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An investigation into the minimum energy requirements for transforming end-of-life cotton textiles into carbon fibre in an Australian context

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ABSTRACT

The work presents on the relationship between temperature, time and carbon content in the preparation of a carbon fibre cloth from end-of-life cotton textiles. The aim of this investigation was to identify the minimum energy requirements for this textile recycling opportunity. The composition of the carbon fibre was studied using elemental combustion instruments and X-ray fluorescence. The structure of the carbon fibres was studied through scanning electron microscopy and X-ray diffraction. Through this process it was determined that the optimum temperature and time requirements were 1150 °C and 30 min to prepare a carbon fibre under the conventional definition, which requires carbon content to be in excess of 90%. The minimum temperature and time requirements for a lower grade carbon fibre of 80% carbon content are 650 °C and 30 min. This research can support efforts to improve circularity of cotton textiles for high value applications in environmental management or electronic markets. This in turn could support these industries to reduce their carbon footprint and meet their sustainable procurement targets for greater uptake of recycled content materials.

1. Introduction

The global fashion industry accounts for 4% of greenhouse gas emissions, which is equivalent to the annual emissions of the United Kingdom, France and Germany combined (McKinsey & Company, 2020, p. 3). This industry is also responsible for 20% of global industrial water pollution, from textile treatment and dying processes (World Bank, 2014, p. 13). This pollution has a direct impact on the health of workers, neighbouring communities and threatens water security (World Bank, 2014). Despite the enormity of this environmental and social sacrifice, this resource is rarely recycled at end-of-life, and the cycle repeats indefinitely to growing international demand (Ellen Macarthur Foundation, 2017).

Australia is the third largest cotton exporter (Cotton Australia, 2020) and farmers persevere through frequent and prolonged drought only to see 7% of their end product recycled and the balance (725,000 tonnes) disposed to landfill (Blue Environment, 2020, p. 45). This resource is worth more than a mass burial (Lamb, 2016).

Domestic resource recovery is a government priority and incentivised by the landfill levy. However, Australian recycling efforts are constrained by limited local demand for materials due to the small manufacturing market, and competition with inexpensive imported goods.

Recent global supply chain issues, highlighted Australia's reliance on importation of manufactured goods. According to analysis by Dr Jim Stanford and the Australia Institute's Centre for Future Work, "*Australia has one of the most underdeveloped manufacturing sectors of any industrial country in the world*" (Pupazzoni, 2020).

As shown in Fig. 1, many of the processes in the cotton textile supply chain occur overseas, which means system interventions are limited to the primary production, retailing and consumption phases only. With little local manufacturing, efforts to improve circularity will require working with international spinners, mills and garment factories. A handful of Australian clothing and bedding brands already have these relationships to meet growing consumer demands for cotton textile circularity. The more common recovery pathways are through re-use, repair and re-purposing for other applications such as bags, rags and animal bedding (Fig. 1). As previously noted, this amounts to 7% recovery of all textile waste generated. Contextually appropriate and financially viable solutions are required to increase the circularity of

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textiles in the Australian economy and divert more textile waste from landfill.

According to the Ellen Macarthur Foundation, the three principles to build a circular economy include:

- Eliminate waste and pollution
- Circulate products and materials (at their highest value)
- Regenerate nature (Ellen Macarthur Foundation, 2022 n.d.)
- The Ellen Macarthur Foundation's ambitions for the textile industry include:
- Phase out substances of concern and microfibre release
- Increase clothing utilisation
- Radically improve recycling
- Make effective use of resources and move to renewable inputs (Ellen Macarthur Foundation, 2017)

In the context of waste cotton textiles in Australia this means maximising use of the clothing or bedding items in the first place. This could be through durable design and branding commitments to extend the life of products and avoid the generation of waste. Fortuna and Diyamandoglu in *Optimization of greenhouse gas emissions in second-hand consumer product recovery through re-use platforms* define re-use as extending the life of a product or material by transferring it to new owners (Fortuna & Diyamandoglu, 2017). According to Sandin and Peters, methods of re-use include "*renting, trading, swapping, borrowing and inheriting, facilitated by, for example, second hand shops, flea markets, garage sales, online marketplaces, charities and clothing libraries*" (Sandin & Peters, 2018). Fortuna and Diyamandoglu categorise entities or platforms involved in re-use activities as 're-use enterprises', 'material exchanges', 'online platforms' and 'direct exchanges' (Fortuna & Diyamandoglu, 2017). Factors influencing their categorisation include ownership or rental arrangements, storage capacity, distances travelled, and vehicles used in the exchange process (Fortuna & Diyamandoglu, 2017). Re-use opportunities should be exhausted through permanent ownership transfer and 'collaborative consumption', which is defined as coordination of the acquisition and distribution of resources for a fee or other compensation (Belk, 2014).

In Australia clothing donation is the predominant means of keeping textile waste out of landfill (Australian Fashion Council, 2022). These clothing items are re-used locally and exported to international second-hand markets (Australian Fashion Council, 2022)

When the items are no longer suitable for re-use, they could be recycled by mechanical, chemical, or thermal means (Sandin & Peters, 2018). The product could be a textile or non-textile product derived from pre or post-consumer textile waste (Sandin & Peters, 2018). Transformation of textile waste into a higher or lower value products is known as 'upcycling' and 'downcycling' (Sandin & Peters, 2018). 'Downcycling' into products such as animal bedding, rags or insulation material is undertaken in Australia. However, these products must compete with cheap imported products for price sensitive consumers.

As Australia is a cotton growing country rather than a textile manufacturing country, mechanical 'textile to textile' recycling requires export and offshore re-spinning as local capacity for fibre-to-fibre recycling is limited. Cotton fibres also shorten over time and therefore cannot be recycled as a fibre indefinitely (Echeverria, et al., 2019).

