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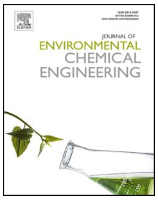

Pyrolysis of textile waste: A sustainable approach to waste management and resource recovery

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ABSTRACT

The textile industry is one of the most polluting sectors globally, with increasing amounts of textile waste posing significant environmental and public health challenges. This study investigates the pyrolysis of pure cotton, polyester, and a 55 % polyester/45 % cotton blend as a sustainable approach to waste management and resource recovery. Pyrolysis was conducted in a tubular reactor at 425 °C, 500 °C, and 575 °C. The resulting char, oil and gas fractions were quantified and characterized. The highest oil yield (over 45 %) was obtained from pure cotton, primarily composed of heavy naphtha. In contrast, pure polyester and the cotton-polyester blend produced predominantly gaseous fraction (over 50 %) with H_2 as the major compound. The char produced, especially from cotton at 575 °C, exhibited significant carbon content (75.6 %) and textural properties suitable for being used as adsorbents. These findings demonstrate the potential of pyrolysis for energy recovery and promoting a circular economy in textile waste management.

1. Introduction

Fast fashion has led to an increase in global textile production, reaching a record 116 million tons by 2022 [1]. Increasingly, this industry is producing clothing at lower costs, a phenomenon that has led to an unprecedented increase in the generation of post-industrial or post-consumer textile waste worldwide, posing serious challenges in terms of management and treatment [2,3].

The origin of this negative impact of textile waste is largely due to the linear model followed by the textile industry based on the production, use and dispose. The main raw materials used are natural fibers, i.e. cotton, leather, wool and silk [4], or synthetic fibers [5], such as polyester, polyamides, acrylics and polypropylene. The textile market is dominated by cotton and polyester, the latter being the most common, accounting for 54 % of global fiber production in 2022 with approximately 63.6 million tons produced [1]. Polyester is often blended with cotton to obtain fabrics with good breathability in the case of cotton, and strength in the case of polyester [6].

Textile waste has to be divided into two categories, pre-consumer waste, which is waste generated during the textile production process before it becomes a garment and reaches the consumer, it also includes production surpluses that have been returned or that have not even been sold in the sales, so they have not reached the consumer. On the other hand, post-consumer waste represents those garments that the consumer discards, generally the latter are those that are found in landfills along with other urban solid waste (USW) [7,8]. It is estimated that in recent years, around 5.8 million tons of textile waste are discarded throughout the European Union (EU), equivalent to 11.0 kg per person [8,9], of which only 22.0 % of waste is collected selectively for recycling, the rest is sent to landfills and in some cases to incinerators that generate pollutants that are harmful to the ecosystem [10] and public health [11]. During the incineration process of textile waste may produce emissions such as dioxins and furans, NOx, HF, HCl, CO, dust and heavy metals among others. The dioxin has long term accumulated in environment and food chain and has an adverse effect on human health such as damage to the immune system, interference with the hormone system, and also in causing cancer [12–14].

The textile industry is considered the second most environmentally polluting sector, followed by the oil industry [15], it is estimated that 990,000 tons of textile products end up in landfills in Spain every year

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[8]. This issue has motivated countries to introduce stricter policies and regulations to promote the reduction of production, reuse, and recycling of textile waste. In 2018, Directive (EU) 2018/851 of the European Parliament and of the Council of 30 May 2018 amending Directive 2008/98/EC on waste was adopted, which has as its main objective to promote the transition to a circular economy and improve waste management in Europe [16]. The European Commission has suggested a specific framework for the textile sector, calls on producers to be responsible for the entire life cycle of textile products and proposes the implementation of a requirement that by 2025 textile products must be collected separately [17].

To meet these new challenges, textile waste management has evolved from a solution based on landfill or incineration to one based on recycling [18], from a circular economy perspective, whose main objective is to convert substances of lower value to products or raw materials of higher value and thus maximize the use of resources to new products of industrial interest and promote long-term environmental sustainability. Depending on the type of textile waste to be treated, different methods or routes can be considered. Generally, wastes made up of only one kind of fiber are easier to treat as the process is simpler. The two main methods of textile waste valorization include mechanical recycling and chemical recycling.

Mechanical recycling is a process where the textile is sorted by fiber type or color, then shredded and re-produced into a new textile or product of interest, without altering the original chemical structure of the polymer. Its main limitation is reflected when the textile waste is formed by a combination of natural and synthetic fibers [18,19]. In search of technologies that respond to this problem, chemical recycling has emerged, including pyrolysis, also known as thermolysis [20]. Pyrolysis is a chemical reaction in an inert atmosphere induced by thermal energy, irreversible, leading to a change in the physical state and chemical composition of matter, i.e. lysis to smaller and less complex chains [21,22]. During the pyrolytic process, the chemical bonds are broken generating three fractions; a non-condensable gaseous product with a high calorific value including hydrocarbons H_2, CO, CO_2, CH_4 and C2-C3 [20,23], carbonaceous solid products that open up possibilities for use as adsorbents or catalytic supports [6,24–26] and liquid products that have important combustible properties [27–29].

Among the most promising advantages of pyrolysis is the ability to use homogeneous and complex materials, transforming them into fractions that can be modified with a certain flexibility [30] depending directly on the operating parameters, temperature [31], heating rate, residence time, use of catalytic converters [32,33], moisture content, carrier gases, the type of reactor [34] and pressure [35,36]. It has been reported that the most commonly used reactors in pyrolysis are batch reactors [37], fixed bed reactor and fluidized bed reactor [38,39].

In recent years, the interest in the study of this technology has increased, from 2023 to date, 794 articles registered in the Scopus database, mention that pyrolysis is promising in the production of oil from different wastes such as plastics [21,40,41], newspaper [42], pomegranate peel [43], treated Wood [44], used tires [45], coffee roasting [46] residues and electronic wastes [5]. As an industrial example of plastic pyrolysis, in 2018, at ReOil's pilot plant, plastic pyrolysis was integrated into OMV's oil refinery. The goal was to feed the refinery unit with the liquid produced from pyrolysis, successfully processing 100 kg of plastic per hour to obtain approximately 100 liters of valuable crude oil [47]. Also, some research reports the use of co-pyrolysis using a mixed feedstocks [48].

Other research groups have chosen to study energy recovery from textile waste in its three fractions, gas, liquid and solid. A recent review of the field shows an increase in the number of publications in three scientific research databases, with China and India having the highest number of publications [49]. Pyrolysis is highlighted as a promising recycling option for textile wastes, in the recovery of monomer from nylon, or as a method of converting these wastes into energy such as fuel gas [50].

