Review

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Textile waste pretreatment for anaerobic digestion: a review and technology feasibility study

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Abstract

The increasing volume of textile waste in landfills and incineration poses severe environmental challenges. Waste valorisation of textile waste via anaerobic digestion (AD) is preferable, as it offers economic and environmental benefits, but it is hindered by textile complexity, necessitating effective pretreatment technologies to improve biogas production. This study aims to evaluate various pretreatment technologies for biogas production from textile fibres via AD. A weighted-scoring analysis (WSA) assessed pretreatment methods based on technical, economic, environmental and operational criteria. Hydrothermal pretreatment emerged as the most technically effective method, scoring 140 owing to its substantial methane enhancement. Economically, shredding was the most viable option, scoring 125, as a consequence of low capital and O&M cost. Environmentally, hydrothermal and deep eutectic solvent (DES) pretreatments were top performers with 100 points owing to low environmental impact and positive heat reactions. In a case study conducted in the Auckland region, the potential environmental impact (PEI) obtained from hydrothermal and DES were 169 and 92 per year, respectively, resulting in minimal environmental impact. Operationally, ultrasonic and biological pretreatments scored highest owing to their ease of operation, and minimal health and safety requirements. Overall, hydrothermal pretreatment achieved the highest WSA score of 340, reflecting its balanced performance across all criteria. Hydrothermal pretreatment is the most promising technology for enhancing biogas production from textile waste. Its technical efficiency, economic feasibility and environmental benefits regarding the WSA score make it suitable for upscaling and providing a viable solution for managing textile waste in the AD plant.

Supporting information may be found in the online version of this article.

Keywords: textile waste; anaerobic digestion; pretreatment; weighted-scoring analysis; biogas

ABBREVIATIONS

AD	anaerobic digestion
BMP	biomethane potential
CAPEX	capital expenditure
CGT	cotton gin trash
CSTR	continuous stirred-tank reactor
DES	deep eutectic solvent
DS	dry solid
EIF	environmental impact factor
GHG	greenhouse gases
GT	Gigatonnes
MC	microbial consortium
MCDA	multiple-criteria decision analysis
OLR	organic loading rate
PE	population equivalent
PFR	plug-flow reactors
sCOD	soluble carbon oxygen demand
TH	thermal hydrolysis
TRL	technology readiness level
TS	total solids
S/I	substrate-to-inoculum
SRL	societal readiness level
TJ	terajoule
VFA	volatile fatty acid
VOC	volatile organic content
VS	volatile solids
WAO	wet air oxidation
WSA	weighted-scoring analysis

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INTRODUCTION

The generation of textile production results in tremendous increments from the fast fashion industry's current business model, characterised by mass production, variety, agility and affordability.[1] Fast fashion causes a global issue because textile companies manufacture enormous amounts of new clothes every week, encouraging overconsumption. Total global textile production is expected to reach 147 million tonnes in 2030, an approximately four-fold increase compared to 1975 amounts.[2] From the latest statistics for the year 2022, the recorded textile production amounted to 116 million tonnes, which increased by 2.65% from 2021.[2] The major textile type production is synthetic (65%), followed by plant, man-made cellulosic, and animal fibres (Fig. 1). In terms of classification, polyester (54%), cotton (22%), viscose (5%) and wool (1%) represent the highest proportions of textile production.[2]

As a consequence of the overconsumption of fast fashion, a significant amount of discarded clothes is produced globally after end-of-life.[3] According to annual textile waste statistics, 92 million tonnes of textile waste are generated, and carbon (C) emissions are expected to reach 3.5 million tonnes of carbon dioxide equivalent (CO_2e) emissions.[4] From the total textile production, ≈60% of garments manufactured are sent to landfills and incineration within a year of production.[5] These two waste disposal methods affect the environment and human health, via land degradation, methane (CH_4) emissions, toxic leachate emissions, air and water pollution, and climate change.[6]

Instead of disposing to landfill, textile waste can be valorised into energy via pyrolysis, gasification and anaerobic digestion to reduce environmental problems.[1,3] Anaerobic digestion (AD) has garnered greatest interest amongst these technologies as a consequence of its ability to generate bioenergy, and its environmentally friendly and cost-efficient nature.[7,8] Natural textile fibres such as cotton and wool, known as C- and protein-rich substrates, respectively, are highly valuable for biogas conversion owing to their cellulose and keratin contents.[9]

Textile fibres have been affected by complex structures and synthetic material, which inhibits biogas production. Therefore, pretreatment helps to improve organic matter degradability in textile fabric, which increases AD's biogas yield.[9,10] Several studies have briefly been conducted on fibre substrate pretreatment.[9,10] No research has been conducted on the economic and environmental aspects of textile fibre pretreatment. This study details the pretreatment progress conducted on fibre substrates, technology, societal readiness level, environmental impact assessment (EIA) and weighted-scoring analysis (WSA) to comprehensively evaluate pretreatment technology commercialisation in biogas plants.

ANAEROBIC DIGESTION OF FIBRE SUBSTRATES

Anaerobic digestionis a cost-effective waste-to-energy technology; it is a sustainable process as it recovers energy from biogas, replaces fossil fuel usage and minimises emission of greenhouse gases (GHG).[11] The four steps of AD are hydrolysis, acidogenesis, acetogenesis and methanogenesis.[12] Hydrolysis is the first step that converts complex polymers (polysaccharides, proteins, lipids) to monomers (sugars, amino acids, fatty acids).[13] Hydrolysis is considered a pretreatment step, and it can produce biogas fermentation more efficiently and faster.[14] Biogas is a renewable energy source produced by the biological breakdown of organic matter under anaerobic conditions. Raw biogas contains ≈55–65% CH_4, 30–45% CO_2 and a small amount of hydrogen sulfide (H_2S).[15] The CH_4 recovery from biogas, called biomethane, is helpful for fuel transportation or injection into the national natural gas grid.[16]

The high cellulose and keratin content of cotton and wool fibre, respectively, and their low lignin levels make natural textile waste a promising feedstock for CH_4 fermentation.[17] The organic content is an essential part of total solids (TS), which evaluates the percentage of total solid mass in a fibre substance for AD. According to the TS contents in the reactor, AD is usually divided into wet (TS <15%) and dry (TS >15%) states, among which wet AD has received considerable popularity in sewage sludge and food waste plants owing to its suitability for high moisture content substrate applications.[18] Wet AD is commonly applicable to industries because it is more manageable in regulating and providing increased efficiency in biogas production.[19] Nonetheless, the main challenge associated with the digestion of cotton and wool fibres is their high dry solid (93.4 and 37.9% TS, respectively)[9] causing clogging during AD,[20] and structural complexity consisting of crystallinity structures and disulfide

