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Review

A review on evaluation of textile waste for ethanol extraction

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Abstract

Today, the growth in textile consumption is influenced by increasing of population. This leads to the generation of a large volume of textile waste, which has a negative impact on the environment. Textile waste is mainly disposed of by landfill or incineration. To reduce the amount of textile waste disposed of, it can be recovered and transformed into valuable compounds, such as ethanol (bioethanol). This article describes ways in which pre- and post- consumer textile waste is used as a feedstock for ethanol.

Keywords: ethanol, pre-treatment, recycling, textile waste, valorisation

INTRODUCTION

Nowadays, according to official report of current legislation from the European Union, in each year approximately 26 Kg/person of clothing and 11 Kg/person of textile waste are disposed. Is known also, that approximately 87% of textile waste is incinerated or landfilled [1, 2]. Taking into consideration this approach, cotton is the most widely used natural textile fibre and polyester is the most widely used synthetic fibre [2, 3]. In addition, textile fibre production is expected to reach around 130 million tons (MT) by 2025 [3] and 145 MT by 2030 [1]. The textile industry is one of the biggest polluters [2÷4], being the second largest polluter on the planet, contributing 10% of total CO₂ emissions and 20% of waste water generated [2]. According to official reports, the textile industry also ranks an unwelcome 4th in the list of polluting industries and climate change, and 3rd in water consumption and land use [2].

In compliance with European Union legislation [5, 6], separate collection of textile waste will be mandatory from 1 January 2025. This will facilitate a faster transition to a circular economy. In order to implement the context of the circular economy, there is also a need for closer cooperation between the manufacturing and recycling companies involved [2]. New strategies for recycling and reusing textile waste are needed, which must be addressed by collecting it for reuse, converting it into virgin fibres for new textiles and producing ethanol from it [7, 8].

The following steps should be taken into account in future strategies for the reuse and recovery of textile waste [7]: collection of textile waste; converting them into virgin yarns and fibres for new textile materials; processing them into ethanol.

For manufacturing process, the following types of textile fibres are used in the textile industry [2, 9]: Naturals, such as vegetables and animals; regenerated (based on natural polymers, but subject to further treatment and processing); synthetics.

Studying the literature data, it was found that the most clothes are made from blends of cotton (a natural polymer) and polyester (a synthetic polymer) [2, 4].

In addition, studding the literature review, textile waste can be classified into the following categories:

pre-consumer (textile waste resulting during the technological processes of textile manufacturing); post-consumer (textile products at the end of their life cycle, with different degrees of wear and tear) [3, 4, 9 \div 24]; industrial (textile waste from technical textiles, carpets, rugs, etc.) [9, 25 \div 28].

Taking into consideration on the Ellen MacArthur Foundation the following methods can be applied for the textile waste recycling:

- a) Mechanical involves the processing of textile waste into new products by cutting, shredding and defibring. This recycling can be applied to all types of fibres, regardless of their nature: vegetable, animal or those obtained by chemical processes (from oil). Fibres obtained by these methods are inferior in quality to those from which they are obtained.
- b) Chemical involves depolymerising textile waste or converting it into valuable chemicals (e.g. ethanol). These methods can be applied to textile waste containing natural and/or synthetic polymers.
- c) Thermal involves the conversion of synthetic textile waste by melt extrusion into plastic (polyethylene terephthalate PET) granules, flakes or chips. These methods are often confused with the incineration of textile waste for energy recovery [20].

Ethanol is the most abundant renewable fuel source and is a renewable substitute for petroleum fuels such as oil and gasoline. Due to the criticism that has arisen as a result of ethanol production from food feedstocks (molasses, cereals, fruits), it is necessary to develop and implement methods to produce ethanol from cellulosic feedstocks [29÷31]. These include cotton, which contains approximately 96% cellulose and a small amount of hemicellulose, but no lignin [31].

Cotton is a textile fibre made from seeds. The structure of cotton is multi-layered, which consists of:

- a cuticle (a very thin wall that is attached to the top of the primary layer (wall) and consists of a mixture of waxes, proteins and pectins);
- a cellulosic and non-cellulosic primary layer (wall);
- a winding layer, which connects the primary and secondary layers and is assimilated by them;
- a cellulosic secondary layer (wall);
- lumen [2, 30, 32],

and is represented schematically in Figure 1.