When fibre-to-fibre recycling are not (or no longer) viable, there are a range of 'upcycling' products that can be developed from end-of-life cotton textiles. These include fibre reinforced composites for building applications (Echeverria, et al., 2019) and acoustic panels (Echeverria, et al., 2018). However, these solutions are not currently being delivered at sufficient scale to meet net demand in Australia.

Sustainable procurement policies and sustainable building rating systems are driving demand for building products which are certified to have recycled content. Increased uptake of building products containing waste textiles is expected in the Australian waste industry. For increased financial resilience, the commodity should service multiple markets to withstand market fluctuations and cycles within the construction sector.

Cotton textile waste can also be composted and returned to land for nutrient cycling (Better Cotton, 2022). However, this solution is only viable when the composition of the textile is known and there are no risks associated with dyes and other contaminants present in the cotton textile waste.

A chemical recycling facility for textile waste is in the Australian infrastructure pipeline but capacity is insufficient to meet demand at a national scale and additional recycling solutions will be required. Thermal textile recycling is limited to energy recovery only through the production of refuse derived fuel typically for export markets.

This research paper examines the viability of end-of-life cotton textile as an alternate precursor to polyacrylonitrile (PAN) carbon fibre for certain applications. The research contributes to knowledge in this field by exploring the minimal energy needs to transform 'end-of-life' cotton into carbon fibre.

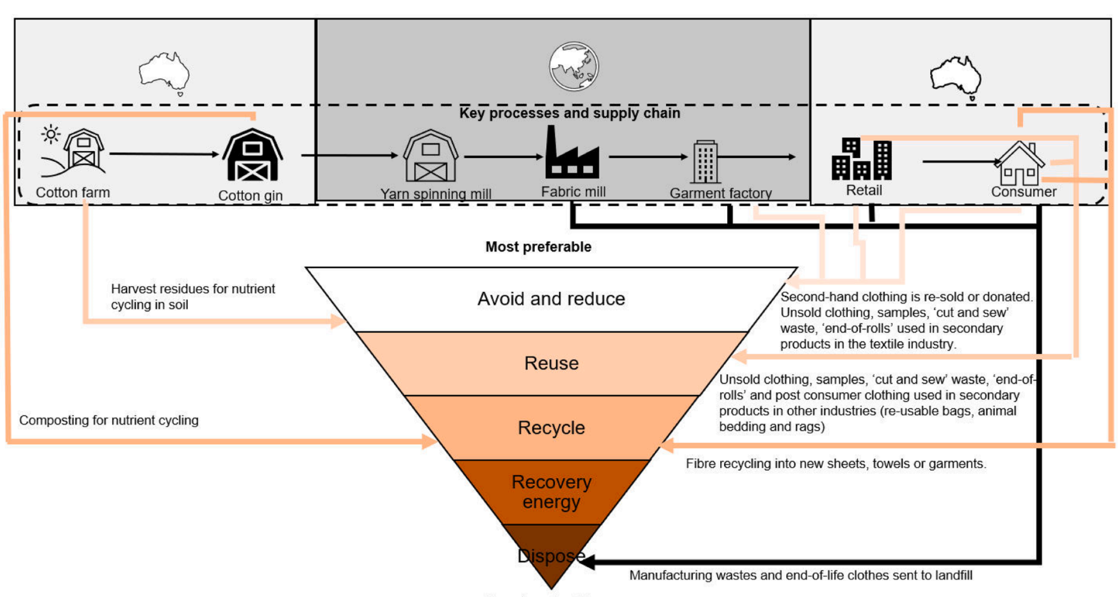

Fig. 1. Cotton textile waste flows and the waste hierarchy.

As previously discussed, this recycling method sits within a myriad of different approaches to cotton textile waste recycling and all higher order resource recovery opportunities should be maximised prior to this more energy intensive recycling process. In the context of the waste hierarchy, this approach is less preferable to avoidance, re-use or nutrient cycling. However, the potential to displace conventional carbon fibre in certain applications could be promising in terms of carbon abatement for the energy intensive carbon fibre market. This recycling method could support certain industries to reduce their carbon footprint and meet their sustainable procurement targets.

It must also be reiterated that only 7% of waste textiles are currently recycled in Australia despite many organistions working tirelessly to support avoidance, re-use and composting efforts. From a market perspective, demand for rags or compost is evidently insufficient to drive diversion of textile waste from landfill. The waste hierarchy is instructive but sacrifices the 'good' in pursuit of 'perfect' when applied to the waste and recycling infrastructure projects. With 725,000 tonnes of textiles being pushed into Australian landfills indefinitely, it is worth challenging conventional thinking in Australia and explore the idea of profit-making recycling endeavours displacing energy intensive materials in other industries. When 'supply push measures' are failing to gain traction, 'demand pull measures' need to be explored to accelerate Australia's progress against their circular economy ambitions.

The carbon fibre market is considered promising for end-of-life cotton textile offtake because it is strong, growing and has high barriers to entry which limits competition.

The carbon fibre market is expanding at a compounded annual growth rate of 9.9%, with the main driver being demand for light-weight materials in aerospace, defence, renewable energy, automotive, recreation and manufacturing industries (Bajpai, 2017, pp. 25–26). Over 90% of carbon fibres are made from polyacrylonitrile (PAN) and the balance petroleum pitch and regenerated cellulose (rayon) (Bajpai, 2017, p. 3). However, the origins of carbon fibre are in baked cotton which were used as filament in the first incandescent lightbulbs (American Chemical Society, 2003). Cotton fibres are the purest form of cellulose (Hsieh, 2006), which converts to carbon making this fibre a suitable precursor material.

