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ORIGINAL PAPER

# Thermal hydrolysis pretreatment for anaerobic digestion of cotton textile waste

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## Abstract

Cotton textile waste is challenging to digest and convert to biogas during anaerobic digestion (AD) due to high crystallinity of cellulose. This study investigated thermal hydrolysis pretreatment (THP) at 120–200 °C for 30 and 60 min to enhance cotton biodegradability prior 21 days of mesophilic AD at a substrate-to-inoculum ratio of 0.5 volatile solids (VS) basis. THP increased cotton solubilisation up to 98% VS removal, at the most sever condition (200 °C, 60 min), crystallinity decreased by 11.84% and COD solubilisation reached 51. However, the highest methane yield was achieved under moderate conditions (140 °C, 60 min), producing 17.92 mL $CH_4$/g VS, representing a 123% increase over untreated cotton, indicating an optimal balance between solubilisation and process inhibition. Gompertz modelling showed excellent agreement ($R^2 \geq 0.99$), with a reduced lag phase (2.3 days) and ~2-fold higher biodegradability than untreated cotton. Digestate pH, ammonia nitrogen, and volatile fatty acid (VFA) remained within non-inhibitory ranges under the optimal THP condition, supporting stable and efficient methane production. These results identify cotton-specific THP operating conditions and demonstrate THP-AD as a pathway for valorising cotton textile waste.

**Keywords** Anaerobic digestion · Thermal hydrolysis pretreatment · Cotton textile waste · Biogas · Methane

## Abbreviations

| | |
|---|---|
| AD | Anaerobic digestion |
| ATHP | Advanced thermal hydrolysis process |
| COD | Chemical oxygen demand |
| C/N | Carbon-to-nitrogen ratio |
| CrI | Crystallinity index |
| EMP | Experimental methane production |
| FAN | Free ammonia nitrogen |
| FT-IR | Fourier transform infrared spectroscopy |
| GC-FID | Gas chromatograph with flame ionisation detection |
| HTP | Hydrothermal treatment process |
| $NH_3$-N | Ammonia–nitrogen |
| S/I | Substrate-to-inoculum ratio |
| sCOD | Soluble chemical oxygen demand |
| SEM | Scanning electron microscopy |
| STP | Standard temperature and pressure |
| TAN | Total ammonia nitrogen |
| TBMP | Theoretical biochemical methane potential |
| tCOD | Total chemical oxygen demand |
| THP | Thermal hydrolysis pretreatment |
| TS | Total solids |
| SF | Severity factor |
| VFA | Volatile fatty acids |
| VS | Volatile solids |
| VSS | Volatile suspended solids |
| XRD | X-ray diffraction |

## Introduction

Global textile consumption poses a critical environmental challenge, intensified by accelerating population growth and the proliferation of fast-fashion models (Juanga-Labayen et al. 2020). In 2024, cotton textiles accounted for 19% of global production, making it the second-most-produced textile material after polyester (Exchange 2025). Massive cotton production results in substantial waste (Abtew et al. 2025). On a global scale, approximately 92

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million tonnes of textile waste are generated annually from post-consumer use, largely flowing to developing countries for second-hand and total waste, although consumption originates predominantly in high-income countries with higher per capita waste generation. The majority of this waste (57%) is disposed of in landfills, with the remainder incinerated or recycled (UNEP 2025; Waste 2024).

Cotton accounts for approximately 25% of global textile waste annually, representing the largest pure textile waste stream worldwide. This cotton textile waste primarily comprises apparel and household textiles characterised by low durability and short lifespans, which rapidly reach the end of life (LIDA 2024; Sharma et al. 2025). Cotton textile waste in landfills contributes to greenhouse gas emissions (Hu et al. 2018; Jin et al. 2022; Tharmarajah et al. 2024b), releasing an average of 1.5 kg $CO_2$-equivalent per kilogram of textile waste (Directory 2025; Hu et al. 2018), owing to the inefficient collection of the landfill gas collection system. Therefore, a sustainable solution for cotton textile waste management is needed as a mitigation strategy.

Compared with conventional textile recycling methods, anaerobic digestion (AD) is a promising waste-to-energy technology because of its benefits in renewable energy production, greenhouse gas mitigation, and scalability (Jin et al. 2022; Tharmarajah et al. 2024a, 2024b). AD is a biological process in which microorganisms convert organic substrates into methane and carbon dioxide through a series of sequential steps (hydrolysis, acidogenesis, acetogenesis, and methanogenesis). Owing to their high cellulose content (~80-98%), cotton textiles, organic substrates that are a promising feedstock for AD, provide an energy-rich substrate for microbial conversion (Hasan et al. 2025; Marinho 2025). Cotton cellulose, derived from the Gossypium plant species, is an ordered biopolymer of β-D-glucose units linked by β-1,4-glycosidic bonds (Cai et al. 2025; Huang et al. 2021). As a carbon-rich polymer, cellulose is an important source for the hydrolysis stage, where complex polymers are broken down into soluble sugars that are accessible to microbes and drive subsequent acidogenesis, acetogenesis, and methanogenesis (Y Wang et al. 2023).

Despite the advantageous properties of cotton textiles for AD implementation, the intrinsic crystalline architecture of cellulose limits microbial accessibility, thereby reducing biogas production efficiency (Juanga-Labayen et al. 2020; Scherzinger & Kaltschmitt 2021). In addition, cotton textile substrates are classified as carbon-rich when their carbon-to-nitrogen (C/N) ratio exceeds 30 (Rajlakshmi et al. 2023). Carbon-rich substrates typically demonstrate elevated total solids (TS) concentrations, where values surpassing 15% indicate dry AD conditions, resulting in operational challenges including inadequate mixing, mass transfer limitations, and acidification, which cause a reduction in methane yields (Edwiges & Kaparaju 2025; Gao et al. 2023; Wangliang Li et al. 2023; Motte et al. 2014).

To address these constraints, pretreatment methodologies are critically needed to increase cotton cellulose biodegradability during AD processes (Hasanzadeh et al. 2018; Jeihanipour & Taherzadeh 2009). While numerous investigations have examined cotton pretreatment for AD enhancement through biological, physical, and chemical approaches, these techniques remain limited by suboptimal methane production efficiency, adverse environmental impacts, and economic constraints (Tharmarajah et al. 2024b).

Therefore, thermal hydrolysis pretreatment (THP), a hydrothermal pretreatment process (HTP), has garnered significant attention as a clean and energy-efficient technology that enhances biogas production before AD. This technology employs liquid water heated at moderate temperatures (120–200 °C) under elevated pressure (9–15 bar) in the absence of oxygen (Guangyin & Youcai 2017; Ngo et al. 2023b; Thompson et al. 2020). The THP effectively reduces cellulose crystallinity while increasing the proportion of amorphous cellulose, thereby increasing accessibility for enzymatic hydrolysis during AD. This structural modification facilitates the conversion of complex polysaccharides into fermentable sugars, ultimately improving the methane yield.