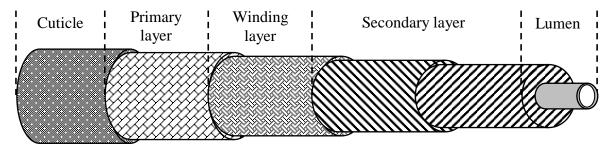


Fig. 1. Schematic representation of cotton fibre

Flax is another source of bioethanol. The structure of flax fibre is similar to that of cotton [2, 32] and consists of a primary wall, three secondary walls and a lumen. Flax fibre is composed of cellulose, hemicellulose, wax, lignin and pectin [2, 33, 34]

Hemp fibre is another source of bioethanol. It is also structurally similar to flax. It consists of a primary wall, a middle lamella, an outer secondary wall, an inner secondary wall, a tertiary wall and a lumen. Hemp fibre is composed of cellulose, hemicellulose, pectin, lignin and wax [35, 36].

Cotton cellulose has a high degree of polymerisation and crystallinity [37, 38]. The high crystallinity of cotton cellulose significantly reduces the yield of the enzymatic hydrolysis process, resulting in glucose which, when subjected to the fermentation process, results in bioethanol [37, 39]. The high crystallinity is due to the composition of the primary cell wall, which consists of two layers, the outer one consisting mainly of pectin substances and the inner one of hemicellulose, composed mainly of (xylo)glucan and cellulose [31]. In order to decrease the crystallinity, it is necessary to break the

existing hydrogen bonds between the glucan chains [39] by performing a pre-treatment process on textile materials [37].

In this paper are summarised chemicals methods used for recovery valuable compounds from textile waste. Is the recycling is applied a low impact to environmental pollution will done.

At the same time, by applying environmentally friendly methods, they lead to valuable compounds such as ethanol, biogas and others such as ethylene glycol and terephthalic acid.

Literature methods for obtaining ethanol (bioethanol)

a) Methods for production from pre-consumer textile waste

Jeoh et al. (2001) [40] and Vignesh et el. (2021) [41] studied the production of ethanol (bioethanol) from cotton gin waste. The steam explosion method was used $[40\div44]$, and steam-exploded waste samples were overlimed to precipitate microbial inhibitory compounds [40]. Fermentation was performed using *Escherichia coli*. The theoretical maximum ethanol yield was 83.1%, corresponding to 270 L ethanol per metric ton of cotton ginning waste (64.9 gal/ton) [40, 45].

Khandaker et al. and Agblevor et al. have used cotton gin residues for the production of ethanol [32, 46]. Agblevor et al. [46] used the same method of steam explosion that was used by Jeoh et al. [40]. The theoretical maximum ethanol yield was 92.5%, which corresponds to 191 L (50 gallons) ethanol per metric ton of cotton gin residue [46]. The pH of the medium studied increasing the theoretical maximum ethanol yield.

Another method for producing ethanol is obtaining from cotton gin trash [32, 38, 47÷49]. Placido et al. [47] applied three pre-treatment methods individually or in combination: ultrasonication, liquid hot water and ligninolytic enzymes.

The fermentation process was carried out to obtain ethanol in the presence of Saccharomyces cerevisiae. The best result was obtained by combined pre-treatment with ultrasonication, liquid hot water and ligninolytic enzymes. The maximum theoretical yield of ethanol was 31.6% [47].

A method similar to the one studied by Placido et al. [47] is mentioned by the authors [41, 48, 50, 51]. Placido et al. applied the pre-treatment method with ultrasound, liquid hot water and lignolytic enzymes in the presence of 15% NaOH [47]. The yield of enzymatic hydrolysis was increased by the addition of NaOH. Fermentation was carried out in the presence of Saccharomyces cerevisiae. The maximum theoretical yield of ethanol was 63% [50].

The methods described above are shown in Table 1.