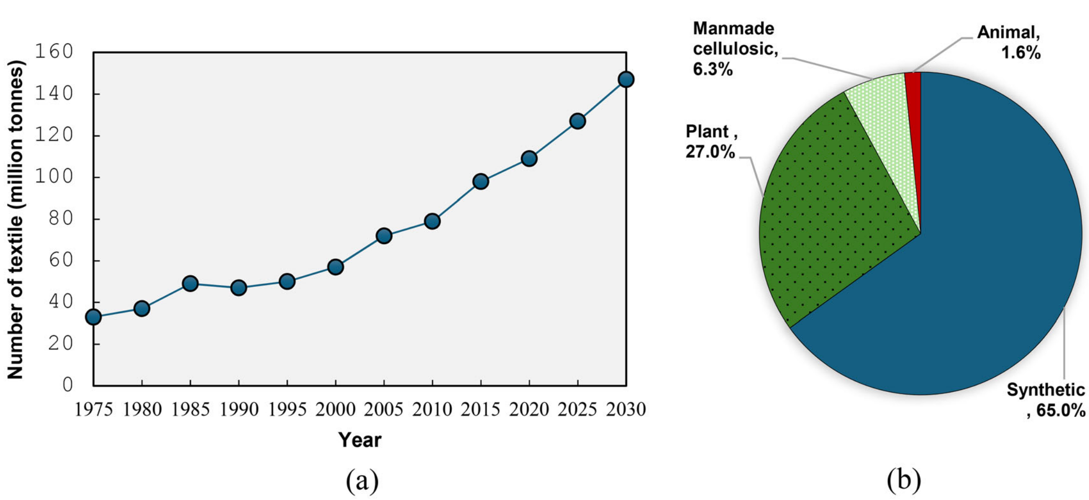

Figure 1. (a) Textile production and expected textile waste between 1975 and 2030, and (b) global fibre production share in 2022.[2]

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bonds, respectively,[10] resulting in low bioconversion. To address such challenges, pretreatment methods are required to break down the complex organic matter into monomers for biogas enhancement.[21]

PRETREATMENTS OF FIBRE SUBSTRATES

Various pretreatment techniques have been implemented to address this challenge effectively in response to the low CH_4 productivity posed by natural textile fibres owing to their structural complexity. Pretreatment increases the bioavailability of organic matter for microbial hydrolysis, thereby shortening the hydraulic retention time (HRT) and improving biogas formation.[22,23] Additionally, this treatment enhances the hydrophilicity of the textile materials, eliminates surface contaminants and decreases their structural compactness so that they may be readily processed further.[24] Pretreatment techniques, including mechanical, chemical, biological, chemical and combination methods, have been applied to fibre substrates with different results.

Mechanical pretreatment

In mechanical pretreatment, the biomass structure gets altered, and the particle size is reduced by applying mechanical force. This increases the particle surface area and resultant effectiveness of enzymatic and microbial assaults, which speeds up the AD process that produces CH_4.[25] Several mechanical pretreatments have been conducted on textile waste and other similar substrates on CH_4improvement: shredding, ultrasonic, nanobubble water, and grinding (summarised in Table 1).

Shredding is the common recycling method used for textile waste in the industry as a result of its convenient usage, environmentally friendly nature and low cost, facilitating efficient scale-up.[31,32] Despite its advantages, shredding produces shorter, weaker fibres that cannot be reused to make clothes.[33] No comparison was conducted on bio-methanation enhancement from the shredding process, but it has been shown individually on cotton waste. Azcona *et al.*[17] cut the cotton fabric into 4×4 cm^2 pieces with a substrate-to-inoculum (S/I) ratio of 1.6 in a batch process, resulting in a CH_4 yield of 105 mL gV^{-1}. By contrast, Jin *et al.*[26] cut the fabric into 2×2 cm^2 pieces generating a CH_4 yield of 343 mL gVS^{-1} with S/I ratio of 1 and organic loading rate (OLR) of 10 g L^{-1}, which increased CH_4 yield by three-fold. Grinding pretreatment is performed to decrease the size of the substrate and increase the biomass's specific surface area (SSA),[34] which is relatively comparable to shredding. Sołowski *et al.*[30] ground the cotton and achieved 653 mL gVS^{-1} under the mesophilic conditions. Likewise, grinding pretreatment on wool textiles with liquid nitrogen (N_2) helped to enhance the CH_4 yield by 80%, which effectively increased the microbial accessibility of wool proteins that possess amino acid group linkages.[35] Cutting into smaller sizes helps increase fabric SSA and the suitable S/I ratio required for efficient microbial degradation to improve biogas generation.[36]

Ultrasonication, a highly efficient mechanical pretreatment technique, has the potential to enhance biomass biodegradability. This technique produces ultrasonic waves that travel across liquid media and create microbubbles, producing shear solid forces that can damage biomass cell walls.[37] Hanif *et al.*[27] reported that a combined ultrasonication and hot water contributes to a higher CH_4 yield than hot water (11 mL gVS^{-1}) and ultrasonic (25 mL gVS^{-1}). At the highest power amplitude (100%), the cotton gin trash biomethane potential (BMP) improved by 160% from its original feedstock. Another experiment conducted on cellulose used nanobubble water technology, which is comparable to ultrasonic. This pretreatment involves production of nanobubbles which adhere to the surface of biomass, facilitating enhanced mass transfer, chemical reactions and metabolic processes.[38,39] Three gases are injected into the nanobubbles to pretreat the cellulose: oxygen (O_2), air and N^2. The addition of O_2 into

Table 1. Mechanical pretreatment of textile fibre for biogas production

Technique	Substrate	Treatment conditions	AD conditions	CH_4 yield (mL gVS^{-1}) Untreated	Treated	Change in BMP (%)	References
Shredding	Cotton	Cut into 4×4 cm^2	S/I ratio: 1.6 Temp.: 37 °C HRT: 40 days	–	105	–	Azcona *et al.*[17]
	Cotton	Cut into 2×2 cm^2	S/I ratio: 1:1 Temp.: 37 °C HRT: 40 days	–	343	–	Jin *et al.*[26]
Ultrasonic	Cotton gin trash	100% amplitude, 30 min	S/I ratio: 1:1 Temp.: 37 °C HRT: 91 days	210	370	+76	Hanif *et al.*[27]
Nanobubble water (NBW) with O_2, N_2, air	Cellulose	NBW treatment, 25 °C, 20 min	S/I ratio: 1:1 Temp.: 35 °C HRT:18 days	179	O_2: 233 N_2:196 Air: 193	O_2: +13 N_2: +9 Air: +8	Wang *et al.*[28]
Nanobubble water (NBW) with air, CO_2	Cellulose	NBW treatment, 25 °C, 20 min	S/I ratio: 3.5:1 Temp.: 55 °C HRT:35 days	224	Air: 264 CO_2: 246	Air: +18 CO_2: +10	Wang *et al.*[29]
Grinding	Cotton	Grinding	S/I ratio: – Temp.: 38 °C HRT:30 days		653	–	Sołowski *et al.*[30]

Abbreviations: AD, anaerobic digestion; BMP, biomethane potential; HRT, hydraulic retention time; S/I, substrate-to-inoculum; VS, volatile solids.

nanobubbles peaked the electron transfer system activity resulting in better CH_4 yield (233 mL gVS^{-1}) compared with air and N_2 (196 and 193 mL gVS^{-1}, respectively) in mesophilic AD conditions.[28] This shows that oxidative conditions are much more reliable for cellulose content and crystallinity breakdown, achieving 14% and 21% (repectively) by degrading cellulose structure glycoside bonds. A micro-oxygen environment could improve CH_4 yield by enhancing volatile fatty acid (VFA) production during acidogenesis.[40,41] Under thermophilic conditions, cellulose produces a higher CH_4 yield with the addition of air compared with CO_2 into nanobubbles with a BMP increment of 18%, possibly as a result of air and thermophilic conditions enriching the microbial community for cellulose crystallinity reduction.[29]

Chemical pretreatment

Chemical pretreatment aims to break down complex organic compounds into simple ones[50] using alkali, acid, ionic liquid (IL) and deep eutectic solvent (DES). The impact of these chemicals on different textile fibres is shown in Table 2..