The THP process has been widely investigated for biogas yield enhancement in the anaerobic digestion of various substrates, including sewage and waste-activated sludge (WAS) (Ngo et al. 2023b), brown seaweeds (Thompson et al. 2020), food waste (Thompson et al. 2021a, b) and lignocellulose substrates (Adl et al. 2012; Passos et al. 2018). Specifically, several studies have demonstrated the effectiveness of THP for methane production from carbon-rich substrates with lignocellulosic characteristics similar to those of cotton, as documented in Table 1. For example, the THP of cotton stalks, lignocellulosic residues remaining in agricultural fields following cotton fibre harvest, resulted in a substantial 172% increase in methane yield compared with that of untreated substrates (Adl et al. 2012). Similarly, Hmeekong et al. (2025) reported that Napier grass, which contains 31.5% cellulose (dry weight basis), achieved the highest methane production of 184.5 $m^3$ $CH_4$ per tonne of dry biomass, representing a 65% improvement in biodegradability compared with that of untreated grass. These findings suggest considerable potential for using THP to valorise cotton textile waste.

As shown in Table 1, THP has demonstrated technical capabilities across various carbon-rich substrates and is therefore promising for the treatment of cotton textile waste. Cotton textiles are distinct from other lignocellulosic materials because of their high cellulose content, minimal lignin content, presence of finishing agents and dyes, and tightly woven structure that creates mass-transfer barriers. The rigid crystalline structure of cellulose in cotton can be disrupted through the THP process, increasing accessibility for microbial degradation. However, to the authors' knowledge, no previous studies have systematically evaluated optimal THP

**Table 1** Thermal hydrolysis pretreatment of carbon-rich substrates before anaerobic digestion

| Substrates | C/N | Total solid (%) | Optimal temperature (°C) | Optimal reaction time (min) | $CH_4$ changes (%) | Reference |
|---|---|---|---|---|---|---|
| Coffee husk | 52.22 | 89.4 | 120 | 60 | +37 | (Passos et al. 2018) |
| Kraft pulp solids | – | 35.2 | 70 | 60 | 0 | (Strong & Gapes 2012) |
| Pelagic Sargassum seaweed | 21.67 | 79.3 | 140 | 30 | +56 | (Thompson et al. 2020) |
| Orange peels | – | 39.7 | 140 | 30 | – | (Saadatinavaz et al. 2021) |
| Cotton stalk | 64.42 | 92.28 | 100 | 30 | +172 | (Adl et al. 2012) |
| Napier grass (*Pennisetum purpureum*) | 66.71 | 25.2 | 150 | 30 | +53 | (Hmeekong et al. 2025) |
| Pulp and paper sludge | – | 1.3 | 165 | 45 | +159 | (Goycoechea et al. 2023) |
| *Typha angustifolia* | – | 18.35 | 170 | 60 | +37 | (Kist et al. 2017) |
| Lignocellulosic residues | 59 | 92 | 180 | 5 | +27 | (Allegue et al. 2021) |
| *Ludwigia grandiflora* | – | 11.95 | 180 | 30 | +132 | (Bhatia et al. 2024) |

operating conditions for cotton textile waste, which represents a critical research gap.

This study's objectives explore the effects of THP operating conditions on the morphology, biogas production, and composition of cotton fabric waste. It also examines the impact of THP on organic matter reduction and the release of potential process inhibitors, specifically volatile fatty acids (VFAs) and ammonia–nitrogen compounds, during AD. This work provides novel insights by establishing cotton-specific optimal THP conditions for a previously unexplored feedstock. The findings reveal substrate-specific responses in solubilisation efficiency, methane production potential, and biogas quality parameters that deviate significantly from those reported for conventional lignocellulosic materials.

Furthermore, this study has environmental significance: it diverts cotton waste from landfill/incineration into a value-generating process (AD), yields renewable energy in the form of biogas, and could reduce the carbon footprint of textile waste management. Establishing cotton-specific THP conditions is important because cotton's high crystallinity and extreme C/N ratio make it one of the most challenging textile wastes for AD; even modest absolute methane yields can translate to meaningful gains when applied to high-volume textile waste streams.

## Materials and methods

### Materials

A 100% labelled, white, woven cotton textile was obtained locally and cut into sizes ranging from 0.1 to 0.5 cm. The fabric was pre-washed overnight in a washing machine with detergent to remove any sizing agents, finishes, or impurities. Table 2 shows the characteristics of the cotton textile waste.

The inoculum was sourced from the full-scale mesophilic anaerobic digester at the Mangere wastewater treatment plant in Auckland, New Zealand, which continuously processes municipal wastewater. The inoculum was refrigerated at 4ºC. For the inoculum, the initial pH was 7.2 ± 0.05, and the total solid (TS), volatile solid (VS), ammonia nitrogen ($NH_3$-N) and total chemical oxygen demand (tCOD) contents were 3.27 ± 0.09 (wt.%), 2.14 ± 0.13 (wt.%), 1026.25 ± 53.75 mg/L and 23,838 ± 12.5 mg/L, respectively.

Test kits for measuring chemical oxygen demand (COD) and ammonia nitrogen ($NH_3$-N) were sourced from Hach Pacific (Auckland, New Zealand). Reference standards for volatile fatty acids (VFAs), including acetic, propionic, butyric, and valeric acids, were purchased in chromatographic grade from Sigma-Aldrich.

### Thermal hydrolysis pretreatment

The thermal hydrolysis of cotton textiles was conducted via a 1 L autoclave reactor (Amar Equipment Ltd., Mumbai,

**Table 2** Characterisation of cotton fabric

| Parameter | Units | Values |
|---|---|---|
| Proximate analysis: | | |
| Moisture content | wt.% | 4.9 ± 0.37 |
| TS | wt.% | 95.1 ± 0.37 |
| VS | wt.% | 93.85 ± 1.12 |
| VS/TS ratio | % | 98.68 ± 1.11 |
| Ash | wt.% | 1.25 ± 0.06 |
| Ultimate analysis: | | |
| C | % of TS | 41.94 ± 0.22 |
| N | % of TS | 0.11 ± 0 |
| H | % of TS | 6.69 ± 0.02 |
| S | % of TS | 0.09 ± 0.13 |
| O | % of TS | 50.95 ± 0.27 |
| C/N ratio | | 381.27 ± 0.11 |
| Theoretical $O_2$ demand | mg $O_2$/g VS | 434.30 |
| TBMP | mL/g VS | 397 |

