

Closing the loop: Analysis of biotechnological processes for sustainable valorisation of textile waste from the fast fashion industry

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# Closing the loop: Analysis of biotechnological processes for sustainable valorisation of textile waste from the fast fashion industry

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## ABSTRACT

The textile industry currently stands as one of the most polluting sectors globally. The proliferation of fast fashion has led to an unprecedented increase in textile production and waste generation, marked by mixed material compositions and significant reduction in the lifespan of each garment. These factors contribute to the creation of complex mixed waste streams, with a majority ending up in landfills. In agreement with international sustainability directives, the textile sector has emerged as a prime candidate for harnessing valuable raw materials from waste.

This review specifically targets the transformation of the prevailing linear production model into a more circular one. It focuses on utilizing biotechnological processes to convert textile waste into secondary raw materials to produce platform chemicals and added-value products, able to replace petrochemical-derived materials. The review begins with an extensive analysis of the state-of-the-art and the determination of technically feasible, economically viable, and environmentally sustainable waste valorisation techniques.

The focus is on the pre-treatment phases of hydrolysis and fermentation of textile waste to produce industrially promising building blocks. Cotton and cotton-polyester blends, the two most common waste materials in fast fashion, were selected as the primary research materials. Significant variables affecting the efficiency of pre-treatment and hydrolysis methods are identified, highlighting the importance of pre-treatment and the potential use of enzymes for textile hydrolysis.

Following the selected studies, the review defines the environmental and economic interests of the projects. These assessments provide essential insights into the sustainability and financial feasibility of the proposed waste valorisation methods.

## 1. Introduction

In a world where the speed of change holds paramount importance, the fast fashion market has responded to the consumers' demand for large quantities of low-cost clothing in very short periods. However, this has overshadowed the escalating environmental costs associated with waste management in the industry.

The textile sector follows a linear production model, and its adverse environmental impact has been accentuated by the reduced time between textile fibre production and garment disposal. Furthermore, clothing is predominantly composed of a mix of synthetic

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and natural fibres. Such mixed-waste textiles are not only pollution-intensive but are also challenging to dispose of adequately. These factors justify the industry's high pollution impact, positioning fashion manufacturing as the second most environmentally harmful sector for agricultural land, following only the oil industry (Gupta et al., 2022).

The escalation of textile waste is not solely influenced by the nature of materials but also by the staggering volume of generated waste. Over the past two decades, there has been a substantial increase in the average annual textile consumption per person, doubling from 7 to 13 kg. In 2020 global textile consumption reached an astonishing 100 million tons (Shirvanimoghaddam et al., 2020).

From a circular economy perspective, the prospect of reutilizing waste from this sector to manufacture new products becomes particularly engaging. Such an approach holds the potential to diminish the environmental impact of the textile industry and other sectors that can leverage these valorised products as starting points for production. Among these sectors are those providing widely consumed goods, such as the ethanol and biogas industry (Jeihanipour et al., 2010), lactic acid production, bioplastics like polylactic acid (PLA) and polyhydroxyalkanoate (PHA) (Ribul et al., 2021), and the manufacturing of reagents like succinic acid (Mihalyi et al., 2023). This shift towards repurposing textile waste not only addresses environmental concerns but also contributes to a more sustainable and resource-efficient industrial landscape.

Moreover, it is estimated that world textile consumption for clothing and footwear will increase by 63% by 2030 (Manshoven et al., 2019).

A significant portion of fast fashion purchases contributes to the exploitation of extensive water resources, raw materials, and energy, resulting in the production of low-quality goods (Niinimäki et al., 2020, Copyright © Ellen MacArthur Foundation, 2017). Approximately 75% of global textile waste ends up in landfills or is incinerated, leading to the generation of toxic fumes and solid pollution, including substantial amounts of microplastics harmful to ecosystems (Juanga-Labayen et al. 2022). Merely 1% of these textiles are recycled into new clothing, as the technologies required to recycle clothes into virgin fibres are still in their nascent stages (Šajn 2019).

From a circular economy perspective, the idea of reusing waste from this sector to create new products of interest is particularly compelling. The reason behind the extensive waste generated in this sector cannot be only attributed to population growth. Gupta et al. have demonstrated that the majority of post-purchase textile waste is discarded due to size incompatibility with the buyer (Gupta et al., 2022). This finding is corroborated by the National Institute of Standards and Technology (NIST), which reports a significantly lower growth rate in the number of garments produced between 2000 and 2020 compared to the production of textile fibres (Schumacher and Foster, 2022). During this period, the global population increased from 6.144 to 7.821 billion, while the number of fibres produced surged from approximately 60 million to 100 million tons (Schumacher and Foster, 2022).

Enhancing the circularity of this process would substantially reduce its environmental impact by implementing the three fundamental principles of global policies for transitioning to a circular economy: production reduction, waste reuse, and recycling (Shirvanimoghaddam et al., 2020).

This approach could mitigate the environmental footprint of the textile sector and serve as a starting point for other productive industries.

Another critical parameter for reusing textile products is related to the nature of their fibres. The proportion of synthetic fibres within clothing items is on the rise, contrasting with cotton, which has remained relatively constant over time (Niinimäki et al., 2020). The utilization of mixed fibres proves to be more complex due to the necessity for a greater number of treatments to separate the plastic fraction from the organic one. For this reason, there is a portion of textile waste that cannot be directed towards the formation of new regenerated yarns. This material fraction serves as a starting point for an alternative recycling process.

Both synthetic and natural fibres are polymer-based, and their monomers hold significant industrial value as building blocks for various production processes. Reusing this waste is not only environmentally advantageous for textile production but also economically beneficial, particularly for reducing the costs of raw materials in other industries, often reliant on petrochemical sources (Subramanian et al., 2022).

Depending on the nature of the raw material, it is possible to recover the building blocks from which it is composed, such as terephthalic acid (TPA) and glucose, and subsequently manufacture industrially valuable goods from textile waste, including ethanol, biogas, lactic acid, bioplastics like polyethylene terephthalate (PET) and Polyhydroxyalkanoate (PHA), and succinic acid (Jeihanipour et al., 2010; Ribul et al., 2021; Mihalyi et al., 2023).

To obtain these products, textile waste can be valorised through the utilization of many techniques, including glycolysis, hydrothermal treatment, ammonolysis, hydrolysis, and pyrolysis (Damayanti et al., 2021).

These are all chemical and thermochemical depolymerization techniques that allow to produce energy and products with lower molecular weight, including sugars, char, aldehydes, ketones, and plastic monomers.

Many publications focus on viable techniques for reusing textile waste. Among these, enzymatic hydrolysis emerges as one of the most promising strategies with a high Technology Readiness Level (TRL), ranging between 7 and 8, associated with pre-commercial to commercial-scale implementation (Biddy et al. 2016).

However, despite significant variations among these methods, three common stages emerge within the valorisation process: a pre-treatment phase, a hydrolysis phase, and a fermentation phase. Among these, the initial two stages are the most critical in terms of yield, costs, and environmental considerations, as noted by Vera et al., in 2023. (Vera et al., 2023). While many techniques have been developed for repurposing textile scraps, especially in recent years, there remains a noticeable lack of uniformity in presenting the experimental results. This lack of consistency makes it challenging to compare and evaluate the most promising methods. Moreover, the majority of these studies were conducted at a laboratory scale.

Therefore, this review seeks to address these challenges by systematically gathering and reorganizing the most significant information in the field, facilitating an assessment of the economic and environmental aspects of these processes concerning their perfor-

mance. This systematic review is intended to provide clarity and enhance the comparability of results for further advancements in the repurposing of textile waste.

**2. Methodology: literature studies inclusion criteria and material organization**

The review is based on a systematic analysis of the data reported in the literature. Key terms were selected to conduct the bibliographic research. These terms encapsulate the main topics addressed in the study and the phases of the process under focus. The research was primarily conducted within the libraries of Web of Science, Google Scholar, and Scopus. A literature review was conducted by searching scientific papers containing the following keywords: “Textile waste valorisation”, “enzymatic hydrolysis”, “fast fashion valorisation”, “saccharification”, and “fermentation”.

The reference publications include those published in the period 2013–2023. Publications preceding 2013 were cited only if deemed relevant by articles within the specified time range. For each article, its contribution to the research topic was identified and integrated into the review organization, following the framework illustrated in Fig. 1.

**3. Temporal trends in public interest and engagement with textile waste**

From the analysis of publications, it was possible to identify a growing interest in recent years (Fig. 2).

A general initial finding is depicted in the incremental trends graph of the number of publications related to textile waste, as found through the analysis of three different research databases: Web of Science (blue), Google Scholar (yellow), and Scopus (green). In general, the number of publications related to “textile waste” has tripled in the last decade.