Alkali pretreatment improves the cotton fabric lignin solubilisation and reduces cellulose crystallinity because the mercerisation effect accelerates cellulose depolymerisation,[51] because low inhibitor development during biomass hydrolysis increases cellulose digestibility.[52,53] An experiment conducted on cotton textile waste with a S/I ratio of 1:1 shows a good result of CH_4 improvement by 103% from the untreated substrate as pretreated with Na_2CO_3 for 3 h at 150 °C.[42] Hasanzadeh *et al.*[43] experimented with 60/40 cotton/polyester jeans pretreated with 0.5 M Na_2CO_3 at 150 °C, significantly incrementing CH_4 two-fold. The authors also concluded that increased alkali concentration at high temperatures affects the hydrolysis of textile waste. Although jeans yielded lower CH_4, the CH_4 enhancement was higher than pure cotton as a reuslt of the increased surface contact of cotton cellulose during enzymatic hydrolysis. Chicken feathers are similar to wool textiles rich in keratin for protein-based comparison.[54] Sumardiono *et al.*[55] pretreatment of 25% TS with NaOH at 30 °C for 3 h helped to maximise yield by 40% because of its ability to hydrolyse the sulfide bonds in the keratin structure, which has significant biogas generation capability. Additionally, Forgács *et al.*[56] supported this finding through pretreating chicken feathers with $Ca(OH)_2$ (calcium hydroxide) at 100 °C for 30 min, enhancing CH_4 yield by 105% as a result of the strong alkali effect for protein breakdown.

Acid pretreatment can hydrolyse the hemicellulose's polymeric linkages to produce its monomers, which increases the cellulose's

Table 2. Chemical pretreatment of textile fibre for biogas production

Technique	Substrate	Treatment conditions	AD conditions	CH_4 yield (mL gVS^{-1})		Change in BMP (%)	References
				Untreated	Treated		
Alkali	Cotton textile waste	0.5 M Na_2CO_3, 150 °C, 3 h	S/I ratio: 1:1 Temp.: 35 °C HRT: 15 days	151	307	+103	Juanga-Labayen *et al.*[42]
	Jeans and pure cotton	0.5 M Na_2CO_3, 150 °C, 2 h	Temp.: 37 °C HRT: 40 days	Jeans: 170 Cotton: 200	Jeans: 330 Cotton:360	Jeans: +92 Cotton: +80	Hasanzadeh *et al.*[43]
	Cotton straw	K_2FeO_4, 10 min	S/I ratio: 10:1 Temp.: 35 °C HRT: 60 days	83	109	+31	Wang *et al.*[44]
	Cotton stalk	NaOH, 100°C, 1 h	S/I ratio: 4:1 Temp.: 35 °C HRT: 21 days	224	296	+32	Cheng and Zhong[45]
Acid	Wool scouring waste	NH_4Cl	S/I ratio: 1:3 Temp.: 35 °C HRT: 35 days	300 mL	266 mL	−11	Othman[46]
	Cotton yarn	2% H_2SO_4 and 2% H_3PO_4, 140 °C, 2 h	Temp.:35 °C HRT: 30 days	-	H_2SO_4: 196 H_3PO_4: 278	-	Binczarski *et al.*[47]
	Cotton +5% wool	2% H_3PO_4, 140 °C, 2 h	S/I ratio: 1:2 Temp.: 35 °C HRT: 37 days		466	-	Binczarski *et al.*[48]
	50/50 Polyamide/ cotton +5% wool	2% H_3PO_4, 140 °C, 2 h	S/I ratio: 1:2 Temp.: 35 °C HRT: 37 days		339	-	Binczarski *et al.*[48]
	50/50 Polyester/cotton +5% wool	2% H_3PO_4, 140 °C, 2 h	S/I ratio: 1:2 Temp.: 35 °C HRT: 37 days		204	-	Binczarski *et al.*[48]
Ionic liquid	50/50 Blended Polycotton 40/60 Blended Poly-viscose	85% NMMO, 120 °C, 2 h	S/I ratio: 1.25:1 Temp.: 35 °C HRT: 3 days	Polycotton: 1 Poly-viscose: 8	Polycotton: 131 Poly-viscose: 128	Polycotton: +13 000 Poly-viscose: +1500	Jeihanipour *et al.*[49]

Abbreviations: AD, anaerobic digestion; BMP, biomethane potential; H_2SO_4, sulfuric acid; H_3PO_4, phosphoric acid; HRT, hydraulic retention time; K_2FeO_4, potassium ferrate; Na_2CO_3, sodium carbonate; NaOH, sodium hydroxide; NH_4Cl, ammonium chloride; NMMO, *N*-methyl morpholine-*N*-oxide; S/I, substrate-to-inoculum; VS, volatile solids.

availability and the biodegradability of its crystalline and amorphous structures in cotton textiles.[31] Hitherto, the disadvantages of pretreatment formation of inhibitors affect biogas production, high cost and corrosion effect defects.[57] This is proven by testing NH_4Cl and cationic flocculent on wool scouring waste, which negatively affects CH_4 yield and shows that acid is inappropriate for keratin decomposition.[46] Binczarski *et al.*[47] experimented that H_3PO_4 has higher CH_4 yield production by 42% compared to H_2SO_4 owing to phosphorus, a crucial nutrient for buffer capacity to enhance microbial activity during AD.

Ionic liquids which contain anions, and cations can be tuned to generate various liquids.[58] The primary benefit of IL pretreatment is its ability to dissolve different types of textile waste in an environmentally friendly and mildly processed method. However, the disadvantages of this pretreatment are its high cost, low biodegradability and nonrecyclability.[59,60] As a recent advancement to address these issues, *N*-methyl morpholine-*N*-oxide (NMMO), a nontoxic and biodegradable solvent, was synthesised to dissolve cellulose effectively in mild conditions.[61,62] For instance, Jeihanipour *et al.*[49] concluded that NMMO has a good ability to dissolve cellulose and separate synthetic material. A blended textile of polycotton and poly-viscose pretreated with 85% fresh NMMO showed impactful CH_4 percentages of 31.28% and 30.85%, respectively. However, the significant issue for NMMO solvent is its high capital cost, in which the makeup cost increased by 5.4-fold for a 1% recovery rate decrease.[63] For this case, an economically feasible solvent (e.g. DESE) is required to solve this issue.[64]

Deep Eutectic Solvent, a solvent that contains hydrogen (H)-bond acceptors (HBA) and H-bond donors (HBD), can degrade the mechanical structure of the biomass with minimal energy consumption during pretreatment.[65,66] DES has a few advantages, such as low cost, easy preparation, low volatility, biodegradability and a nontoxic environment.[67] Little research has been conducted on cellulose substrates, comparable to cotton textiles, pretreated via DES for biogas production. An example of ammonium thiocyanate: urea with a 1:2 ratio DES was experimented on a corn stover with a loading rate of 35 g L^{-1} for 21 days of digestion. It showed a 48% BMP increment with the final CH_4 yield of 44.4 mL gVS^{-1}.[68] Acidic HBD has efficient degradation on lignocellulose substrates. Kang *et al.*[69] experimented on willow with chlorine chloride: lactic acid (1:2 ratio), which showed a significant CH_4 yield enhancement of 1.4-fold. Bagder Elmaci *et al.*[70] also concluded that pretreating cork dust with formic acid HBD enhanced CH_4 gas by ≈125%. Acidic HBD shows efficient cellulose degradation for biogas production.