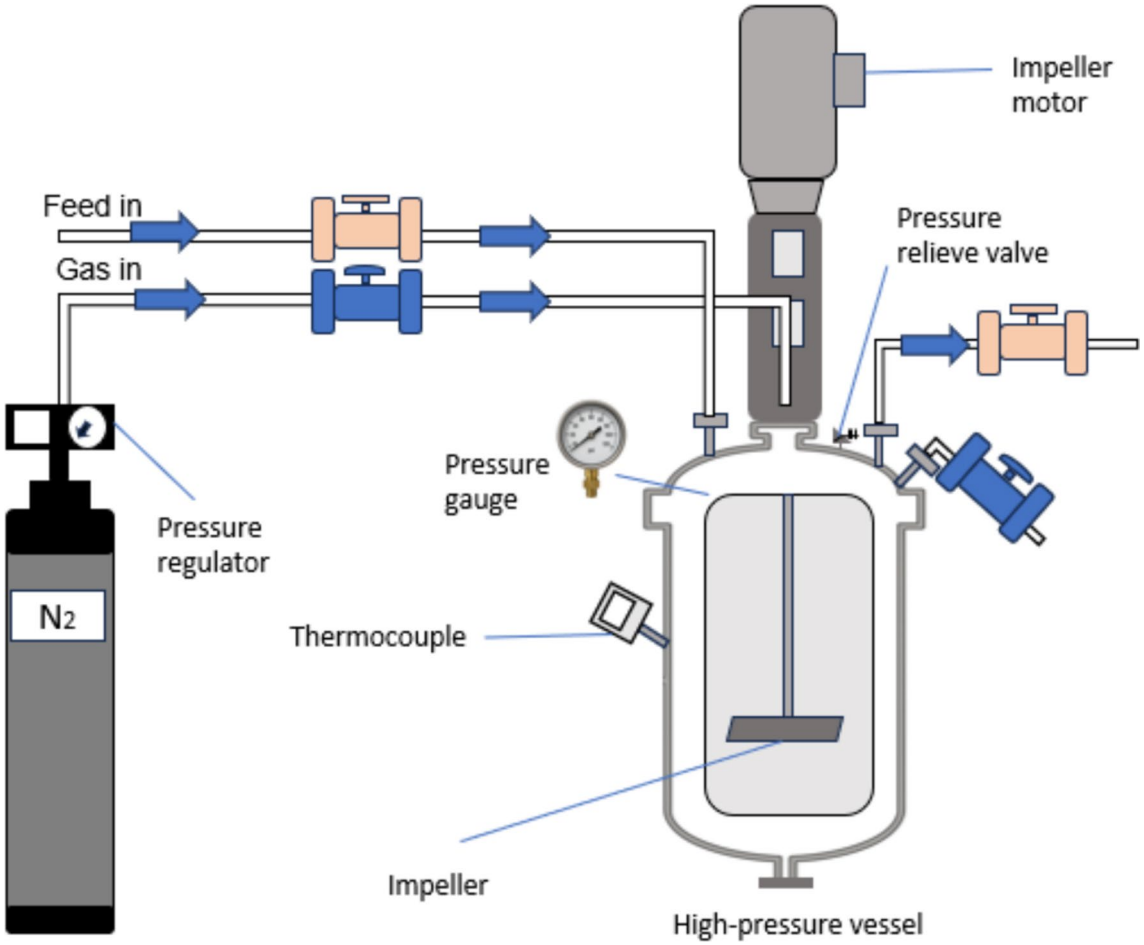



**Fig. 1** Experimental setup for thermal hydrolysis pretreatment

India) equipped with a 1 L flash tank, impeller, thermocouple and pressure gauge (Fig. 1). In each experimental run, cotton fabric and deionised water were combined in the reactor at a solid-to-liquid ratio of 1:99 (w/w), resulting in 1% total solids (TS). This lower substrate concentration was selected to improve the methane yield for textile waste, as reported by Azcona et al. (2023). The reactor was pressurised with 30 bar $N_2$ gas and stirred at 300 rpm. Pretreatment was tested at 120, 140, 160, 180 and 200 °C for 30 and 60 min. Pretreatment began once the reactor reached the target temperature. After each run, the reactor was cooled to room temperature, and the solid–liquid slurry was collected for analysis and microbial conversion.

## Anaerobic digestion

Figure 2 shows the setup of the AD system, which includes three connected Schott bottles: (1) a 100 mL digestion bottle, (2) a 1 L water displacement bottle, and (3) a 500 mL water collection bottle with Tygon tubes (Mazdak Rasapoor et al. 2016; Thompson et al. 2021a, b).

The solid and liquid-treated cotton textiles were added to the digestion bottle at a similar solid-to-liquid THP ratio. The pH of the liquid THP cotton was subsequently adjusted to 7.0 ± 0.1 with 0.5 M NaOH and 0.02 M HCl solutions to improve the bioconversion efficiency under optimal pH AD conditions (6.8–7.2) (Cioabla et al. 2012; Gonde et al. 2023; Ward et al. 2008). The experiment was conducted on the basis of a substrate-to-inoculum (S/I) ratio of 0.5 (VS basis) (Juanga-Labayen et al. 2020). The working mass in each 100 mL digestion bottle was 80 g.

After mixing and sealing, the samples were purged with nitrogen for 1 min to ensure an anaerobic environment and then sealed with a metal cap and a plastic cover. Then, the samples were incubated for 21 days at 35 ± 1 ℃. Daily biogas production was measured using the water-displacement method, and the cumulative yield was calculated at standard temperature and pressure (STP). Gas volumes were corrected to STP (0 °C, 1 atm), and net values were obtained by subtracting inoculum-only blanks. AD was performed in triplicate, and the results are expressed as the mean ± standard deviation (SD). The inoculum blanks were conducted

