

Chemical Treatment for Textile Waste

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Trends in the textile industry show a continuous increase in the production and sale of textile materials, which in turn generates a huge amount of discarded clothing every year. This has a negative impact on the environment, on one side, by consuming resources—some of them non-renewables (to produce synthetic polymers)—and on the other side, by polluting the environment through the emission of GHGs (greenhouse gases), the generation of microplastics, and the release of toxic chemicals in the environment (dyes, chemical reagents, etc.). When natural polymers (e.g., cellulose, protein fibers) are used for the manufacturing of clothes, the negative impact is transferred to soil pollution (e.g., by using pesticides, fertilizers). In addition, for the manufacture of clothes from natural fibers, large amounts of water are consumed for irrigation. According to the European Environment Agency (EEA), the consumption of clothing is expected to increase by 63%, from 62 million tonnes in 2019 to 102 million tonnes in 2030.

textile waste valorization

environmental pollution

Chemical Treatment

1. Introduction

One way to overcome the time-consuming separation of natural and synthetic fibers by manual selection is the use of a chemical treatment [\[1\]\[2\]\[3\]\[4\]\[5\]\[6\]](#). Waste jeans represent an important fraction of the total textile waste. They are a major source of cellulose. Natural cellulosic fibers are a renewable biomass source to produce higher-value products via cellulose hydrolysis. The cellulose contained by the cotton fibers has high crystallinity, being inefficient in the conversion to biofuels. Alkali pre-treatment (with NaOH or Na₂CO₃) at moderate temperatures transforms the crystalline structure of cellulose into an amorphous form, which is more easily biodegradable [\[7\]](#).

Palme et al. (2017) used a treatment with NaOH at temperatures ranging from 70 to 90 °C to separate cotton and PET (poly (ethylene terephthalate), polyester) from mixed textiles. In this step, the PET is degraded to terephthalic acid (TPA) and ethylene glycol (EG). Three product streams are generated from the process: cotton, TPA and the filtrate containing EG, and the process chemicals [\[1\]](#).

The separated materials can be further used to produce new chemical products. For example, Gholamzad et al. (2014) studied the production of ethanol from the cellulose recovered from a polyester-cotton textile material [\[2\]](#). In previous studies, it was found that the addition of urea, thiourea, or a mixture of these two enhanced the dissolution of cellulose in an alkaline solution [\[3\]\[4\]\[5\]\[6\]](#). The alkali pre-treatment is followed by enzymatic hydrolysis, in the presence of cellulase and glucosidase. Alkali pre-treatment was found to improve the production of ethanol from the cellulosic part of a polyester-cotton blend [\[2\]](#).

Hasanzadeh et al. (2018) used Na_2CO_3 as the reagent for the alkali pre-treatment. The waste textile consisted of used jeans made of cotton and polyester with about 90% cellulose. The alkali-pre-treated textile waste was subjected to anaerobic digestion, enzymatic hydrolysis, and fermentation to produce biogas, sugars, and ethanol [7].

It was shown that substituting conventional fuels by green fuels offers the advantages of disposal of waste material that is used for fuel production and reduction of GHG (greenhouse gases) emissions. One example is the use of cotton ginning wastes as an alternative energy source to replace part of the heavy fuel oil used for the thermal needs (up to 52%) of a textile plant located in northern Greece [8].

Another possibility for valorization of the textile waste is the synthesis of different chemicals, after the recovery of natural or the synthetic polymers from the clothing material, using environmentally friendly solvents. Jeyhanipour et al. (2010) developed a process for the separation of the cellulose, cotton, and viscose blended fibers [9]. The recovered polymers were used in the synthesis of ethanol or biogas. The separation of the polyester/cotton fibers was made for a 50/50 mixture of polyester/cotton. In the first step, the cellulose was separated using an environmentally friendly cellulose solvent, N-methylmorpholine-N-oxide (NMMO). The cellulose was separated by precipitation, by adding water to the mixture solvent-cellulose. In this way, the recovered cellulose fibers are more accessible to the hydrolyzing process by enzymes or bacteria. To obtain the ethanol, the cellulose was hydrolyzed by cellulase enzymes, followed by fermentation, to produce the ethanol. The biogas was produced through fermentation of the cellulose.