Biological pretreatment

The biological pretreatment process is based on the function of multiple forms of heterotrophic microbes. Complex biopolymers such as protein and carbohydrates can be transformed into more straightforward end products owing to the action of various enzymes which the bacteria produce.[21] The biological pretreatment process can occur either aerobically or anaerobically, improving hydrolysis and bio-methanation.[71]

Several studies have been conducted on maximising biogas production using biological pretreatment for fibre feedstocks (Table 3). Jin *et al.*[26] used Clostridium *sensu stricto* for cellulosic fibre pretreatment and showed good BMP results after 50 days of digestion in mesophilic conditions, successfully breaking down

Table 3. Biological pretreatment of textile fibre for biogas production

Technique	Substrate	Treatment conditions	AD conditions	CH_4 yield (mL gVS^{-1}) Untreated	CH_4 yield (mL gVS^{-1}) Treated	Change in BMP (%)	References
Bacteria	Cotton Kapok Rayon White denim Blue denim Flax Ramie Hemp Jute Abaca Sisal	Clostridium *sensu stricto*	S/I ratio: 1:1 Temp.: 37 °C HRT:50 days	Cotton:195 Kapok: 168 Rayon: 193 White denim: 189 Blue denim: 112 Flax: 200 Ramie: 193 Hemp: 193 Jute: 193S Abaca: 159 Sisal: 200	Cotton:343 Kapok: 295 Rayon: 327 White denim: 330 Blue denim: 160 Flax: 356 Ramie: 333 Hemp: 335 Jute: 318 Abaca: 266 Sisal: 327	Cotton: +76 Kapok: +76 Rayon: +70 White denim: +75 Blue denim: +43 Flax: +78 Ramie: +73 Hemp: +74 Jute: +65 Abaca: +67 Sisal: +64	Jin *et al.*[26]
Bacteria	Cellulose	Clostridium *sensu stricto*	S/I ratio: 2.5:1 Temp.: 35 °C HRT:40 days	225	248	+10	Liu *et al.*[72]
Bacteria	Cotton stalk	Microbial consortium (MC1)	S/I ratio: 1:1 Temp.: 35 °C HRT:30 days	50	118	+136	Yuan *et al.*[73]
Enzyme	70/30 Wool/nylon 6	Alkaline endopeptidase	S/I ratio: 2:1 Temp.: 37 °C HRT:50 days	6% TS: 48 12% TS: 35 21% TS: 61 30% TS: 3	6% TS: 108 12% TS: 131 21% TS: 40 30% TS: 3	6% TS: +125 12% TS: +274 21% TS: −34 30% TS: 0	Kabir *et al.*[74]

Abbreviations: AD, anaerobic digestion; BMP, biomethane potential; HRT, hydraulic retention time; S/I, substrate-to-inoculum; TS, total solids; VS, volatile solid.

the substrate's crystalline structure. The hydrolysis of substrates was most effective for three materials: flax (78%), kapok (75.7%) and cotton (75.6%).[26] Liu *et al.*[72] confirmed that cellulose pretreatment with a similar bacterium indicates an increment of CH_4 yield by 10%, which shows a good fermentation process and low VFA accumulation. However, using a different bacterium, *Bacillus subtilis*, on chicken feathers resulted in diminished CH_4 productivity. This is attributed to its inefficiency in breaking down the disulfide bonds within the keratin structure.[75]

A study showed that alkaline endopeptidase on wool textiles with 13% TS comprises the highest BMP increment by 274% from the nonpretreated substrate under the optimum conditions compared with 6%, 21% and 30% TS.[74] The significant increase in BMP can be attributed to the enzyme's efficacy in efficiently breaking down the wool substrate compared to bacterial pretreatment.

Thermal pretreatment

Thermal pretreatment is usually performed over a wide temperature range of 50–250 °C and can be divided into two categories according to temperature: high-temperature pretreatment (>140 °C) and low-temperature pretreatment (<140 °C).[76,77] Thermal pretreatment, which involves hydrothermal and autoclave, increases the bioavailability of biomass materials by aiding in the partial or complete dissolving of refractory components. Several studies have been conducted using thermal pretreatment methods on the bioconversion of textile fibre (Table 4).

The autoclave, subjected to supplied steam and pressure, represents one of the most effective heating processes. Autoclaving also is very effective in degrading the recalcitrant structure of lignocellulose biomass, which is inhibitory for chemical action.[8,80] Autoclaving cotton textiles at 120 °C for 10 min in mesophilic AD increases CH_4 yield by 29% compared to the nonpretreated textile.[43] However, wool textiles with a similar pretreatment in thermophilic AD improve by 855% compared to the nonpretreated textile.[78]

Hydrothermal pretreatment, using pressurised hot water, substitutes traditional thermal processing. Hydrothermal pretreatment helps to enhance enzymatic hydrolysis, which can break down the complex structure and make it more accessible to microorganisms and enzymes that convert it into CH_4.[81,82] During hydrothermal pretreatment, a water molecule can act as an organic solvent at a high-performance temperature (>100 °C), thereby increasing the solubility of organic chemicals in the pretreatment system.[83] An experiment conducted on hydrothermal pretreatment with ammonia additives at 100 °C for 30 min, substantially increased the CH_4 yield by 172% as a consequence of the ability of the alkali medium, which can cause significant degradation in the crystalline structure of cellulose. The cellulose structure degradation results from the mercerisation effect that breaks the breaks H-bonds in cellulose, leading to fibre swelling and increased accessibility of hydroxyl groups.[51] Strong and Gapes[84] concluded that a longer pretreatment time is effective, in which thermal hydrolysis (TH) and wet air oxidation (WAO) are more responsive at 140 °C for 60 min, producing CH_4 yield increments of 274% and 195%, respectively, from kraft pulp. To date, no study has explored the effect of this pretreatment on textile fibre.

Combined pretreatment

Several pretreatment combinations have improved biomass enzymatic hydrolysis (Table 5). They are more effective than the standard treatment procedures but are highly complex.[87]

Thermo-biological pretreatment is the first combination studied for textile wool fibre. The CH_4 has a substantial increment from the original fabric after the pretreatment combination with pretreatment conditions of autoclaving at 120 °C for 10 min, with alkaline endopeptidase enzyme in reaction for 8 h. After 46 days of HRT, the CH_4 yield recovery stood at 430 mL gVS^{-1}, showcasing a two-fold enhancement compared to thermal pretreatment alone. Additionally, the process yielded a six-fold increase in soluble chemical oxygen demand (sCOD).[78] Likewise, Forgács *et al.*[56] used chicken feathers in the same conditions and achieved 122% CH_4 yield enhancement. The combination of thermal and biological benefits of keratin decomposition. Hitherto, NaOH and biological pretreatment showed no CH_4 gas enhancement despite a high solubilisation rate (96%) when pretreating chicken feathers at 90 °C and 1.27 bar as a consequence of metabolite accumulation.[86]

A combination of micro-aeration and acid was studied by Wysocka *et al.*[88] to treat medical cotton waste. However, an increase of micro-aeration organic flow rate to 7.8 mL h^{-1} led to decreased CH_4 production owing to an increase in the loading rate of O_2 and H_2SO_4 addition, leading to poor breaking of the crystallinity structure of the cellulose in the cotton textile waste, which reduced the BMP to 62%.

Table 4. Thermal pretreatment of textile fibre for biogas production

Technique	Substrate	Treatment conditions	AD conditions	CH_4 yield (mL gVS^{-1}) Untreated	Treated	Change in BMP (%)	References
Autoclave	Cotton textile	120 °C, 10 min	Temp.: 37 °C HRT: 40 days	158	204	+29	Hasanzadeh *et al.*[43]
	70/30 Wool/nylon 6	120 °C, 10 min	S/I ratio: 2.5:1 Temp.: 55 °C HRT:46 days	22	210	+855	Kabir *et al.*[78]
Hydrothermal	Cotton stalk	Ammonia, 100 °C, 30 min	OLR: 1.6 g/L Temp.: 38 °C HRT:40 days	53	144	+172	Adl *et al.*[79]

Abbreviations: AD, anaerobic digestion; BMP, biomethane potential; HRT, hydraulic retention time; OLR, organic loading rate; S/I, substrate-to-inoculum; VS, volatile solid.