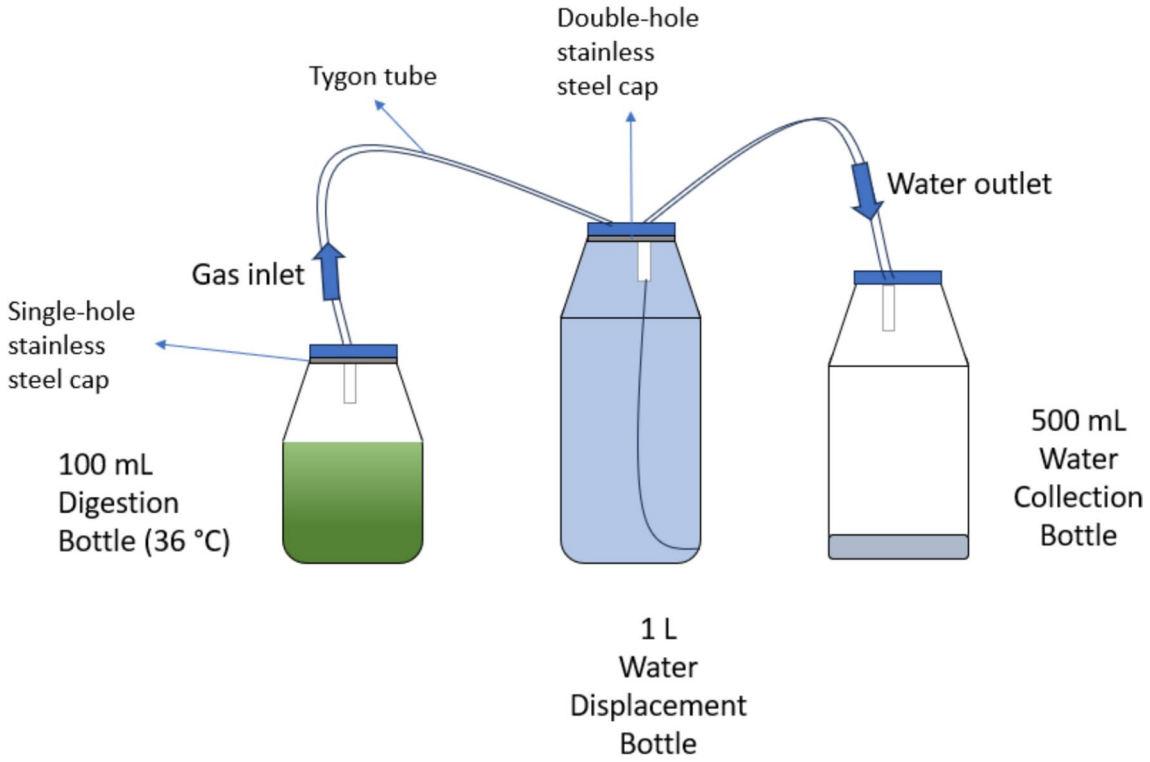



**Fig. 2** Experimental setup for anaerobic digestion

in triplicate, and used as a control, and the net biogas and methane contents were computed by subtracting the blanks.

At the end of AD, the net methane production obtained under each experimental condition was compared with the feedstock's theoretical biochemical methane potential (TBMP) for biodegradability ($B_D$), which was calculated with minimal error. The TBMP was calculated via Buswell and Boyle's formula (Eq. (1)) and deduced as (Eq. (2)). The $B_D$ was subsequently calculated via Eq. (3). This equation is based on elemental analysis of the substrates.

$$\begin{aligned} &C_xH_YO_zN_aS_b + \left(x - \frac{y}{4} - \frac{z}{2} + \frac{3a}{2} + \frac{b}{4}\right)H_2O \\ &\rightarrow \left(\frac{x}{2} - \frac{y}{8} + \frac{z}{4} + \frac{3a}{8} + \frac{b}{4}\right)CO_2 \\ &+ \left(\frac{x}{2} + \frac{y}{8} - \frac{z}{4} - \frac{3a}{8} - \frac{b}{4}\right)CH_4 + aNH_3 + bH_2 \end{aligned} \quad (1)$$

$$\begin{aligned} &\text{Theoretical Biomethane Potential}\left(\text{mL}\frac{CH_4}{\text{gVS}}\right) \\ &= \frac{22.4 \times 1000 \times \left(2x + \frac{y}{2} - z - \frac{3a}{2} - \frac{b}{4}\right)}{12x + y + 16z + 14a + 32b} \end{aligned} \quad (2)$$

where $x$, $y$, $z$, $a$ and $b$ represent moles of C, H, O, N and S, respectively; 22.4 (L) is the volume of 1 mol of gas at standard temperature and pressure, and 1000 is the conversion factor from litres to millilitres.

In addition, by multiplying the measured methane concentration (%) in biogas by the total volume of biogas produced, the biogas methane content was calculated, yielding the experimental methane production (EMP). Biodegradability (BD) was calculated using Eq. (3).

$$B_D(\%) = \left(\frac{EMP}{TBMP}\right) \times 100 \quad (3)$$

The THP and AD digestor performance was determined by the solid reductions in TS (%) and VS (%), as shown in Eq. (4) and Eq. (5), respectively.

$$\text{TSreduction}(\%) = \frac{\text{InitialTS} - \text{FinalTS}}{\text{InitialTS}} \times 100 \quad (4)$$

$$\text{VSreduction}(\%) = \frac{\text{InitialVS} - \text{FinalVS}}{\text{InitialVS}} \times 100 \quad (5)$$

where the initial and final TS and VS are represented in (wt.%).

The level of inhibition (free ammonia nitrogen (FAN)) was calculated via Eq. (6) and Eq. (7) (Capson-Tojo et al. 2020):

$$K_a = K_{a_25} \times e^{\frac{51965}{R}\left(\frac{1}{298.15} - \frac{1}{T}\right)} \quad (6)$$

$$FAN = \frac{K_{a\times TAN}}{K_a + 10^{-pH}} \quad (7)$$

where $K_a$ is the acid dissociation constant at temperature, $R$ is the ideal gas constant (8.314 J $mol^{-1}$ $K^{-1}$), $K_{a_25}$ are the acid dissociation constant at 25 °C ($10^{-9.25}$), total ammonia nitrogen (TAN), temperature $T$ in K and pH of the digestate.

## Solubilisation degree

Chemical oxygen demand solubilisation is the ratio of soluble chemical oxygen demand (sCOD) to total chemical oxygen demand (tCOD). This method was employed to determine the impact of THP cotton substrate solubilisation. Equation (8) was used to compute the solubilisation degree (Ngo et al. 2023b):

$$COD\ solubilisation\ \% = \frac{sCOD}{tCOD} \times 100 \quad (8)$$

## Severity factor

The reaction time and temperature were combined into a single metric, the "reaction ordinate" ($R_o$), to quantify the impact of various treatment conditions during THP. When processing is performed with different equipment/reactor scales under varying reaction conditions, $R_O$ helps integrate data from complex reaction systems and removes obstacles to data comparison. The reaction ordinate parameter is determined via Eq. (9):

$$R_O = texp\frac{\left(T_r - T_b\right)}{14.75} \quad (9)$$

where t is the THP treatment time (min), $T_r$ is the reaction temperature (℃), and $T_b$ is the base temperature (℃) at which the solubilisation mechanism occurs. In this study, the base temperature was set at 100 °C because, above this, the impact of THP on the solubilisation of cotton was observed. The severity factor (SF), which has been used to represent the severity of THP, is defined via Eq. (10):

$$\mathrm{SF} = \log_{10} R_O \quad (10)$$

Previous modelling studies of the THP of lignocellulosic biomass have utilised the severity factor. The SF is also reminiscent of other parameters employed for pulp and paper processes (Thompson et al. 2020). In this study, cotton was selected as the substrate, which is well-suited for evaluating the severity factor. The SFs of the different pretreatment conditions employed in this study are shown in Table 3.

## Gompertz model

The cumulative methane production was fitted via a modified Gompertz model for kinetic analysis (Eq. (11)):

$$B = P \times \exp\left\{-\exp\left[\frac{e \times R_{max}(\lambda - t)}{P} + 1\right]\right\} \quad (11)$$

where $B$ (mL/g VS) represents the cumulative biogas production at time $t$ and $P$ (mL/g VS) represents the final biogas production. $R_{max}$ (mL/d·g VS) refers to the maximum biogas production rate, $\lambda$ (d) is the lag phase time in days, and $e$ is Euler's number (2.718). $t$ (d) is the digestion time in days (Ajayi-Banji et al. 2021). The Gompertz model was fitted via nonlinear least squares regression implemented in the SciPy library (Python 3.12.x, Google Colab environment). The fitting was performed via the curve_fit function, which applies the Levenberg–Marquardt algorithm as the solver. Model performance was evaluated via the coefficient of determination ($R^2$) and the root mean square error (RMSE). The fits were made to net cumulative biogas production, expressed per gram of volatile solids (VS) added.