The separation of the polyester/viscose blended textiles was made for materials having the proportion 40/60 polyester/viscose. The polyesters resulted in fibers after treatment with NMMO solvent.

Another method for textile waste valorization is the production of biogas. Jeyhanipour et al. (2013) tested cotton/polyester and viscose/polyester fiber blends with no pre-treatment or milling [10]. The textile waste (jeans) was grounded and mixed with solvent, N-methylmorpholine-N-oxide (NMMO) to dissolve the cellulose. The steps employed for cellulose recovery were described in a previous study [9]. In the two-stage process, the lag phase was shorter than in the single-phase CSTR. Comparing treated and untreated jeans textiles wastes, the semi-continuous two-stage process can treat higher organic load rates.

The alkali and organic solvent treatment methods have several advantages compared with incineration:

allows fast and easy separation of natural and synthetic polymers from blended fibers;

the resulting cellulosic fibers are much shorter and therefore more degradable for further transformation in glucose, ethanol, and biogas;

the reaction conditions are relatively mild (temperature below 100 °C and atmospheric pressure).

Treatment with NMMO solvent also allows the recovery of the solvent and its immediate use.

Cellulose hydrolysis using concentrated acid (H_2SO_4) allows the production of hydrogen from cellulose hydrolysate via subsequent fermentative process [11]. Ouchi et al. (2010) developed a method consisting of acid treatment of an uncut textile piece, followed by mechanical stirring, filtering, washing, and drying, to produce cellulose powder [12]. The recovery of sulphate ion from the hydrolysate was made by anionic exchange. The conversion of a towel to glucose, and further to ethanol, was made by using microwave-assisted treatment of the towel impregnated in concentrated sulfuric acid [13].

To obtain a good conversion of recovered glucose in subsequent valuable products, the glucose yield in the hydrolysate must be sufficiently high. It has been reported that a single-step acid hydrolysis (using sulfuric acid) does not achieve a high glucose content. When using a two-step method, combining hydrolysis with concentrated and diluted sulfuric acid, it was possible to get 90% glucose yield [14].

Yousef et al. (2020) used the nitric acid treatment to recover the cellulose contained in jeans waste [15]. The dyes are removed together with the spent acid that is regenerated with activated carbon. Polyester was dissolved and separated from cotton, by using a green switchable hydrophilicity solvent. The polyester and the solvent were regenerated by adding CO_2 to the solution. The solidified polyester is separated by filtration. The recycling rate of the technology was more than 96% for jeans waste containing 84% (wt.) cotton and 16% (wt.) polyester [15]. To select the leaching media among the strong acid reagents (HNO_3 or H_2SO_4), preliminary tests showed that the nitric acid is leaching only the dye, while the sulfuric acid solubilizes all the components of the jeans waste [16][17].

The major drawback when using concentrated acid pre-treatment is the necessity of acid recovery. In addition, most of the studies are performed with textile waste that is pre-washed, dried, and cut, which cannot be done on a large scale.

Kuo et al. (2014) found that pre-treatment of waste textiles with ortho-phosphoric acid resulted in an improved rate of enzymatic hydrolysis, reducing the sugar yield [18]. This is because the acid dissolves the crystalline cellulose structure, which is resistant to enzymatic processes. When saccharification and fermentation steps are performed simultaneously, the ethanol concentration is higher, and the process takes place in one single reactor. Dyed textile waste did not show any inhibitory effect on the ethanol fermentation activity of *Zymomonas mobilis*. Phosphoric acid-pre-treated cellulose has been proven to have a higher initial hydrolysis rate and glucose yield [19]. This method was applied for cotton textile waste and for mixtures of 40/60 polyester/cotton textile waste. Furthermore, the ortho-phosphoric acid is characterized by non-corrosivity and non-toxicity, being safer compared to NaOH , H_2SO_4 , or HNO_3 [20][21]. The researchers optimized the hydrolysis conditions so that they could efficiently recover 100% of the polyester with a maximum sugar recovery of 79.2% (when using 85% phosphoric acid, 50 °C, 7 h, ratio of 1:15) [19].

