencia (Spain). Pure cotton fibres (Sigma-Aldrich) and commercial PET (NOVAPET) were acquired as reference materials.

The ILs used were: 1-allyl-3-methylimidazolium chloride ([Amim][Cl], >99 % w/w purity), 1-ethyl-3-methylimidazolium methylsulfate ([Emim][$MeSO_4$], >99 % w/w purity), 1-ethyl-3-methylimidazolium ethyl sulfate ([Emim][$EtSO_4$], >98 % purity), 1-ethyl-3-methylimidazolium acetate ([Emim][OAc], >95 % w/w purity), 1-butyl-3-methylimidazolium acetate ([Bmim][OAc], >98 % w/w purity), 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide ([Emim][NTf_2], >99.5 % w/w purity) and 1-ethyl-3-methylimidazolium dicyanamide ([Emim][DCA], >98 % w/w purity). All ILs were purchased from IoLiTec Ionic Liquids Tecnologies GmbH (Germany) and used without further purification.

The commercial enzyme blend Cellic® CTec2 (Novonesis, Denmark) was used for the enzymatic hydrolysis. The cellulase activity was determined using the standard procedure of the National Renewable Energy Laboratory (NREL) [27], yielding a value of 150.1 FPU/mL.

Lactic fermentation of the hydrolysates was carried out using the bacterium *Heyndrickxia coagulans* DSM 2314 (formerly *W. coagulans* and *B. coagulans*), which was obtained from the German Collection of Microorganisms and Cell Cultures (DSMZ, Germany). The culture was maintained in 20 % (v/v) glycerol with DSMZ medium 1 (5 g/L peptone and 3 g/L meat extract) at −80 °C. For seed inoculum, the bacteria were cultured at 50 °C and 150 rpm overnight in 250-mL conical flasks with a working volume of 50 mL. The seed medium was (g/L): glucose, 20; yeast extract, 20; sodium acetate, 0.5; triammonium citrate, 0.2; NaCl, 0.01; KH_2PO_4, 0.2; $MgSO_4{\cdot}7\,H_2O$, 0.2; $MnSO_4{\cdot}7\,H_2O$, 0.05.

2.2. Screening of ILs for the dissolution of cellulose

The maximum solubility of cellulose in different ILs was determined using pure cotton fibres as a model substrate. Seven imidazolium-based ILs were evaluated: [Amim][Cl], [Emim][$MeSO_4$], [Emim][$EtSO_4$], [Emim][OAc], [Bmim][OAc], [Emim][NTf_2] and [Emim][DCA]. Solubility tests were performed at 100 °C in a paraffin bath with constant magnetic stirring (250 rpm). The pure cotton fibres were gradually added into the IL in 1 % w/w increments of the total solution until no further dissolution was observed. The dissolution of cotton was monitored visually by observing the disappearance of turbidity in the solution, which was taken as the endpoint of the dissolution process. The saturation limit was considered to be reached when no further changes were seen in the solution after one hour of adding the sample. To validate this qualitative approach, preliminary optical microscopy observations were conducted in a few experiments, confirming the visual observation of cellulose fraction dissolution. The IL with the highest cellulose solubility was chosen for further optimisation of the pretreatment.

2.3. IL pretreatment of polycotton waste

Before IL pretreatment, the textile waste was subjected to a bleaching process to remove impurities following the procedure proposed by Xia et al. [9] with minor adjustments. Briefly, the samples were cut into 20 × 20 mm pieces and treated at 2 % w/w solid loading with a solution of commercial bleach (3.3 % NaClO, diluted 1:1 v/v with deionised water) at 60 °C for 20 min under gentle agitation. After bleaching, the textiles were thoroughly washed with deionised water until neutral pH was reached, dried in a ventilated oven at 50 °C overnight, and stored in a dry place at room temperature before use.

Pretreatment of the polycotton waste with the chosen imidazolium-based IL was performed at 8.5 % w/w of solid loading in a glass vessel placed in a paraffin bath under magnetic stirring (250 rpm). Four temperatures, i.e., 40, 60, 80, and 100 °C, were assessed by recording the time needed for the cotton fraction to dissolve in each case. After pretreatment, water was added as an antisolvent (at 2.5 % w/w relative to the IL solution) to precipitate the dissolved cotton fraction. The solid fraction (containing the PET and the regenerated cotton) was washed in five sequential washes with deionised water and separated by centrifugation (4000 rpm, 2934 g, 5 min) and vacuum filtration. The recovered solid was dried at 50 °C and weighed to determine its recovery following the pretreatment process.

2.4. Recovery and reuse of the selected IL

The recyclability of the solvent was evaluated for the selected IL and pretreatment conditions. After each pretreatment cycle, the IL-water mixture from the first wash was collected and subjected to vacuum distillation using a rotary evaporator until the residual water content was reduced to 10 % w/w. The IL was recycled up to four times, with 20 % w/w of fresh IL added in each new cycle. The effectiveness of the recycling process was assessed through enzymatic hydrolysis of polycotton, followed by lactic acid production via microbial fermentation.

2.5. Enzymatic hydrolysis

Enzymatic hydrolysis of untreated (control experiment) and treated polycotton was conducted at 50 °C and 150 rpm for 72 h in an orbital incubator (G25, New Brunswick Scientific, USA). Samples were suspended in 0.05 M sodium acetate buffer (pH 5.5) with a textile waste loading of 5 % w/v and an enzyme dosage of 20 FPU/g. After saccharification, the reaction mixture was centrifuged (2934 g, 5 min) and vacuum filtered. The recovered solid fraction, which is expected to contain all the PET from the textile waste, was stored at room temperature before analysis. The glucose-rich hydrolysate was analysed by high-performance liquid chromatography (HPLC) to assess the efficiency of the saccharification process. The saccharification process was evaluated based on glucose yield, considering solid losses during pretreatment and the cellulose digestibility of the pretreated samples, as described by the following equations:

$$\text{Glucose yield}(\%) = \frac{\text{Glucose}(g/L)\cdot 0.9^{*}\cdot \text{Solid recovery}(\%)}{\text{Textile waste loading}(g/L)\cdot \text{Cellulose content in untreated polycotton}}\cdot 100 \quad (1)$$

$$\text{Cellulose digestibility}(\%) = \frac{\text{Glucose}(g/L)\cdot 0.9^{*}}{\text{Textile waste loading}(g/L)\cdot \text{Cellulose content in pretreated polycotton}}\cdot 100 \quad (2)$$

*Conversion factor glucose-to-cellulose.