Recycling by pyrolysis offers an alternative that could contribute to meeting the circular objectives. This technology does not require prior feedstock preparation, has the potential to treat contaminated waste, and can be used with any type of material [51]. It is a promising method for energy recovery and decreasing environmental pollution; however, most studies report pyrolysis analysis using a single type of textile material and a single set of parameters. A few investigations have been carried out with different textile wastes varying chemical composition, a condition that brings us closer to the reality of USW arriving at the landfill and different reaction parameters.

In this research, we worked under three pyrolysis temperatures, 425 °C, 500 °C, and 575 °C with three types of textile material, pure cotton, pure polyester, and a mixture of 55 % polyester and 45 % cotton. Our objective is to evaluate the influence of temperature on the performance of the fractions (solid or char, liquid or oil and gaseous) and to characterize them by analytical techniques. The results open the possibility of valorizing textile waste in products of industrial value and the fuel market.

2. Materials and methods

2.1. Raw materials

Textile waste was collected from the residual fraction of the Ecocentral municipal solid waste treatment plant located in the metropolitan area of Granada (Alhendín, Granada, Spain). 100 kg of textile waste was collected from the rejection line and classified based on the information reported by the manufacturer on their labels. Once received in the laboratory, the textile waste was washed and dried for disinfection before handling. Additionally, the moisture and dirt content of the textile waste was determined by measuring the weight difference between the original textile waste and the waste after washing and drying. The materials were divided into four types; 100 % cotton, 100 % polyester, polyester/cotton blends, and other materials, after which they were quantified. For textile materials that did not report information on their labels, a Fourier transform infrared spectrophotometer (FTIR) Perkin Elmer, model Spectrum 65, was used. The spectra generated were compared with bibliographic references and with the database available in the research group [52].

In addition, the previously washed and dried selected materials were characterized. Elemental analysis was performed on a Thermo Scientific, model Flash 2000 and the proximate analysis was carried out by thermogravimetry (TGA) in a Perkin-Elmer STA 6000 thermobalance.

2.2. Pyrolysis reactor and operation conditions

The pyrolysis experiments were carried out with a laboratory-scale fixed tubular horizontal reactor made of 316 stainless steel (internal diameter: 4 cm and length: 34.25 cm) inserted in a Naberthem furnace, model R50/250/12. Approximately 20–30 g of the textile sample, previously cut to an approximate size of 1–2 cm, were placed in a stainless-steel crucible under an inert atmosphere of N_2 (100 mL/min), heating rate being 10 °C/min and a residence time of 90 min. The pyrolysis temperatures used were 425, 500, and 575 °C. These temperatures have been chosen taking into account the thermogravimetric analysis of the samples (data not included) and the results obtained in the study of other similar materials [53]. The volatile gas generated by the pyrolysis passes through a condensation system. The condensable fraction (oil) was collected in bottles, the non-condensable (gaseous) in inert tedlar bags, and the residual solid was collected in sealed bags for subsequent analysis, as described in previous work [54].

2.3. Analysis of pyrolytic products

2.3.1. Solid fraction

Elemental analysis (CHNS-O) was performed on a Thermo Scientific, model Flash 2000. The combustion temperature was 1400 °C and the gases generated passed through a copper bed at 860 °C, to capture excess O_2. The fraction components are separated in a chromatographic column and transferred to a thermal conductivity detector. To determine the percentage of moisture, volatiles and ash present in the different chars, a proximate analysis was carried out by thermogravimetry (TGA) in a Perkin-Elmer STA 6000 thermobalance. Between 20 and 30 mg of solid samples were deposited in an alumina crucible. The flow rate used throughout the analysis was 20 (mL/min). At the end of the heating at a temperature of 800 ºC, O_2 was introduced to provoke combustion and determine the final residue or ash.

Textural and surface properties were evaluated by adsorption-desorption isotherms in a static physisorption analyzer, model Sync 200 from 3 P Instruments©. First, the samples were degassed at 110 °C for 12 h in a 3 P Instruments© Prep J4 unit under vacuum. For the determination of the total specific surface area of the material (S_{BET}), the Brunauer-Emmett-Teller (BET) method was used [55], with N_2 as an adsorbent gas at a temperature of −196 °C. The total pore volume (V_T) was quantified from the N_2 uptake at $p/p_0 \approx 0.99$. The t-Plot method [56] was applied to determine the specific surface area of the micropores (S_{MP}) and the volume of the micropores (V_{MP}).

2.3.2. *Oil fraction*

To design the simulated distillation curves following the ASTM D2887 procedure [57], was used in a PerkinElmer Clarus 590 gas chromatograph (GC) coupled to a flame ionization detector (FID). The sample was injected directly into an ELITE 2887 dimethylpolysiloxane capillary column (Length: 10 m, internal diameter: 0.53, film thickness: 2.65 µm). The identification of the compounds contained in the pyrolysis oil was performed on a gas chromatograph coupled to triple quadrupole mass spectrometry (GC-MS) Agilent brand, model 7890 A, with a Phenomenex ZB-5MS capillary column (Length: 30 m, internal diameter: 0.25 mm, film thickness: 0.25 µm). An injection temperature of 250 °C, split mode, and a flow rate of the mobile phase (carrier gas: HE) of 1 mL/min was set. Detector conditions were set at an interface temperature of 250 °C and electron ionization energy of 70 eV. Compound identification was based on the National Institute of Standards and Technology mass spectral library using NIST MS Search 2.0 software integrated with MassLynx V4.1 software with NIST 08 mass spectral library.