Lopatina et al. (2021) used ionic liquids to prepare a cellulose-based ultrafiltration membrane from white cotton waste [22]. Zhong et al. (2020) used ionic liquid (IL) 1-butyl-3-methylimidazolium chloride ([Bmim]Cl) to extract wool keratin. The extracted keratin was used to prepare a keratin/polyacrylonitrile (PAN) composite nanofibrous membrane with good antibacterial effects and high moisture permeability [23]. This method was also applied for the

separation of blended natural and synthetic polymers. The polyester-cotton blended textiles were treated with ionic liquid 1-allyl-3-methylimidazolium chloride (AmimCl). The recovered cellulose was transformed into transparent cellulose films and high-purity polyester [24]. It was found that the cellulosic fibers extracted from textile cotton waste have increased mechanical properties compared to cellulosic fibers produced from wood pulp. This is linked to a higher degree of polymerization of waste cotton [25]. The ionic liquid is recovered by washing the cellulose or keratin with water.

Another aspect that must be considered when developing a plan for textile recycling is the removal of dyes. Generally, the dyes used in the textile industry are synthetic organic colorants (e.g., azo dyes) with properties that make them resistant to destruction by conventional treatment methods. For cellulose fibers, the following types of dyes are used: reactive dyes, direct dyes, naphthol dyes, and indigo dyes; for protein fibers, acid dyes and Lanaset dyes are used. Dispersed dyes, basic dyes, and direct dyes are used to dye synthetic fibers [26]. The removal of dyes needs to be done before the recovery stage. Even small concentrations of dyes in the water receptor affects the environment, by their toxicity and by blocking both the penetration of light and oxygen transfer [27]. The dyes can be present in very low concentrations of 1 mg L^{-1} in the receptor waters, and they can enter the food chain through aquatic organisms [28]. Among the methods employed for the dyes' removal, enzymatic degradation is more effective [29][30]. Other parameters that affect the stability of dye complexes are temperature and acidity. The results showed that yellow or orange chromium complex containing an aromatic carboxyl group could be easily destroyed by either of them. The destruction of more stable black chromium complex is achieved by both heating and acidification [31].

The sludge resulting after the treatment of textile waste contains Pb, Cd, Cr, and other toxic elements that can leach into the environment [32][33]. Several studies have tested the efficiency of vermicomposting to reduce the metal content of the sludge [34][35][36][37][38]. The tests showed that, for the *Eudrilus eugeniae*, the bioaccumulation preference was in the order: $\text{Zn} \geq \text{Fe} > \text{Mn} = \text{Cu} > \text{Cr} = \text{Pb} = \text{Cd}$. In addition, vermicomposting can be applied to reduce the spread of fungal pathogens, inoculums, and so on [39]. The resulting compost is suitable for use as a soil supplement because the earthworms produce the humus essential in the crop growth. The main disadvantage of the composting is the long time needed to transform the waste in compost, although the introduction of earthworms accelerates the process. Abbas et al. (2013) applied composted waste textile (cotton), alone and in combination with a commercial fertilizer, on sunflower crop. The highest significant improvement in plant growth were obtained for soils treated with cotton waste compost and those treated with cotton waste compost plus commercial fertilizer [40].

Araujo et al. (2007) tested the use of composted solid sludge from a textile mill in the growing of soybeans and cowpeas. According to the researchers, the sludge was not harmful for the plant's growth. However, more studies are necessary because of the short time scale of this one (up to 63 days after plant emergence) [41].