Jagdale et al have demonstrated that waste cotton textile is a suitable precursor for carbon fibre through microstructural and physical property investigations (Jagdale et al., 2017). However, they have only demonstrated its viability for energy storage applications (Jagdale et al., 2021).

In the first study of their two studies, Jagdale et al transformed cotton textile of 100% purity into a carbon fibre through pyrolysis in argon at temperatures of 400 °C, 600 °C and 800 °C for 60 min. They undertook a number of tests to understand the microstructural and physical properties of the carbon fibres including thermo-gravimetric analysis, micro-raman analysis, nano-indentation, surface wettability, X-ray photoelectron spectroscopy and field emissions scanning electron microscopy. This study provided a framework for further testing of the relationship between temperature, time and carbon content, as well as for testing as a substitution for common carbon fibre applications

In the second study, the researchers found that cotton waste is a suitable precursor for carbon fibre lithium (Li^+)-ion batteries because of its favourable structure. The fibrous structure changes with increasing temperature but the two-dimensional structural orientation remains intact, and this intra and inter layer spacing is suitable for storing Li^+ ions (Jagdale et al., 2021). In addition to these promising electrochemical results, the product is foldable and therefore suitable for shapeable energy storage applications (Jagdale et al., 2021).

This study seeks to contribute to their research by identifying the minimum temperature and time parameters for thermal transformation to reduce the energy requirements of this novel textile recycling solution. This study also discusses the strengths and weaknesses of the carbon fibre for certain applications based on available literature and research findings.

Similar to built environment applications, a recycled carbon fibre would also need to compete in price and performance with other materials. In the renewables sectors, carbon fibre wind turbine blades need to compete in performance and price with glass fibre polyester blades (Wood, 2012). In the automotive sector, the competition between metals and plastics (Gardiner, 2014). Recycled carbon fibres are already on the market and they are typically derived from virgin carbon fibre from previous applications such as aircrafts. The fasted growing segment of the recycled carbon fibre market is 'chopped carbon fibre' (Wood, 2022). Industry requires continuous or discontinuous carbon fibres, and it makes sense to use waste rather than primary continuous carbon fibre to service the market for discontinuous or 'chopped' carbon fibre (Francis, 2019). Recycled carbon fibres are attractive from both an environmental and financial perspective, with suppliers charging 20–40% less than virgin product (Francis, 2019). The quality of the chopped recycled carbon fibres is proven to be of equivalent quality to the virgin product, so the only remaining barrier to overcome is supply at scale (Francis, 2019).

During the global pandemic, prices for all recycled commodities soared due to supply chain issues. These logistical challenges show that there is benefit in having both local and international suppliers for key resources. It enables business and consumers to have more options available to them in times of supply crises or peak pricing.

Implementation of this research in Australia could solve a lot of problems. It could:

- Satisfy increasing demand for locally made recycled content product.
- Increase Australia's local recycling and re-manufacturing capacity, which in turn increases domestic supply chain resiliency.
- Divert textile waste from Australian landfills.
- Increase circularity in the textiles industry.

This research initiative aligns with the principles of a circular economy because it maximises the value of the material after all other higher order resource recovery pathways have been exhausted. The process creates an advanced material from an end-of-life product, which is key to the financial sustainability and resilience of the initiative. Recycled products in Australia have had to compete with cheaper imported virgin products and this has limited the circularity of a number of material streams including glass and plastic bottles. The European regulators and the international business community are driving change and this will impact Australian markets. For the first time, the European price of recycled PET exceeded virgin PET due to increasing consumer demand for recycled content product, European Union (EU) taxes on virgin plastics and the United Kingdom (UK) levy on virgin plastics (Brooks, 2022). However, during this time supply chain challenges associated with the global pandemic would have also contributed to the higher prices. This buoyant market for recycled plastics driven by regulatory and voluntary initiatives serves as an example for other recycled commodity markets. Both regulatory and non-regulatory initiatives have the power to develop markets for recycled commodities such as waste textiles. This research is novel because it adds to the body of knowledge of use of waste cotton as precursor to carbon fibre by examining the relationship between thermal parameters and carbon content. Findings from this study may support research investigations by others into potential applications for carbon fibres in an Australian market.

2. Material and methods

The purpose of this section is to gain insights into the thermal transformation process of waste cotton textiles to carbon fibre. This information can be used by other researchers to optimise production methods for producing carbon fibres for appropriate applications.

2.1. Materials

The input material selected for this analysis were second hand personal protective uniforms. Workplace uniforms are not always suitable for re-use because they are branded or too worn out. Unforms were also selected because they offer homogeneity in composition which was needed for the analysis. The end-of-life work shirts were donated by a Sydney council. They were of 100% cotton and of a brand commonly used by councils, construction, mining and industrial organisations.

The shirts were processed as a woven fabric cut into the appropriate sizes to fit the different crucible types.

2.2. Thermo-gravimetric analysis

In order to understand how waste cotton reacts to different temperatures, Thermo-Gravimetric Analysis (TGA) was undertaken. The aim of this test was to measure the temperatures where the majority of reactions take place. In this case the dominant reactions are dehydration, volatisation and carbonisation.

Thermo-Gravimetric Analysis (TGA) is a process that measures the weight of a sample under increasing temperatures.

The TGA unit used for this analysis is a Perkin Elmer STA 800. The analysis was performed between 30 °C and 900 °C at a heat ramp rate of 20 °C/minute and under a nitrogen purge of 20 ml/minute.