2.6. Lactic acid fermentation

Fermentations were carried out using 23 mL of the hydrolysate in 30 mL serum bottles with a working volume of 25 mL. The fermentation media comprised (g/L): yeast extract, 5; sodium acetate, 0.5; triammonium citrate, 0.2; NaCl, 0.01; KH_2PO_4, 0.2; $MgSO_4{\cdot}7\,H_2O$, 0.2; $MnSO_4{\cdot}7\,H_2O$, 0.05 and $CaCO_3$, 10. The carbon source was 23.0 ± 1.4 g/L for the fermentation assays and 23.0 g/L of glucose for the blank control. The fermentations were performed at 50 °C and 150 rpm with an inoculum size of 5 % (v/v) for 72 h. All media were sterilised before use at 121 °C for 20 min in an autoclave. All experiments were performed in triplicate. Liquid samples were withdrawn at the desired time points for analysis of pH, optical density (OD_{600}), glucose, and lactic acid. For the evaluation of the fermentation process, the following equations, which are widely used in biomass pretreatment and fermentation studies, were applied to calculate process yields and conversion ratios:

$$\text{Lactic acid yield}\left(g\Big/g\right) = \frac{\text{Lactic acid produced}\ (g/L)}{\text{Glucose consumed}\ (g/L)} \quad (3)$$

$$\text{Lactic acid}\Big/\text{Raw Textile Ratio}\left(g\Big/kg\right) = \frac{(\text{Lactic acid produced}(g/L) * V_{\text{Fermentation}}\ (L))/V_{\text{Hydrolysate}}(L)}{[\text{Textile waste loading}(kg/L)/\text{Solid recovery}(\%)]\bullet 100} \quad (4)$$

2.7. Analytical methods

The cellulose content in the untreated and pretreated polycotton waste was determined according to the standard protocol reported by NREL [28]. The fraction designated as acid-insoluble lignin in the NREL protocol was referred to as non-cellulosic residue [29], and it was assumed to comprise the PET fraction along with other additives. The chemical changes in the recovered PET after saccharification were analysed by Fourier Transform Infrared Spectroscopy (FT-IR) (Nicolet 6700, Thermo Scientific) over the range of 400–4000 cm^{-1}, using 32 scans with a spectral resolution of 1 cm^{-1}. The software utilised for estimating the similarity of the spectra to commercial PET was MestRenova. The water content in the recycled IL was quantified by Karl Fischer titration (787 KF Titrino, Metrohm, Switzerland). The morphology of the polycotton waste before and after pretreatment, as well as the solid recovered after saccharification, was analysed by scanning electron microscopy, SEM (S-4800, Hitachi, Japan), operating at an acceleration voltage of 20 kV. Samples were sputter-coated with gold palladium before SEM analysis. Thermogravimetric analysis (TGA) was also performed in a TA Instruments TGA550 device from 25 to 700

Table 1
Maximum capacity of the IL tested to dissolve pure cotton fibres at 100 °C.

Entry	IL	Abbrev.	Solid loading (% w/w)
1	1-ethyl−3-methylimidazolium bis (trifluoromethylsulfonyl)imide	[Emim] [NTf_2]	0
2	1-ethyl−3-methylimidazolium dicyanamide	[Emim] [DCA]	0
3	1-ethyl−3-methylimidazolium methylsulfate	[Emim] [$MeSO_4$]	8
4	1-ethyl−3-methylimidazolium ethyl sulfate	[Emim] [$EtSO_4$]	0
5	1-ethyl−3-methylimidazolium acetate	[Emim] [OAc]	15
6	1-butyl−3-methylimidazolium acetate	[Bmim] [OAc]	11
7	1-allyl−3-methylimidazolium chloride	[Amim] [Cl]	7

°C at a heating rate of 10 °C/min with a nitrogen flow rate of 60 mL/min.

The concentrations of glucose and lactic acid were determined using an HPLC system (Agilent 1100 Series, Agilent Technologies, USA). The HPLC was coupled with an Aminex® HPX-87H column (300 mm × 7.8 mm, Bio-Rad Laboratories Inc., USA), operating at 50 °C, and a refractive index detector (RID) operating at 35 °C. A 5 mM sulfuric acid solution was used as the mobile phase at a flow rate of 0.6 mL/min.

3. Results and discussion

3.1. Assessment of the cellulose dissolving capacity of imidazolium ILs

To identify the most suitable ionic liquid for processing polycotton waste, a screening was performed to evaluate the cellulose dissolution capacity of the seven imidazolium ILs, using pure cotton fibres as a model substrate for cellulose. The maximum solubility of cellulose in each IL at 100 °C is listed in Table 1, which indicates the maximum percentage of cotton fibres dissolved relative to the ionic liquid. The solubility of pure cotton fibres in ILs when using 1-ethyl-3-methylimidazolium ($[Emim]^+$) as cation (entries 1–5) showed the strong influence of the anion in the dissolution of cellulose. In this regard, several studies have suggested that the formation of hydrogen bonds between anions and the hydroxyl groups of cellulose is the main driving force for cellulose dissolving in ILs, which relates to the hydrogen bond basicity of the anion [30,31]. Therefore, the weak hydrogen bond basicity of the anions $[NTf_2]^-$ and $[DCA]^-$ [26] may explain why the ILs [Emim][NTf_2] and [Emim][DCA] (entries 1–2) failed to dissolve pure cotton fibres, owing to their restricted capacity for interacting with cellulose. In addition, the large size of the anions $[NTf_2]^-$ and $[DCA]^-$ also contributes to their inability to dissolve pure cotton fibres. The midrange basicity of the anion $[MeSO_4]^-$ [32] led to the dissolution of 8 % w/w of pure cotton fibres when using the IL [Emim][$MeSO_4$] (entry 3). However, increasing the alkyl chain length of the sulphate anion (entry 4 vs. entry 3) resulted in no dissolution of cellulose with the IL [Emim][$EtSO_4$] (entry 4). This result shows that cellulose dissolves better in ILs when smaller anions are used, which is related to the improved self-diffusion ability of the ions and thus the enhanced hydrogen bond interaction between the IL and cellulose [33]. Furthermore, increasing the alkyl chain length of the anion can significantly increase the viscosity of the resulting IL [34], which strongly hinders the mass transport of ions during the dissolution process. All these reasons support that the anion $[MeSO_4]^-$ is a better choice compared to $[EtSO_4]^-$ for dissolving pure cotton fibres.

From the results listed in Table 1 corresponding to the ILs containing the cation $[Emim]^+$ (entries 1–5), the highest solubility of cotton fibres (15 % w/w) was obtained with the anion $[OAc]^-$ (entry 5). The high hydrogen basicity and small size of the $[OAc]^-$ anion [35] make it the strongest hydrogen bond acceptor, thereby being the most effective in disrupting the intra- and intermolecular hydrogen bonds between the cellulose chains. Indeed, the solubility of pure cotton fibres in either of the two tested $[OAc]^-$-based ILs (entries 5 and 6) was higher than that of all other ILs used (Table 1), corroborating the key role of the anion on the disruption and dissolution of cellulose with ILs. On the other hand, the results obtained for the $[OAc]^-$-based IL (entries 5 and 6) indicated that the structure of the imidazolium cation also influences the solvation process. Specifically, the cation can impact the solubility of cellulose by forming hydrogen bonds with hydroxyl groups or ether oxygen atoms in cellulose [33]. As mentioned above for the anions, the smaller size of the IL components would facilitate interaction with cellulose due to improved transport properties, such as reduced viscosity, thereby enhancing the solvation process [31]. Consequently, the use of the cation $[Emim]^+$ instead of $[Bmim]^+$ increased the solubility of pure cotton fibres from 11 % to 15 % w/w (Table 1).