The application of hydrogel-based materials has been extended in the last years to the removal of heavy metals from aqueous solutions [42][43][44]. One of the applications of the extracted cellulosic solutions via hydrolysis with NaOH is the adsorption of heavy metals from aqueous solutions [44]. A double network hydrogel is synthesized via

crosslinking of natural polymer with synthetic polymer (polyacrylamide). The cellulosic hydrolysate is treated with KPS (potassium persulfate, which is the initiator) and crosslinked with MBA (N-methylene bis acrylamide). Different quantities of epichlorohydrin were added to this solution to obtain several hydrogel materials. These materials were tested in the retention of Cd, Cu, Pb, Zn, and Fe.

2. Thermal Treatment

Natural and synthetic polymers are a resource for recovering carbon. By using the tertiary recycling concept (turning waste into a completely new product), the carbon contained in the textile waste can be recovered as a new chemical substance. The pyrolysis process is the thermal decomposition of materials at high temperatures, in an inert atmosphere, and it involves changes in chemical composition. In this way, textile waste can be transformed after pyrolysis into three products: char, pyrolytic oil, and syngas. This method has been studied extensively in an attempt to find an efficient method of recycling textile waste [45][46][47][48][49][50][51][52][53][54][55][56][57][58][59][60][61][62][63]. The mechanisms and the kinetics of cotton pyrolysis are well known [64][65].

Another method studied for the reuse of textile waste is the synthesis of porous carbon through pyrolysis [66][67][68][69]. This method uses an additive: CaCO₃, calcium acetate [69], or a natural source of CaCO₃ (such as oyster shells) [70]. At high temperature, CaCO₃ decomposes to CaO, releasing the CO₂ and helping in the formation of the microporous carbon structure. The pyrolysis process follows the next steps: (a) raising the temperature of the material to be pyrolyzed using an external source; (b) initiation of pyrolysis reactions at high temperature; (c) release of volatile compounds; (d) formation of residues containing carbon.

(1) 30–300 °C: calcium acetate (CA) is dispersed on the surface of cotton polyester waste, and water contained by CPW is evaporated.

(2) 300–600 °C: CPW degradation to furans, sugars, ethylene, and aldehydes. At 400 °C, CA decomposes into acetone and CaCO₃. Formation of aromatic structure through the cross-linking and cyclization reaction; construct preliminary carbon skeleton of porous carbon.

(3) 600–800 °C: CaCO₃ decomposes into CaO and CO₂, which helps in the formation of the mesoporous structure.

(4) CaO is washed by HCl solution, and porous carbon is obtained.

By changing the operating parameters (temperature, textile waste composition, residence time), the composition of the pyrolysis gas (H₂ and CO) can be adjusted.

Another product that can be used after the pyrolysis is pyro-oil. Its composition can be improved by using catalysts (catalytic pyrolysis) [56]. ZnO was found to have the highest catalytic effect in the pyrolysis of mixed textiles, while Fe₂O₃ had the highest catalytic effect in the process of char gasification. The waste textiles were cut into very small pieces, with a particle size range of 0.8–1.2 mm, and the catalyst (single or composite) was loaded onto waste

textiles. The major disadvantage of this method is the difficulty in pre-processing large quantities of textile materials to be pyrolyzed [47][56].

Comparing non-catalytic and catalytic pyrolysis, to minimize the production of benzene derivatives and polycyclic aromatic hydrocarbons, the addition of CO₂ leads to three-fold higher H₂ and eight-fold higher CO production [47].

Pyrolysis of flax textile waste can be used to produce, in a first step, furans, and further, monocyclic aromatic hydrocarbons. Furans are produced by catalytic dehydration of cellulose and/or hemicellulose in biomass [55][71][72][73].