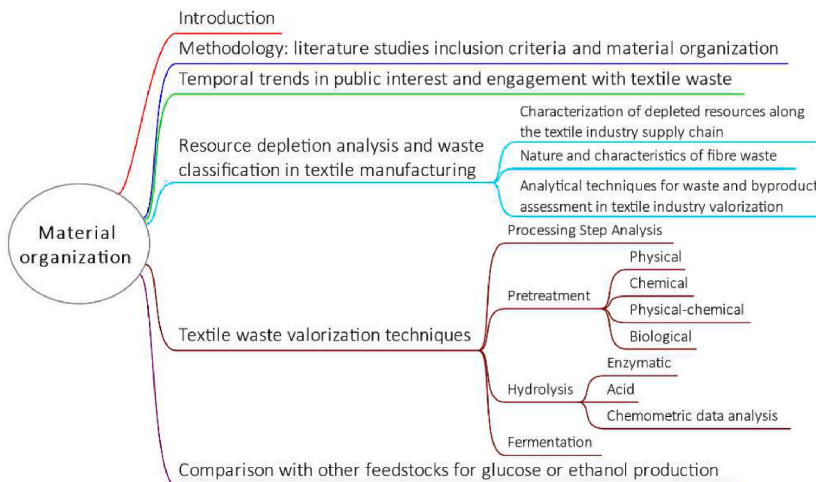


Fig. 1. Research flowchart describes the sequence regarding the selection of the articles through the PRISMA method for systematic review and meta-analysis.

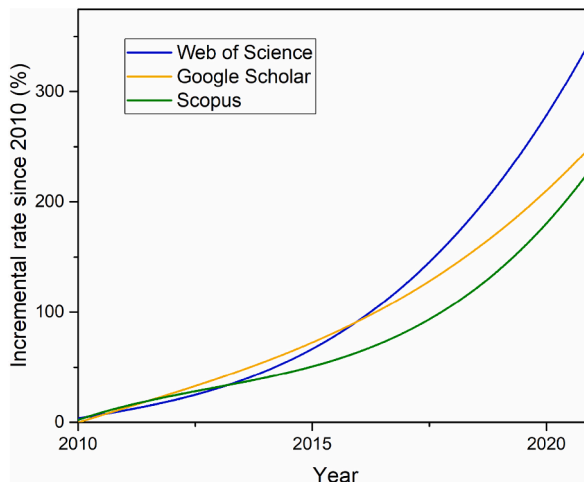


Fig. 2. Publication increasing on the “textile waste” topic: incremental ratio of publication number found by the analysis of three different research databases: Web of Science (blue), Google Scholar (yellow), and Scopus (green). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

The interest in this topic was also assessed with consideration to geographical regions, with the aim of identifying the countries more actively engaged in research and exploration of new methods for waste valorisation.

The publications data were collected from Scopus, selecting the period January 2013–October 2023. The collected results were subsequently transformed into graphical representations using the open-source platform Flourish (<https://flourish.studio/>). As observed, the preeminent numbers of publications are ascribed to China and India, which have been exhibiting publication counts in the order of thousands over the past decade. Following closely are the United States, Turkey, and Brazil, each with publication counts of approximately half a thousand.

The international policies of the last years have focused on regulating the textile sector to enhance its sustainability. Examples of these policies include China's proposed plans under "Made in China (2025)", European initiatives such as the "Circular Economy Action Plan" (Bennis et al. 2022; Calisto Friant, Vermeulen, and Salomone, 2021; Chen et al., 2021), and the establishment of concepts like Extended Producer Responsibility (EPR) by the Organization for Economic Cooperation and Development (OECD). The European Commission has suggested a specific framework for the textile sector to be applied to member states (European Commission 2023; OECD 2001). All these measures increase the interest of the Western world and, consequently, the importing entities in safeguarding the textile market. Therefore, one would expect a noticeable shift in publication percentages towards an increase in publications from importing countries.

Despite the economic policies for environmental support implemented in recent years for more sustainable production, the majority of scientific publications still originate from regions involved in textile goods manufacturing (Fig. 3).

In 2013, the top 10 states with the highest number of publications, listed from most active to least, were India, China, the United States, Turkey, Brazil, Pakistan, Indonesia, and Malaysia. In 2023, the most active states remain the same, with India, China, Indonesia, Australia, Saudi Arabia, and Egypt emerging as the states exhibiting the highest growth rate in the number of publications.

The highlighted mismatch suggests that the discussion on this subject has not received the substantial attention it deserves in nations with the potential to catalyse transformative changes in the textile production sector.

#### 4. Resource depletion analysis and waste classification in textile manufacturing

##### 4.1. Characterization of depleted resources along the textile industry supply chain

The textile sector entails the consumption of different resources for its production, and the environmental and economic impact of the supply chain depends on their utilization. These resources can be categorized as follows: water resources used, energy resources, the quantity of water utilized, quantity of water contaminated by the supply chain (e.g., the use of solvents or mordants), solid waste and micro components, and toxic chemical reagents (Shirvanimoghaddam et al., 2020).

To make the supply chain more sustainable upstream, efforts are made to manage the primary waste streams. Water is purified to remove pesticides and dyes, ensuring that it does not pollute the environment. Solid waste is initially considered for reuse. The simplest form of reuse occurs when the garment is made entirely of cotton. Cotton has a high valorisation capacity, primarily composed of cellulose, suitable for biofuel, biochemical, and bioenergy production using microorganisms as cell factories (Li et al., 2019).

The nature of the raw materials used and their assembly is, therefore, a key stage to focus on to reduce the quantity of non-recyclable waste (Ranjithkumar et al., 2017). The waste generated by the textile sector can be categorized into pre-consumer, post-consumer, and industrial textile waste (Juanga-Labayen et al., 2022; Mishra et al., 2022).

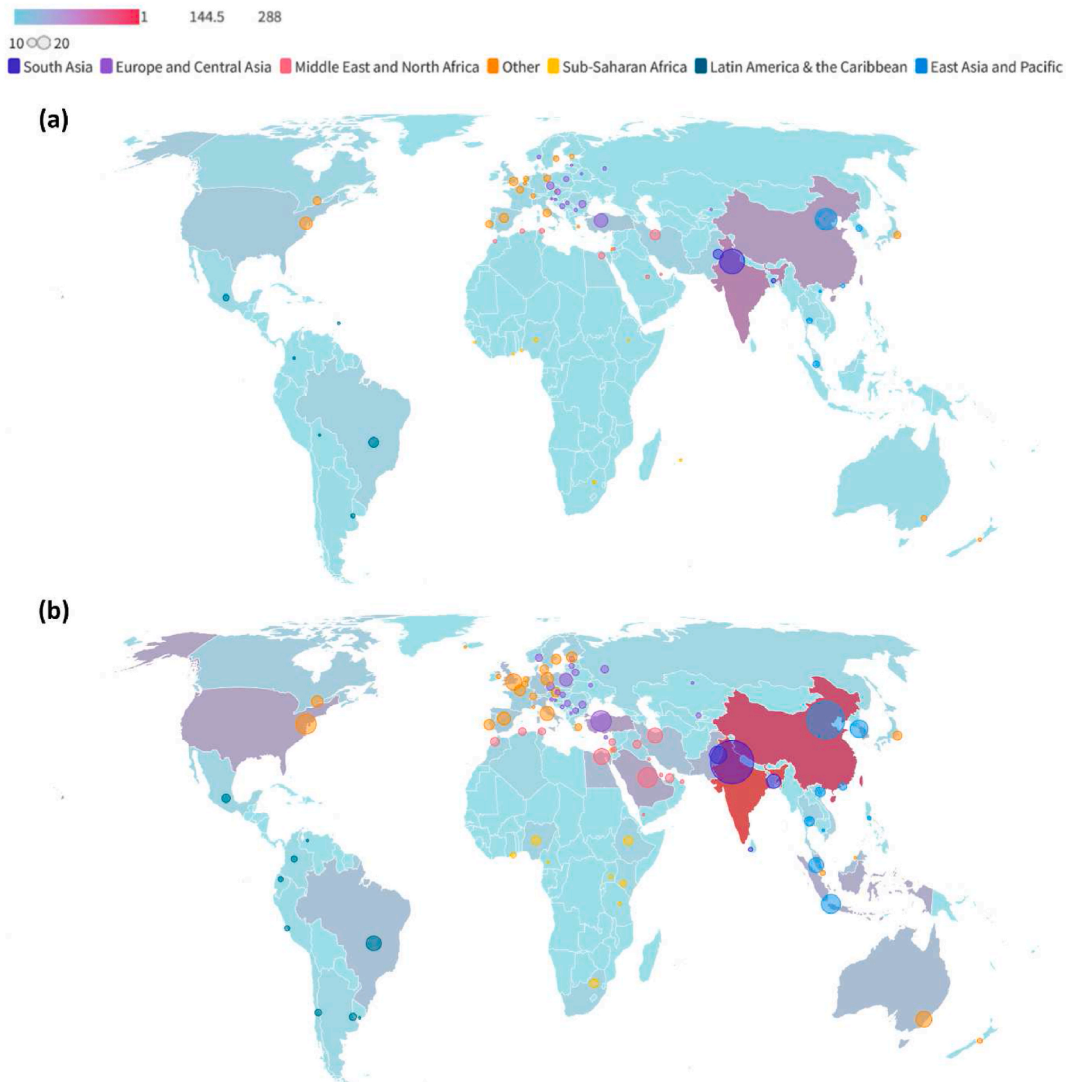
Within manufacturing industries, solid waste materials are already identified and stored based on their nature through a chain process outlined by Damayanti et al. The process involves the use of specific sensors capable of characterizing the nature of the waste, such as buttons, zippers, and decorations, and directing it towards appropriate disposal methods. This constitutes a step towards reducing waste disposed of in landfills by companies operating in the sector (Damayanti et al., 2021).