## Analytical methods

The American Public Health Association's standard techniques 2540D and 2540E were used to measure the moisture content, ash content, total solids (TS), and volatile solids (VS) content (APHA 2014). The weight loss resulting from drying the sample in an oven at 105 °C for 24 h and igniting it in a muffle furnace at 550 °C for 2 h was used to compute TS and VS, respectively. The ash content of the remaining sample is measured after the furnace ignition. The elemental analysis results (C, N, H, and S) were tested via an elemental analyser, a Vario elementary cube system (Elemental Analyser, GmbH, Germany) and the O content was calculated

**Table 3** Severity factors of different pretreatment conditions

| Temperature (°C) | Time (min) | Severity factor (SF) |
|---|---|---|
| 120 | 30 | 2.07 |
| | 60 | 2.37 |
| 140 | 30 | 2.65 |
| | 60 | 2.96 |
| 160 | 30 | 3.24 |
| | 60 | 3.54 |
| 180 | 30 | 3.83 |
| | 60 | 4.13 |
| 200 | 30 | 4.42 |
| | 60 | 4.72 |

via the equation (C + N + H + O + S = 100%) (Thompson et al. 2020; Yuen & Lau 2023).

A Hach DR3900 spectrophotometer (method 8000) was used to measure the digestate chemical oxygen demand (COD) content via a high-range COD test kit (20–1500 mg/L) from Hach. Similarly, for THP and AD digestate samples, the Hach salicylate low range (method 10,023) and high range (method 10,031) were used to assess the ammonia–nitrogen ($NH_3$-N) content. A Hach DR3900 spectrophotometer was used to take measurements for each test kit. The sample pH was measured via a Thermo-Scientific Orion Star A211 pH meter. Volatile fatty acids, such as acetic acid, propionic acid, butyric acid, and valeric acid, were detected via a Shimadzu QP2010 Plus gas chromatograph (Kyoto, Japan) with flame ionisation detection (GC-FID). The column used in the instrument was a nitro terephthalic-acid-modified polyethylene glycol (PEG) column (DB-FFAP, 30.0 m × 0.53 mm × 0.5 µm, Agilent Technologies, US). The carrier gas was helium. The column was first heated to 40 °C and maintained at this temperature for 2 min after sample injection. A temperature ramp-up rate of 10 °C/min was used, and the column was held at 180 °C for 26 min. By calibrating reference standard solutions to sample peak retention durations, VFA peaks were identified. Before injection into the GC, the samples were centrifuged at 5000 rpm for 5 min.

For the solid samples, Fourier-transform infrared (FT-IR) spectroscopy, scanning electron microscopy (SEM), and X-ray diffraction (XRD) were performed. Before FT-IR, XRD and SEM analysis, the solid cotton after THP was separated via a sieve, washed with distilled water, and dried in an oven at 50 °C overnight. FT-IR was performed under pre- and post-maximum THP conditions on cotton substrates using a PerkinElmer Spectrum 100 FT-IR spectrophotometer (USA) at a resolution of 4 cm-1 to evaluate changes in chemical composition and functional groups. Structural changes were analysed through SEM using a ZEISS Crossbeam 350 (Germany). The crystallinity index (CrI) of the cotton was calculated via Eq. (12) on the basis of the XRD results (Rigaku SmartLab, Japan).

$$CrI = \frac{\left(I_{002} - I_{amorphous}\right)}{I_{002}} \times 100 \quad (12)$$

Where CrI is the crystallinity index (%); $I_{002}$ is the maximal intensity at the 002-lattice plane, and $I_{\text{amorphous}}$ describes the intensity of the background peak at 2θ = 18° (Jin et al. 2022).

The biogas composition ($CH_4$, $CO_2$, and $H_2S$) from anaerobic digestion was determined at the end of the process via an OPTIMA7 portable biogas analyser (MRU Instrument, USA).

### Statistical analysis

Statistical analysis was conducted using two-way ANOVA to assess the effects of temperature and reaction time on thermal hydrolysis pretreatment outcomes, using the statsmodels library in Python 3.12.x through Google Colab. The main effects were considered significant if $p \leq 0.05$. Additionally, Tukey's honest significant difference (HSD) post hoc test was conducted via Python 3.12.x in Google Colab, and the analysis was performed via the pairwise_Tukeyhsd function from the statsmodels library package. For example, it was used to compare COD solubilisation, biogas yield and methane composition across different HTP operating conditions (e.g., temperature and time) and to identify which conditions differed significantly.

## Results and discussion

### Thermal hydrolysis pretreatment of cotton textile waste

#### Physical changes in cotton textile waste

Total solids (TS) and volatile solids (VS) are critical parameters in HTP for quantifying organic matter content and assessing biodegradability potential, both of which directly influence the efficiency of subsequent AD (Lee et al. 2023). Figure 3 presents the TS and VS reductions in THP in cotton substrates, calculated using Eqs. (4) and (5), respectively.

The results demonstrate that THP significantly reduces both the TS and VS contents in cotton substrates, as shown in Fig. 3a and b, respectively. Both parameters decreased substantially, indicating extensive organic solubilisation. Generally, elevated temperature and extended reaction time conditions enhanced organic solubilisation with reductions reaching 98% for both TS and VS, efficiently converting solid matter to the liquid form. For example, Strong and Gapes (2012) reported that THP reduced volatile suspended solids (VSS) by 6% in Kraft pulp, indicating successful solubilisation of organic matter. The reduction in solids effectively transforms cotton fibres from insoluble polymeric components into low-molecular-weight, water-soluble organic compounds, thereby facilitating the enzymatic hydrolysis by microorganisms during AD. To further investigate this phenomenon, VS/TS is depicted in Fig. 3c to indicate the fraction of biodegradable solids in the total solids. Overall, the untreated and pretreated cotton samples presented high VS/TS ratios (> 70%), with a steady increase observed up to 140 °C and a further increase above 160 °C. There was a reduction in VS/TS from untreated (98.68%) to THP at 120 °C for 30 min (74.92%), which decreased the measured VS/TS because a portion of particulate organics was solubilised or volatilised during treatment, influenced by the TS reduction. Even though THP lowered the VS/TS ratio, slurry conditions improved mass transfer, accelerated