The presence of $[Cl]^-$ ions in imidazolium-based ILs has also been recognised as a potential option to dissolve cellulose, since the $[Cl]^-$ anion is a strong proton acceptor that interacts effectively with the hydroxyl groups of cellulose [36]. The interest in using ILs for cellulose processing was initially sparked by the discovery of the ability of [Bmim][Cl] to dissolve cellulose, as reported by Swatloski et al. [37]. Further studies support that including electron-withdrawing groups in the cation of $[Cl]^-$-based ILs can improve their interaction with cellulose, as seen with an allyl group that can form hydrogen bonds with cellulose [38]. Despite its promising characteristics, the use of [Amim][Cl] for dissolving pure cotton fibres resulted in a maximum solubility of only 7 % w/w (entry 7, Table 1), which was significantly lower than the values achieved with $[OAc]^-$-based IL (entries 5 and 6). The results above are linked to the increased basicity of the $[OAc]^-$ anion compared to $[Cl]^-$, which enables more effective disruption of the inter- and intramolecular hydrogen bonds in cellulose [35]. Additionally, other benefits of [OAc]-based ILs over $[Cl]^-$ ones include lower viscosities and melting points, as well as reduced corrosiveness and toxicity due to the absence of halides [30,35]. Moreover, chloride-based ILs have been shown to promote enzyme deactivation and cellulose degradation under harsh conditions, which can limit their practical application in integrated bioprocesses [39].

Based on the results in Table 1, [Emim][OAc] was chosen as the best-performing IL, among those tested, for dissolving pure cotton fibres, achieving the highest cellulose solubility (up to 15 % w/w). This IL is considered non-toxic, non-corrosive, and biodegradable [40], making it an environmentally friendly option for treating cotton-based waste.

3.2. Effect of the dissolution temperature on IL pretreatment of polycotton waste

The waste used in this study showed a cellulose content of 57.7 ± 1.6 % w/w, accompanied by a non-cellulose residue content of 43.6 ± 0.2 % w/w (Table S1, Supplementary Information). These results are consistent with the composition indicated on the manufacturer's label, namely, 60/40 cotton/polyester. The polycotton waste was pretreated with [Emim][OAc] (best-performing IL in the former screening experiment) to enhance its susceptibility to enzymatic hydrolysis. Although [Emim][OAc] can solubilise up to 15 % w/w of pure cotton fibres (Table 1), using such high solid loadings may lead to the formation of highly viscous polymeric aggregates or coagulates, which could complicate the regeneration and washing of the dissolved cotton fraction [12]. Therefore, it was decided to reduce the solid loading to a maximum of 8.5 % w/w to ensure easier handling and processing. The dissolution of cellulose is affected not only by the chemistry of the

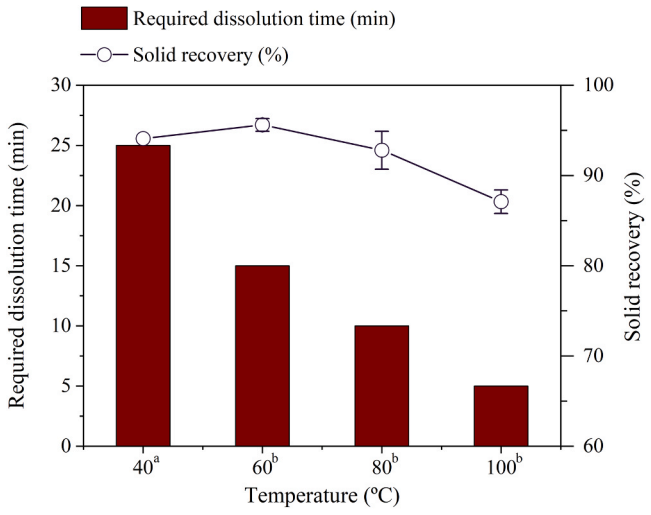

Fig. 1. Influence of the four tested temperatures on the required time to dissolve cotton from the polycotton blend using [Emim][OAc] (bars) and on the solid recovery after the process (points). [a]For a textile loading of 5.5 % w/w; [b]for a textile loading of 8.5 % w/w.

chosen IL but also by process parameters. Among these, temperature is one of the most important factors influencing both the rate of cellulose dissolution and its efficiency [31]. Therefore, the impact of temperature on the dissolution of cotton/polyester blends was examined. Fig. 1 shows the time needed to fully dissolve the cotton fraction from the polycotton blend using [Emim][OAc] at the four tested temperatures (40, 60, 80, and 100 °C), as well as the solid recovery achieved after the process at each temperature.

At the lowest temperature (40 °C), the [Emim][OAc] was unable to fully dissolve the cotton fraction contained in the textile blend within 1 h, forming a highly viscous polymer with visible cotton and polyester fractions. The maximum textile loading processable at 40 °C was found to be 5.5 % w/w, which took 25 min to dissolve the cotton fraction (Fig. 1). Temperatures above 40 °C allowed the use of a textile loading of 8.5 % w/w, resulting in the complete dissolution of the cotton fraction. As shown in Fig. 1, increasing the temperature shortened the time needed to dissolve the cotton fraction, due to the enhanced attack on the cellulose structure. Specifically, the increase in temperature decreased the required dissolution time to 15 min (60 °C), 10 min (80 °C), and less than 5 min for the highest temperature (100 °C), all of which are short-term dissolution processes. Generally, higher temperatures promote a destabilisation of the hydrogen bonds in the three-dimensional structure of cellulose, accelerating its swelling and dissolution [41].

Due to the enhanced dissolution of cellulose at higher temperatures, regenerating the dissolved cotton became more difficult. Consequently, the solid recovery decreased with temperature (Fig. 1), falling from about 95 % at 40 °C to a minimum of 87.1 % at 100 °C, resulting in the loss of valuable substrate for further processing. Higher temperatures are also linked to increased energy consumption, degradation of both cellulose and the IL, and the intensification of side reactions, which can compromise the recyclability of the IL [35,42]. Although [Emim][OAc] is generally considered a non-derivatising solvent, Clough et al. [19] reported that side reactions may occur under harsh conditions, such as 120 °C for 24 h. However, the same study demonstrated that lowering the dissolution temperature to 100 °C significantly reduced byproduct formation, shortening the reaction time from 24 h to 6 h. Based on these findings, a temperature of 60 °C was selected for dissolving polycotton waste in subsequent experiments. This was the lowest tested temperature that achieved an optimal balance between energy efficiency and dissolution performance, requiring only 15 min to dissolve the cotton fraction and yielding a maximum solid recovery of 95.6 %. Additionally, this mild condition minimises degradation of both the ionic liquid and the cotton fraction, enhancing the overall practicality and potentially sustainability of the process.