2.3.3. *Gaseous fraction*

The pyrolysis gases produced at 575 °C were analysed, and the calorific values were calculated using the gas composition data from the

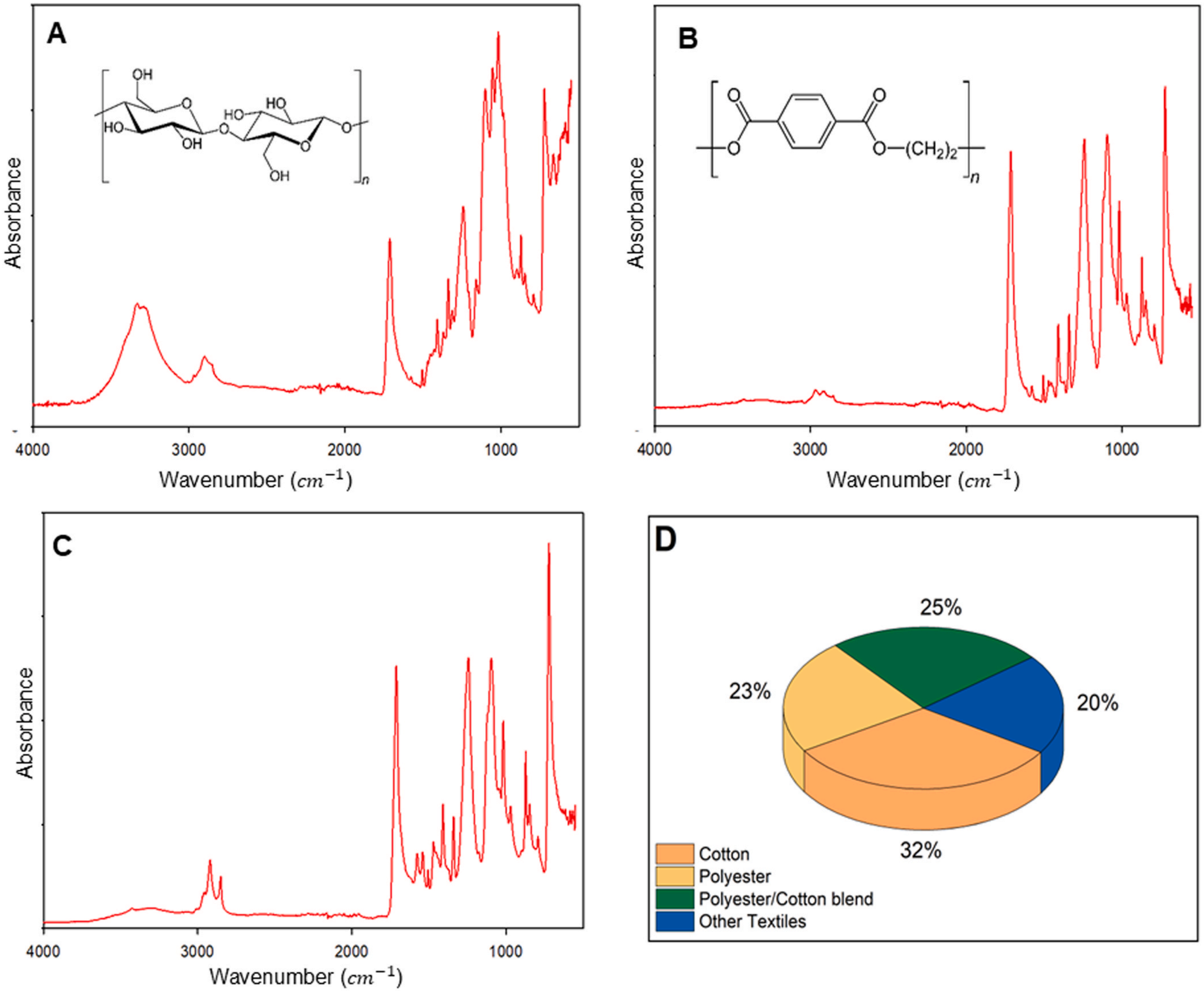

Fig. 1. . FTIR characterization and classification of the materials studied. (A) 100 % cotton, (B) 100 %polyester, (C) polyester/cotton blend (D)the quantification of material (wt%).

experiments. Analyses of the non-condensable gases were performed using an Agilent 990 microGC equipped with two channels and thermal conductivity detection. The first channel, which quantifies O_2, N_2, CO, and CH_4 gases, was connected to a 5 Å Molsieve column coated with a molecular sieve (length: 20.0 m internal diameter: 0.25 mm, film thickness: 0.3 μm). The second channel, which determines ethane, ethylene, CO_2, propane, n-butane, acetylene, and methyl acetylene, was connected to a PoraPLOT column (length: 10 m, internal diameter: 0.25 mm, film thickness: 8 μm). The results of the gas analysis were expressed as a volume percentage of each compound with respect to the total number of compounds analyzed on an N_2 free basis.

3. Results and discussion

3.1. Quantification and characterization of raw material

The raw material was classified based on the information recorded by the manufacturer on the textile labels and the FITR analysis, see Fig. 1 A-C, resulting in the classification of four categories; cotton, polyester, polyester/cotton blend, and other textiles. Typical bands of these materials are evident in the IR spectra. For example, the bands between 3500 and 3000 cm^{-1} are attributed to O-H stretching, and those between 1100 and 1000 cm^{-1} are assigned to C-O stretching, typical of cellulose fibers [58]. In the IR spectra of polyester, the characteristic bands are observed between 1750 and 1715 cm^{-1} assigned to C=O stretching (ester) and between 1250 and 1150 cm^{-1}, assigned to C-O stretching (ester) [58].

Next, the quantification according mentioned category is depicted in Fig. 1D, showing that 32 % corresponded to textiles made in their vast majority with cotton (>90–95 %), 23 % were polyester (>90–95 %), 25 % were polyester/cotton blends and the remaining 20 % were other textiles, which include a variety of less common but equally important materials in the manufacture of textile products, including polyamide, elastane, nylon, linen and wool among others. The Granada municipal waste treatment plant receives approximately 18,000 tons of textile waste per year, an approximate average value of 19 kg/inhabitant and year of textile waste. This value coincides with the average for Spain [8] and it is slightly higher than the European average estimated at 11 kg/inhabitant and year [59].

Taking into account this amount of textile waste, around 5760 tons per year of cotton textile waste, 4040 tons of polyester textile waste and 4500 tons per year of cotton-polyester blend textile waste could potentially be treated by pyrolysis.

Regarding the moisture and dirt content of the textile waste, an average moisture content of 16.5 % and dirt content of 9.4 % were obtained.

According to the results found, three types of textile material, pure cotton, pure polyester, and a mixture of 55 % polyester and 45 % cotton have been selected for the pyrolysis test. Textile waste with a fiber ratio of 45 % cotton and 55 % polyester (shown on the label) was the most frequently found in the quantification carried out. Other researchers have used similar cotton and polyester mixtures [60,61].

Table 1 summarizes the results of elemental and proximate analyses of textile waste feedstock. The elemental analysis showed that the textile waste samples mostly was composed of carbon (cotton, 41.57 % and polyester, 60.15 %) and oxygen (cotton, 49.39 %, and polyester, 33.80 %). Significant amounts of hydrogen and a low ash content (>2 %) were also found. The contents of nitrogen and sulfur were negligible. The proximate analysis confirmed that the textile wastes mostly consisted of volatile matter (>82 %).