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Table 5. Combined pretreatment of textile fibre for biogas production

Technique	Substrate	Treatment conditions	AD conditions	CH_4 yield (mL gVS^{-1}) Untreated	Treated	Change in BMP (%)	References
Thermal +Biological	70/30 Blended Wool/nylon 6	Thermal: Autoclave at 120 °C, 10 min Biological: alkaline endopeptidase, 8 h	S/I ratio: 2.5:1 Temp.: 55 °C HRT: 46 days	22	430	+1855	Kabir *et al.*[78]
Mechanical + Chemical	Cotton waste	Mechanical: Micro-aeration Chemical: H_2SO_4	OLR: 5 gVS Temp.: 38 °C HRT: 30 days	653	247	−62	Sołowski *et al.*[30]
Chemical +Thermal	Medical cotton waste	Thermal: Autoclave at 120 °C, 20 min Chemical: $Ca(OH)_2$	Temp.: 35 °C SRT: 90 days	13	18	+38	Ismail and Talib[85]
Chemical +Biological	Chicken feather	Alkali: NaOH Biological: *Flavobacterium pennivorans* bacteria	S/I ratio: 2:1 Operating Temp.: 65 °C HRT: 80 days	123	123	0	Costa *et al.*[86]

Abbreviations: AD, anaerobic digestion; BMP, biomethane potential; HRT, hydraulic retention time; OLR, organic loading rate; S/I, substrate-to-inoculum; VS, volatile solids.

HOLISTIC ASSESSMENT OF PRETREATMENT TECHNOLOGIES FOR TEXTILE WASTE

A holistic assessment involves considering the technology readiness level (TRL) and societal technology level (SRL) to understand the technology maturity and public and stakeholders' acceptance of existing technology. Environmental impact assessment (EIA) was conducted on pretreatment technologies to assess the environmental implications by quantifying C footprint, particularly concerning textile waste disposal in Auckland. Then, a weighted scoring analysis (WSA) was conducted to assess the feasibility of pretreatment technology on fibre substrate.

Technology readiness level of pretreatment technologies

The TRL assesses the critical technology elements (CTE) development stage throughout the programme research, development and deployment phases. TRL uses a nine-point scale system developed by NASA,[89] where TRL 1–3 is defined as the laboratory scale, TRL 4–6 as the pilot scale, and 7–9 as the commercial scale according to the literature (Fig. 2).[90] Table S1 in the Supporting information shows the explanation of each TRL criterion.

The methodology involved gathering information from Loo *et al.*[91] and Damayanti *et al.*,[32] applying the pretreatment technologies above for recycling. Mechanical technology ranks the highest, at TRL 7–9, for textile waste recycling owing to scalability, low

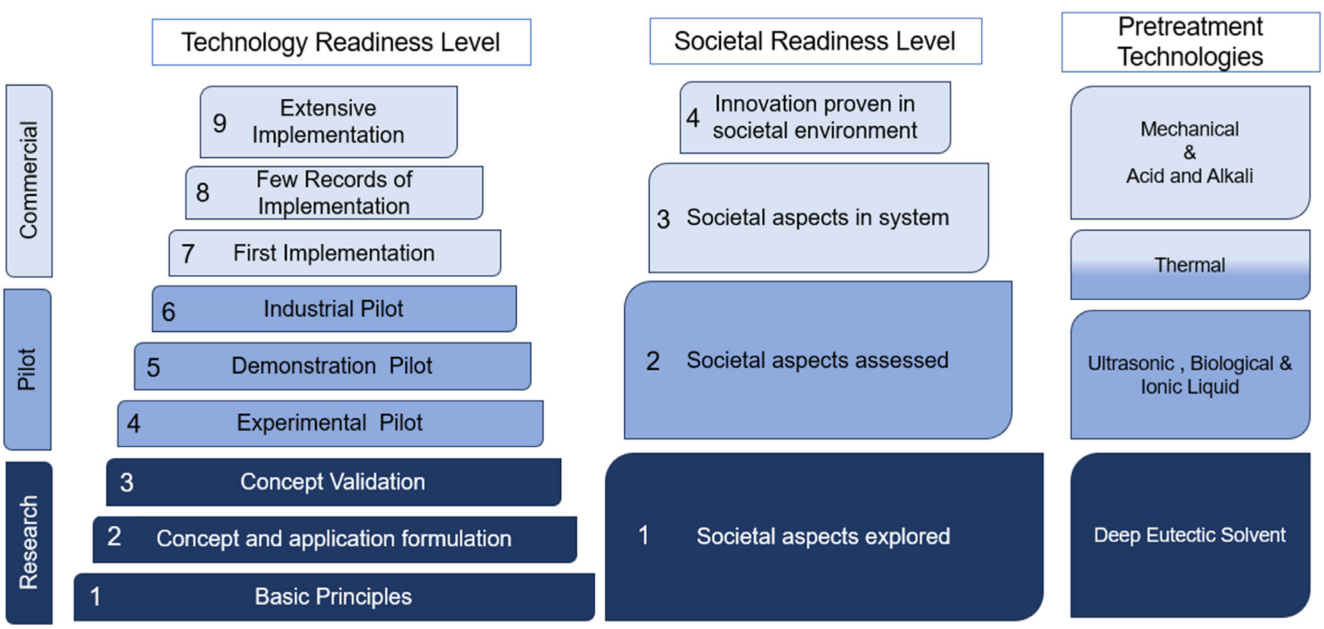

Figure 2. Technology readiness level (TRL) and societal readiness level (SRL) status of textile waste pretreatment technologies.

cost, low energy demand and easy operation. Chemical recycling ranks second, with TRL 5–6 for blended textiles utilising ionic liquid (e.g. NMMO) for separation, and TRL7–9 for pure cotton textiles primarily employing acid and alkali. Chemical recycling is on a commercial scale as a result ofefficient dissolution, hydrolysis and glycolysis methods. This method can recycle textiles into monomers, a potential closed-loop recycling method. There is limited research on DES, primarily owing to ongoing experimentation at the laboratory scale, indicated by TRL 1–3. Hydrothermal, categorised as thermal technology, at TRL 6–7 falls between pilot and commercial scales because of efficient pure and blended fibre pretreatment, on the one hand, high capital cost and heavy water consumption, on the other. Enzymatic hydrolysis, a biological technology, is categorised at a pilot scale because of milder reactions with lower operational costs, that are neverthless hindered by low recycling efficiency and duration.

Societal readiness level of pretreatment technologies

The SRL assesses the societal adaptation of a technology or innovation to be integrated into society. SRL ranges from 1 (lowest) to SRL 4 (highest), which is related to the TRL decision (Fig. 2). At SRL 1, the technology is under exploration and at level SRL 2, technology development is accessed with the cooperation of relevant stakeholders. The solution is refined in the end stages (SRL 3&4), and the technologies are implemented commercially.[92] Table S2 shows the explanation of each SRL criterion.