Previous studies have also explored the use of ILs for the separation and recovery of cellulose from textile blends. For instance, [Amim][Cl] has shown high dissolving efficiency when DMSO is used as the solvent, but the temperatures and time required were found to be slightly higher than those selected in this work. Villalta et al. [43] achieved complete dissolution of a polycotton fabric at a load of ~6 % w/w after 3 h working at 90 °C. Zhang et al. [44] proposed a 1:1 [Amim][Cl]:DMSO ratio to pretreat coloured and white cotton fabrics with a load of 6 % w/w at 110 °C for 120 min. A superbase-based IL, such as [DBNH][OAc], was successfully proven for the separation of cotton and polyester (13 % w/w) at 80 °C over 1 h [25]. More recently, other strategies for recycling polycotton textiles have been applied to reduce operational temperature, including the use of novel deep eutectic solvents. Indeed, Yang et al. [45] were able to dissolve after 24 h up to 15 % w/w of polycotton waste at room temperature using a system based on $ZnCl_2$:H_3PO_4:H_2O [1:3:0.5 molar].

Table 2
Main results of the enzymatic hydrolysis of untreated and pretreated polycotton textile waste.

Sample	Glucose (g/L)	Acetic acid (g/L)	Cellulose digestibility (%)	Glucose yield (%)
Untreated polycotton	1.03 ± 0.01	4.46 ± 0.12	3.12 ± 0.06	3.12 ± 0.06
Pretreated polycotton[a]	29.77 ± 1.01	3.30 ± 0.18	95.12 ± 2.66	86.54 ± 2.42

[a] Pretreated with [Emim][OAc] at 60 °C, 15 min and 8.5 % w/w.

3.3. Effect of IL pretreatment on the enzymatic hydrolysis

The untreated and recovered polycotton after IL pretreatment ([Emim][OAc] at 60 °C for 15 min and 8.5 % w/w) was subjected to enzymatic hydrolysis to produce glucose as a substrate for microbial LA fermentation, while recovering the PET fraction. After pretreatment, the composition of the polycotton sample was found to be 54.6 % cellulose and 44.0 % non-cellulose residue (Table S1). These findings show that 91 % of the cellulose was recovered in the pretreated solid, making it available for further saccharification. Meanwhile, 97 % of the PET fraction was retained in the pretreated polycotton.

The results from the enzymatic hydrolysis of untreated and pretreated polycotton samples are shown in Table 2. The lower acetic acid concentration in the hydrolysate from the pretreated polycotton may indicate the absence of IL moieties in the pretreated solid, supporting the effectiveness of the regeneration and washing process used after IL pretreatment. Without pretreatment, enzymatic hydrolysis produced only 1.03 g/L of glucose, which reflects just 3 % saccharification of the cellulose in the original polycotton waste (3.12 % glucose yield/cellulose digestibility). The low digestibility of untreated polycotton confirms that a pretreatment process is essential before breaking down the polysaccharide content in polycotton with cellulases. As shown in Table 2, the glucose concentration increased from 1.03 to 29.77 g/L after pretreating polycotton with [Emim][OAc], indicating that 95 % of the cellulose in the pretreated solid was successfully saccharified (95.12 % cellulose digestibility). Considering the losses involved in the pretreatment, the processing of polycotton with [Emim][OAc] followed by enzymatic hydrolysis resulted in the saccharification of 87 % of the cellulose (86.54 % glucose yield). These results confirmed the effectiveness of the selected pretreatment with [Emim][OAc] in disrupting the recalcitrant structure of cellulose, thereby increasing its accessibility to cellulases. In this regard, several studies have shown that pretreating cellulose with [Emim][OAc] can facilitate the conversion of highly crystalline cellulose I (native cellulose) into cellulose II, which becomes more amorphous and easier for cellulases to access [46,47]. For instance, a previous study reported a decrease in the crystallinity of rice straw from 51 % to 19 % using [Emim][OAc] as a pretreatment reagent, which enabled the complete saccharification of cellulose in the pretreated solid [21]. However, achieving this effectiveness required a more energy-intensive process (120 °C for 5 h) combined with a lower solid loading (5 % w/w) compared to the conditions selected herein (60 °C for 15 min, 8.5 % w/w). Similarly, Guo et al. [48] suggested using the IL [Amim][Cl] as an effective method for pretreating cotton-based waste. In particular, the pretreatment of purple bed sheet made of pure cotton fabrics with [Amim][Cl] achieved a similar digestibility (98 %) to that in the present study (95.12 %), although it was also influenced by the use of more severe conditions (110 °C, 75 min, 10 % w/w) compared to those used here. Conversely, the selected pretreatment might provide a more environmentally friendly and gentler approach compared to conventional chemical pretreatments of waste, such as the process used for waste blue jeans (60/40 cotton/polyester) with Na_2CO_3 1 M (88.9 % glucose yield, 150 °C, 20 min, 5 % w/w) [17] or the pretreatment of jeans (86/14 cotton/polyester) with 85 % H_3PO_4 (79.2 % glucose yield, 50 °C, 7 h, 5 % w/w) [49]. Additionally, although imidazolium ILs are shown to inhibit cellulase activity at high concentrations (> 0.5 M) [50], the saccharification of the pretreated polycotton exceeded 95 % of cellulose digestibility. This high efficiency indicates that the washing process was able to remove [Emim][OAc] from the recovered polycotton, preventing negative effects on enzymatic

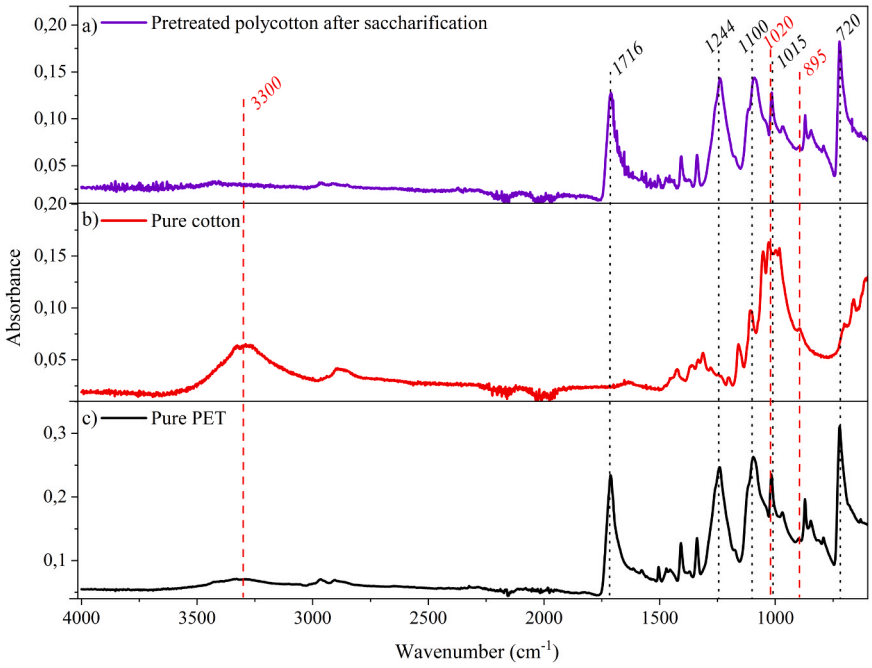

Fig. 2. FT-IR spectra of (a) solid residue of pretreated polycotton after saccharification, (b) pure cotton and (c) commercial pure PET. Characteristic absorption bands of cellulose are indicated with red dashed lines, while black dashed lines represent those for commercial pure PET.

performance. Therefore, the proposed dissolution process stands out as not only effective but also promising for the sustainable circularity of polycotton waste.