3.2. Effect of pyrolysis conditions on the products' distribution

The main fractions obtained from the pyrolysis of any material are gas, oil, and char. However, the amount of each of them depends directly on the pyrolysis conditions and raw material used. It has been reported that at temperatures above 600 °C, the majority fraction is the gaseous one, and at temperatures below 300 °C it generates high percentages of solid [18]. During the pyrolysis process, the cotton, polyester, and cotton/polyester blend are subjected to chemical reactions that impact the product yields. At lower temperatures, the initial thermal decomposition produces mainly char solid and heavy oils due to the breaking of weaker bonds and the formation of complex intermediate structures [62]. As the temperature increases, the breaking of stronger bonds and the complete depolymerization of macromolecules due to the greater thermal energy available, produces an increase in the yield of liquids and gases [63]. In the case of polyester, pyrolysis at elevated temperatures promotes the formation of volatile monomers and oligomers, as well as light gases such as CO, CO_2, and CH_4 [64]. On the other hand, cotton, composed mainly of cellulose, also produces more oil and gases due to the decomposition of cellulose into volatile compounds and subsequent thermal cracking [65]. Fig. 2A shows the results of the pyrolysis of 100 % cotton. The majority fraction obtained was the oil, with yield over 45 %, followed by a 30 % gaseous fraction and the minority was the solid fraction, i.e. less than 20 %. When evaluating the quantities obtained as a function of the working temperature, it is observed that as the temperature increased from 425 °C to 575 °C, the solid fraction decreased from 22 % to 16 %, while the gaseous fraction increased from 29 % to 34 %. These results are in accordance with the literature [66]. The higher amount of oil is due to the chemical composition of cotton. This material is mainly cellulosic, a polymer formed by repeated glucose units.

Fig. 2B shows the pyrolysis results at the three working temperatures, using pure polyester as the feedstock. For the three working temperatures, the predominant fraction was the gaseous fraction, yielding over 50 %, while the oil and gaseous fractions did not exceed 30 %. The temperature increase favors the production of gaseous fraction, rising from 49 % (475 ºC) to 54 % (575 ºC), unlike oil whose yield did not vary under the change of this parameter.

The results obtained from the pyrolysis of polyester/cotton blends, see Fig. 2C, show that the predominant fraction was the gaseous fraction, around 49 %, followed by the oil fraction (30 %) and finally the solid fraction (20 %). It is noticeable that the temperature rise promotes the gas yield and decreases the solid yield, i.e. 23 % (475 ºC) to 19 % (575 ºC), while the oil fraction maintains its yield stabilized for the three working temperatures. These results are supported by the literature, which indicates that increasing the pyrolysis temperature increases the pyrolytic gas yield and decreases the residue yield [15,67].

In this context, Joo et al. [67] studied the pyrolysis of denim jeans waste composed of approximately cotton (~90 %), polyester (~8 %), and elastane (1–2 %), varying the pyrolysis temperature from 400 to 800 ºC. The authors found that an increase in temperature produces a

Table 1
Proximate and elemental analysis of the textile wastes.

Material	Proximate Analysis (%)				Elemental Analysis (%)				
	Moisture	Volatiles	Fixed Carbon	Ash	C	H	N	S	O*
Cotton	3.72	87.76	6.90	1.61	41.57	6.02	0.66	0.75	49.39
Polyester	0.00	84.05	14.12	1.83	60.15	4.22	0.00	0.00	33.80
Cotton/Polyester blend	2.52	82.70	13.04	1.71	48.78	5.63	0.23	0.00	43.65

*By difference

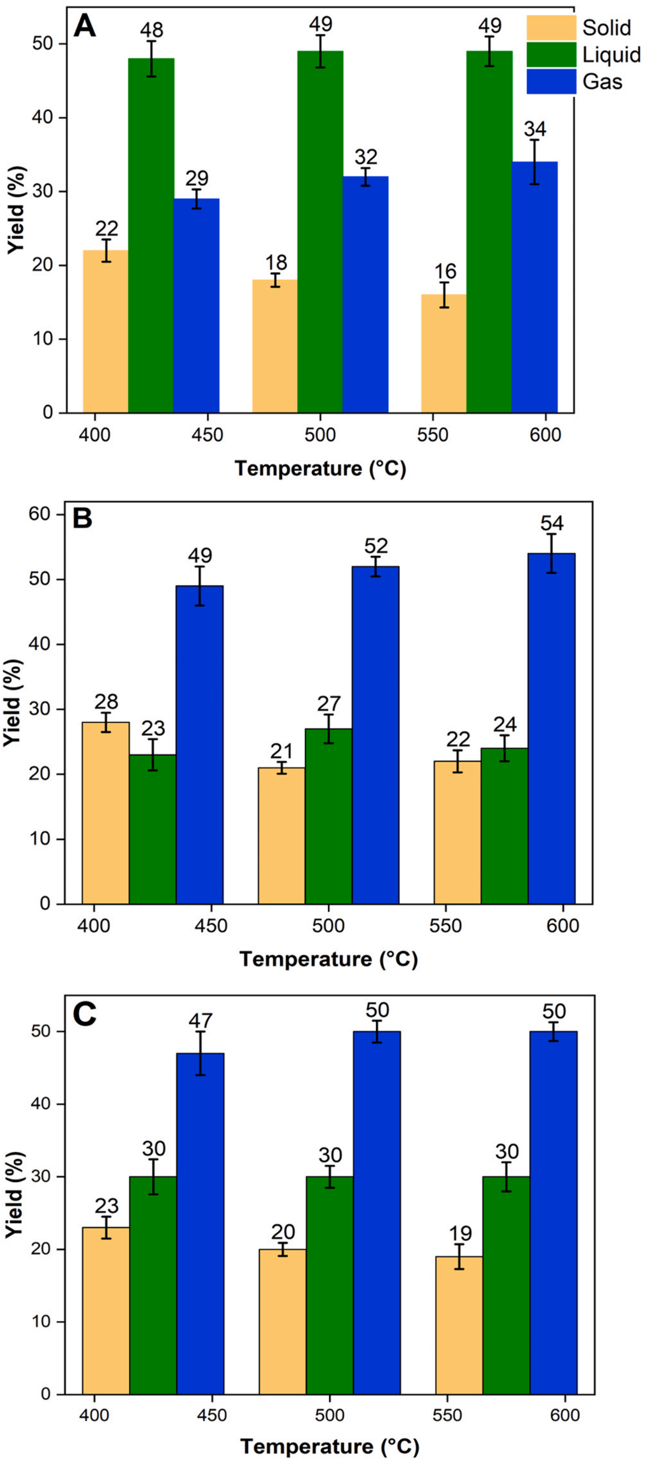

Fig. 2. Pyrolysis product yields of textile waste composed of (A) 100 % cotton, (B) 100 % polyester, (C) polyester/cotton blend.