The composition of waste in the textile supply chain exhibits significant variability. Therefore, understanding the origin of waste in the yarn's life cycle is crucial for defining its composition in terms of cellulose, hemicellulose, lignin, or synthetic fibres (percentage value showed in Fig. 4). For instance, waste generated during the initial stages of cotton processing contains higher percentages of lignocellulose and hemicellulose, ranging between 20% and 30%. In contrast, cotton present in yarns is predominantly composed of cellulose, with percentages ranging from 88% to 99%. (Ranjithkumar et al., 2017).

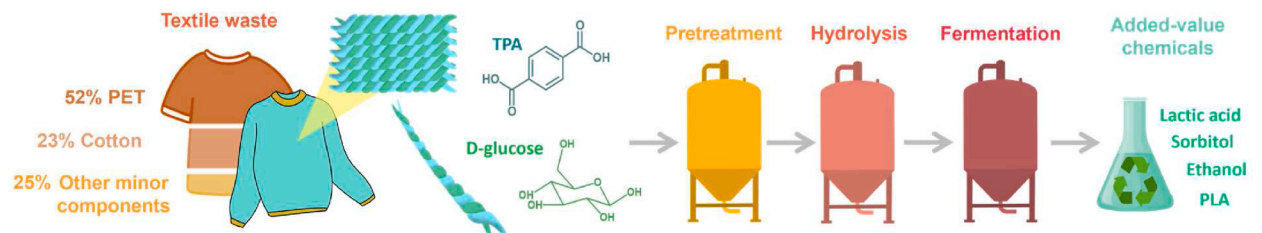
Among the types of waste, post-production waste also represents the highest quantity and is the most challenging to dispose of due to its mixed nature and management, which entirely falls into the hands of the consumer. Fast fashion has incredibly increased the number of toxic mixed waste. This is attributed to the rise in the percentage of plastic and nylon in both intact and damaged garments, which are often discarded and predominantly destined for incineration.

When confronted with blended textiles, a common scenario in most cases, the need arises to employ additional methodologies such as fabric crushing, carding, or solvent-based techniques. In the former, both fractions are co-mingled and processed to yield materials suitable for various applications, whereas in the latter, a separation of the two fractions occurs. The resulting natural fibres are then incorporated into virgin fibres for the manufacturing of novel garments, while the liquid fraction finds application in the production of new polymers.

Effectively recycling mixed fibres often presents considerable complexity. The treatment process frequently entails numerous steps with a substantial environmental footprint and associated high costs. Consequently, it becomes imperative to explore alternative modes of recycling for the production of novel commodities, particularly when the conventional spinning route proves economically and environmentally prohibitive (Mishra et al., 2022; Subramanian et al., 2022).



**Fig. 3.** World map obtained by performing a number of articles searched in the Scopus database with the keyword “textile waste” from every state for the year 2013 (A) and from January to October of 2023 (B). The shaded states in color gradients, from light blue to red, represent those that have been published on textile waste in the last ten years. Darker and red shades are associated with states that have a higher number of publications. In the same manner, the size of the points is a function of the publication number, while their color depends on the geographic region in which they are located. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)



**Fig. 4.** Schematic representation of the composition of textile waste and key steps involved in their valorisation.

#### 4.2. Nature and characteristics of fibre waste

The nature of fibres can be identified by ISO 1833–1, which employs a dissolution technique to discern the composition of the material (Damayanti et al., 2021).

Among the most prevalent mixed yarns, those composed of cotton and polyester stand out. The precise composition may vary, but it commonly comprises 35–40% cotton blended with a synthetic fibre, typically polyester (Hu et al., 2018; Juanga-Labayen et al., 2022; Srichola et al., 2023).

Cotton is the natural fibre containing the highest concentration of cellulose, approximately 98%. Cellulose is composed of glucose monomers linked together through  $\beta$ -(1,4)-glycosidic bonds. This characteristic is particularly interesting as it results in a material's higher susceptibility to hydrolysis compared to other natural fibres, which typically entail elevated concentrations of lignocellulose, a more complex biopolymer that is harder to degrade (Vera et al., 2022).

Lignocellulose has a three-dimensional polymeric structure that renders it less accessible to enzymes. Consequently, residues from the cotton processing at initial stages are more challenging to degrade due to their higher content of this polymer. Simultaneously, cellulose exhibits both amorphous and crystalline regions, with the latter being present in substantial amounts, making the polymer resistant to attack by hydrolytic agents, albeit to a lesser extent than lignocellulose (Sasaki et al., 2020; Yang et al., 2014).

The crystallinity of cellulose decreases with exposure to mechanical treatments of fibres. The first among these treatments is induced by their utilization, which is why post-production and post-use textile waste emerge as promising substrates for material recycling.

Glucose is regarded as a valuable product in its own right, serving as the raw material for various productions of economic interest. It is a key component in the production of bioethanol, lactic acid (LA) (a constituent of bioplastics), sorbitol, ethylene glycol (EG), and terephthalic acid (TPA) (Jin et al., 2023).

The synthetic component of the fabric often comprises the semi-aromatic polyester known as Polyethylene terephthalate (PET). PET is synthesized through a condensation reaction involving Terephthalic Acid (TPA) and ethylene glycol (EG), both derived from the petroleum supply chain. This polyester is particularly noteworthy due to its excellent physical, thermal, optical, and mechanical properties, coupled with its ready availability (Li et al., 2022). Despite PET susceptibility to degradation through various chemical processes, such as glycolysis and methanolysis, only hydrolysis proves effective in breaking down PET into its constituent monomers, TPA and EG, which can be subsequently utilized in the production of new PET polymers (Boondaeng et al., 2023).

As hinted at in the previous chapter, two additional factors significantly influence reaction yields and must be considered in the assessment of treatment efficiency. These factors are the presence of colorants and the level of fibre crystallinity. Both these factors negatively impact reaction yields because they make the fibre less susceptible to attack by external agents (Gupta and Prakash 2015; Vera et al., 2022).

#### 4.3. Analytical techniques for waste and byproduct assessment in textile industry valorisation

Monitoring the progress of degradation and transformation reactions of textile waste is of fundamental importance. The nature of the material changes both in terms of chemical and physical states. For this reason, various quantitative and qualitative methods are applied in order to characterize the material throughout the valorisation process. These methods enable the determination of the yarns' condition, facilitating the identification of treatments that yield higher results, as listed in the table below (Table 1) (Damayanti et al., 2021).

**Table 1**  
Characterization techniques for analytes.

Technique Name	Type	Physical state of the analyte	Technique and interesting output
<b>TGA</b> thermogravimetric analysis	qualitative	Solid state	Analyse the thermal properties of textile waste. Identification of thermal stability and degradation degree of the fibres (Yousef et al., 2020)
<b>NIR</b> Near infrared spectroscopy	Qualitative	Solid state	Spectrometer techniques, able to describe the sample surface composition and percentage of materials (Wojnowska-Bacaryła et al. 2022).
<b>ATR-FTIR</b> Attenuated Total Reflection–Fourier Transform Infrared	Qualitative quantitative	Solid state	Spectrometer techniques to determine the chemical structures and the functional group based on the recognition of textiles matrix based on the type of polymer materials in that textile with an accuracy of up to 100%. Mathematical modeling was applied to predict the data more accurately from the IR (Binczarski et al., 2022; Damayanti et al., 2021).
<b>SEM</b> Scanning electron microscope	qualitative	Solid Semi-solid	Microscopy techniques to analyse the matrix morphology, to see a significant changes in of the fibres (Binczarski et al., 2022; Nikolić et al., 2017)
<b>Raman spectroscopy</b>	quantitative	liquid	To examine the percentage concentrations of the analyzed compounds. Often employed in conjunction with FTIR spectroscopy (Binczarski et al., 2022).
<b>Gas chromatography</b>	Qualitative quantitative	Gas state	to identify and quantify the gas generated through the chemical reactions (Kuo et al., 2014).
<b>HPLC</b> high performance liquid chromatography	qualitative	Liquid state	Samples analysis from various steps of enzymatic hydrolysis and fermentation. Glucose and ethanol were analyzed on a hydrogen-base ion exchange column (Jeihanipour et al., 2010).
<b>X-ray spectroscopy</b>	qualitative	Solid and liquid state	Spectroscopic techniques to recognise and study the compound shape (Li et al., 2022).

Beyond the characterization techniques of the matrix, it is crucial to quantify the enzymes ability to degrade the substrate after extraction. For this purpose, the following assays are primarily employed: the Ximenes method is useful to measure the enzyme activity for  $\beta$ -glucosidase. This technique assesses the reactivity of  $\beta$ -glucosidase, calculated as the ability to react with *p*-nitrophenyl- $\beta$ -D-glucopyranoside to generate *p*-nitrophenol (Ximenes et al. 1996). Another equally important assay is the cellulase activity test, among which one of the most recognized is the method developed by Adney and Baker. This method evaluates cellulase activity following the International Union of Pure and Applied Chemistry (IUPAC) guidelines. The procedure has been formulated to quantify cellulase activity in terms of “filter-paper units” (FPU) per milliliter of the original (undiluted) enzyme solution (Adney and Baker 1996).