Commercially, stakeholders prefer mechanical and chemical technologies as a result of low energy demand and easy operation, which are assigned SRL 3–4.[32,91] Ultrasonic pretreatment is considered to be at the pilot scale for fibre pretreatment,[93] because of the high cost and energy consumption,[60] and expensive upscaling.[25] Hydrothermal pretreatment technologies are globally commercialised in wastewater treatment plants (WWTPs).[94] However, textile waste is still at the development stage owing to high capital costs, which hinder textile management decisions. Biological pretreatment technology is of lower priority because of low efficiency and long pretreatment time,[60] which affects scalability decisions. Ultrasonic, hydrothermal, IL and biological systems can be regarded as SRL 2 because stakeholders' decision-making regarding technology implementation is hindered by cost and performance. DES is limited in pretreatment research owing to the research stage, distinguished in SRL 1. Table 6 describes the designated TRL, SRL and stakeholders involved in each pretreatment technology.

Environmental impact analysis of pretreatment technologies in Auckland, New Zealand

The textile waste pretreatment technology's EIA was simulated using the waste reduction algorithm (WAR). This algorithm is used to quantify the potential environmental impact (PEI) of chemical and energy process simulation and calculate the possible effects of chemical processes on the environment.[96,97] The WAR algorithm evaluates eight impacts categories: human toxicity potential by ingestion (HTPI), human toxicity potential by exposure (HTPE), aquatic toxicity potential (ATP), terrestrial toxicity potential (TTP), global warming potential (GWP), ozone depletion potential (ODP), photochemical oxidation potential (PCOP) and acidification potential (AP) stated in Table S3. The eight PEI categories listed above were totalled in a single PEI index expressed per year (PEI year^{-1}).[97–99] The calculations are only a 'gate-to-gate analysis'.[96]

Figure 3 depicts the total PEI of pretreatment technologies applied to the textile waste disposed of by Aucklanders, regarding the values obtained for each category. On average,

Table 6. Comprehensive description of each technology for technology readiness level (TRL) and societal readiness level (SRL)criterion[32,91,93,95]

Pretreatment technology	TRL level	Description	SRL level	Description	Company/Institution
Shredding and grinding	7–9	Mature, scalable, low cost, low energy demand, easy operation	3–4	High societal acceptance and commercial use	Andritz, Valmet Technologies
Ultrasonic	5–6	Pilot scale for fibre pretreatment, high cost, and energy consumption challenges	2	Societal readiness is hindered by high-cost upscaling, which is attributed to assessing stage	Centre of Biological Engineering (CEB), University of Minho
Acid and alkali	7–9	Mature, efficient recycling into monomers	3–4	High societal acceptance and commercial use	EVRNU, BlockTexx, Renewcell, Worn Again Technologies, Sodra, Lenzing, Infinited Fiber
Ionic liquid	5–6	Pilot scale using ionic liquids for blended fibre separation	2	The development stage is being assessed because of high cost and nonrecyclability	Tencel, Ioncell
Deep eutectic Solvent	1–3	Effective for blended fibre pretreatment but lacking in maturity	1	Exploration stage owing to limited research	Beijing Key Laboratory of Ionic Liquids CleanProcess
Hydrothermal	6–7	Effective for pure and blended fibre depolymerisation, limited by high capital costs.	2	The development stage is being assessed as hindered by high capital costs	Circ, Tyton BioScience
Biological	5–6	Pilot scale, mild reactions, lower operational costs, but low recycling efficiency.	2	Less prioritised owing to low efficiency and scalability challenges, allocated in accessing stage	Carbios, HKRITA

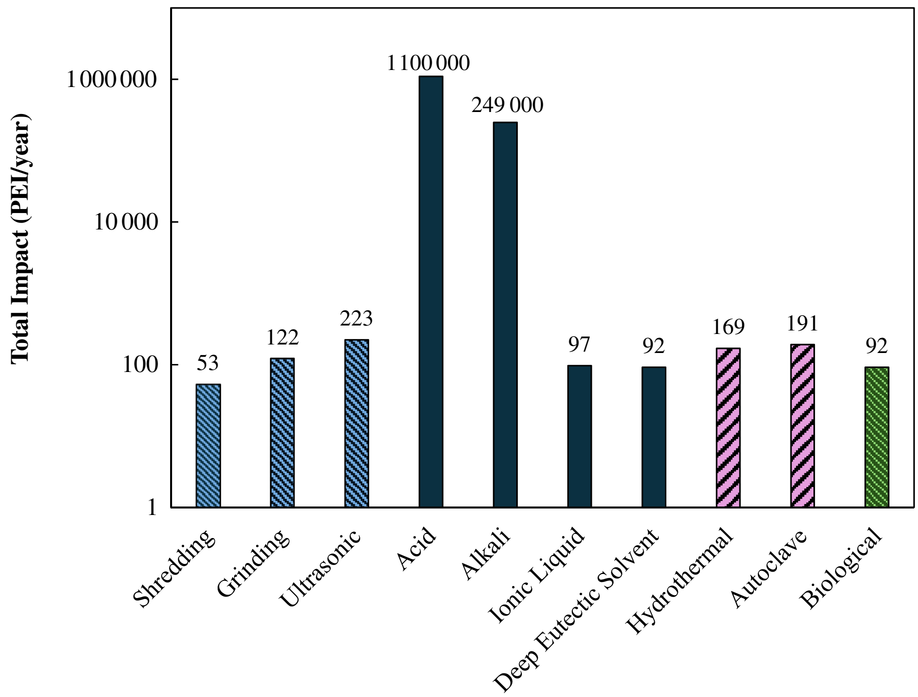

Figure 3. Potential environmental impact scores for pretreatment technologies of textile waste in Auckland with energy generation.

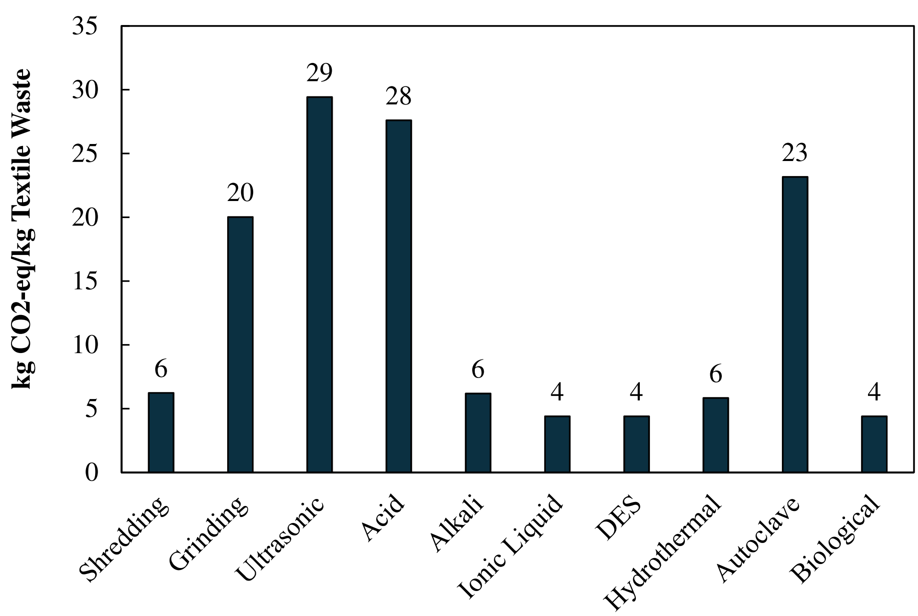

Figure 4. CO_2 emissions of each textile waste pretreatment process in Auckland, New Zealand.