Textile	Pre-treatment phase	Hydrolysis phase	Fermentation phase	Maximum theoretical	Reference
waste type	requirements	requirements	requirements	yield of ethanol, %	
Cotton gin	Steam explosion	Enzymatic	Escherichia coli	83.1	[40]
waste	$t = 237^{\circ}C$	cellulase	$t = 35^{\circ}C$		
	Residence time = 20 s	pH = 5.3	time = 24 h		
	Severity $log(R_0) = 3.56$ (reaction conditions)	$t = 50^{\circ}C$			
Cotton gin residues	Steam explosion $t = 237^{\circ}C$	Enzymatic cellulase	Escherichia coli strain KO11	92.5	[46]
	Residence time $= 20$ s	pH = 4.7	$t = 35^{\circ}C$		
	Severity $\log(R_0) = 3.56$	$t = 50^{\circ}C$	time = 18 h		
	(reaction conditions)				
Cotton gin	Ultrasonication + hot	Cellulase	Saccharomyces	31,6	[47]
trash	water + enzyme	pH = 4.8	cerevisiae		
	Hot water 121°C,T=1h	$t = 50^{\circ}C$	$t = 32^{\circ}C$		
	Enzyme: time = $96 h$	time = 96 h	pH = 4		
	$pH = 6, t = 30^{\circ}C$		time = $72 h$		
Cotton gin	Ultrasonication + hot	Cellulase	Saccharomyces	63	[50]
trash	water + enzyme	pH = 4.8	cerevisiae		
	Hot water 121°C,T=1h	$t = 50^{\circ}C$	$t = 32^{\circ}C$		
	Enzyme: time = $96 h$	time = 96 h	pH = 4		
	pH=6, t=30 °C		time = $72 h$		
	NaOH 15%				

Table 1. Ethanol production phase requirements

b) Methods for production from post-consumer textile waste

Jeihanipour et al. studied ethanol production from cotton linter and blue jeans textile waste applying alkaline pre-treatment [52]. The alkaline pre-treatment method with NaOH solution was used in the following studies [31, 37, 44, 52÷55]. In addition, the simultaneous saccharification and fermentation (SSF) method was used to produce ethanol. Glucose obtained by enzymatic hydrolysis was subjected to fermentation in the presence of Saccharomyces cerevisiae to produce ethanol. In this case, the maximum theoretical ethanol yield was 92%, obtained in the presence of 12% NaOH [52]. The ethanol production process applying SSF requirements is shown in Table 2.

Textile	Pre-treatment phase	SSF phase requirements		Maximum theoretical yield
waste type	requirements	Hydrolysis phase	Fermentation phase	of ethanol, %
		requirements	requirements	
Cotton linter	NaOH 12%	Cellulase	Saccharomyces	92
Blue jeans	t= 0 °C;	ß-glucosidase	cerevisiae	
waste	time = $3 h$	t = 45 °C, $pH = 4.8$	$t = 37^{o}C,$	
		time = 4 days	time = 4 days	

Table 2. Ethanol production phase requirements using NaOH as cellulose solvent and SSF method

In the following papers $[2, 21, 32, 39, 55 \div 62]$, another method for obtaining ethanol from cellulose, using an environmentally friendly solvent N-methylmorpholine-N-oxide (NMMO). The technological process used is presented.

The technological process for cellulose extracting is described below:

(i) first this purpose, the cellulose was separated and subsequently precipitated in aqueous medium; (ii) subsequently, the medium obtained was filtered, resulting in a mixture of NMMO/cellulose/water; (iii) the next step was to wash the cellulose again, from which the mixture was filtered. The solution of NMMO and water was subjected to the evaporation process in order to recover NMMO. After the last filtration step: the solution of NMMO and water was subjected to evaporation to recover NMMO; and the separated cellulose was subjected to enzymatic hydrolysis followed by anaerobic fermentation to produce ethanol.

The by-product was biogas, resulted from anaerobic digestion. The maximum theoretical yield of ethanol was 96% [56]. The technological process is shown schematically in Figure 2.

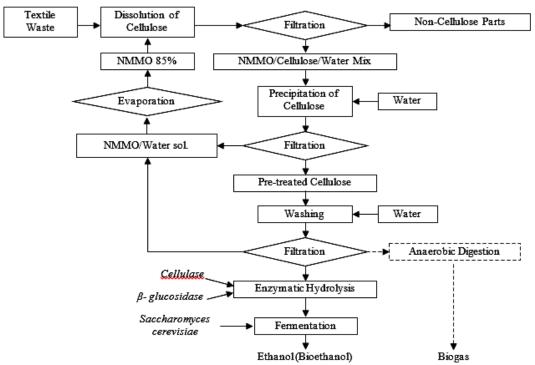


Fig. 2. Method of obtaining ethanol using NMMO as a cellulose solvent from textile waste composed of cotton/polyester and viscose/polyester blends (adapted according to references [7] and [56])

In another papers [2, 31, 44, 63÷66] are presented studied to obtain ethanol using sodium carbonate for pre-treatment of textile waste to dissolve cellulose.