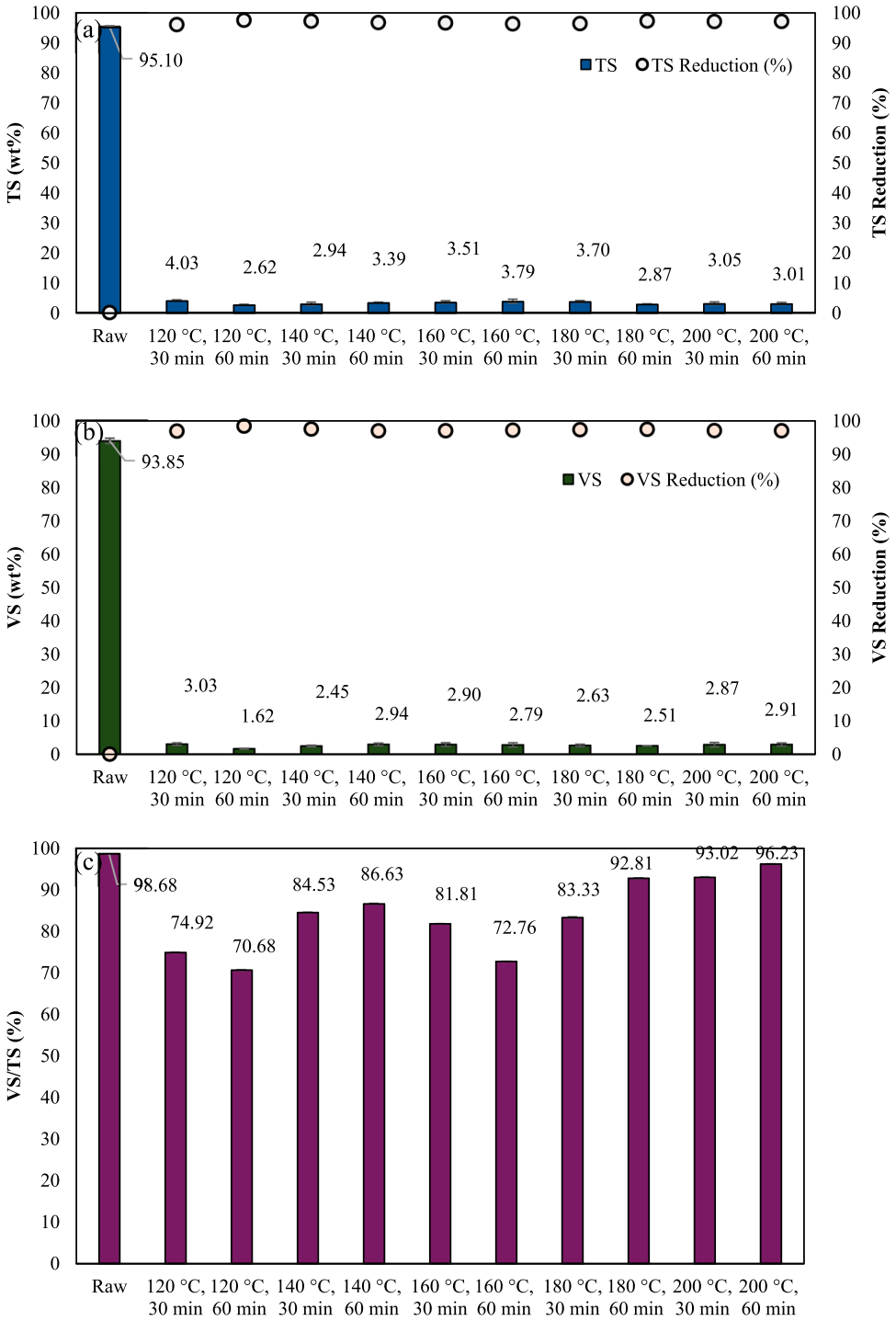



**Fig. 3** Changes in **a** total solids, **b** volatile solids and **c** VS/TS of THP cotton

hydrolysis, and reduced potential inhibitors (da Silva et al. 2020; Martin et al. 2022). Notably, the maximum condition (200 °C, 30 min) resulted in the highest VS/TS (96.23%), likely due to the efficient solubilisation of cotton organics. However, VS/TS determines the organic availability in the substrate, but it does not indicate direct biogas conversion (Kuo & Dow 2017; Wanwu Li et al. 2020; Rakić et al. 2023). A previous study by J Xu et al. (2021) reported that the THP of pig carcasses decreased TS from 25% to 16.6% as temperature increased from 140 to 180 °C, increasing the amount of biogas from untreated to approximately 29%. Nevertheless, the VS/TS ratio remained 98% of that of the untreated sample. In addition, Thompson et al. (2020) reported that the maximum THP conditions for brown seaweed resulted in the highest VS/TS, but inhibited the methane yield due to recalcitrant formation. This suggests that high VS/TS does