Regarding the PET fraction, the average solid recovery after separating the enzymatic hydrolysate was found to be 48 %, resulting in an average PET recovery of 98 %. The recovered solid was analysed by FT-IR to examine the changes in its functional groups compared to commercial pure PET and pure cotton samples. The FT-IR spectra for (a) the solid residue of the pretreated polycotton after saccharification, (b) the pure cotton and (c) the commercial pure PET are shown in Fig. 2. The spectrum of pure cotton exhibits three distinct absorption bands characteristic of cellulose at 3300 cm^{-1} (hydroxyl group stretching), 1020 cm^{-1} (C-O stretching), and 895 cm^{-1} (C-O-C stretching) [10,20]. These bands were not detected in the spectrum of the recovered solid, indicating the effective removal of cellulose by cellulases. Furthermore, the spectrum of this solid closely resembled that of commercial pure PET. In this regard, the characteristic absorption bands of PET at 1716 cm^{-1} (C═O stretching), 1244 cm^{-1} (C-O-C stretching), 1100 and 1015 cm^{-1} (C-O stretching), and at 720 cm^{-1} (C-H bending) [51] were retained in the recovered polycotton after enzymatic hydrolysis. Indeed, the similarity of its functional groups in the recovered polycotton to those in commercial pure PET was found to be 96 %, which closely aligns with the gravimetric recovery value of 98 %. TGA also confirmed this similarity, with ~90 % of recovered PET corresponding to the main decomposition peak of commercial PET (Figure S1, Supplementary Information). Additionally, morphological changes in the polycotton were examined by SEM images for the original polycotton waste (Fig. 3a), the pretreated polycotton with [Emim][OAc] (Fig. 3b) and the solid residue of the pretreated polycotton after saccharification (Fig. 3c). The untreated polycotton (Fig. 3a) presented a packed structure where cotton fibres (twisted shape and rough surface) and polyester fibres (cylindrical shape and smooth surface) can be distinguished. After pretreatment with [Emim][OAc] (Fig. 3b), the structure appeared more open, with polyester fibres more exposed and cotton fibres became more aggregated. The solid residue recovered after saccharification (Fig. 3c) was composed mainly of polyester fibres, in agreement with the high similarity with commercial PET determined by FT-IR and TGA. Leenders et al. [52] reported similar observations, finding polyester as the remaining fraction after acid-hydrolysis of a 44/56 cotton/polyester textile waste.

Therefore, the pretreatment of polycotton with [Emim][OAc] followed by enzymatic hydrolysis has been demonstrated to be an effective method for separating and recycling polycotton waste into its main components: cellulose and PET, thereby removing the need for harsh temperatures or harmful reagents.

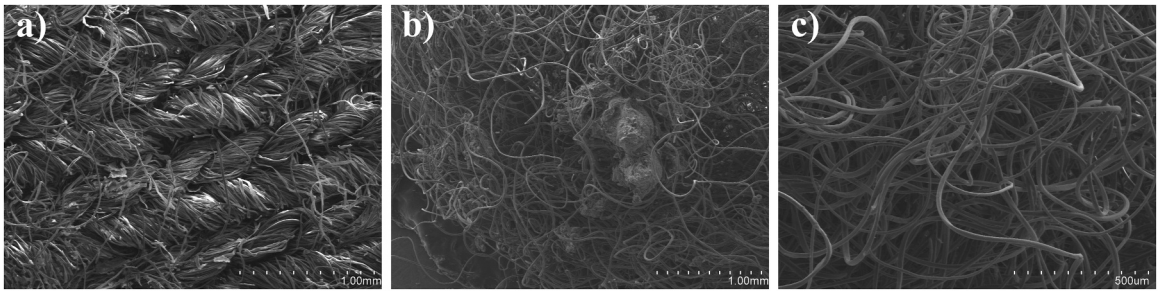

Fig. 3. SEM images of the polycotton: (a) untreated (x45 magnification), (b) pretreated (x35 magnification) and (c) solid residue of pretreated polycotton after saccharification (x90 magnification).

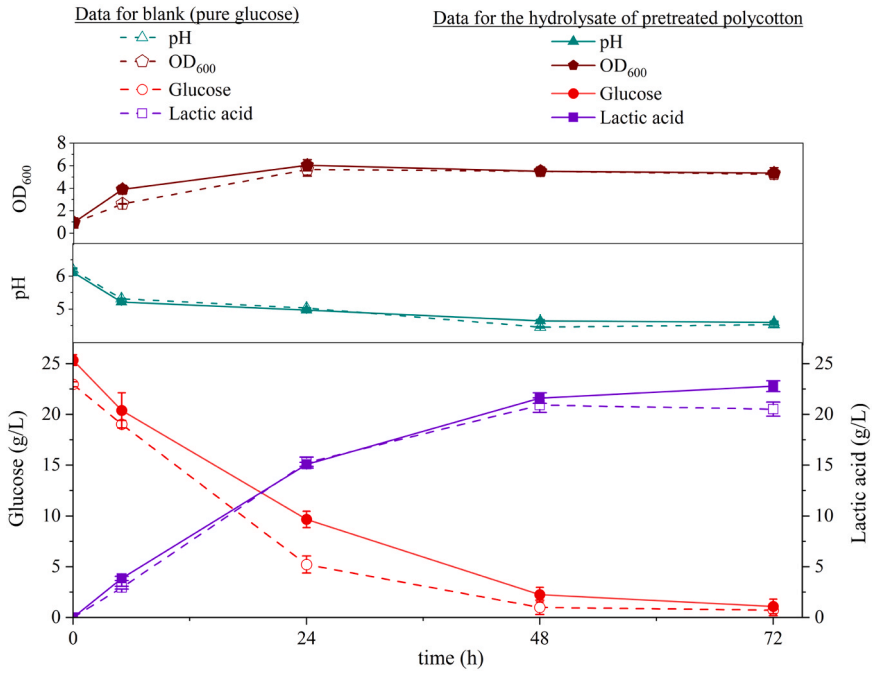

Fig. 4. Fermentation profile by *H. coagulans* using pure glucose as substrate (dashed lines) and the enzymatic hydrolysate from pretreated polycotton with [Emim][OAc] (solid lines).