In this regard, the technological process is as follows:

- (i) for this purpose, the textile waste was pre-treated with Na₂CO₃ solution under thermal conditions, preceded by a cooling step, resulting in a pre-treated cellulose solution;
- (ii) subsequently, the pre-treated cellulose solution was filtered, resulting in the following filtration phases: filtered pre-treated cellulose and a pre-treatment liquor, composed of a mixture of polyester and ethylene glycol;
- (iii) in the next step, the filtered pre-treated cellulose was rinsed with water, followed by its drying;
- (iv) dried pre-treated cellulose was then subjected to enzymatic hydrolysis followed by anaerobic fermentation to produce ethanol.

The by-product was biogas, which resulted from the anaerobic digestion process. The maximum theoretical yield of ethanol was 70% [64].

The technological process is shown schematically in Figure 3.

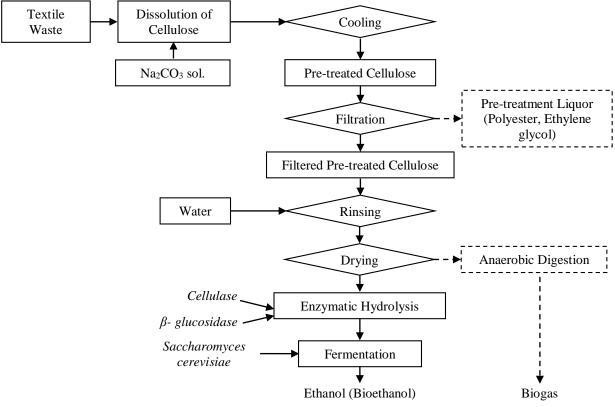


Fig. 3. Method of obtaining ethanol by using Na₂CO₃ as a cellulose solvent from textile waste composed of cotton and polyester blends. (adapted according to references [64] and [65])

In literature data are presented study about ethanol production using phosphoric acid for pre-treatment of textile waste to dissolve cellulose. SSF method were applied to obtain ethanol [2, 55, 67÷70]. The technological process described by Anacleto et al. [67] is as follows:

- (i) in the first step, the textile waste was subjected to a bleaching process in the presence of Na₂S₂O₄ and Na₂CO₃ under thermal conditions;
- (ii) then, the bleached textile waste was subjected to the cellulose dissolution process in the presence of H₃PO₄;
- (iii) followed the textile waste regeneration process by washing with deionised water;
- (iv) the next step was to remove excess water (by centrifugation) from the regenerated textile waste, followed by washing with deionised water, which was carried out 3 times;

- (v) the filtration step followed, from which the resulting filtration solution was neutralized with NaOH and the regenerated textile waste was collected on filter paper;
- (vi) the next step was to remove the salt by washing the regenerated textile waste collected by the filter paper with water;
- (vii) in the last step, the wet pre-treated textile waste was subjected to the SSF process, in order to obtain ethanol.

Enzymatic saccharification was carried out in the presence of *cellulase*, and fermentation in the presence of Zymomonas mobilis, yeast extract and the following mixture of chemicals: KH_2PO_4 , $(NH_4)2HPO_4$ and $MgSO_4 \cdot 7H_2O$. The maximum theoretical yield of ethanol was 92% [67].

The technological process described by Anacleto et al. [67], is shown schematically in Figure 4.

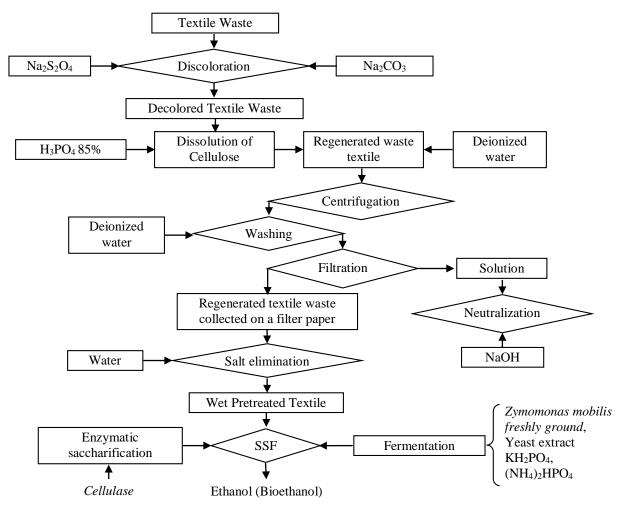


Fig. 4. Method of obtaining ethanol using H₃PO₄ as a cellulose solvent from textile waste composed of cotton linters and blends of cotton and polyester (adapted according to reference [67])