2.3. Thermal transformation of waste cotton textile into carbon fibre

To enable testing of the different temperature and time parameters, three different furnaces were used in this study including a horizontal tube side furnace, muffle furnace and a dual chamber furnace. All thermal transformation processes took place in an inert environment of argon gas.

The horizontal tube was used for higher temperatures (>900 °C). Samples were placed in ceramic crucibles prior to loading into the ‘cold zone’ of the horizontal tube furnace. The samples were kept in the ‘cold zone’ for 10 min prior to transfer to the ‘hot zone’. After spending 10 – 30 min in the ‘hot zone’ the samples transferred back to the cold zone for a further 10 min.

For both muffle furnace and the dual chamber furnace, samples were placed between two ceramic crucibles. According to Jagdale et al “the controlled stress allows the fibres to shrink uniformly but also protects the shape and orientation of the cotton fabric during the pyrolysis process”.

The heat ramp was 5 °C/minute to the target temperatures of testing. The samples were then held for periods of 30 min for the first phase of sampling, which involved optimising the temperature parameters. The second phases of sampling involved modifying this period for the purposes of optimisng time parameters. The samples remained in the furnace as the temperature reduced down to the ambient temperature.

In one experiment, the samples were inserted into the dual chamber furnace at 300 °C to see if time could be reduced without causing carbon losses.

2.4. Compositional analysis

The intent of this study is to understand the concentration of carbon in samples transformed under different temperature and time parameters. The major elements of thermally transformed samples and untreated samples were identified through X-ray fluorescence spectroscopy, using Epsilon and Omnian units.

The proportion by weight of oxygen was determined by the Elementar Rapid OXY cube.

The NHCS analysis was undertaken using Vario MACRO CUBE. The results were normalised by subtracting the O, N, H, C and S proportions by weight from 100%. The balance was understood to be the major elements. The reported proportions of the major elements were multiplied by the balance.

2.5. Scanning electron microscopy

In order to observe changes to the waste cotton textile after carbonisation, scanning electron microscopy was undertaken. The samples were coated in 15 nm of gold in a sputter coater, Quorum Q300T Sputter Coating. The samples were analysed at magnifications of 30, 100, 1000, 2000 and 4000 using scanning electron microscope, TM4000Plus.

2.6. X-ray diffraction

XRD analysis was undertaken to understand the extent to which the atoms were ordered, or ‘crystalline’. The unit used to measure the crystallinity was the XRD Empyrean II.

2.7. Life cycle assessment

Life Cycle Assessment (LCA) was used to demonstrate the benefits of this process compared to conventional methods of preparing carbon fibres from PAN. The LCA focused on production only which involved thermally treating cotton waste in an inert environment. Additional processing for particular applications was not included as these have not been defined at this time. Impacts associated with collection and transport were not included for the same reasoning. As the target feedstock is a waste, the production phase of cotton growing, and textile manufacturing were not included. This is because the cotton textile was not produced for the purpose of producing carbon fibre, rather it was prepared for the purpose of clothing or bedding. As discussed in Section 3.5, the added carbon abatement benefits of landfill avoidance were not included as the viability of this material substitution was proven without this, and to allow for the unlikely possibility this material would have been recycled by other means.

According to Muralikrishna & Manickam “Life cycle assessment is an analysis technique to assess environmental impacts associated with all the stages of a product’s life, which is from raw material extraction through materials processing, manufacture, distribution, and use” (Muralikrishna & Manickam, 2017). The LCA was undertaken in accordance with the ISO 14040:2006 standard. The software used for the analysis was SimaPro 9.0.0.49. The selected method for analysis was TRACI 2.1 V1.05 / US 2008. The database used was Ecoinvent 3.

3. Results and discussion

As outlined in the materials and methods section, a case study was undertaken involving the transformation of waste cotton textiles to carbon fibre. This involved the cutting of textile waste to fit into the various crucibles. The samples were then processed under different temperature and time settings to understand how it effects the carbon content of the carbon fibres. Additional tests were performed to understand the form of the material including microscopy and XRD. The intent of this analysis is to support other researchers to develop suitable applications for this material. The potential benefits of the material were considered through a preliminary LCA, with opportunity to refine as particular applications for this product are defined and studied in due course.

In this study ‘carbon fibre’ refers to the carboneous material that is generated through thermal transformation of cotton fibres under controlled conditions without oriented graphitic structure. Therefore, potential applications for this material will not include conventional mechanical applications that require a particular structure.

3.1. Thermal transformation

The process of thermal transformation of cotton waste involves three phases, dehydration, volatisation and carbonisation. Between ambient temperature and 200 °C the dominant reaction is dehydration, with the majority of weight loss occurring at these temperatures. Between 200 °C and 300 °C is the volatisation phase, where volatile products including levoglucosan ($C_6H_{10}O_5$) are emitted (Hsieh, 2006). Thermal dehydration and the formation of levoglucosan are competitive reactions (Huang, 2009). Formation of the levoglucosan reduces the carbon yield (Huang, 2009) and therefore this reaction should be minimised to the greatest extent possible by optimising pre-treatment temperatures or using flame retardant chemicals. After 450 °C, the dehydration and volatisation reactions have been exhausted and only char remains (Hsieh, 2006).

The impacts of these reactions are displayed in Fig. 2, which shows the changes in weight of a sample with increasing temperature.

As shown in Fig. 3, the pyrolytic products include CO, CO_2 and CH_4 in the gaseous phase, levoglucosan in the liquid phase and char (Hsieh, 2006).

3.2. Activation energy

According to Balluffi et al, kinetic analysis is the study of the rates at which various process occur in materials (Balluffi et al., 2005). In other words, kinetics is the measurement of how quickly a reaction occurs. The energy required for the reaction to occur is known as the 'activation energy'. The 'activation energy' can be used as a basis to judge the difficulty of a reaction (Zhao et al., 2021).