## 5. Textile waste valorisation techniques

The re-use process invariably involves a state of breakdown and the formation of new bonds. This is precisely what occurs in the preferred technique for reusing fibres in second-generation yarns. In this case, the degradation of waste material is induced by the mechanical and chemical treatment of fibres, which are subsequently blended with virgin yarns to produce regenerated fibres.

However, this process is not infinite: regenerated fabrics contain increasingly shorter fibres that, at a certain point, become impractical for weaving (Sun et al., 2021).

Hence, it becomes useful to explore other techniques for the reuse of textile waste. The most well-known techniques include hydrocracking, pyrolysis, and gasification, which are typically conducted in the presence of catalysts at high temperatures, along with hydrolysis. (Damayanti et al., 2021).

Among these techniques, enzymatic hydrolysis stands out as one of the most promising pathways for waste valorisation due to the high specificity of the enzymes used and the environmentally friendly reaction conditions (Fig. 4) (Ribul et al., 2021; Quartinello et al., 2018).

### 5.1. Processing step analysis

The analyzed articles present three main phases necessary for carrying out the hydrolysis and reuse of textile waste: (1) a pretreatment, (2) a hydrolysis process, (3) a specific conversion process to the material of interest. Table 2 summarizes the common variables found in various studies, enabling a comparison between different methods. The conditions under which the treatments take place may appear highly diverse, but they often refer to these variables for yield evaluation: type of substrate, type of pretreatment and its operation conditions, type of Hydrolysis and its operation conditions.

### 5.2. Pretreatment

The pretreatment step is essential to increase hydrolysis yields (Damayanti et al., 2021) by modifying the chemical and physical conditions of the fabric (Vera et al., 2022). Literature findings indicate that the glucose production yield from hydrolysis is generally below 30% in studies without textile waste pretreatment (Boondaeng et al., 2023; Hasanzadeh et al. 2018; Jaihanipour et al., 2010; Vera et al., 2022). Despite appearing as a costly step, constituting around 30–32% of the costs for bioethanol production, it remains necessary (Klein-Marcuschamer et al. 2011).

Pretreating textiles is particularly useful for enhancing the accessibility of hydrolytic agents to the fibres of the treated yarn, allowing an increase in the contact surface and resulting in a smaller degree of crystallinity (Nikolić et al., 2017). This reaction step is essential to reduce the structural compactness of the fabric, remove surface impurities, and increase the hydrophilicity of the material. Cotton becomes substantially easier to attack, more hydrophilic, and cellulose more susceptible to attack. Consequently, the products of subsequent treatments, such as the glucose concentration resulting from hydrolysis or other raw products, also increase (Subramanian et al., 2022).

Reducing the matrix size is a crucial aspect of pretreatment, further increasing hydrolysis yields (Austad 2018). The advantages of performing these steps include increased enzyme digestibility, low production of degradation products, and low energy demand (Alvira et al., 2010; Binod et al., 2012; Giakoumakis et al., 2021; Hasanzadeh et al., 2018; Ranjithkumar et al., 2017; Yang and Wyman 2008).

The most effective type of pretreatment depends on the nature of the substrate. Therefore, defining the most promising technique is challenging, and it is more informative to observe common behaviours in the treatment of various types of textiles. Conversely, for all types of pretreatments, the process yield is influenced by the presence of colorants. Dye and mordant bonds with the fibre protect intrapolymeric covalent bonds, making their hydrolysis more challenging. Vera et al.'s review extensively explains these effects and classifies them to characterize the chemical nature of major colorants. However, it is not yet clear which pretreatment is more effective in minimizing the impact of color presence (Vera et al., 2022, 2023).

Pretreatments can be categorized as: (a) physical, (b) chemical, (c) physical-chemical, (d) biological, as synthesized in Table 3.

#### 5.2.1. Physical pretreatment

Hydrolysis, an age-old and cost-effective method, involves a physical pretreatment step in every process, albeit on a small scale. These methods focus on reducing the size and crystallinity of the matrix to increase surface area and enhance enzyme digestibility. Physical pretreatments hold promise by circumventing or minimizing the use of chemical pretreatments. However, they have inherent limitations, notably their inability to remove dyes, leading to an inhibitory effect on enzymes. Additionally, the intensive energy consumption associated with these methods raises environmental concerns and poses high costs for large-scale implementation (Gupta and Prakash 2015; Sasaki et al., 2020; Vera et al., 2022).

**Table 2**

Summary Table of Reaction Conditions and Yields. The considered substrate types made with different textile materials, including 100% cotton fibres (C), cotton/polyester composite fibres (CP), cotton stacks production residues (CS), textile fibres from jeans composed of cotton/polyester fibres (CPJ), cotton fibres only (CJ), cotton towel (CT), cotton denim (CD), and cotton gin waste production residues (CG). Different types of pretreatments were evaluated, including physical (F), base (B) with known composition, organic base (BO) or inorganic base (BI), acidic (A), organic acidic (AO), or inorganic acidic (AI), as well as treatments involving ionic liquids (IL). The comparison of experiments primarily focused on enzymatic hydrolysis (E), although some studies involving acid hydrolysis (A) were also included to facilitate a comprehensive analysis of operational conditions and achieved yields.

Reference	Substrate	Pretreatment	Pretreatment temperature (°C)	Pretreatment Time (h)	Hydrolysis	Hydrolysis temperature (°C)	Hydrolysis Time (h)	Yield (%)
Asaoka and Funazukuri (2011)	C	F	240	0,06	A	250	1	36,6
Binczarski et al. (2022)	C	F	60	0,5	AI	140	2	39,57
Boondaeng et al. (2023)	CP	BI BO	-20	6	E	50	96	48,72
	C	BI BO	-20	6	E	50	96	34
	CP	BI	121	15	E	50	96	66,75
	C	BI	121	15	E	50	96	56,67
	CP	F	121	15	E	50	96	9,79
	C	F	121	15	E	50	96	16,78
Dimos et al. (2019)	CS	F	175	2	E	50	6	23
	CS	F	175	2	E	50	6	27
	CS	AO	80	1	E	50	6	25
	CS	BI	121	0,5	E	50	6	25
Giakoumakis et al. (2021)	C	AI	200	0,5	E	50	48	95
Guo et al. (2016)	C	IL	110	1,25	E	50	0,17	80
Hasanzadeh et al. (2018)	CPJ	F	20	0	E	45	72	28
	CPJ	B	150	2	E	45	72	81,7
	C	F	20	0	E	45	72	36,9
	C	B	150	2	E	45	72	88,9
Hong et al. (2012)	C	IL	110	1,5	E	50	24	81,6
Hu et al. (2018)	CP	BI BO	-20	6	E	50	96	70,2
Jeihanipour and Taherzadeh (2009)	CJ	BI	0	96	E	45	96	99
Jeihanipour et al. (2010)	CJ	BI	0	96	E	45	24	85
Jeihanipour et al. (2010)	CP	F	120	2	E	30	48	16,4
	CP	F	120	2	E	30	24	11,2
	CP	F	120	2	E	30	12	6,8
	CP	F	120	2	E	30	6	5,3
	CPJ	F	120	2	E	30	48	88,6
	CPJ	F	120	2	E	30	24	49,8
	CPJ	F	120	2	E	30	12	22,6
	CPJ	F	120	2	E	30	6	8,8
	CP	BI	120	2	E	30	48	94,4
	CP	BI	120	2	E	30	24	93,7
	CP	BI	120	2	E	30	12	88,8
	CP	BI	120	2	E	30	6	78
	CPJ	BI	120	2	E	30	48	90,6
	CPJ	BI	120	2	E	30	24	90,9
	CPJ	BI	120	2	E	30	12	86,8
	CPJ	BI	120	2	E	30	6	74
Jin et al. (2023)	C	BI	25	2	E	45	120	99,1
	CT	BI	25	2	E	45	120	98,45
	CD	BI	25	2	E	45	120	62,95
Kaabel et al. (2022)	CP	F	20	1	E	55	168	33
Kuo et al. (2014)	C	BI	50	48	E	37	48	81
Kuo, Lin, & Lee, n.d.	C	AO	50	1	E	50	72	95
Li et al. (2019)	CP	BI BO	-20	6	E	50	24	98
Mcintosh et al. (2014)	CG	AI	180	0,2	E	50	96	89
Nikolić et al. (2017)	CS	F	20	2	E	50	192	31,65
Quartinello et al. (2018)	CP	E	50	48	E	50	70	95
Sahu and Pramanik (2018)	CG	AO	150	0,75	E	50	40	68
	CG	AI	140	0,75	E	50	40	67
Sanchis-sebastiáRuuth et al., 2021	C	AI	80	3,5	A	30	1	91
Sasaki et al., 2020	CT	F	25	0	E	50	72	80
Shen et al. (2013)	CPJ	AI	50	7	E	50	96	79,2
Gholamzad et al. (2014)	CP	BI	-20	3	E	45	72	30

(continued on next page)

Table 2 (continued)

Reference	Substrate	Pretreatment	Pretreatment temperature (°C)	Pretreatment Time (h)	Hydrolysis	Hydrolysis temperature (°C)	Hydrolysis Time (h)	Yield (%)
	CP	BI	0	3	E	45	72	20
	CP	BI	23	3	E	45	72	20
	CP	BI	100	3	E	45	72	18
	CP	BI	20	3	E	45	72	12
	CP	BI	-20	72	E	45	72	91
	CP	BI	0	72	E	45	72	88
	CP	BI	23	72	E	45	72	86
	CP	BI	100	72	E	45	72	80
	CP	BI	20	72	E	45	72	45
	CP	BI BO	-20	3	E	45	72	32
	CP	BI BO	0	3	E	45	72	20
	CP	BI BO	23	3	E	45	72	20
	CP	BI BO	100	3	E	45	72	22
	CP	BI BO	20	3	E	45	72	10
	CP	BI BO	-20	72	E	45	72	90
	CP	BI BO	0	72	E	45	72	84
	CP	BI BO	23	72	E	45	72	70
	CP	BI BO	100	72	E	45	72	76
	CP	BI BO	20	72	E	45	72	46

Table 3

Pretreatment method description.