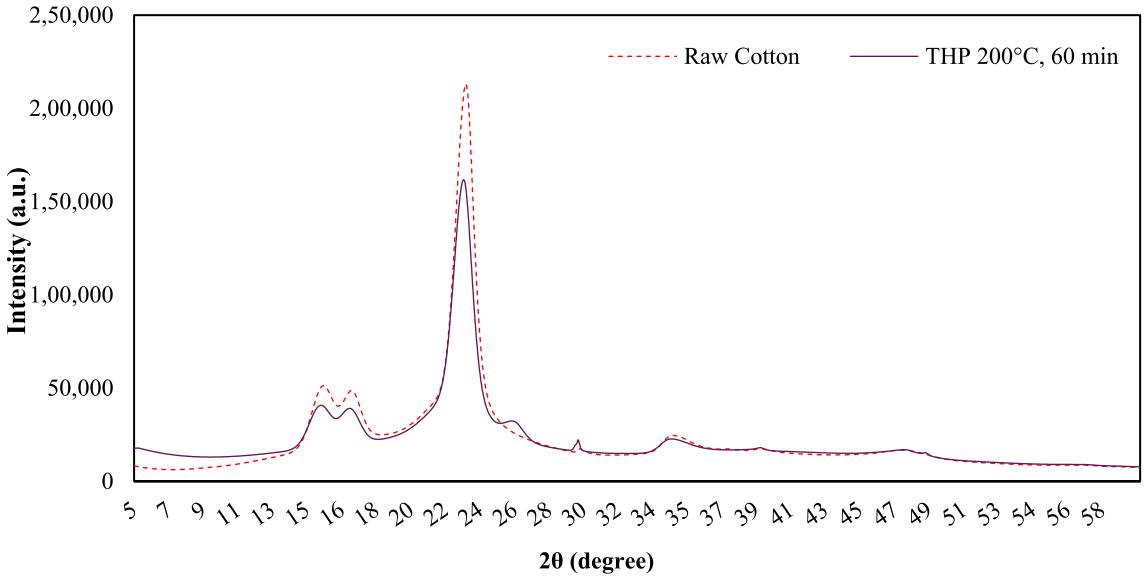



**Fig. 4** X-ray diffraction analysis of untreated and THP-treated cotton samples at 200 °C for 60 min

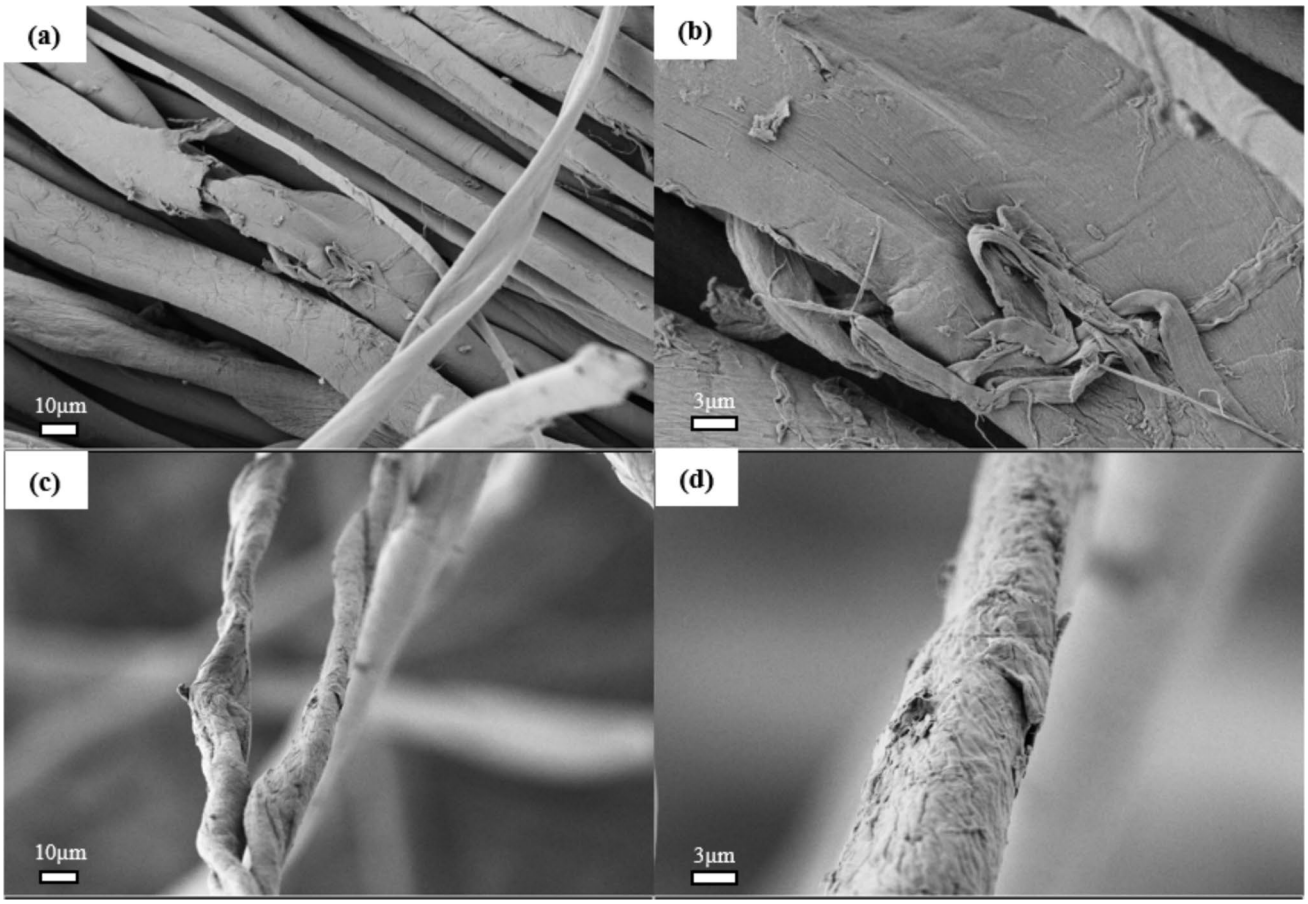



**Fig. 5** SEM images of cotton: **a** raw cotton 600×, **b** raw cotton 2500×, **c** cotton after thermal hydrolysis pretreatment at (200 °C and 60 min) 600× and **d** cotton after thermal hydrolysis at (200 °C and 60 min) 2500× magnification

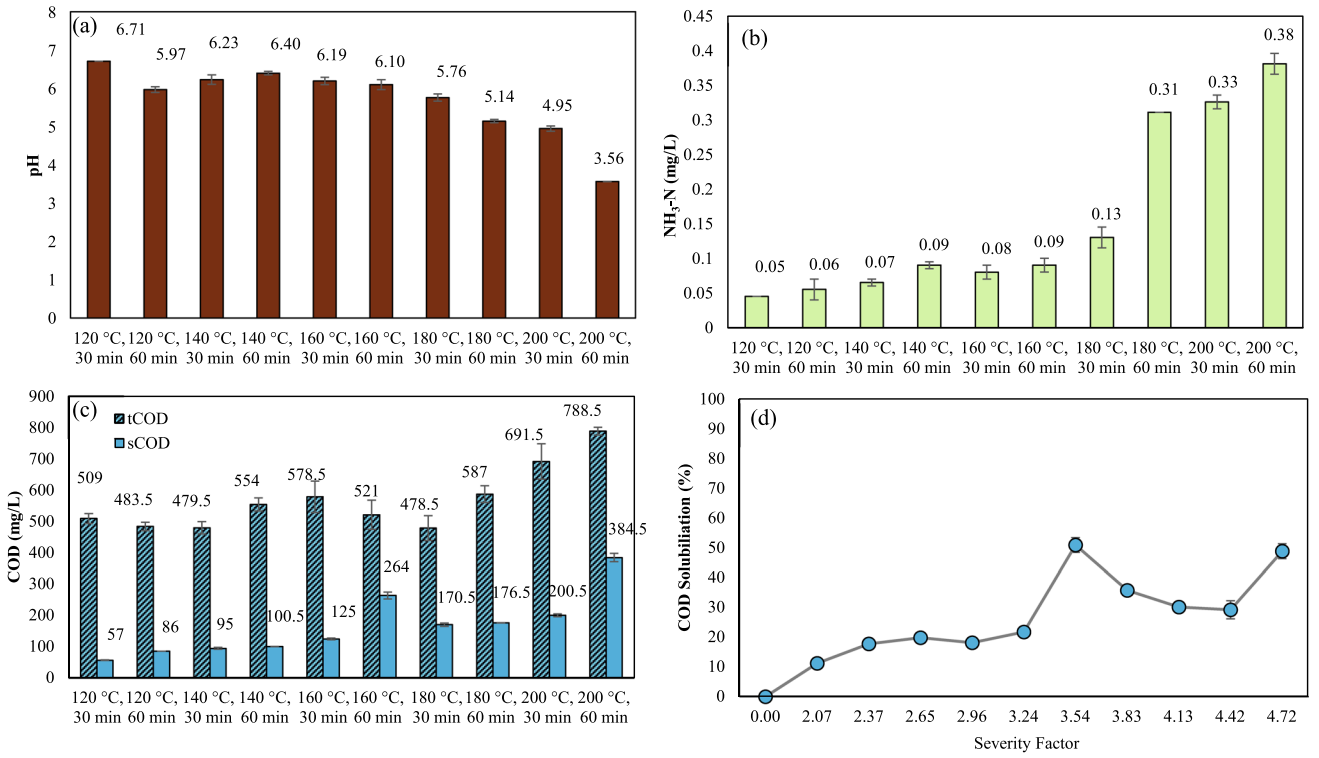



**Fig. 6** Influence of THP on **a** pH, **b** ammonia nitrogen, **c** COD and **d** COD solubilisation of cotton

not necessarily indicate an abundant availability of organic components that can be directly converted to methane.