The Arrhenius equation underpins analysis into 'activation energy' and represents the temperature dependence of reaction rates. Table 1 steps out the variables involved in this exponential function.

Thermogravimetric analysis is a common method used for kinetic analysis where temperature is increased at a constant rate and mass loss is measured. Through analysis of the mass loss a reaction conversion curve (scale of reaction versus time) can be obtained (Cai & Bi, 2008). Integral analysis of the reaction conversion curve can be used to estimate the activation energy. The most common integral method is the Coats and Redfern Method which is used for non-isothermal degradation studies (Cai & Bi, 2008). Table 2 shows the equations used in the Coats Redfern Method.

Fig. 4 shows the derivative thermogravimetric analysis (DTG) for waste cotton textile sample at a heating ramp rate of 20 °C/minute. This is the first derivative with respect to temperature in mg/minute. Fig. 4shows that most of the change takes place between 340 °C and 430 °C.

Only the first equation (n=1) within Coats Redfern method is considered in this analysis. The degree of conversion is calculated by subtracting the instantaneous mass from the original mass and dividing this by the instantaneous mass minus the final mass. The degree of conversion α is then applied to the first equation (n = 1) and plotted

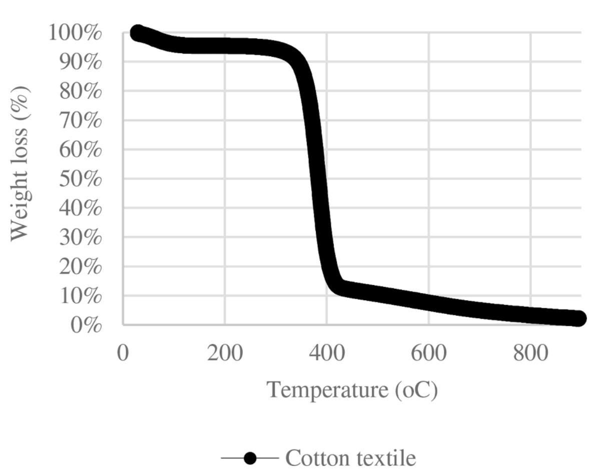

Fig. 2. Thermogravimetric analysis of cotton textile.

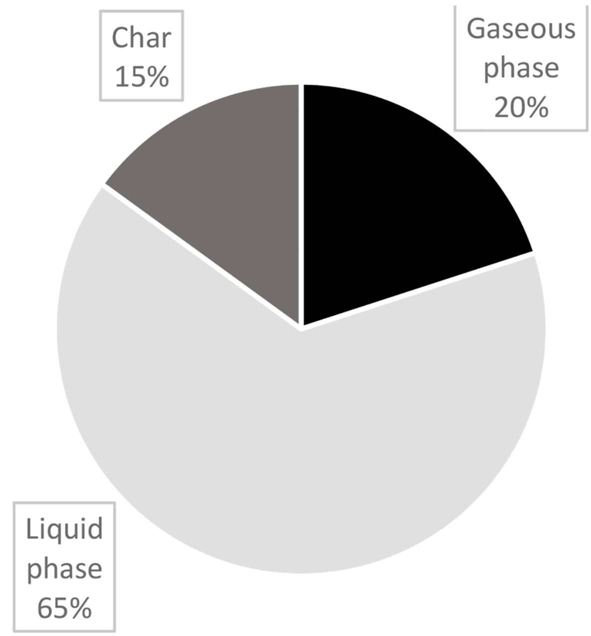

Fig. 3. Pyrolytic products (Hsieh, 2006).

Table 1
Arrhenius equation (Britannica, The Editors of Encyclopaedia, 2021).

Equation	Variables
$k = Ae^{\frac{-E_a}{RT}}$	k is the reaction rate constant A is the frequency at which atoms and molecules collide in a way that leads to a reaction e is exponential $-E_a$ is the activation energy R is the ideal gas constant T is the absolute temperature

Table 2
Coats and Redfern method (Cai & Bi, 2008).

Equations	Variables
$\ln\left\{\frac{-\ln(1-\alpha)}{T^2}\right\} = \ln\left(\frac{AR}{\beta E_a}\right) - \frac{E_a}{RT}$ when n =1 $\ln\left\{\frac{1-(1-\alpha)^{1-n}}{T^2.\,(1-n)}\right\} = \ln\left(\frac{AR}{\beta E_a}\right) - \frac{E_a}{RT}$ when n ≠ 1	α is measure of the change of the sample, which is determined by comparing the initial mass of the sample, the instantaneous mass of the sample (t) and the final mass of the sample (f). The following equation can be used to estimate α $\frac{W_0 - W_t}{W_0 - W_f}$ A is the frequency at which atoms and molecules collide in a way that leads to a reaction R is the ideal gas constant B is the heating rate E_α is the activation energy T is the absolute temperature

against 1/T. By linear fitting the slope (y = mx+c, where the slope mx = - $-\frac{E_a}{R}$ and the intercept c = $\ln\left(\frac{AR}{\beta E_a}\right)$) the activation energy (Ea) can be determined. For this particular sample, the activation energy was determined to be approximately 138 kJ.