higher percentage of gaseous fraction and decreases the formation of oil and char, which indicates that the release of volatiles and their thermal degradation is promoted by increasing the temperature of the process. Lee et al. [18] reviewed textile waste recycling using pyrolysis technology. They highlight that, the yields of pyrolysis products depend mainly on the raw material and operating conditions. The authors included a table in their work summarizing the pyrolysis product yields reported by various researchers studying different textile waste feedstock. They noted that gas yields vary from 2 % to 80 %, oil yields range between 14 % and 74 %, and solid yields are between 30 % and 50 % in most cases depending on factors such as the type of textile waste, the pyrolysis conditions, the use of catalyst and the type of reactor.

Moreover, if the product yields percentages and the amount of textile waste available in the province of Granada are considered, approximately 5162 tons of oil and 2004 tons of char could be produced annually. These quantities could be further increased by processing textile waste generated in other regions of Spain.

3.3. Characterization of chars

The proximate and elemental analyses of the chars are shown in Table 2. The behavior of volatiles and fixed carbon in the three materials is similar. In the case of volatiles, their percentage tends to decrease as the working temperature increases, while fixed carbon is inversely proportional to temperature. The literature reports similar results in hemicellulose, cellulose, and lignin materials [68]. The humidity results show different behavior in the three materials. In the case of cotton, the humidity decreased as the pyrolysis temperature increased and, in the polyester/cotton blends it increased initially with temperature and then decreased again. This could

be explained by a possible post-pyrolysis moisture reabsorption produced by the additives and stabilizers that some polymeric materials such as polyester may contain [69]. The highest amount of carbon was obtained with cotton textile residues at 575 °C leading to 75.6 %, while the lowest was performed by the polyester/cotton mixed samples at the same temperature. A residual amount of nitrogen content is generally observed for all materials at all three working temperatures. The hydrogen content remained low regardless of the temperature at which pyrolysis was carried out. This could have been due to the easy volatilization into compounds such as H_2, CH_4, and water during the pyrolysis process, especially as the temperature increased in the early stages while the carbon content decreased. This decrease in carbon content for the cotton-polyester blend can be explained by the appearance of new oxygenated functional groups [70], or that at elevated temperatures more complex bonds can be broken and generate carbon-containing gaseous products such as CO, CO_2, and other hydrocarbons [71]. The sulfur content in polyester char and polyester/cotton blends is directly proportional to temperature. This result is attributed to the presence of additives and impurities contained in the synthetic fibers [72].

The results of the textural characterization by N_2 adsorption-desorption isotherms are shown in Fig. 3. According to the IUPAC classification of physisorption isotherms [73], the three fractions of solids can be classified as type I. It is a concave isotherm concerning the p/p_0 axis and the adsorbed amount approaches a limiting value. This result suggests the presence of narrow micropores, opening up possibilities of being used in various areas, including pollutant gas elimination and water treatment [52,74–76]. Table 3 shows that the pyrolysis temperature significantly impacts the textural properties of chars derived from cotton, polyester, and a 45 % cotton-55 % polyester blend. For cotton chars, increasing the temperature enhances the surface area (S_{BET}), micropore area (S_{MP}), and pore volumes (V_T and V_{MP}), with the highest values observed at 575°C. Polyester chars exhibit minimal textural properties at 425°C, but these properties increase substantially at 575°C. The 45 % cotton-55 % polyester blend chars display negligible properties at 425°C, yet at 500°C and 575°C, they surpass the textural properties of both pure cotton and polyester chars. This suggests that higher temperatures and blending materials can enhance the development of porous structures in pyrolyzed chars.

3.4. Characterization of oil

Fig. 4 shows the simulated distillation curves of the oil for the three types of materials. In addition, distribution of fractions obtained from distillation curves of pyrolysis oils are shown in Fig. 5. The results reflect

Table 2
Proximate and elemental analysis of the char fraction.

Sample	Temperature (°C)	Proximate analysis (%)				Elemental analysis (%)			
		Moisture	Volatile	Fixed carbon	Ash	C	H	N	S
Cotton	425	2.5	40.7	55.0	1.7	65.1	0.4	0.3	2.0
	500	1.5	30.6	64.3	3.6	67.8	2.5	0	0
	575	0.7	12.2	84.7	2.4	75.6	2.2	0	0
Polyester	425	11.0	18.6	68.7	1.7	67.1	2.5	0.3	3.9
	500	1.1	18.4	69.9	10.6	68.9	3.3	0	7.8
	575	12.2	8.3	76.6	2.8	55.9	2.2	0.2	7.8
Polyester/cotton blend	425	7.3	37.0	52.2	3.5	72.6	3.6	0.3	3.2
	500	15.4	13.4	70.0	1.2	58.5	2.4	0.4	5.2
	575	12.6	9.7	73.5	4.1	54.5	1.7	0.3	6.8

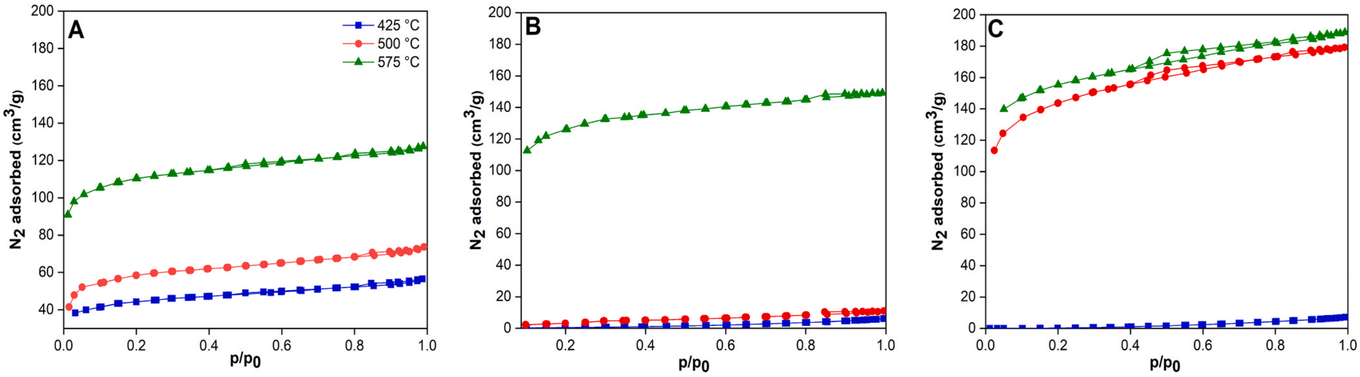

Fig. 3. Adsorption-desorption isotherms with N_2 at −196 °C of chars obtained in the pyrolysis of (A) 100 % cotton, (B) 100 % polyester, (C) polyester/cotton blend.