Methods	Types	Advantages	Disadvantages	References
<b>Physical</b>	Milling, grinding, irradiation, sonication maceration, hydrothermal treatment	Low chemical usage, high enzyme loading capacity	Low chemical usage, high enzyme loading capacity	(Giakoumakis et al., 2021; Sasaki et al., 2020)
<b>Chemical</b>	Organic acids (oxalic acid, malic acid, acetic acid) Inorganic acids (H <sub>2</sub> SO <sub>4</sub> , H <sub>3</sub> PO <sub>3</sub> ) Organic bases (NaOH) Inorganic bases (urea, [amim]Cl, NMMO)	It prevents the formation of species inhibiting fermentation  High yields, limited treatment times	High cost of chemicals, their recovery, high water consumption, high costs of usage and maintenance of the plant due to corrosive substances and high operative temperatures  High capital cost associated with long residence times, solvents costs and expensive solvents recovery, expensive catalysts, difficult by-products treatments	(Binczarski et al., 2022; Subramanian et al., 2022; Vera et al., 2023)  Vera et al. (2022)
	Ionic liquid	High yield	Energy cost, high enzyme dosage	(Hong et al., 2012; Vera et al., 2022)
<b>Physical-chemical</b>	Microwave/ionic liquid	Reduced treatment times, low water consumption compared to acid-alkaline treatments.	High energy use, moderately high chemical use, high enzyme load	Ranjithkumar et al. (2017)
	Steam explosion	Reduced treatment times	Low ability to increase enzymatic yield, high cost of acid catalysts, and high thermal energy cost	Vera et al. (2023)
	Liquid hot water	Low environmental impact	Low ability to increase enzymatic yield, and high thermal energy cost	(Dimos et al., 2019; Hong et al., 2012)
	Solvent-based	High yields	High cost of solvents, high capital investment associated with solvent recovery, and high energy cost associated with solvent recovery	Elsayed et al. (2020)
<b>Biological</b>	Fungal degradation	Moderate-low energy consumption, good enzyme loading capacity	High chemicals use	Ribul et al. (2021)

Due to these limitations, mechanical treatment alone is insufficient to achieve the desired products. However, it proves valuable in reducing the need for other chemicals, thereby maximizing the yields of associated degradation processes.

Microwave irradiation, widely used for bioethanol production, serves as an alternative to conventional heating methods. The radiation penetrates the sample, disintegrating the polymeric structure of the matrix through direct interaction with the applied electromagnetic field. The advantage lies in its high reproducibility and low energy consumption. However, a drawback is the relatively modest yields, approximately around 60% (Sanchis-sebastiáRuuth et al., 2021).

### 5.2.2. Chemical pretreatment

This is the most widely used method in the conversion process of textiles through enzymatic hydrolysis (Vera et al., 2022). This step aims to reduce the crystallinity of cellulose, thereby increasing the hydrolytic agent's ability to come in contact with the substrate. This leads to an increase in glucose yield and facilitates the removal of the synthetic component from mixed fabrics. After the dissolution of the fabric, it is subsequently regenerated (Gholamzad et al. 2014).

Chemical pretreatment allows for an increase in enzymatic hydrolysis yield and a reduction in the amount of enzyme required for hydrolysis (Shen et al., 2013). Variables that can influence this step include (i) the nature of the solvent, (ii) solvent concentration, (iii) process temperature, (iv) process time, and (v) agitation (Shen et al., 2013; Vera et al., 2022).

When the treated fibre consists of a blend of pet and cotton, the dissolution phase allows the separation of cellulose from the synthetic fraction. Only after a filtration step, cotton or polyester is regenerated by removing the solvent (Vera et al., 2022). This is the most expensive step in both economic and environmental terms. Solvents are used in large quantities, and their use necessitates multiple substrate washes before the hydrolytic phase begins.

**5.2.2.1. Alkaline pretreatment.** Alkaline pretreatment involves the use of sodium, potassium, calcium, and ammonium hydroxides, inducing swelling of the porous structure in the biomass, thereby increasing accessibility to enzyme action (Gholamzad et al., 2014; Jehanipour and Taherzadeh 2009; Vera et al., 2022). These bases enhance lignin solubility and reduce the crystallinity of the cellulose structure, making it more digestible (Subramanian et al., 2022). Due to these reasons, alkaline hydrolysis exhibits high reaction yields. Furthermore, it prevents the formation of inhibitor species of hydrolysis or fermentation (Nikolić et al., 2017). Consequently, recent studies conduct experiments using urea as well, aiming to identify a base with sufficiently high yields but with a lesser environmental impact (Damayanti et al., 2021).

Alkaline solvents yield higher reactions when used at elevated temperatures, around 120–150 °C, or at low to moderate temperatures ranging between 20 °C and –20 °C. As demonstrated in Tables 2 and it is observed that temperatures within these ranges are crucial for achieving high yields. Ambient temperatures or less extreme conditions, in fact, yield lower values in terms of obtained monomers. High hydrolysis yields are achieved at lower alkaline concentrations but with an elevated contact time with the matrix. The primary variation lies in the effect on the percentage of synthetic fibre content. Alkaline pretreatment with the addition of a phase-transfer catalyst has been reported as effective in depolymerizing PET at temperatures of 70–95 °C and alkalinity in the range of 5–15% NaOH. PET undergoes hydrolysis at an ester bond, forming disodium terephthalic salt and EG in the liquid phase. The process yields three products: cotton cellulose, TPA, and an aqueous phase containing EG (Boondaeng et al., 2023).

**5.2.2.2. Acid pretreatment.** Acids such as sulfuric acid and phosphate acid are employed for pretreatment. The acid concentration, temperature, pretreatment duration, and the ratio of acid concentration to substrate are closely interrelated. Consequently, literature data predominantly report studies either managing to decrease the hard reaction conditions while maintaining high acid concentrations, or lower percentages are associated with conditions such as higher temperatures (Shen et al., 2013).

Typically, a preference exists for working with low solvent concentrations ranging from 4% to above 0.5%, but at elevated temperatures between 160 and 250 °C for several minutes (up to an hour) (Sasaki et al., 2020). Dilute acid pretreatment offers advantages such as higher reaction rates and improved cellulose hydrolysis. However, the high temperatures and acidic nature result in elevated environmental impacts and the formation of degradation by-products during the neutralization step (Jung and Kim 2015). So far, more than 100 compounds can be generated from acidic pretreatments, including aldehydes and ketones, weak acids. These compounds are inhibitory of the next degradation step mediated by enzymes (Liu and Blaschek, 2010).

Acids hydrolyse hemicellulose, breaking polymeric bonds and leaving cellulose more suitable to hydrolysis, leading to the degradation of the microstructures of cellulose fibres. Shen et al. demonstrated pretreatment with phosphoric acid to recover polyester and fermentable sugars, achieving 100% polyester recovery and 79.2% sugar yield (Shen et al., 2013).

**5.2.2.3. Ionic liquid pretreatment.** Ionic Liquids (ILs) are defined as compounds entirely composed of ions with a melting point below 100 °C (Lei et al., 2017). These liquids, at moderate temperatures, cause the dissolution of cellulose without degrading it and without degrading the solvent. The solvent can be recovered through low-pressure thermal treatment. One advantage presented by this method is the ability to dissolve various types of materials, including cellulose, lignocellulose, and hemicellulose, under mild conditions (Subramanian et al., 2022). For example, pretreatment of cotton waste is carried out using an organic solvent such as N-methylmorpholine-N-oxide (NMMO) and 1-allyl-3-methyl-imidazolium chloride ([AMIM]Cl), followed by enzymatic hydrolysis. Ionic solvents prove to be more sustainable than basic treatments (Damayanti et al., 2021; Hong et al., 2012).

### 5.2.3. Physical-chemical pretreatment

They represent some of the most effective and widely employed treatments. Physical treatment induces alterations in the regularity of cellulose structures, thereby enhancing susceptibility to external agents. Conversely, chemical treatment demonstrates a broader capacity to impact the chemical bonds of cellulose polymers, PET, and mordants involved in dye binding (Subramanian et al., 2022).