Wang et al. (2018) studied the mechanisms of the catalytic fast pyrolysis of cellulose, cellobiose, and glucose in the presence of the zeolite catalyst NaY to decrease the activation energies of cellulose, cellobiose, and glucose [55]. The contents of furans after cellulose, cellobiose, and glucose pyrolysis in the presence of NaY were more than doubled (from 17.48%, 18.79%, and 28.77% to 46.71%, 52.11%, and 67.81%, respectively).

Catalytic degradation of flax waste (FW) to generate furans followed by Diels–Alder transformation to monocyclic aromatic hydrocarbons over USY zeolite (Si/Al molar ratios of 5.3 and 11.0) resulted in a 5.5-fold increase in furans production [73]. Three types of waste were tested: polyethylene (PE), flax waste (FW), and polypropylene (PP). PE co-fed with FW yielded almost two times higher aromatic hydrocarbons than PP. The selectivity to aromatic hydrocarbons was 81.6% for a mixture of 20% PE co-fed with 80% FW, in which benzene, toluene, and xylenes (BTX) were predominant products, with a maximum selectivity of 68%.

Textile waste has low calorific value. One method to remedy this is co-pyrolysis of textile dyeing sludge with high-calorific plastic waste (medical plastic wastes from syringes, medical bottles). Polyolefin plastics with high calorific value and low ash content have an optimum pyrolysis temperature in the range of 400–550 °C [46].

Alongi et al. (2013) studied the synthesis of char from cotton, poly(ethylene terephthalate), and their blends by thermal oxidative degradation. The researchers proposed two mechanisms for the transformation of cotton and polyester, respectively [66]. In the case of cellulose, its thermal decomposition takes place at temperatures ranging from 300 to 400 °C through two concurrent steps: depolymerization and dehydration. The aliphatic structures generated after dehydration are further transformed into aromatic structures at about 400–600 °C. In a first step, the polyester undergoes chain scission, and at about 400–500 °C, it can either undergo depolymerization or decompose into char. The obtained char is thermally stable up to temperatures of 800 °C.

The resulting product can be used either as an adsorbent material or as an additive to improve the properties of clothing. Cay et al. (2020) investigated the use of textile waste-based biochar as additives, to improve clothing performance by changing the surface properties of textile materials. Cotton, cotton/polyester, and acrylic textile wastes were carbonized at low temperature, and the resulting products were applied to cotton by a printing method. This gives a slight hydrophobicity to the material, by increasing the water spreading speed and radius.

Therefore, the acceleration of moisture transfer and drying has been improved. In addition, it was found that the resulting material has odor-masking properties [68].

Torrefaction has also been studied as an alternative method to produce quality fuels from textile waste [74][75][76]. Torrefaction is a mild form of pyrolysis at temperatures in the range of 200–320 °C. The resulting product has a lower moisture content and a higher heating value. The fuel characteristics of obtained biochar are comparable to those of coal [76]. The torrefaction temperature has a significant effect on biochar yield, while the fiber type influences the energy densification ratio (ratio of the HHV of biochar to the HHV of raw waste textile) [74]. Different types of textile waste (natural, synthetic, and their blends: cotton/polyester, acrylic/wool, acrylic/polyester, acrylic/viscose) were torrefied at temperatures in the range of 300–400 °C. The resulting material has low ash and sulphur content [74]. The 100% polyester fiber has high thermal stability, and is therefore not suitable for the torrefaction process. Torrefaction of polyester-containing blends, however, produced energy-dense biochar. Torrefied materials obtained from acrylic waste materials have properties similar to bituminous coal, while cellulosic polyester-based biochars have a similar structure to lignite. The biochar obtained from acrylic materials has a high nitrogen content (12–23 wt.%), which makes it unusable as a fuel.