Table 3
Textural properties of chars obtained in the pyrolysis of 100 % cotton, 100 % polyester and 45 % cotton-55 % polyester blends at different temperatures.

Sample	Temperature (°C)	S_{BET} (m^2/g)	S_{MP} (m^2/g)	V_T (cm^3/g)	V_{MP} (cm^3/g)	V_{MP}/V_T (%)
Cotton	425	138.5	109.4	0.087	0.057	65.5
	500	192.8	157.1	0.114	0.075	65.8
	575	357.1	309.9	0.197	0.151	79.0
Polyester	425	2.7	0	0.009	0.000	0.0
	500	29.8	4.3	0.032	0.001	3.1
	575	404.6	321.8	0.231	0.161	69.7
Polyester/ cotton blend	425	0.8	0.0	0.011	0.000	0.0
	500	442.6	319.4	0.277	0.179	61.3
	575	488.0	385.4	0.292	0.197	67.4

S_{BET}: total area by BET method; S_{MP}: micropore area by t-plot method; V_T: total pore volume obtained at p/p_0~0.99; V_{MP}: micropore volume by t-plot method

that the volatilization temperature is higher in the fractions from polyester waste at all three working temperatures. The shift to the left of the three curves (425 °C, 500 °C, and 575 °C) indicates the reduction of light naphtha and the increase of kerosene (> 70 %) compared to the baseline product distribution observed at lower pyrolysis temperatures.

In the distribution graph of the fractions corresponding to the three types of material portrayed in Fig. 5, the polyester sample at 575 °C is the only one where the presence of Heavy vacuum Gas Oil (HVGO) is observed with 4 % of the total of this fraction. Also, the oil from polyester stands out with a high percentage of kerosine, decreasing with the pyrolysis temperature, and Light Vacuum gas oil is generated. On the other hand, the majority fraction, in the curves from cotton and polyester/cotton blends, corresponds to heavy naphtha (>30 %) and a small amount (<5 %) to distillate fuel oil. It is observed that at higher temperature there is an increasing trend in the production of heavy fractions. The results indicate that by adjusting the pyrolysis temperature, the production of specific fractions can be optimized, which is useful for industrial applications requiring products of interest. These results are consistent with similar investigations of pyrolysis of cotton/polyester blends in which aliphatic hydrocarbons have been detected [6].

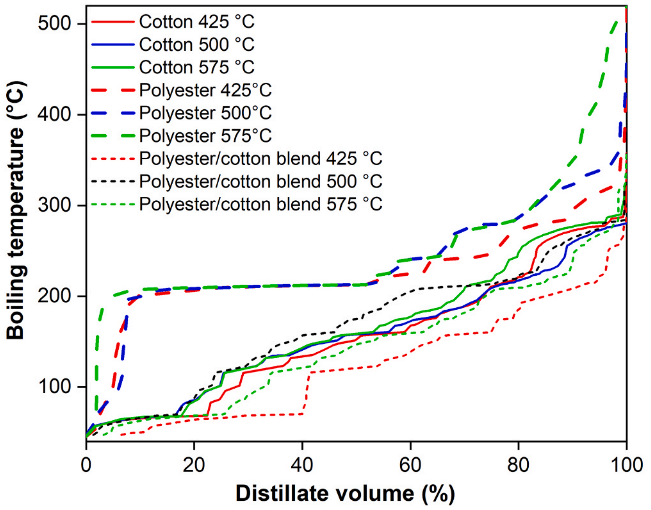

Fig. 4. Simulated distillation curves for pyrolysis oils.

The compounds contained in the pyrolysis oil were identified by GCMS, see Fig. 6. D-Glucosamine was the analyte with the highest percentage area (>50 %) for the oils obtained from cotton textile samples at the three working temperatures (Fig. 6**A**). 2,6-pyridinadicarboxylic acid or also known as dipicolinic acid was detected in polyester oil samples (Fig. 6**B**), at low temperatures (425 °C) the percentage exceeded 50 % and a decrease was observed when the temperature increased. The remaining major compounds present are primarily benzene derivates. In the case of oils obtained from polyester/cotton blends, the analyte with the highest percentage area was D-allose (49.6 %), indicating thermal

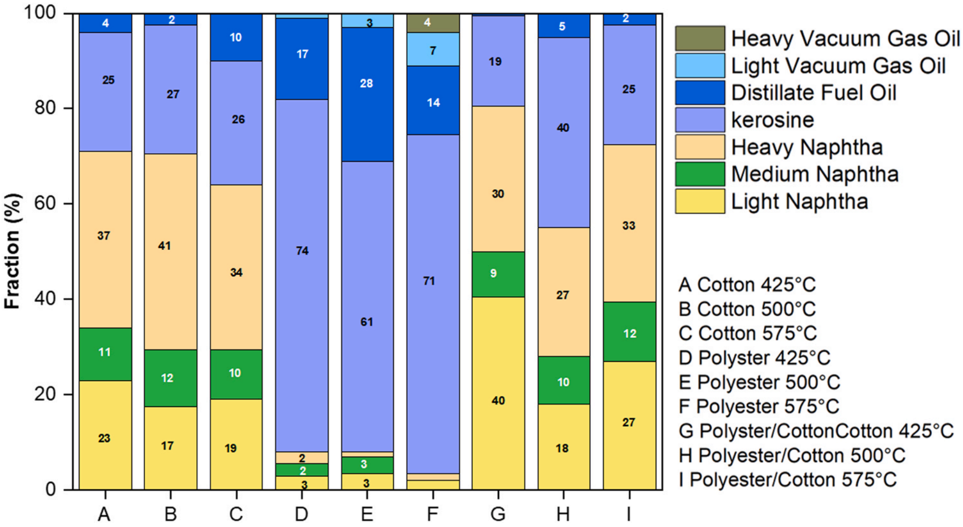

Fig. 5. Distribution of fractions obtained from distillation curves of pyrolysis oils.