Between them, steam explosion is a thermos-mechano-chemical process that involves heating with steam at elevated temperatures and pressures (between 160 and 270 °C, 20–50 bar) for several minutes (Silverstein et al., 2007). It induces the expansion of moisture through applied shear forces and the hydrolysis of glycosidic bonds within cellulose (Ranjithkumar et al., 2017). While causing minimal material loss, this pretreatment exhibits a limited ability to increase enzymatic yield, accompanied by a high cost of acid catalysts and elevated thermal energy expenses (Vera et al., 2022).

### 5.2.4. Biological pretreatment

Useful and already known for the treatment of lignocellulose, it consists of the microbial degradation of feedstock. They do not cause the formation of fermentation inhibitors, making them competitive methods compared to classical acid-base pretreatments. Additionally, they generate a low quantity of by-products and involve low energy consumption (Sharma et al., 2020). On the downside, these methods have longer processing times and higher costs (Ranjithkumar et al., 2017).

Brown rot, white rot, and soft rot fungi may be employed for the degradation of the lignocellulosic complex to liberate cellulose (Gupta and Prakash 2015).

### 5.3. Hydrolysis

This phase involves the degradation of cellulose to obtain its monomers. It is based on the use of a hydrolytic agent that comes into contact with the tissue, hydrolyzes the matrix, and is then removed from the product. The limiting step of these three phases varies depending on the hydrolytic agent. Crucial for a good reaction yield is the contact area between the hydrolytic agent and the matrix.

Cotton proves to be a promising substrate, thanks to its high percentage of cellulose and low amounts of lignin and hemicellulose, which make the attack on the glycosidic bonds of cellulose more challenging. When hydrolysis occurs on pure cotton, the result is D-glucose, while the hydrolysis of lignin and hemicellulose yields both C5 and C6 saccharide monomers (Vera et al., 2022).

The hydrolysis of polyester leads to the production of TPA and ethylene glycol (EG), its monomers (Gluth et al., 2022). When degradation occurs on a mixed matrix like cotton/PET, various hydrolytic processes are employed to modify the physical state of each component. The aim is to recover the monomeric product in a liquid state and thus facilitate its separation from the solid fraction, with a different nature, of the mixed fibre (Kaabel et al., 2022).

Hydrolytic agents can be acidic, basic, or with enzymatic nature. In each case, the hydrolysis yield is influenced by the concentration of the hydrolytic agent, the available surface area of the substrate and its nature, the physical properties of the hydrolytic agent, and the hydrolysis environment (i.e., pH, salt concentration, temperature) (Ghose and Bisaria 1979).

As seen in the previous chapters, high yields are influenced not only by the chemical composition of the substrates but also by their molecular conformation. Mixed fibres involve the interweaving of synthetic and natural fibres, which are spaced apart and more easily susceptible to enzymatic action. This theory is supported by the studies conducted by Eun Jin scientific group, which already demonstrated improved results after the impregnation of the fabric, causing a separation between the fibres (Jin et al., 2023). Polymer additives and chemicals used in the textile processes could act as fermentation inhibitors and impede the production of biofuels (Binczarski et al., 2022).

The industrial sector currently prefers the use of acidic and basic catalysts, mostly of inorganic type, for conducting these reactions. However, the current conditions, coupled with the increased knowledge of biocatalysts and their reduced production costs, are leading to a transition towards their use in many sectors. Microorganisms containing enzymes capable of producing desired products are already used for the transformation of agro-food waste. Therefore, this chapter compares the potential of the two catalysis methods, emphasizing their challenges and advantages (Ghose and Bisaria 1979).

The hydrolysis yield (Eq. (1)) is calculated as the grams of extracted glucose per gram of initial cellulose multiplied by 1.111. The factor 1.111 accounts for the dehydration of cellulose chains based on the number of monomers, namely glucose (Jeihanipour et al., 2010). This dehydration factor is applied to consider the water added to the cellulose chains (Gholamzad et al., 2014). The hydrolysis yield equation reported by (Boondaeng et al., 2023):

$$\text{Hydrolysis yield (\%)} = \frac{\text{Amount of glucose released (g)}}{\text{Amount of initial glucose in substrate (g)} \times 1.111} * 100\% \quad \text{Eq. 1}$$

To allow a consistent treatment of results, the yield considered in various studies is the production of glucose expressed as a percentage. In reality, many studies prefer alternative expressions of results, such as g/L or the percentage of sugars obtained from cellulose hydrolysis, not necessarily in monomeric form.

#### 5.3.1. Enzymatic hydrolysis

Enzymatic hydrolysis is the final degradation process employed to obtain building blocks such as TPA and glucose from textile fibres. The enzymes used, due to their high selectivity, can degrade only one of the two considered fractions, making the separation of phases easier in different production steps. For the hydrolysis of the natural fibre component, an enzymatic complex induces a morphological change in the textile fibre. Addressing such factors is crucial to increase the availability of cellulose chains for reaction with enzymes (Vera et al., 2022).

Cellulases are multi-enzyme mixtures that integrate three different enzymes to complete the specific conversion of cellulose into glucose: endoglucanases, exoglucanases, and beta-glucosidases. It finds applications in various industries, including food, paper and pulp, textiles, pharmaceuticals, alcoholic beverages, starch processing, biofuel, and production. Most commercial cellulases are derived from fungi, especially *Trichoderma* and *Aspergillus* species (Boondaeng et al., 2023).

In the first conversion step, endoglucanases cleave individual  $\beta$ -glycosidic bonds in the cellulose chain to yield oligosaccharides with polymerization degrees of 2–6. These oligosaccharides are then converted by exoglucanases into small oligosaccharides (cellobiose), and finally hydrolyzed by beta-glucosidases into glucose (Ghose and Bisaria 1979; Vera et al., 2022). In many cases, the hydrolysis yield does not vary significantly when conducted for 24 or 48 h (Jeihanipour et al., 2010). Similarly, enzymatic hydrolysis can occur on the synthetic fraction of the fabric. Enzymes such as PETase and cutinase from *Humicola insolens* (Hic) can depolymerize the crystalline structure of PET to obtain TPA (Kaabel et al., 2022). Although PET can be degraded by different chemical processes, including glycolysis and methanolysis, only hydrolysis can yield TPA and EG, the monomers used to generate new PET polymers (Boondaeng et al., 2023). This reaction follows a first-order kinetics (Eq. (2)):

$$\frac{dC_a}{dt} = k(C_a - C_s) \quad \text{Eq. 2}$$

where  $C_a$  is the concentration of adsorbed enzyme,  $t$  is the reaction time,  $C_S$  is the saturation concentration of adsorbed enzyme, and  $k$  is the adsorption rate constant (Walker and Wilson 1991). To overcome the structural characteristics of cellulose (mainly the high crystallinity, amorphous regions, and refractory lignin-containing sites) engineered cellulases have been developed (Vera et al., 2022). A disadvantage of enzyme hydrolysis is its cost. The most commonly used commercial enzymes with higher yields are Celluclast (Novozymes®), and Cellic CTec2, also from the same company. The use of  $\beta$ -glucosidase is often combined with them, which is responsible for splitting cellobiose into two glucose units (Kahoush and Kadi 2022).

The current trend is to devise a production process that regenerates enzymes through the use of microorganisms capable of generating these enzymes. Alternatively, enzymatic synthesis can be carried out within the laboratory to reduce process costs. Another negative aspect of their use is the reaction time: compared to hydrolysis operated by strong bases or acids, enzymatic hydrolysis times are longer. A positive aspect of this saccharification is the mild conditions, resulting in lower energy and plant cost. This eliminates the need for machinery capable of withstanding high temperatures, pressures, or corrosive solutions.

Some studies are exploring the possibility of degradation using higher organisms such as worms. These organisms already possess metabolic pathways for plastic degradation, or specific genes are added to them through recombinant DNA technology to enable the degradation of the target matrix (Spínola-Amilibia et al., 2023).

### 5.3.2. Acid hydrolysis

The acid hydrolysis of cotton fibres resulted in the generation of glucose in the hydrolysate. This glucose could serve as a favorable medium for microorganisms and a potential source of bio-based products, such as bioethanol or biogas. Acid hydrolysis is one of the oldest methods employed, primarily conducted with sulfuric acid and phosphoric acid, the same acids found in some pretreatments. This type of hydrolysis exhibits very high yields, but the associated environmental costs are equally significant (Binczarski et al., 2022).