Hydrothermal carbonization is also a thermochemical process used to pre-treat waste with high moisture content, under hot compressed water. Subsequent treatment of the obtained product is required to achieve the final product (e.g., activated carbon) [77][78][79][80][81][82][83][84]. Compared to a chemical treatment that uses strong reactants, in hydrothermal carbonization, under the effect of the pressurized water, the polymers contained in the textile waste are hydrolyzed and degraded into smaller molecules [85].

Another advantage of hydrothermal carbonization as a pre-treatment for textile waste, besides allowing the transformation of a waste into a valuable product, is the improvement of activation performance of the textile waste. This, in turn, reduces the quantity of activation agent (FeCl_3 , ZnCl_2 , KOH , CO_2), which is needed in a subsequent step. Among the cotton, polyester, and cotton/polyester blends subjected to hydrothermal carbonization, the cotton-based ZnCl_2 -activated carbon exhibited the highest surface area (1906 m^2/g), and an anoxytetracycline adsorption capacity of 621.2 mg/g, whereas the polyester-based adsorbent had a lower surface area (554 m^2/g) and lower oxytetracycline adsorption performance [77]. FeCl_3 was found to lower the initial processing temperature, catalyze dehydration and decarboxylation, and facilitate the formation of furfural derivatives, which are subsequently transformed into lignin structure [80].

Hydrothermal carbonization applied to solid waste containing textile waste has also been investigated. Wood, paper, and food have been used to perform simple hydrothermal carbonization or co-hydrothermal carbonization [79]. Synergistic effects have been reported during co-hydrothermal carbonization: negative synergistic effects between textile, wood, and paper waste; positive synergistic effects between textile and food waste. Increasing the oxygen content of hydrochars decreased the HHV.

Co-hydrothermal carbonization of cotton textile waste and polyvinyl chloride waste (PVC) was carried out to recover the energy contained in the waste as an alternative solid fuel. During the thermal treatment of PVC, HCl is

generated from the dechlorination process. HCl release allows the formation of the porous structure in the hydrochars [82].

When using hydrochloric acid, it is possible to extract the cellulose in its crystalline form [81][86]. Sheng et al. (2018) studied the extraction of microcrystalline cellulose by the hydrothermal method and compared the extracted product with a commercial one (Avicel PH101 microcrystalline cellulose). The microcrystalline cellulose was extracted under hydrothermal conditions, in the presence of HCl: solid–liquid ratio 1:30, HCl concentration 0.6 mol/L, 150 °C [81].

Waste cotton textiles were subjected to hydrothermal pre-treatment at 240 °C–340 °C, to obtain bio-crude oil and biochar [84]. The optimum conditions (320 °C for 60 min) allowed the highest bio-crude oil fraction of 23.3%. The obtained biochar has a rich carbon content (76.86%) and low oxygen content, and it can be used as an alternative fuel. Furthermore, the biochar was evaluated as an electrocatalytic material with good results (the biochar enhanced the conductivity of the electrode and improved the electrochemical surface area, which could speed up charge transfer rate).

3. Enzymatic separation

Compared to the other methods presented above, enzymatic degradation allows the degradation process to operate under mild conditions [87][88][89][90]. Furthermore, enzymes are biodegradable, and therefore harmful chemicals are replaced by materials that are easier to dispose of. Enzymes act as catalysts and, at the end of the process, can be reused. Enzymatic degradation is specific to natural polymers, allowing the separation of natural and synthetic polymers.

The separation of wool fibers from mixed wool and polyester fabrics by enzymatic digestion allowed the recovery of keratin hydrolysate (obtained from wool) and polyester [87]. Analysis of polyester by scanning electron microscopy showed that the enzymatic treatment had no significant change on the quality of recovered polyester fibers. Enzymatic degradation can also be applied to blends of several natural fibers with polyester. For that, two types of enzymes are used: protease, for the extraction of amino acids from wool components, and cellulases, for the recovery of glucose from cotton fibers. The glucose resulting from the cotton fibers is subsequently converted into ethanol, by fermentation with *Saccharomyces cerevisiae* [88].

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