3.3. Carbon content

Cotton fibres are approximately 88%–96.5% cellulose (Hsieh, 2006) and cellulose has a theoretical carbon yield of 45% (Huang, 2009). As

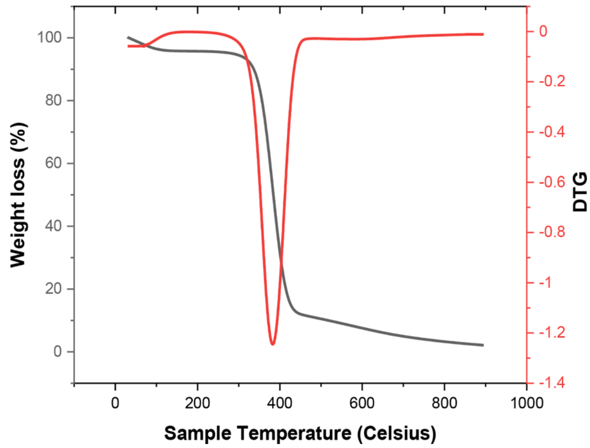

Fig. 4. DTG of carbon samples.

discussed in the previous section, the release of carbon gases and levoglucosan during the volatisation stage reduces actual carbon yield. After these reactions have been exhausted, only char remains and the proportion of carbon by weight increases with temperature. Fig. 5 shows the carbon content of thermally transformed cotton textiles increasing with temperature in an inert environment. A target line of 90% is included in this figure. This is because the technical definition of carbon fibre is a fibre that:

- Contains at least 90% carbon
- Obtained through pyrolysis
- High strength
- A micro graphite crystal structure

The results show a high carbon content for samples thermally transformed at temperatures greater than 650 °C. Whilst the samples with a carbon content of around 80% may not meet the technical definition of a carbon fibre, they still contained a significant amount of carbon and were less brittle than samples with a higher carbon content.

The carbon fibre that exceeded the 90% is promising in the context of specifications for conventional carbon fibres. However, it would need to demonstrate a high-modulus to be considered suitable for some of higher value industrial applications. The carbon fibre with 90% carbon content largely retained its cloth form but appeared to be more brittle than the lower temperature samples. The potential limitations for this

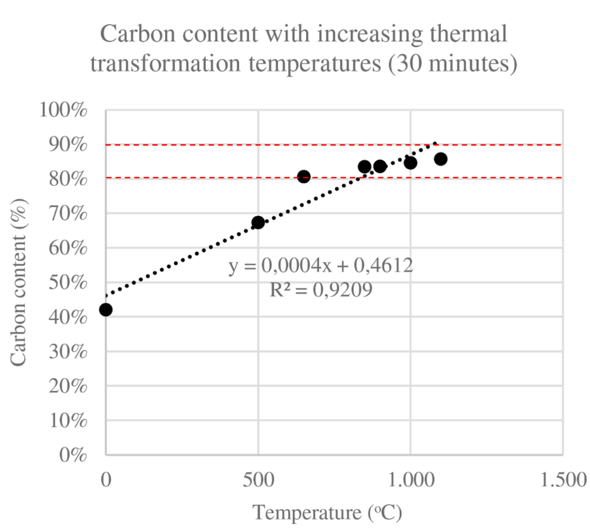

Fig. 5. Percent by weight of carbon in thermally transformed cotton textile samples.

material for mechanical applications is discussed in Section 3.6.

Fig. 6 shows that processing times can be shortened by inserting samples into the furnace at 300 °C instead of at ambient temperatures. This results in only modest loss in carbon. This could only offer energy savings if the carbon fibres were produced in a sequence of batches. Otherwise, there is no advantage as the furnace would need to heat up anyway.

3.4. Structure

As previously discussed the samples all retained their textile structure albeit shrunken and more fragile. Fig. 8 shows the woven structure that remains intact after pyrolysis at 850 °C. The woven quality of the shirts was very high to begin with which may have been a contributing factor to the retention of structure.

Another reason for this could be application of fire-retardant chemicals in the work shirts. Cotton is a naturally fire-resistant material, but it may have been enhanced with additional chemical treatments for safety. The shirts are not designed to be fireproof they are designed for outdoor wear. However, a light flame resident or flame retardant coating may still have been applied. Information on chemical treatments is not readily available and may be 'trade secret'. However, the presence of N, S and P in the major elements assessment may indicate a polymeric flame retardant was applied. Similarly, the presence of Si could be indicative of a non-polymeric flame retardant. Further investigations into the benefit of these flame retardants in the carbonisation process is an opportunity for further research.

Consistent with the literature, the cotton fibres hollowed through the dehydration process. This is shown in the comparison of thermally transformed cotton textiles and raw cotton textiles (Fig. 7).

The samples on the left (of Fig. 7) are all more moisture rich, whereas the samples on the right are dehydrated and hollow. XRD of samples at 650 °C and 850 °C show the structure is almost entirely amorphous.

3.5. Environmental benefits

LCA has been undertaken for the thermal transformation of waste cotton textiles to a carbon fibre to determine whether this process is associated with less environmental harm than the conventional production process. Over 90% of carbon fibre is produced from polyacrylonitrile (PAN) which is a petroleum based material, with few producers using renewables to power the production (Malnati, 2020)

Table 4 shows the LCA results of conventional carbon fibre compared to the LCA results of the carbon fibre cloth prepared through this recycling process. The LCA results of the conventional carbon fibre is from a study by Janssen et al which considered promising precursor alternatives such as lignin (Janssen, et al., 2019).

The applications for the waste cotton textile carbon fibre has not

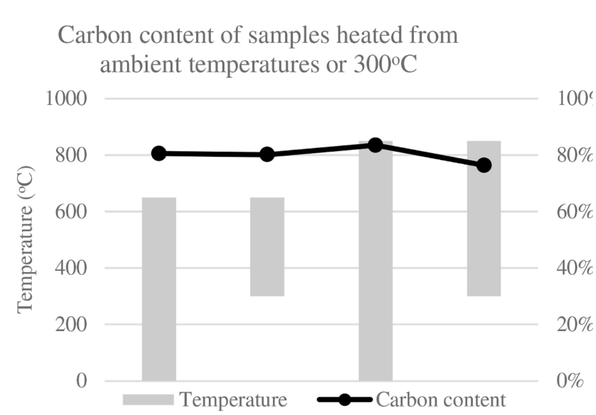

Fig. 6. Heating starting and finishing temperatures (primary axis and carbon content (secondary axis).

been defined in this study. However, the thermal transformation process would mostly likely be the most energy intensive phases of any carbon fibre product. Future researchers can add to this preliminary LCA to tailor it to particular applications.