In the following scientific articles [9, 11, 28, 31, 32, 55, 58, 63, 69, 71 \div 74] alkaline NaOH, NaOH/urea, NaOH/thiourea, NaOH/urea/thiourea solutions were used for pre-treatment of textile waste. Analysing the result presented, the best yield was obtained using the mixture between the NaOH/urea mixture at temperature (T), T= -20 °C [63]. To obtain ethanol, as in the study by Kuo et al. [68], the SSF step was applied.

In this aims the technological process described by Gholamzad et el. [63] is the following:

- (i) in the first step the textile waste, composed of a polyester/white cotton blend, was subjected to the cellulose dissolution process in the presence of NaOH/urea;
- (ii) followed by the washing stage of pre-treated textile waste in order to lower the pH to 7, and they were subjected to a drying process at ambient temperature;

(iii) the pre-treated, dried samples were subjected to the SSF step. In this step, the dry pretreated samples were suspended in a mixture of sodium citrate buffer solution, yeast extract, (NH₄)2SO₄, K₂HPO₄, MgSO₄·7H₂O and CaCl₂·2H₂O and subjected to a thermal autoclaving process. Enzymatic saccharification was carried out in the presence of *cellulase* and β -glucosidase, and fermentation in the presence of *Saccharomyces cerevisiae*. Polyester was obtained as a by-product of the enzymatic hydrolysis process.

The maximum theoretical yield of ethanol was 70%. The technological process described by Gholamzad et al. [63] is shown schematically in Figure 5.

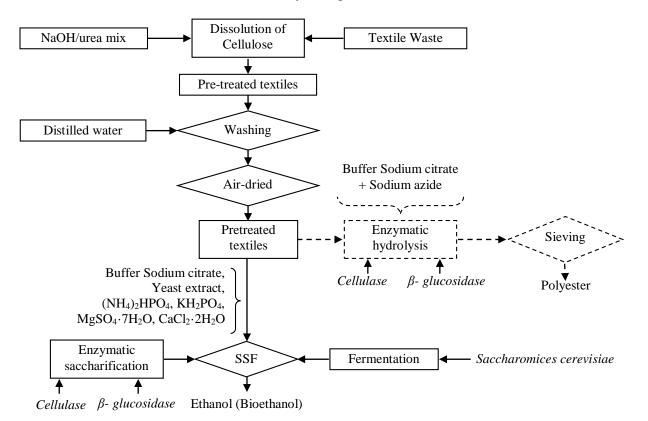


Fig. 5. Method of obtaining ethanol using NaOH/urea as a cellulose solvent from textile waste composed of cotton and polyester blends [63]

Another methodology for ethanol obtained is presented by Cho et al. [37]. In this aims were used the following types of textile waste as raw materials: white (uncoloured) cotton, coloured cotton and cotton/PET blend for ethanol extraction. They used three different technologies in order to obtain ethanol.

The technological process to obtain ethanol using white (uncoloured) cotton-based textile waste was: (i) uncoloured cotton-based textile waste was subjected to alkaline pre-treatment with NaOH;

(ii) followed by filtration and washing with distilled water until a pH = 7;

(iii) in the next step the pre-treated textile waste was subjected to enzymatic hydrolysis, which was carried out in the presence of sodium citrate, sodium azide and cellulase;

(iv) the last step was that of fermentation in order to produce ethanol. Fermentation took place in the presence of yeast.

The technological process for obtaining ethanol using coloured cotton-based textile waste was identical to that of uncoloured cotton-based textile waste, with the specification that, at first, the decolourization of the coloured cotton waste was carried out using hydrogen peroxide and acetic acid. *The technological process to obtain ethanol using textile waste based on a blend of cotton and PET* was as follows:

(i) cotton/PET blend textile waste was pre-treated in the presence of NaOH and C₂H₅OH;

(ii) the next step was filtration, which resulted in pre-treated textile waste;

(iii) this was followed by the enzymatic hydrolysis step, which was carried out in the presence of cellulase and the recombinant WCCG gene;

(iv) the last step, fermentation, is identical to that applied to cotton-based textile waste.