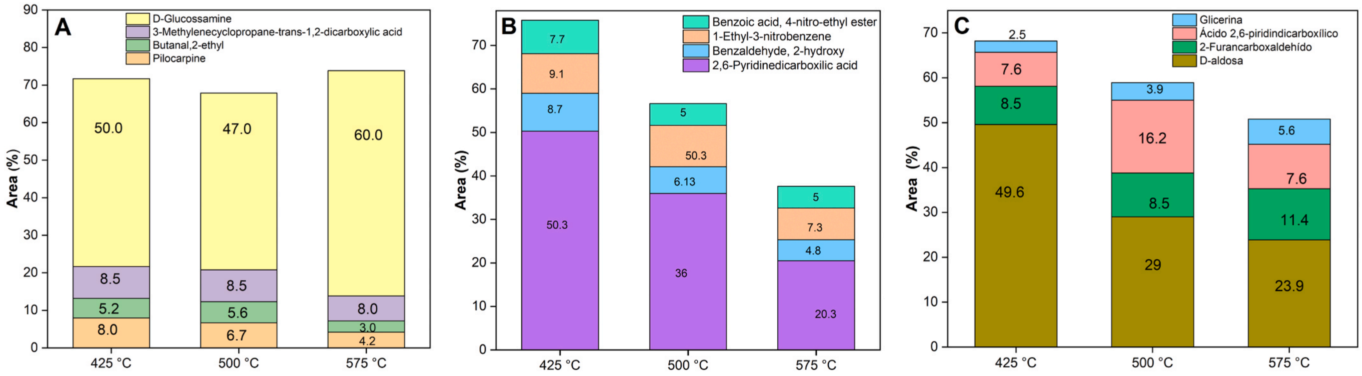

Fig. 6. Compounds with higher percentage area in oils of (A) 100 % cotton, (B) 100 % polyester, (C) polyester/cotton blend.

decomposition of cotton cellulose. Other important compounds include furfural derivatives (such as 2-furancarboxaldehyde), which are typical products of hemicellulose decomposition. These results are consistent with the literature on the pyrolysis of cellulosic materials and cotton, where GC-MS analysis is widely used to identify and quantify degradation products [77,78]. Rittfors [6] found that the chromatography analysis of the pyrolysis oil of cotton textile waste revealed compounds derived from furans, anhydrous sugars, and phenolic compounds. Likewise, benzoic acid and its derivatives are among the main compounds found in the pyrolysis oil of polyester.

Yousef et al. [79] identified compounds such as alcohols, furfural, ethanol, ethane, and acetophenone in the pyrolysis oil of cotton and polyester blends. Similarly, Kwon et al. [50] valorized synthetic textile waste through pyrolysis and found that the main constituents of the condensable gases were benzene, toluene, styrene, benzoic acid, and naphthalene. Joo et al. [67] studied the pyrolysis at various temperatures of denim jeans waste formed by cotton (~90 %), polyester (~8 %), and elastane (1–2 %) and found that the pyrolysis liquid is a mixture of compounds such as acids, alcohols, aldehydes, ketones, esters, furans, dioxolanes, hydrocarbons, sugars, among others and this composition is independent of the pyrolysis temperature.

Other authors have demonstrated the effectiveness of pyrolysis for the recovery of valuable chemicals from textile waste [80,81]. For example, Yang et al. [80] studied the pyrolysis of nylon stockings for the recovery of caprolactam (nylon monomer), among other products, using γ-Al_2O_3 supported metal catalysts (Ni, Cu, Fe, or Co). The authors found yields in obtaining caprolactam close to 95 % concerning the nylon content, demonstrating that it is possible to convert a textile waste into a high-added value product.

3.5. *Gaseous fraction*

The temporal evolution composition of the non-condensable pyrolysis fraction (N_2 free basis) for the three materials is shown in Fig. 7. In addition, Fig. 8 shows the number of non-condensable gas products generated by the degradation of textile waste expressed in mmol after 1 hour. The gas composition trends differ between the materials. The gases with the highest molar percentage correspond to $CO_2>CO>H_2>CH_4$ and other light hydrocarbons in the polyester and cotton/polyester blend pyrolysis. However, the methane produced by cotton pyrolysis is higher than hydrogen. The production of CO_2 and CO is high in the three materials and consistent with the decomposition of cellulose and polyester, which tend to decompose into these gases due to the oxygen-rich structure, as has been indicated in its elemental analysis. Methane formation using cotton samples is higher than polyester samples, as opposed to H_2 which in fractions from polyester exceed cotton samples over time. This is mainly due to the organic structure. Cotton, being a natural fiber composed of cellulose, a glucose polymer, has a greater tendency to decompose into simple compounds, due to its greater availability of C-H atoms. Polyester, however, is a synthetic fiber composed of polymers derived from organic acids such as terephthalic acid and the alcohol ethylene glycol, with ester bonds that are more complex to break than cellulose. Therefore, when subjected to high temperatures, it generates H_2. Fig. 7 shows that, in general, as time

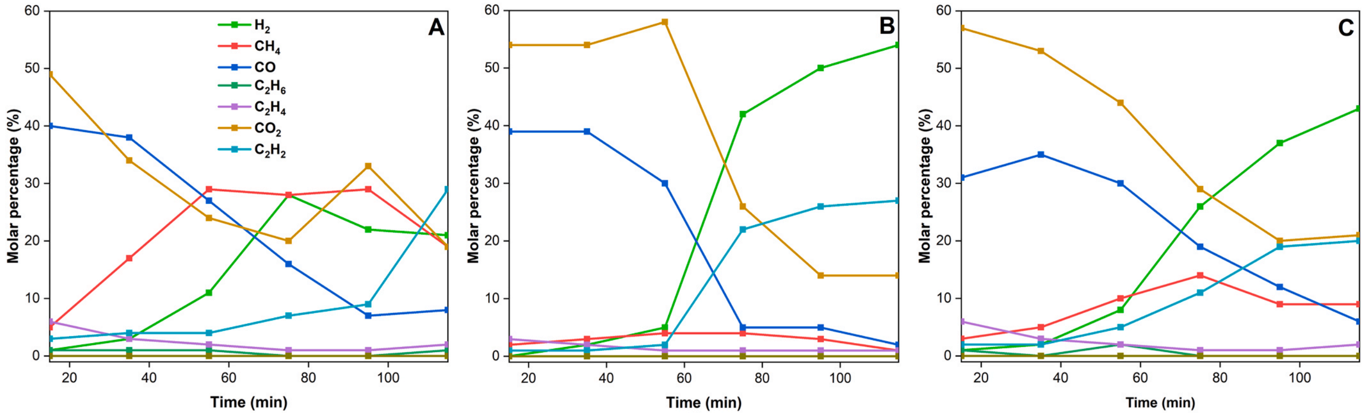

Fig. 7. Temporal evolution of gas composition (% molar) at 575 ºC for (A) 100 % cotton blend, (B) 100 % polyester, (C) polyester/cotton blend.