As the predominant energy source for Australian electricity is coal fired power, only analysis with a 'non-renewable background' has been included. The results presented in Table 4 use the European CML method for LCA. The results for recycled carbon fibre make the following assumptions –

- The target carbon content is 80% as this material had advantageous physical properties and required less energy to produce. Therefore the temperature needs to be raised to 650 °C only.
- It is assumed that energy will be needed for 2.6 h only to raise the temperature to 650 °C and hold it at this temperature for 30 min. It is assumed that no energy is required for the 'cool down'.
- It is assumed that the process will take place in a medium size furnace with a temperature capacity of 650 °C, volume of 4.8 m^3 and a power of 75 kW.
- It is assumed that the furnace will be 90% full of textiles (432 kg) to produce 26 kg of carbon fibres (at 80% carbon content).
- It is assumed that 5 kg of argon gas will be required to provide an inert environment for the thermal transformation.
- As the preferred form of the carbon fibre is cloth, no shredding activities have been included in this life cycle. It is assumed the textiles will be cut into sheets by hand, which would in-turn add to the operational cost of the carbon fibre.

These results show that it is a better outcome for the environment to recycle the waste cotton textiles and convert them into a carbon fibre cloth, which displaces the need for carbon fibres produced from PAN. The only circumstance where conventional carbon fibre performs better than recycled carbon fibre is when it is produced from renewable energy, and only in the field of 'Human Toxicity Potential'. However, this is currently not the status quo but expected to be negated if the recycled carbon fibres were also produced from renewable energy.

Further benefits that have not been quantified in this LCA include:

- Avoided emissions associated with transport of carbon fibres from the handful of countries that manufacture PAN carbon fibre.
- Avoided emissions associated with the spinning process to produce a carbon fibre cloth.
- Avoided emissions associated with disposal of the textile to landfill.

3.6. Application considerations

Carbon fibre products have traditionally been made for the aerospace industry, which is the most profitable market for this advanced material. Many industry commentators believe that the automotive industry has the potential to supersede the aerospace sector and this emerging market does not have the same performance requirements (Williams & Calder, 2015). Other growing markets include renewable energy, recreation and the construction sector.

The price points and the quality requirements of these markets are very different thus creating demand for carbon fibre products with lower, or non-aerospace grade performance standards (Williams & Calder, 2015). It is predicted that the automotive industry will consume more carbon fibre than the aerospace industry but at a lower unit cost, serviced by less expensive precursors and processing methods (Williams & Calder, 2015). These industry observations and predictions provide a promising context for the development of carbon fibres by non-conventional means.

Mechanical applications are not considered the most promising application for carbon fibre produced from waste cotton textiles. This is because the carbonised textiles are unlikely to offer the oriented graphitic structure required for structural applications. Furthermore, to compensate for less optimum fibre orientation and density, more resin would be required to produce a product that could compete with PAN carbon fibre composites. The carbon fibres made from waste textiles may also need to be reprocessed to make them straight for mechanical applications which would add to energy requirements for the process. If these challenges were resolved, at best the product would likely only compete with low cost and low strength commercially available carbon fibres. Additional processing requirements to produce a comparable carbon fibre for mechanical applications would need to be accounted for in life cycle assessments and business cases. For the aforementioned reasons, the viability of this application is considered less promising.

More promising applications are likely to take advantage of the natural properties of the carbon fibre produced from waste textiles. The exploitable properties for applications include high carbon content, woven textile structure, spacing between carbon (or porosity) and conductivity. The membrane like structure and porosity may be suitable for pollution control, particularly when enhanced through chemical activation processes to increase porosity. Australia is reliant on imports of activated carbon for various environmental management applications.

The energy storage potential demonstrated by Jagdale et al may also represent a promising application, particularly in relation to wearable devices (Jagdale, et al., 2021).

3.7. Implementation considerations

The following mechanisms could support implementation of textile recycling system in Australia:

- Engagement with Australian owned clothing and bedding companies in voluntary extended producer responsibility for end-of-life cotton textiles.
- Governments could consider 'return and earn' initiatives. Textiles end up in landfill as 'unusable donations' to charities or through households disposing of their unwanted or end-of-life clothes in the residual waste bin. With over 725,000 tonnes of textiles disposed annually, the cost to the community is in the tens of millions (excluding levies).
- Hypothecation of waste levies to support planning, construction, and delivery of this recycling initiative.

The following principles should influence the selection of products that could be developed from these carbon fibres –

- **'Do no harm'** (Engineers Without Borders Australia, 2018). Product applications must not contribute to release of fibres in the environment. The contexts in which products are used must take into consideration potential exposure pathways for users and risk of deterioration over time. For example, conventional carbon fibres are commonly used as a structural component in composites. Within a composite, the carbon fibres are secure and cannot be released into environment.
- **'Circulate products and materials (at their highest value)'** (Ellen Macarthur Foundation, 2022 n.d.): Any carbon fibre products developed must not be single use and must hold ongoing value within the local materials economy. To support further recycling at end-of-life, the product should be designed for disassembly. For example, Zhang et al in *Current status of carbon fibre and carbon fibre composites recycling* propose use of degradable thermosets and thermoplastic matrices to improve recyclability of carbon fibre reinforced polymer matrix composites. A degradable thermoset can depolymerize on demand under mild conditions (Wang, et al., 2019), facilitating the recovery of the original materials. This would facilitate further circulation of the carbon fibre and plastics in the economy, after all product re-use opportunities have been exhausted.