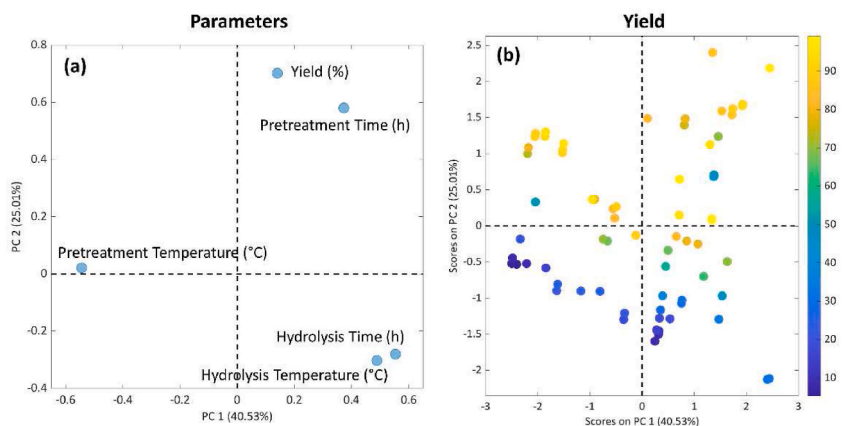
### 5.4. Chemometric data analysis based on the obtained results

To efficiently explore the numerical data gathered from the literature regarding the pretreatment, hydrolysis conditions, and obtained yields, a principal component analysis (PCA) was built (Fig. 5, Fig. 6). This mathematical model was obtained using the PLS-Toolbox (version 8.9, Eigenvector Research Inc. Manson, WA, USA) for multivariate data analysis, operated under MATLAB (2021a, Mathwork, Natick, MA, USA).

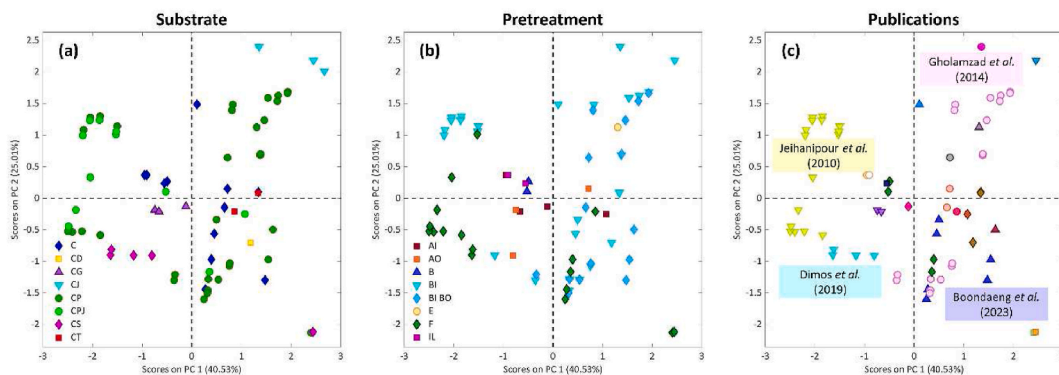
The PCA model describes the similarities and dissimilarities among the different experiments reported in literature, based on the correlation patterns among the descriptors the experiments. In the first plot (Fig. 5a) related to the distribution of variables (the “loadings” plot), the yield appears highly correlated with the pre-treatment time, while the hydrolysis time and temperature are found to be anticorrelated with the yield and correlated with each other.

The corresponding score plot (Fig. 5b) provides information regarding the yield of the considered experiments on a color scale from blue, indicating lower yields, to yellow, indicating higher yields. Each point on this plot represents an experiment reported in the reviewed literature, and the position of the points is determined by their behaviour based on the considered descriptors. From these plots, it is possible to conclude that higher yields were generally obtained for reaction conditions with long pretreatment times and very low pretreatment temperatures (these are indeed inversely correlated parameters). The markers in the scores plot represent the different evaluated experiments. Coloring the markers based on substrate type, pretreatment type, and article author provided additional useful information about the data.

From the point of view of the substrate type, the experiments conducted with gin waste and on cotton stalks as a substrate (Fig. 6a) appear to be similar, likely because they have similar compositions richer in lignocellulose and hemicellulose than post-consumer



**Fig. 5.** PCA Results for the different considered studies. The loadings plot (a) illustrates the relationships among the considered experimental parameters, while the corresponding scores plot (b) provides information regarding the similarities among the considered studies. The scores are colored according to the reported yields on a color scale from blue (lower yields) to yellow (higher yields). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)



**Fig. 6.** Sample coloring based on substrate characteristics. In the first scores plot (a), the markers are colored according to the composition of the samples used in the experiments: cotton (C), cotton as denim (CD), cotton gin waste (CG), cotton from jeans (CJ), cotton-pet blended (CP), cotton-pet blended from jeans (CPJ), cotton stalks (CS), cotton towel (CT). In the second scores plot (b), the markers are colored according to the type of pretreatment: inorganic acids (AI), organic acids (AO), bases (B), inorganic bases (BI), organic bases (BO), enzymes (E), physical treatment (F), treatment with ionic liquids (LI). The third scores plot (c) shows the literature source of the considered experiments. For clarity, only the four main sources were labelled on the plot.

products. These two textile production wastes have the following compositions: cotton gin waste is composed of approximately 30% cellulose, 10% hemicellulose, and 19% lignocellulose (Jordan et al., 2019), while cotton stalks are composed of 40% cellulose, 10% hemicellulose, and 30% lignin (Anon n.d.). Higher yields are obtained for treatments carried out on 100% cotton jeans (CJ) and samples composed of cotton and PET (CP).

Depending on the conditions in which pretreatment is carried out (Fig. 6b), higher yields are observed when pre-treatments use basic solvents. To improve process sustainability, efforts are made to replace or at least complement inorganic solvents (-I) with organic solvents (-O). However, pre-treatment with organic acids results in a decrease in hydrolysis yield, while treatment with organic bases along with inorganic bases shows promising results. In this case, the yield usually decreases compared to that obtained with pre-treatment with only inorganic bases but often remains higher than yields obtained with inorganic acids.

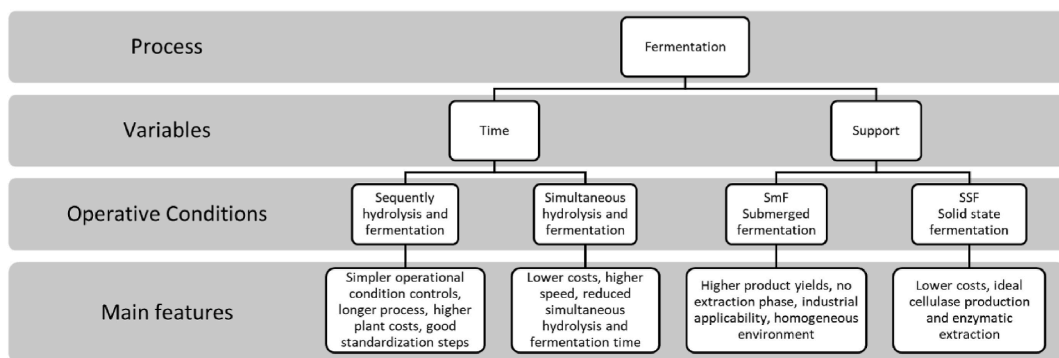
To verify the reliability of the data, it is important to find studies from different research groups that found similar results for the same reaction conditions (Fig. 6c). Despite the significant variability in the number of experiments for which the yield is indicated depending on the research group, similar treatment conditions occupy similar spaces in the scores plot, regardless of the research group that conducted the experiment. There is proximity between experiments from the same research group, but even in this case, their orientation follows the trend described for the variables.

### 5.5. Fermentation

Fermentation is the metabolic process that enables the synthesis of organic compounds of interest through the utilization of enzymes. These enzymes are derived from microorganisms such as bacteria, fungi, and yeasts (Ribul et al., 2021).

When discussing fermentation on textile waste, various types can be defined based on the stage of the process and the type of support on which the reactions take place (see Fig. 7). This metabolic step can occur either after the hydrolysis step or simultaneously. In the first case, fermentation and hydrolysis occur at distinct moments. This allows for better control of the reactions but involves higher overall plant costs. Therefore, simultaneous saccharification and fermentation, which offers numerous economic and environmental advantages, is often preferred (Kuo et al., 2014; Mihalyi et al., 2023).

Depending on the support for fermentation, it is possible to distinguish solid-state fermentation (SSF) and submerged fermentation (SmF). SSF occurs in the absence or near absence of free water. The utilized support contains all the essential components to mimic the ideal environment for the growth of fermenting microorganisms (Thomas et al. 2013).



**Fig. 7.** Synthesis of fermentation processes in comparison, with particular attention to the applicative advantages of different techniques.

Considering the two variables—simultaneous saccharification and fermentation or not—and the use of a support or not, different outcomes can be obtained. For instance, in the case of simultaneous saccharification and SSF, a fungus-like *Trichoderma reesei* may grow on the solid support. It produces the necessary enzymes for cellulose degradation in the cotton substrate. These enzymes hydrolyse the tissue, producing a solution of single or short-chain sugars, accompanied by the synthetic fraction in a solid state if the starting substrate was of mixed nature (Hu et al., 2018; Ribul et al., 2021).

However, SmF offers very interesting advantages from an industrial perspective: better control of pH, temperature, uniform gas concentration, and solution concentration. In this case, the growth medium of microorganisms is dissolved within the solution. With SmF, it is also possible to synthesize cellulases. In this initial production stage by the microorganism, the textile waste serves as a resource for the necessary organic carbon (C) synthesis. Glucose, in contact with specific microorganisms, can direct the reaction towards the production of bioethanol, succinic acid, lactic acid, and sorbitol. In these cases, the microorganism used exhibits a dual capability, namely, the synthesis of cellulase and the fermentation of glucose. Co-culture systems could replace this method, but they are rarely selected due to higher costs, more steps, and greater system management difficulties. Therefore, the preference is often for a single microorganism capable of performing both functions or one that can be modified through recombinant DNA techniques to acquire the missing competencies in the wild type (Ribul et al., 2021).