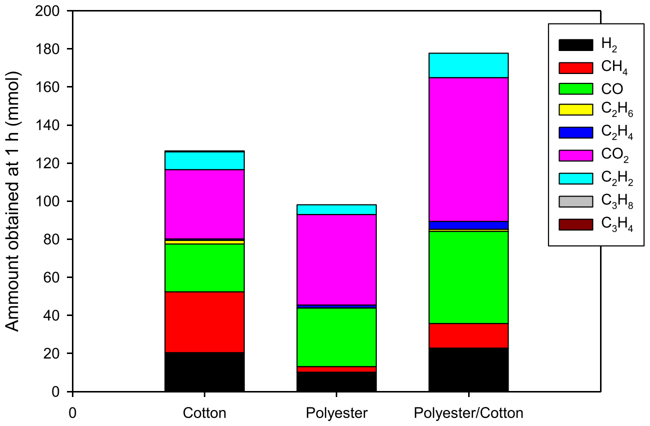

Fig. 8. Amount of gas products obtained by thermal pyrolysis of textile waste at 575°C.

evolves the percentage of CO and CO_2 decreases and the formation of light hydrocarbons such as CH_4 and acetylene is triggered, as reported in other investigations 83–84]. In general, high amounts of CO_2, CO and H_2 are observed after 1 h which can justify the gaseous by-product of pyrolysis as one of high added value.

Pyrolytic gas can be used as direct fuel and/or raw material for the synthesis of other hydrocarbons and alcohols [14]. In this work, the pyrolytic gas obtained had a higher heating of 16.25 MJ/m^3 for cotton, 8.89 MJ/m^3 for polyester, and 12.09 MJ/m^3 for the cotton-polyester mixture. Lee et al. [18] reviewed the literature and reported that the pyrolysis of textile waste typically produces gas with higher heating values ranging from 2 to 10 MJ/m^3. Czahczyriska et al. [82] showed in a study that the pyrolysis of textile waste from municipal solid waste yields a gas with an average heating value of 15 MJ/m^3.

Fig. 8 shows the gas amounts produced after 1 hour, measured in mmol, from the thermal pyrolysis of textile waste at 575 ºC. Results show that cotton and polyester/cotton blend tend to produce more valuable fuel gases such as methane (CH_4) and hydrogen (H_2), among others, than polyester. Hydrogen (H_2) is produced in higher amounts by the polyester/cotton blend, followed by cotton, with polyester generating the least. Carbon monoxide (CO) is predominantly produced by polyester/cotton blend, with lower levels from cotton. All textile wastes also produce significant carbon dioxide (CO_2), which aligns with its higher oxygen content and other published works [78–81,83,84,82,85]. Lastly, polyester and the blend generate higher amounts of acetylene (C_2H_2), propane (C_3H_8), and propylene (C_3H_4) compared to cotton.

Some studies have shown that co-pyrolysis of textile materials can reduce the formation of oxygenated products and increase the production of monocyclic aromatic hydrocarbons (MAHs), which is beneficial for obtaining valuable chemicals. For example, Peng et al. [86] investigated the interaction between the volatiles of polyester and viscose over ZSM-5 and found an improvement in the selectivity and quality of the products obtained.

In the case of pyrolysis of the cotton and polyester blend, synergistic interactions can also occur that improve the quality and performance of the products. Thus, perhaps the free radicals generated during the decomposition of cotton can induce the fracture and depolymerization of the polyester, reducing its initial decomposition temperature [87]. On the other hand, the presence of polyester can catalyze the decomposition of cellulose, resulting in greater production of valuable products [88].

4. Conclusions

This study highlights the potential of the pyrolysis process as a method for recycling textile waste that is commonly sent to landfills.

Pyrolysis generated three main fractions, char, oil, and gas. The yield distribution depended on the temperature condition and the type of textile waste used. Pure cotton predominantly yielded oil, varying very little with the process temperature. However, polyester and cotton/polyester blend yielded more gas, especially at higher temperatures.

The chars produced were classified as type I according to IUPAC physisorption isotherms, indicating the presence of narrow micropores. The pyrolysis temperature significantly affected the textural properties with higher temperatures improving the textural properties of the chars.

The analysis of the pyrolysis oils showed that the oil from polyester had a higher volatilization temperature at the three working temperatures and was mainly composed of kerosene (>70 %). In contrast, oils from cotton and cotton/polyester blend were rich in heavy naphtha (>30 %) and presented a small amount (<5 %) to distillate fuel oil. The distribution of the components of the oil was strongly related to temperature, so at high pyrolysis temperatures there is a tendency to produce heavy fractions, therefore, the composition of the oil can be tailored by adjusting the pyrolysis temperature.

The gas fraction from cotton and polyester/cotton blend contained higher valuable fuel gases such as methane (CH_4) and hydrogen (H_2), among others than that from polyester. In general, high amounts of CO_2, CO, H_2, and CH_4 were observed after 1 h, making the gas a high-value product.

The obtained results open the possibility of using pyrolysis as a textile recycling method, contributing to energy recovery and the circular economy. However, the process requires significant energy and can produce harmful emissions if not properly managed. Therefore, the process must be carefully managed to minimize energy consumption and emissions. In addition, future research should focus on optimizing reactor design, scaling up the process for industrial applications, and exploring a wider variety of textile waste compositions.

CRediT authorship contribution statement

Lourdes Arjona: Writing-original draft, Methodology, Investigation; **Irene Barrós:** Investigation, Data curation; **Álvaro Montero:** Investigation, Data curation; **Rafael R. Solís:** Writing-original draft; Writing-review & editing, Investigation, Methodology, Formal analysis; **Antonio Pérez:** Writing-review & editing; Methodology, Formal analysis; **María Ángeles Martín-Lara:** Writing-review & editing, Supervision, Investigation, Funding acquisition; **Gabriel Blázquez:** Supervision, Methodology, Investigation, Conceptualization; **Mónica Calero:** Writing-review & editing, Supervision, Resources, Funding acquisition, Formal analysis.

Declaration of Competing Interest

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

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Data availability

Data will be made available on request.

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