Table 3
Glucose consumption and lactic acid production by *H. coagulans*.

Sample	Initial glucose (g/L)	Lactic acid (g/L)	Glucose conversion (%)	Lactic acid yield (g/g)
Blank	22.97 ± 0.25	20.51 ± 0.69	96.91 ± 2.32	0.92 ± 0.04
Enzymatic hydrolysate from IL-pretreated polycotton[a]	25.35 ± 0.54	22.79 ± 0.55	95.71 ± 2.84	0.94 ± 0.04

[a] Pretreated with [Emim][OAc] at 60 °C, 15 min, 8.5 % w/w.

3.4. Production of L-lactic acid from the enzymatic hydrolysate

The enzymatic hydrolysate from [Emim][OAc]-pretreated cotton/polyester textile, as well as pure glucose as model substrate (blank), was fermented with *H. coagulans* to assess the feasibility of the overall process in producing optically pure lactic acid. The fermentation profiles are shown in Fig. 4, where the variation with time of the produced lactic acid concentration, glucose concentration, optical density, and pH is plotted for both the model substrate and the hydrolysate of pretreated polycotton. In addition, the main parameters of the fermentation process are listed in Table 3. As shown in Fig. 4, the variation in pH of the fermentation medium was consistent, regardless of whether the assay used the pure glucose blank or the enzymatic hydrolysate. Additionally, the bacteria exhibited the same growth profile, as reflected in the evolution of the OD_{600}. Similarly, glucose consumption followed the same pattern in both cases, achieving almost complete conversion of the available monosaccharide (>95 % glucose consumption, Table 3). Furthermore, lactic acid production occurred concurrently with glucose uptake, exhibiting identical trends across all experiments (Fig. 4). The hydrolysate from the discarded T-shirts showed the same fermentation behaviour as the control, and the high cellulose digestibility (>95 %) achieved during hydrolysis indicates that the residual dyes in this type of textile waste did not adversely affect either the enzymatic or the microbial process. Although ILs have been recently proposed as agents for dye solubilization [53], in the case of valorising multi-coloured textile waste, a detoxification method would probably be necessary to remove dyes and additives from the hydrolysate. Among the most widely applied techniques could be highlighted physicochemical treatment such as adsorption, ion exchange or membrane filtration [54].

At the end of the fermentation (72 h), the lactic acid concentration reached 20.51 g/L for the glucose blank and 22.79 g/L for the enzymatic hydrolysate, indicating a lactic acid yield of 0.92 and 0.94 g/g, respectively (Table 3). The similar fermentation performance to that of pure glucose confirms the feasibility of using the enzymatic hydrolysate from pretreated polycotton waste as a substrate for microbial lactic acid production. In a previous study, Mihalyi et al. [6] also described the fermentation of the enzymatic hydrolysate of a textile blend (viscose/-PET 58/42) by *H. coagulans* as being identical to that of the pure glucose counterpart, indicating equivalent substrate quality.

Furthermore, the lactic acid yields obtained in this study are comparable to or even surpass those reported for easily fermentable substrates in batch fermentations using homofermentative *H. coagulans*. For instance, Ou et al. [55] reported that *Bacillus coagulans* 36D1 was able to produce lactic acid from pure sugars with yields ranging from 0.86 to 0.96 g/g, while López-Gómez et al. [56] demonstrated that pretreated organic municipal waste could be fermented by *B. coagulans* A166, achieving a yield of 0.94 g/g. Other studies have focused on lactic acid production from lignocellulosic biomass pretreated with IL. For example, Qureshi et al. [57] obtained a yield of 0.81 g/g during fermentation by *L. casei* ATCC 334 of sugarcane bagasse at a load as high as 25 % w/w, after its pretreatment with a combination of diluted sulfuric acid and [Emim][Cl]. Similarly, Yang et al. [58] showed that the conversion of corn stalk pretreated with [Ch][Gly] by *Bacillus* sp. strain P38 achieved a lactic acid yield of 0.96 g/g.

Regarding the valorisation of textile waste into lactic acid, Table 4 summarises the results of this study along with those reported in the literature for textile-derived substrates. Studies using viscose [6,15] achieved relatively high yields in fermentation after only an enzymatic step. However, the valorisation of cotton-based textiles or cotton/PET blends required an additional chemical pretreatment to enhance cellulose accessibility, as also demonstrated in this work (Table 2). For example, Cho et al. [18] pretreated white T-shirts (100 % cotton) with a 15 % NaOH solution for 30 min, which were subsequently

Table 4
Comparison of lactic acid production from textile-derived substrates.

Textile	Pretreatment	Batch fermentation method[a]	Microorganism	Lactic acid, (g/L)	Lactic acid yield, (g/g)	Reference
Viscose/PET (58/42)	Enzymatic hydrolysis	SSF	*Heyndrickxia coagulans* DSM 2314	12.8	n.d.	[6]
Post-consumer viscose	Enzymatic hydrolysis	SHF	*Pediococcus acidilactici* ZP26	42.4	0.97	[15]
Cotton textile waste	NaOH pretreatment + enzymatic hydrolysis	SHF	*Actinobacillus succinogenes* ATCC 55618	12.3	0.84	[18]
Cotton textile waste	NaOH pretreatment + enzymatic hydrolysis	SHF	*Saccharomyces cerevisiae* strain CEN.PK m850	53.0	0.83	[59]
Cotton/PET (60/40)	[Emim][OAc] pretreatment + enzymatic hydrolysis	SHF	*Heyndrickxia coagulans* DSM 2314	22.8	0.94	This study

[a] SHF: separate hydrolysis and fermentation; SSF: simultaneous saccharification and fermentation.

enzymatically hydrolysed at a textile waste loading of 1 % w/v for 5 d. Fermentation of hydrolysate using *A. succinogenes* ATCC 55618 was conducted for 36 h, resulting in a lactic acid concentration of 12.3 g/L with a yield of 0.84 g/g. Simonetti et al. [59] employed an engineered yeast strain, *Saccharomyces cerevisiae* CEN.PK m850, which was able to produce a final concentration of 53 g/L (yield of 0.83 g/g) in the valorisation of post-industrial 100 % cotton patches pretreated with a 16 % NaOH solution for 2 h and 24-h enzymatic hydrolysis at a loading of 7.5 % w/v. Compared with these previous studies, the yield obtained here for cotton/PET blends (60/40) is higher than those reported for 100 % cotton waste and slightly lower than that obtained for viscose-based textile. Therefore, the proposed methodology has proven to be an efficient pathway for producing L-lactic acid from blended textile waste.