New Zealanders dispose of ≈220 800 t of textile fabric annually.[100] Auckland's population is 32% of New Zealand's population, estimated at 89600 t annually. PEI ranks in decreasing sequence shredding >DES & biological > IL > grinding > hydrothermal > autoclave > ultrasonic > alkali > acid. Chemical pretreatment using H_3PO_4 was chosen owing to its efficient pretreatment ability on fibre substrate (Table 2), contributing the highest total PEI leaving the system. The highest PEI accounted for acid pretreatment owing to the HTPE category's significant contribution of 40 500 mg m^{-3} causing toxicity and corrosion, contributing to human health risks and water pollution. However, DES and biological pretreatments depict the lowest environmental impact owing to their environmentally friendly nature.

Figure 4 shows the CO_2 emissions from the pretreatment of textile waste collected for disposal in Auckland, New Zealand. CO_2 emission conversion factors were calculated based on Intergovernmental Panel on Climate Change (IPCC) guidelines for National GHG inventories. For example, 56 100 kg CO_2-eq per terajoule (TJ) applicable for shredding, grinding, ultrasonic and autoclave as a consequence of electricity utilisation; acid (5.34 kg CO_2-eq kg^{-1}); alkali (0.42 kg CO_2-eq kg^{-1}); IL and DES solvents (0.01 kg CO_2-eq kg^{-1}); water (0.34 kg CO_2-eq cm^{-3}) for hydrothermal pretreatment and biological (0.01 kg CO_2-eq kg^{-1}).[101] The chemical pretreatments used in this assessment were acid (H_3PO_4), alkali (Na_2CO_3), IL (NMMO) and DES (chlorine chloride: lactic acid). They were selected due to thir higher efficiency in terms of BMP production.

The basis was 89 600 t of textile waste generated in Auckland annually. The CO_2 emission factors for the textile waste involved are 60/40 blended polycotton (3.16 kg CO_2-eq kg^{-1}), cotton (5.34 kg CO_2-eq kg^{-1}) and wool (14.07 kg CO_2-eq kg^{-1}).[102,103] From Fig. 3, the C emission ranks in decreasing order: ultrasonic > acid > autoclave > grinding > shredding & alkali > hydrothermal > IL, DES & biological. Ultrasonic has the highest energy consumption (84 kg CO_2-eq kg^{-1} textile waste) compared to others. This is because of high electricity consumption of ≈14 400 kJ h^{-1},[104] generating a substantial amount of C emissions.

Weighted score analysis

A WSA, known as multicriteria decision analysis (MCDA), was used to evaluate and select a suitable pretreatment for textile waste before the AD process. WSA determines how well those alternatives rate against a chosen set of structured and weighted criteria.[105] This method makes subjectivity explicit in decision-making processes and combines objective measurements with value judgments. The decision involves more than just selecting the best option; it involves learning about, investigating and comprehending the issue and its priorities, values and potential outcomes.[106] The objective measurements involved are pretreatment duration, CH_4 gas enhancement and production, and PEI in the environmental impact factor category (EIF), which are measurable, whereas other criteria are subjective (Table S4). The measurable ranges are based on subjective manipulation.

Each criterion was assigned a numeric scoring value based on the publicly available information and literature discussed.[107] From Table S5, selection was attempted by critically assessing pretreatment for four different aspects: (i) technical, (ii) economic, (iii) environmental and (iv) operational aspects were adapted from these journals as outlined in Table 7.[107,108] As for the numerical importance based on criteria, technical was weighted as the highest (40%), followed by economical (25%), environmental (20%) and operational (15%). Based on the scenarios, Saaty[109] assigned numerical scoring values of 0 (unstudied), 1 (least important), 3 (important) and 5 (most important) to each subcriterion.

The pretreatment's weighted scores were calculated by multiplying the corresponding numerical scores by the criterion's priority values. In this direction, the pretreatment that meets the priorities highlighted in a scenario can be chosen. The pretreatment that most closely matched the priority criteria would get the highest weighted scores.[110] The outcomes of the WSA for each pretreatment for fibre substrate are depicted in Fig. 5.

Under technical criteria, hydrothermal pretreatment (scoring 140) demonstrated superior performance. Hydrothermal technology was ranked as the most promising in terms of technical aspects. For example, by comparing hydrothermal and autoclave methods, the CH_4 enhancement achieved 180% BMP from hydrothermally treated cotton stalk, which is far better than autoclaving cotton textile fibre.[79] The observed prominence of hydrothermal treatment indicates its efficacy in facilitating the efficient hydrolysis of cellulose structure, thereby contributing to an elevated CH_4 gas yield. Contrariwise, shredding is depicted as the lowest score (74), owing to low CH_4 gas production and enhancement during anaerobic digestion. Although the process duration is short and TRL is high, this technology needs additional improvement, such as a combination with other efficient technologies to optimise methane via the AD process.

From an economic perspective, shredding emerges as a highly economically feasible pretreatment option, commanding a weighted score of 125. Subsequently, DES ranks second with a score of 105. This ranking is attributed to the inherent advantage of low capital, operational and maintenance costs associated with the shredding process, irrespective of the specific pretreatment method employed. Literature supports this ranking because shredding is considered the most effective treatment method with low capital and operational costs as a consequence of less energy consumption and no chemical usage.[32,111] Biological pretreatment is considered moderate owing to the expensive cost of enzymes and requires additional chemicals for textile degradation.[91] The least achieved technologies are ultrasonic, grinding and IL pretreatment, which are considered economically unfeasible to scale-up owing to the high machinery and chemical costs.[60,112,113]

For environmental criteria, hydrothermal and DES emerge as the top-performing technologies. This ranking is justified by these treatments' relatively EIF and heat reaction. Hydrothermal pretreatment is industry-friendly, requiring only water, a clean, renewable resource that is generally readily available.[114] Hydrothermal is endothermic and inhibits heat release to the environment.[115,116] DES is also an environmentally nontoxic component, biodegradable, with low vapour pressure, and easy to recycle.[117] Alkali and acid score the lowest in environmental concerns because of GHG emissions by volatile organic compounds (VOCs) and ammonia (NH_4) from the alkali processes, as well as high corrosion, sulfur oxides (SO_X) and nitrogen oxides (NO_X) from acid processes.[31] In addition, acid and alkali are considered exothermic processes,[118] which may contribute to global warming and climate change.

Regarding the operational aspect, ultrasonic and biological are ranked as the highest-scoring technology for operation criteria with 75 points owing to fewer health and safety requirements and operability skills. For ultrasonic pretreatment, no reagent is required for operation, and more accessible operability skills require knowledge of power, frequency and duration to operate the machine.[119] Biological pretreatment has the advantage of mild action conditions and low energy demand, which prevent high risk during operation.[31,119] However, grinding ranks as the lowest (15 points) owing to accuracy and precision requirements in the final production, which requires more operators with proper training.[113] Microfibre/microplastic release during grinding may cause acute and chronic effects on employees' health.[120]

Overall, the ranking is represented in decreasing order: hydrothermal > DES > shredding > biological > ultrasonic & autoclave > alkali > ionic liquid > grinding > acid. Hydrothermal pretreatment, scoring 340, is considered the most feasible method for textile fibre. Hydrothermal pretreatment excels owing to its efficient biogas production, scalable nature, low operational and maintenance cost, environmental benefits and social acceptance, which is beneficial for industrial applications.