Following H_2SO_4 precipitation of the filtrate obtained in the filtration step, terephthalic acid and ethylene glycol, which are monomers of polyethylene terephthalate, were obtained as valuable by-products.

Therefore, applying the previous methodology the maximum theoretical yield of ethanol was 83.5% for white cotton textile waste, follow by 78% for coloured cotton textile waste and 64% for cotton/PET blend waste. Therefore, the maximum theoretical yield of ethanol was obtained for uncoloured cotton textile waste [37].

The advantages and disadvantages of the methods presented in this review can be summarised in Table 3.

Method	Advantages	Disadvantages	References
Steam	Cotton gin waste are used. $\eta = 83.1$ % No polluting chemicals are used.	Severe reaction conditions - Severity $log(R0) = 3.56$ Increased costs by including the grinding operation of raw material (cotton gin waste).	[40]
explosion	Cotton gin residues are used. $\eta = 92.5 \%$ No chemicals are used that pollute the environment and are dangerous to humans.	Severity reaction conditions are	[46]
Ultrasonication	Cotton gin trash are used. No reagents hazardous to the environment and human health are used in the manufacturing process.	$\eta = 31.6 \%$	[47]
+ hot water + enzyme	It is applicable to the raw materials mentioned in reference 47. $\eta = 63 \%$ Reduced operating costs due to the use of NaOH.	operation was required	[50]
Pre-treatment with NaOH	$\eta = 92$ % Low price of NaOH, which reduces manufacturing costs.	It is only used for textile waste with a high cellulose content (93÷98%). The use of NaOH requires neutralization operation.	[52]
Pre-treatment with NMMO	It is used for all types of textile waste. $\eta = 96 \%$ Use of an environmentally friendly solvent - NMMO. Biogas can be obtained from the anaerobic digestion process.		[56]
Pre-treatment with Na ₂ CO ₃	$\eta = 70 \%$ Use of Na ₂ CO ₃ (environmentally friendly). The pre-treated cellulose is subjected to the process of anaerobic digestion, which results in biogas.	Applies only to textile waste with a high cellulose content (approx. 99%).	[64]
Pre-treatment with H ₃ PO ₄	The textile raw materials are identical to reference 56. $\eta = 92$ % Use of a single reactor for SSF (lower	operation. It was necessary to	[68]
Pre-treatment with NaOH/urea	The raw materials referred to in reference 56 may be used. $\eta = 70 \%$ 98% of the polyester contained in the textile waste was recovered following the enzymatic hydrolysis process.	white textile waste.	[63]

Table 3. Advantages and disadvantages of methods presented for pre-treatment of textile waste

Pre-treatment with NaOH	$\eta = 83.5 \ \%$	It is only applicable to uncoloured cellulosic textile waste.	[37]
	$\eta = 78 \%$	It is only used for coloured textile waste containing cellulose. It initially requires a bleaching step in the presence of hydrogen peroxide and acetic acid.	[37]
Pre-treatment with NaOH and C ₂ H ₅ OH	Applies to textile waste mentioned in reference 56. Valuable by-products are obtained: terephthalic acid and ethylene glycol (which are monomers of PET). $\eta = 68 \%$	the neutralization operation.	[37]

Note: the maximum theoretical yield of obtaining ethanol = η

CONCLUSIONS

In this review, are presented methods for treatment and valorisation of pre- and post-consumer textile waste in order to avoid environmental pollution by storing them for long periods of time. Thus, recovery methods (chemical and environmentally friendly) are presented in order to transform them into other valuable products: ethanol, biogas, polyester, terephthalic acid and ethylene glycol. There is concern about the transition from the laboratory phase to the industrial phase.

The textile wastes studied are made from uncoloured cotton, coloured cotton and blends of cotton/PET and viscose/PET.

Coloured textile waste generates the following problems: bleaching them to remove dyes, which inhibit cellulose dissolution; removal of pigments that are part of textile waste dyes. These being organic (e.g. benzoic derivatives) and inorganic (e.g. metal oxides).

Following the assessment, environmentally friendly methods are recommended as they generate minimal environmental pollution, reducing the generation of toxic waste.

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