In the case of SSF, optimal results were observed after 24 h of fermentation, but higher yield outcomes were noticeable after 4 days. The pre-treatment applied to the textile waste can influence higher fermentation yields; hydrolytic treatments using inorganic acids (phosphoric acid, sulfuric acid) lead to the formation of species toxic to microorganisms involved in fermentation, reducing their yield (Jeihanipour and Taherzadeh 2009). McIntosh et al. describe yield rates increasing with the use of strong bases instead of inorganic acids (McIntosh et al., 2014). Another study has shown that the use of organic acids can also be a good solution to reduce the formation of inhibitory species in fermentation, maintaining a low environmental impact but with yields lower than those obtained with NaOH (Sahu and Pramanik 2018).

For this reason, upcoming studies are focused on implementing hydrolysis using organic acids or organic bases, which could provide the best conditions for achieving high yields with low environmental and economic impacts. Comparing various types of textile waste, promising results have been obtained by treating a mixed matrix of polyester/cotton 40/60. In this case, using NaOH at  $-20\text{ }^{\circ}\text{C}$  yielded results that were not achieved with matrices composed entirely of 100% cotton (Ranjithkumar et al., 2017).

#### 5.5.1. Formation of value-added products through fermentative processes

Having reached this stage in textile degradation, the reaction product shares similar characteristics with sugars derived from the valorisation processes of other waste. Consequently, the fermentation processes applied to these products exhibit favorable yields and demand identical reaction conditions to those required for freshly produced sugars. Among the most interesting products obtainable from textile waste are: ethanol, polylactic acid PLA, methane, sorbitol, and succinic acid (Jin et al., 2023).

Alcoholic fermentation for ethanol production is carried out by wild-type (wt) or mutagenic microorganisms. The investigated production processes appear to be significantly influenced by the pre-treatments undergone by the substrate. The research group led by Dimos et al. demonstrated that higher yields were obtained from processes that did not involve pre-treatments with strong acids and bases but utilized organosolvents and hydrothermal processes. Their results were compared with those of other groups operating in the same field (Dimos et al., 2019). The microorganism of choice for fermentation is the yeast *Saccharomyces cerevisiae*. However, other microorganisms are also used, such as *Escherichia coli*, *Clostridium thermocellum*, *Pichia kudriavzevii*, and *Aspergillus terreus* (Brethauer and Michael Hanspeter Studer, 2014; Dimos et al., 2019; Vera et al., 2022). These microorganisms are useful because they find textile waste to be a suitable growth medium.

Fermentation conducted in the presence of microorganisms like lactic acid bacteria, such as *Lactobacillus*, *Lactococcus*, *Actinobacillus succinogenes*, *Leuconostoc*, *Pediococcus*, and *Rhizopus*, can lead to the production of lactic acid, the main monomer of polylactic acid (PLA). PLA is one of the most widely used bioplastics today as an alternative to traditional plastics, especially in packaging (Jin et al., 2023). This polymer is completely biodegradable, being an aliphatic polyester, when placed in a biologically rich environment, such as a composting plant (Mihalyi et al., 2023). The traditional production pathway involves the processing of corn seeds through treatment with enzymes to break down complex sugars into simple sugars, followed by the addition of nutrients to initiate fermentation by the bacteria brought into contact (usually *Lactobacillus* types). The reaction product is three enantiomeric forms of lactic acid, predominantly L. After the purification of the fermentation product, the process involves esterification and polymerization of lactic acid to form a polymer using organic solvents (Botelho et al., 2004).

## 6. Comparison with other feedstocks for glucose or ethanol production

The chemical nature of the matrix shares many common features with wastes already used in valorisation processes, yielding high outputs and higher Technology Readiness Levels (TRL). Therefore, it is interesting to study these methodologies to identify high-yield techniques more effectively for valorising textile waste.

Among the mentioned wastes subjected to the three valorisation steps are wheat straw, wheat bran, pitch pine, pear pomace, rice straw, wheat straw, sweet sorghum, tomato waste, corn stalks, tree leaves, rice straw, willow, pine sawdust, miscanthus, switchgrass, wheat straw, rice straw, corn cob. For each of them, the composition has been identified, and based on it, the most suitable fermentation type and pre-treatment type were determined a decade ago (Sharma et al., 2020; Yousuf 2012).

The number of publications related to the valorisation of textile waste for the production of building blocks and high-value-added products is lower than achieved for other types of waste, such as lignocellulosic, agri-food, or plastic production residues. Many of the substrates used industrially for glucose production contain a lower and often less accessible amount in their matrix. Therefore, apply-

ing the studied techniques in these other sectors is particularly promising for improving cellulose hydrolysis and fermentation processes from textile waste (Jin et al., 2023).

Additionally, the large quantity of waste makes its use interesting for obtaining new products, not necessarily of the highest added value. Even partially reducing the incineration of waste would represent a decrease in the disposal problem.

Textile and other well-known waste substrates present difficulties in raw material reuse, such as the presence of mixed fibres, making reuse more complicated, or higher concentrations compared to lignocellulosic yarns and a protein portion, respectively (Gupta and Prakash 2015).

Pre-treatment is important in various productions, lowering reuse costs (Yang and Wyman 2008). Several studies have compared the ability of pre-treatments to improve hydrolysis efficiency with substrates other than textile waste. As seen in the articles by Yousuf et al. the yields are very high even though the substrate is less rich in cellulose than textile waste. Therefore, studying and attempting to apply pre-treatment techniques already used in other valorisation sectors is an important possibility to reduce research times and identify high-yield processes more quickly.

Based on the considerations just discussed, textile waste can be considered a promising waste material due to its high cellulose composition within cotton fibres, substantial quantity, and the potential for reuse through the application of treatments already verified for similar higher value substrates. For these reasons, textile waste is among the waste materials currently deemed valuable and worth saving from landfilling and incineration. These conventional waste disposal methods are widely employed for waste management, but their unsustainable nature raises significant concerns for environmental protection and resource conservation (Nanda and Franco, 2021).

## 7. Conclusions

In this review, a roadmap of the key research work published in the field of textile waste valorisation and comparable waste streams was explored and outlined. The collected results demonstrate how cotton fibres and cotton/PET fibres can be degraded, once they are no longer suitable for spinning, to serve as raw materials in a secondary production process.

The processing method of enzymatic hydrolysis and fermentation offers clear environmental advantages, such as reducing waste disposal in the sector, and less intuitive benefits, such as a significant reduction in the life cycle assessment (LCA) and life cycle cost (LCC) impact of the textile industry and industries that use the valorised waste as a raw material. It is important to keep in mind that there are still many challenges in achieving high yields and interpreting the results obtained from various studies.

To effectively contribute to the general research effort in this field, this review was written with the aim of standardizing and compiling the literature results, reworking them to express their yields uniformly. This allows us to identify the most promising processes and develop new ideas with a clear understanding of the state of the art.

In addition to this, we believe that the use of multivariate data analysis methods (such as PCA) can be of great help in comparing in a holistic and complete way the different experiments that can be found in literature, based on the actual experimental conditions and the related obtained yields.

The experiments conducted in this field to date allow for a general conclusion regarding the most promising choices for achieving sustainable processes with high yields. However, a key difficulty remains in the partial reporting of results. Generally, there is a tendency to place great emphasis on the tested reaction conditions, reporting only the yields of the most promising experiments. This trend is understandable as it seems logical to report only what has worked. However, presenting the results comprehensively from both the most promising and less promising experiments would represent a significant advantage in understanding the experimental domain and improving knowledge of the relationship between yield and applicable experimental conditions. This approach would enable the generalization of the actual relationship between variables.

As noted, enzymatic hydrolysis appears promising due to its high yields but less so from the perspective of the LCC of the process. Studies supporting their use have been significantly collecting, showing the production of enzymes on-site, reducing process costs. Additionally, the cost of industrially produced cellulases has decreased, enhancing the potential for making enzymatic hydrolysis a mild-condition process useful for transforming the linear model of fast fashion production into a circular system (Pellis et al., 2018; Yang et al., 2014).

The emphasis on pre-treatment in the literature underscores its critical role in the overall process. Companies are actively developing methods for waste recognition, separation, and eco-design to facilitate the detachment of different nature fractions and enhance reusability. This underscores the industry's commitment to addressing the challenges associated with textile waste and transitioning towards more sustainable and circular production systems.

In conclusion, this review operates as a guide for navigating the complex landscape of textile waste valorisation, shedding light on opportunities, challenges, and potential pathways toward a more sustainable future for the textile industry.

### CRediT authorship contribution statement

**Francesca Stella:** Writing – original draft, Data curation, Conceptualization. **Silvia Fraterrigo Garofalo:** Writing – review & editing, Supervision. **Nicola Cavallini:** Validation, Methodology, Data curation. **Debora Fino:** Supervision, Project administration. **Fabio Alessandro Deorsola:** Supervision, Resources, Funding acquisition.

## 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

No data was used for the research described in the article.

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