LIMITATIONS AND AREA FOR FUTURE RESEARCH

The current literature on the effectiveness of pretreatment methods for AD has several inherent weaknesses that can be

Table 7. Decision-making aspects and the scoring guide of weighted-scoring analysis

Subcriterion	Decision-making impact	Scoring guide	Weightage
Technical (40%)			
Duration for pretreatment	Average time for pretreatment	1: Long (>1 day) 3: Medium (1 h–1 day) 5: Short (<1 h)	3
CH_4 gas production	Concentration of CH_4 gas production	1: Low (<100 mL gVS^{-1}) 3: Medium (100–200 mL gVS^{-1}) 5: High (>200 mL gVS^{-1})	13
CH_4 gas enhancement	Average percentage CH_4 enhancement	1: Low (<50%) 3: Medium (50–100%) 5: High (>100%)	20
Technology readiness level	It should be evaluated using the TRL framework	1: Laboratory scale (TRL 1–3) 3: Pilot scale (TRL 4–6) 5: Commercial scale (TRL 7–9)	4
Economical (25%)			
Capital cost	Amount of cost required for equipment	1: High cost 3: Medium cost 5: Low cost	15
Operational and maintenance cost	Amount of cost for employees, raw materials, repairs, and maintenance	1: High cost 3: Medium cost 5: Low cost	10
Environmental (20%)			
Environmental impact factor (EIF)	Evaluated from: (a) Potential environmental impact (PEI) Low: 1–100 PEI $year^{-1}$ Medium: 101–1000 PEI $year^{-1}$ High: >1000 PEI $year^{-1}$ (b) Water and air contamination (c) Waste generation	1: High pollution 3: Medium pollution 5: Low pollution	15
Heat reaction	Evaluate the heat emission produced from the energy consumption.	1–Exothermic 3–Exothermic/endothermic 5–Endothermic	5
Operational (15%)			
Health and safety	Involvement of critical parameters (examples: chemicals, pressure and temperature) to ensure a level of safety.	1–High hazard level 3–Medium hazard level 5–Low hazard level	7
Operation difficulty	Ensure the technology is user-friendly.	1–High operation skills 3–Medium operation skills 5–Low operation skills	5
Societal acceptance	Acceptance by stakeholders for technology application while considering efficiency and environmental criteria.	1–Low acceptance (SRL 1) 3–Average acceptance (SRL 2) 5–High acceptance (SRL 3–4)	3

Abbreviations: TRL, technology readiness level; SRL, societal readiness level; VS, volatile solids.

addressed. One significant area of concern is the impact of AD operation characteristics. Studies often do not adequately differentiate between mesophilic and thermophilic conditions, even though these conditions influence microbial communities and metabolic pathways, affecting degradation efficiency and biogas yield depending on textile fibre types. Additionally, in the diverse

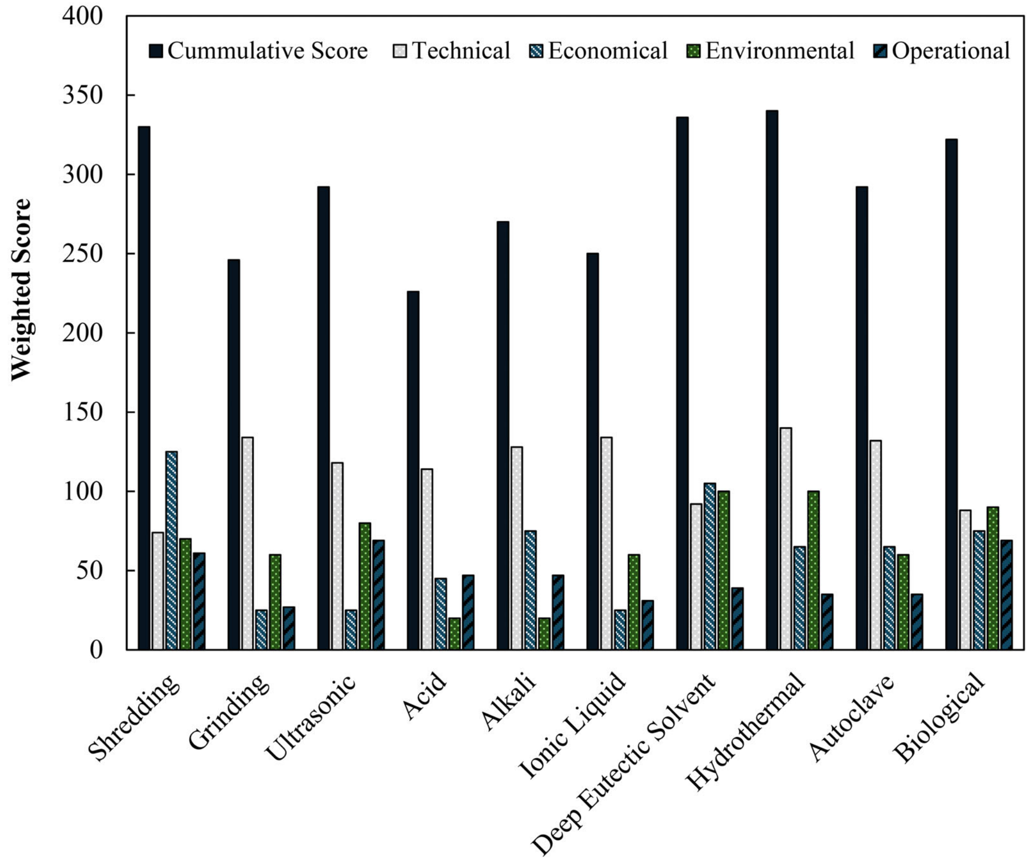

Figure 5. Weighted-scoring analysis for fibre pretreatment.

range of reactor types, such as batch, continuous stirred-tank reactors (CSTR) and plug-flow reactors (PFR), varying operational parameters such as HRT and OLR lead to variable results. This variability makes it difficult to generalise the effectiveness of different pretreatment methods.

Another major gap in the literature is the effect of residual waste post-AD. The literature concluded that mechanical and chemical pretreatment contributes to environmental impact. However, a detailed assessment of the magnitude of the residual waste remaining after AD for each pretreatment method is lacking, which hinders the determination of the disposal cost. The disposal methods and associated costs can vary widely, significantly affecting the overall feasibility and sustainability of different pretreatment technologies. A sensitivity analysis is necessary to compare the residual waste magnitude and disposal cost of each technology for better evaluation.

Lastly, there is still no clarity in AD selection for textile fibre treatment. Emerging innovative technologies such as direct combustion, pyrolysis and gasification offer alternatives to AD by converting organic waste directly into energy or valuable products. In New Zealand's evaluation case, AD remains a preferred option owing to its well-established technology, economic feasibility and environmental benefits.[121] However, AD selection for textile fibre is still lacking. In addition, integration with pretreatment technologies for AD is necessary for textile fibre as a consequence of complex structural challenges that inhibit CH_4 production. Pretreatment evaluation in conjunction with AD is required but varies in technological maturity, cost-effectiveness and environmental aspects.

By addressing these limitations, future research can provide a more complete understanding of the effectiveness of pretreatment methods and potentially identify more efficient and sustainable alternatives.

CONCLUSIONS

The complex structure of textile fibre substrates poses a significant challenge to microbial degradation in AD, necessitating effective pretreatment technologies. Through a comprehensive analysis, hydrothermal pretreatment emerged as the most feasible technology for processing fibre substrates. This conclusion is supported by hydrothermal pretreatment's balanced performance across technical, economic and environmental criteria. The advantages of hydrothermal technology include its high technical efficiency, scalability and reduced environmental footprint.

Despite its promise, the application of hydrothermal pretreatment in the context of textile fibre for biogas production remains underexplored. Future research focusing on optimising hydrothermal pretreatment parameters could significantly enhance biogas yields in the textile industry, paving the way for more sustainable and cost-effective waste management solutions. This study highlights the necessity for continued innovation and investigation into pretreatment technologies to unlock the full potential of fibre substrates in bio-energy production.

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SUPPORTING INFORMATION

Supporting information may be found in the online version of this article.

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