3.5. Assessment of the recyclability of the selected IL in the valorisation of polycotton waste

A key requirement for the widespread use of ILs is their recyclability. After selecting the pretreatment with [Emim][OAc] at conditions of 60 °C for 15 min and 8.5 % w/w, the IL was recovered and reused in four consecutive pretreatment cycles, following the same procedure as for the fresh IL. In all reuse cycles, 20 % w/w of fresh IL was added to simulate industrial conditions and compensate for IL losses during washing. In every case, the recovery of [Emim][OAc] exceeded 80 % across all reuse cycles, resulting in an average IL recovery of 86.9 ± 3.3 %. Furthermore, evaporating large amounts of water after IL pretreatment would require a substantial energy input, so a decision was made to retain 10 % w/w of water in the IL solution as an energy-saving measure. Previous studies have suggested that a water content higher than 10 % w/w would inhibit the dissolution of cellulose using [Emim][OAc] [60]. In this study, the use of recycled [Emim][OAc] containing less than 10 % water by weight did not interfere with the cellulose solubilisation step. Adding water to the dissolution process led to slightly lower average recoveries of the target fractions (86.4 % cellulose; 95.8 % PET) compared to the fresh IL experiment (91.0 % cellulose; 97.1 % PET) (Table S1, Supplementary Information), although the recovery means between fractions were not statistically significant ($p > 0.05$). Since the regeneration of dissolved polymers relies on breaking the bonds formed between the polymers and the pretreatment reagent, using water as both a cosolvent and antisolvent may hamper polymer regeneration compared to using water solely as an antisolvent, as in the case of the fresh IL experiment. In any case, the achieved recoveries in the cellulose and PET fractions suggest that it is not necessary to separate the IL completely from the antisolvent.

The effectiveness of recycling [Emim][OAc] was assessed through enzymatic hydrolysis of pretreated polycotton solids, followed by lactic acid fermentation. Fig. 5 shows the effect of reusing [Emim][OAc] in the pretreatment and saccharification of polycotton (Fig. 5a) and during lactic acid fermentation (Fig. 5b) over five experimental runs (fresh IL plus four reuses). The glucose yield and cellulose digestibility, alongside the final glucose concentration after saccharification, are shown in Fig. 5a. The lactic acid concentration after fermentation, together with the production yield, is displayed in Fig. 5b. As can be observed in Fig. 5a, the trend of glucose hydrolysis displayed a slight U-shape, with a peak during the fresh IL run and a trough in cycle 2, which was somewhat recovered in subsequent cycles due to the addition of fresh IL in each one. Overall, the average quantity of glucose hydrolysed in the recycling experiments was 27.2 ± 0.6 g/L, compared to 29.8 ± 1.0 g/L of glucose released in pretreated polycotton with fresh IL. As a result, the cellulose digestibility remained around 92.1 % in the reuse cycles compared to 95.1 % for fresh IL, indicating no adverse effects on the hydrolysis of the pretreated solids. These findings confirmed a similar pattern in the hydrolysis of polycotton waste, whether using fresh or

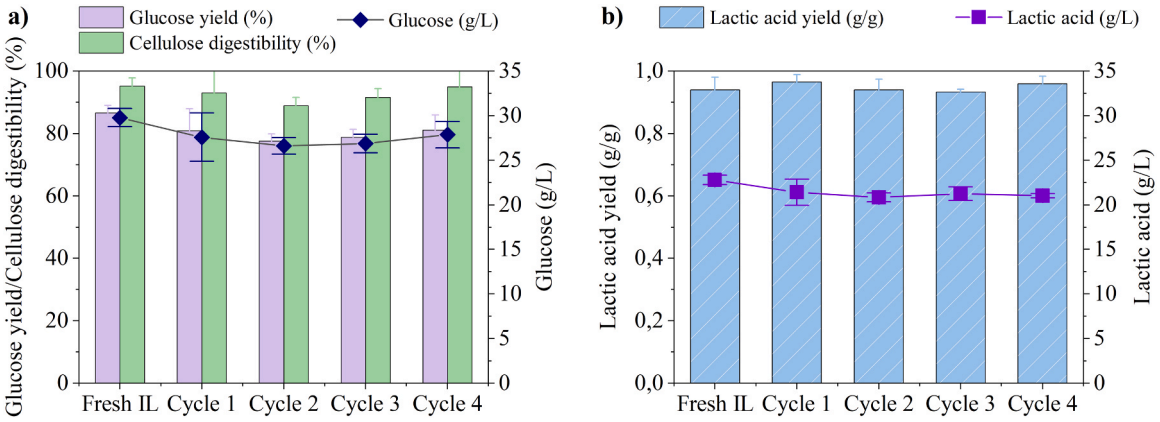

Fig. 5. Evaluation of the recyclability of the IL [Emim][OAc] for pretreating polycotton in terms of: a) glucose yield and cellulose digestibility for enzymatic hydrolysis of the pretreated textile; b) lactic acid concentration and production yield for fermentation of the enzymatic hydrolysate.

recycled IL. Only a small difference was observed when comparing the two sequential processes (pretreatment + enzymatic hydrolysis). In this context, the glucose yield was slightly lower in the reuse cycles (79.6 ± 1.7 % glucose yield, Fig. 5a) compared to the use of fresh [Emim][OAc] (86.5 ± 2.4 %, Fig. 5a), which was attributed to slightly higher cellulose losses during the recycling experiments. Other studies have reported a sharp decrease in the enzymatic hydrolysis of organic waste after reusing the IL [Emim][OAc] without replenishing it with at least a minimal amount of fresh solvent. For example, Qiu et al. [61] demonstrated that the digestibility of pretreated energy cane bagasse declined significantly from nearly 100 % in the fresh [Emim][OAc] to 67 % after employing the recycled IL for up to three cycles. Therefore, the obtained data confirms that replenishing the IL lost during cycles helps maintain consistent cellulose digestibility over extended reuse, thereby enhancing the overall efficiency of the process.

After enzymatic hydrolysis, the produced hydrolysates were subjected to lactic acid fermentation (Fig. 5b). For the recycling experiments, the production of lactic acid was maintained at 21.12 ± 0.25 g/L, compared to 22.79 ± 0.54 g/L achieved for the fresh [Emim][OAc]. The lactic acid yields showed minimal variation among the different assays, ranging from 0.93 ± 0.01 g/g in cycle 3–0.97 ± 0.02 g/g in cycle 1 (Fig. 5b). Therefore, the bacterium's metabolism was not negatively affected by the subsequent reuse of the [Emim][OAc], resulting in fermentation performance comparable to that with fresh IL. In fact, the glucose consumption during the recycling experiments was about 99 %, confirming the excellent performance of the fermentation stage. Overall, these findings validated the effectiveness of the IL recycling strategy for the successive pretreatment of polycotton waste in its valorisation through enzymatic hydrolysis and subsequent fermentation into lactic acid.