Product recycling could also be supported through voluntary extended producer responsibility schemes.

4. Conclusion

The minimum temperature required to produce a carbon fibre under the conventional definition is 1150 °C (90% carbon content). The minimum temperature for a lower grade carbon fibre is 650 °C (80% carbon content). The findings of the LCA show that this recycling initiative is worthwhile when displacing demand for conventional carbon fibres and diverting cotton waste textiles from Australian landfills.

The exploitable properties of the lower grade carbon fibre include its woven structure, high carbon content, space between carbon structures and flexibility to fold. The form of this carbon fibre may lend itself better to non-mechanical applications such as energy storage and pollution control. Chemical activation could further enhance the porosity of material and could be considered in future investigations.

Opportunities to reduce the time parameters of the experiment are limited. This is because the thermal transformation process takes place in an inert environment. When samples are inserted or removed from this environment, combustion reactions take place and carbon is lost. The maximum temperature the sample can be inserted into a furnace with minimum loss of carbon is 300 °C. This is because the majority of thermal transformation activities occur between temperatures of 340°C and 430 °C.

This information is valuable to other researchers and recyclers looking to keep waste textiles out of landfill by creating a valuable carbon fibre product. The need for a variety of different re-use and recycling solutions for waste textiles is apparent with only 7% of textiles recovered in Australia and policy drivers to increase onshore processing of waste.

In addition to diverting waste from Australian landfills, this solution may also support the development of less energy intensive carbon fibres for applications including environmental management.

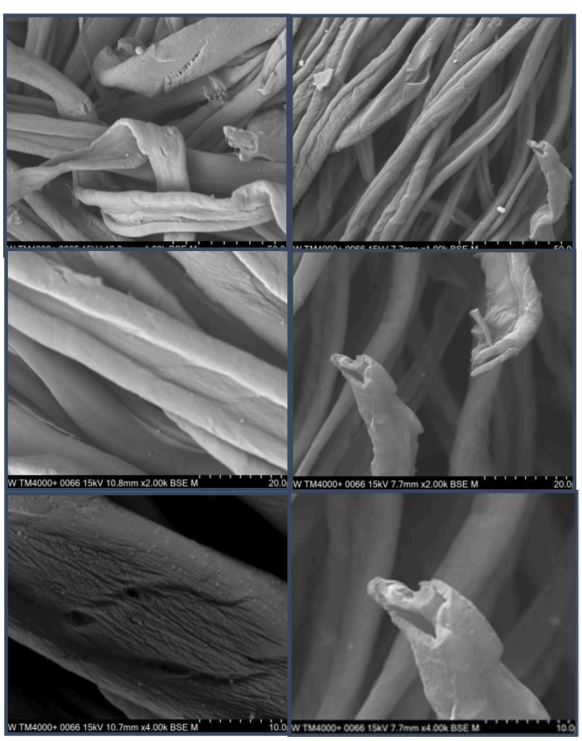

Fig. 7. Raw cotton textiles (left) and thermally transformed cotton textiles (850 °C) (right) at magnifications of 1000 (top), 2000 (middle) and 4000 (bottom).

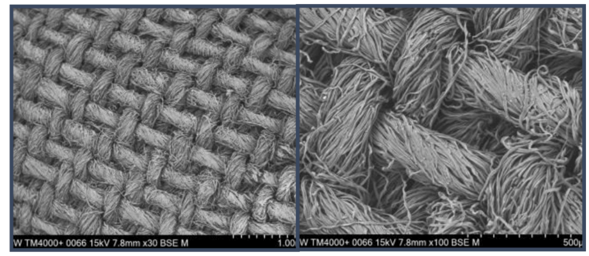

Fig. 8. Thermally transformed cotton textiles (850 °C) at magnifications of 30 (left) and 100 (right) under an SEM.

Table 3
Carbon content of samples thermally transformed from ambient temperature to target temperatures for a period of 30 min.

Temperature (°C)	Carbon content (%)
0	42%
500	67%
650	81%
850	83%
900	84%
1000	85%
1100	86%
1150	91%

Table 4
Comparison of conventional carbon fibre (PAN) with carbon fibre prepared from this.

Energy background	Current energy background (non-renewable energy)	Prospective energy background (renewable energy)	Current energy background (non-renewable energy)
Material type	Carbon fibre (PAN)	Carbon fibre (PAN)	Carbon fibre cloth from waste cotton textile
Global Warming Potential (GWP) (kg CO_{2e})	38.9	19.3	**10.49**
Eutrophication Potential (EP) (kg PO_{4e})	0.1	0.04	**0.05**
Acidification Potential (AP) (kg PO_{4e})	0.3	0.21	**0.04**
Photochemical Ozone Creation Potential (POCP) (Kg $ethylene_e$)	6.7 (10^{-3})	2.7 (10^{-3})	**1.08 (10^{-3})**
Human Toxicity Potential (HTP) (kg 1,4-DCB_e)	11	3.94	**4.98**

CRediT authorship contribution statement

Charlotte Wesley: Investigation, Method, Writing; **Farshid Pahlevani:** Reviewing and Editing; **Md. Shahruk Nur-A-Tomal:** Life Cycle Assessment Review; **Smitirupa Biswal:** Kinetic Analysis; **Veena Sahajwalla:** Conceptualisation.

Declaration of Competing Interest

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

Data availability

Data will be made available on request.

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