3.6. Mass balance of the overall process

The mass balance for the main streams involved in the overall process of producing lactic acid using polycotton waste pretreated with [Emim][OAc] is shown in Fig. 6. The pretreatment of polycotton with [Emim][OAc] enabled the recovery of most of the two building blocks in the pretreated solid, with only traces of cotton and PET in the pretreatment liquor. This liquor was vacuum distilled multiple times to recover the IL and reuse it in subsequent pretreatment cycles, totalling five experimental runs (fresh IL plus four reuses). The enzymatic hydrolysis step separated cotton from polyester by saccharifying the cotton to glucose in the liquid phase, while most of the PET fraction was recovered as an undigested solid. Specifically, 96 % of the original PET was recovered post-hydrolysis, showing a 96 % similarity to pure commercial PET according to FT-IR analysis (Fig. 2). In recycling experiments, approximately 96 % of the PET fraction was recovered after saccharification of the pretreated polycotton, with similarity to pure commercial PET ranging from 87 % to 92 %.

As shown in Fig. 6, most of the cotton fraction was hydrolysed into glucose and then subjected to lactic acid fermentation. Furthermore, most of the glucose was successfully converted into lactic acid, leading to a significant production of 471.5 g of lactic acid per kg of textile waste. In this context, the excellent digestibility of cotton in the textile blend after the [Emim][OAc] pretreatment, combined with the high lactic acid yield of *H. coagulans*, were two key factors for the overall conversion of polycotton into lactic acid. Given that the original polycotton contained 576.6 g of cotton, this result indicates that 81.8 % of the cotton fraction was converted into lactic acid. Additionally, the production of lactic acid remained fairly stable during the recycling of [Emim][OAc], confirming the robustness of the proposed recycling method for processing polycotton into lactic acid. These promising findings highlight the potential to recycle worn-out cotton-blended

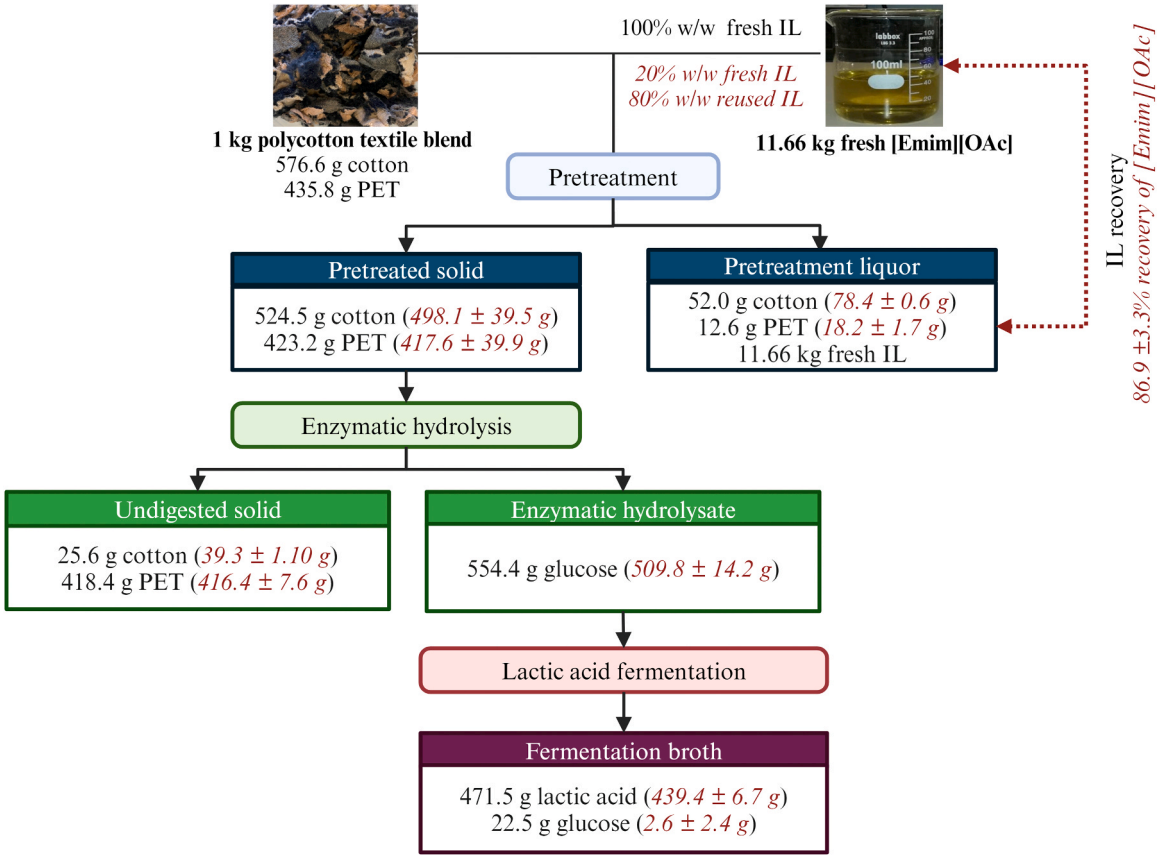

Fig. 6. Mass balance of the entire process for converting polycotton waste into lactic acid using [Emim][OAc]. IL recycling data is shown in parentheses and italics.

textiles into platform chemicals that can be used to create new fibres, as exemplified by lactic acid's conversion into PLA.

4. Conclusions

The sustainability of the fashion industry is at risk due to the significant resource consumption of fast fashion. In this study, we developed a three-stage process for recycling cotton–polyester blends based on ionic liquid pretreatment, enzymatic hydrolysis, and lactic acid fermentation. The use of [Emim][OAc] enabled efficient disruption of the cellulose structure, achieving 95 % cellulose digestibility. Meanwhile, approximately 96 % of the original PET could be recovered after saccharification, exhibiting high FT-IR similarity to commercial PET. Fermentation of the hydrolysates resulted in lactic acid yields of up to 0.97 g/g, corresponding to an overall yield of 471.5 g of lactic acid per kg of processed polycotton. This lactic acid can be used to produce the bioplastic PLA, which would close the loop in accordance with the European Union's fibre-to-fibre policy. This study achieves high cellulose conversion and lactic acid yields under significantly milder conditions (lower temperature and shorter reaction time) for processing textile waste, which, together with the high recyclability of [Emim][OAc], could further enhance the circularity of the process compared to traditional chemical approaches. The technical feasibility of this three-stage process has been confirmed; however, a comprehensive techno-economic and life-cycle assessment would be necessary to evaluate its industrial viability.

CRediT authorship contribution statement

Alvarez-Hornos Javier: Writing – review & editing, Supervision, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization. **P. Ferrero:** Project administration, Funding acquisition. **H. Poy:** Writing – original draft, Visualization, Methodology, Investigation, Formal analysis. **E. Lladosa:** Writing – review & editing, Supervision, Formal analysis, Conceptualization. **M. Capilla:** Methodology, Investigation, Conceptualization. **P. San-Valero:** Writing – review & editing, Visualization, Methodology, Supervision. **B. Taroncher:** Resources, Investigation. **C. Silvestre:** Investigation. **S. Loras:** Methodology.

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.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jece.2025.119892.

Data availability

The authors are unable or have chosen not to specify which data has been used.

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