The observed reduction in the cotton solid content through the THP represents a significant advantage for the subsequent hydrolysis phase of AD. By pre-solubilising complex organic polymers, the THP could increase biogas production efficiency and reduce the overall digestion time, increasing the efficiency of cotton waste valorisation via AD.

Cotton samples were characterised via X-ray diffraction (XRD) and scanning electron microscopy (SEM) to elucidate the physical and structural modifications induced by thermal hydrolysis pretreatment. Figure 4 shows the XRD patterns of untreated and THP-treated cotton at 200 °C for 60 min. According to the XRD results, the crystallinity decreases from 80.5% in untreated cotton to 68.7% in THP-treated cotton heated at 200 °C for 60 min. During this process, pressure-induced hot water disrupts hydrogen bonding, leading to crystallisation and swelling (N Xu et al. 2019). Similarly, when thermal hydrolysis pretreatment (THP) was applied to empty fruit bunches, a lignocellulosic substrate, its crystallinity was reduced from 34% to 22%. This results in THP being highly effective at reducing cellulose crystallinity (Gan et al. 2016). The SEM image shown in Fig. 5 reveals that the damage to the cotton fibre at the highest HTP operating condition is significant at a magnification of 2500×, because the surface is rougher than that of the raw cotton, which is smooth. These findings indicate that THP significantly disrupts the crystalline structure of cotton, thereby increasing its amorphous content and enhancing microbial access for efficient biogas conversion.

## Chemical changes in cotton textile waste

Figure 6 shows the pH, ammonia nitrogen content, chemical oxygen demand (COD) and solubilisation degree of the THP cotton. Elevated free ammonia and low pH can inhibit AD (Y Jiang et al. 2019; Yenigün & Demirel 2013). As shown in Fig. 6a, the pH drops decrease with increasing temperature, and the reaction time increases occur because of increased solubilisation of insoluble organic compounds (e.g., cellulose) in cotton, which are converted to soluble organic compounds, such as glucose, an organic intermediate, due to acidification during HTP. For proteins, the degradation releases inorganic ammonia nitrogen into the liquid phase as either ammonium ions ($NH_4^+$) or free ammonia ($NH_3$) (Chen et al. 2008). Cotton contains low protein levels, releasing only small amounts of amino acids. As shown in Fig. 6b, extending the reaction time and increasing the temperature to ≥ 180 °C resulted in increased ammonia nitrogen production. The maximum operating conditions at 200 °C for 60 min resulted in the highest ammonia nitrogen concentration of 0.38 mg/L, indicating maximum solubilisation of nitrogen compounds in the liquid phase (Zhang et al. 2020). However, all the THP ammonia nitrogen is below

the threshold limit (3000 mg/L), which prevents toxicity to microorganisms and the production of biogas during AD (Nleya et al. 2025a; M Rasapoor et al. 2020). Thermal hydrolysis pretreatment is beneficial for further hydrolysis of polysaccharides to the monosaccharides of cotton textiles to increase the bioconversion efficiency of microbes during AD.

From an organic hydrolysis perspective, the chemical oxygen demand (COD) is a crucial parameter for HTP to measure the presence of total and soluble organic matter in the substrate, which is necessary for microbial degradation of AD. Figure 6c depicts the COD of pretreated cotton substrates. Overall, the COD increased with increasing reaction temperature and time. The sCOD was analysed via the liquid phase of the pretreated slurry, following the same trend as tCOD. At 160 °C for 60 min, COD solubilisation increased sharply due to cellulose breakdown of 5-hydroxymethylfurfural (HMF), an inhibitory compound for AD, and HMF formed through dehydration of fructose after glucose isomerisation (Ma et al. 2021). Notably, the maximum HMF occurs at temperatures between 155 and 165 °C (Guo et al. 2021; Testa et al. 2020). Therefore, the combination of HMF formation and minor protein degradation during THP triggers Maillard reactions, leading to melanoidin formation (Ngo et al. 2021). Therefore, the presence of HMF can affect methane production during AD, altering the community structure of microorganisms and causing process inefficiency (Tan et al. 2021a, b). Additionally, THP at 160 °C for 60 min caused a sudden increase in sCOD, contributing to the highest degree of COD solubilisation of 51% at a severity factor (SF) of 3.54 (Fig. 6d). However, there was a slight drop in COD solubilisation after 180 °C, because of the breakdown of the HMF compound, which was converted to simpler sugars such as levulinic and formic acids (Gwak et al. 2022; Rosenfeld et al. 2020). At 200 °C for 60 min, the thermal severity is sufficient to release a large pool of soluble sugars from recalcitrant biomass, thereby increasing COD solubilisation without generating inhibitory compounds. Further conversion of furans to small acids occurred, and inhibition may persist depending on the concentration. Recent studies on thermal hydrolysis suggest solutions to increase substrate solubilisation. For example, Ngo et al. (2023a) reported that the highest COD solubilisation of sewage sludge was achieved under the highest operating conditions (190 °C for 30 min), with an SF of 3.95, ranging from 5% to 45%; hence, the highest biogas production could not be reached because of Maillard reaction formation. In addition, Thompson et al. (2020) confirmed that the highest operating condition, 180 °C for 30 min, with an SF of 3.83, resulted in the highest COD solubilisation of 96% but a low biogas yield. These findings suggest that THP helps improve the solubilisation of substrates, which cannot contribute to biogas enhancement because of the formation of recalcitrant compounds that inhibit microbial activity. However, further quantification of inhibitors such as furfural and HMF is needed at high THP temperatures (≥ 160 °C) to determine the potential inhibitory effects on anaerobic microorganisms and toxicity threshold concentrations. Furfural and HMF are dehydration products of pentoses and hexoses, respectively, and are known to impair methanogens by inhibiting essential metabolic enzymes, disrupting intracellular redox balance, and prolonging the lag phase of methane production (Ask et al. 2013).

Based on the statistical analysis, a two-way ANOVA revealed a statistically significant interaction effect between THP temperature and time on COD solubilisation ($p = 2.14 \times 10^{-9}$), indicating that changes in solubilisation can be attributed to THP (Table 4). To further explore this relationship, the Tukey HSD test confirmed that most pairwise comparisons between THP temperature and time were statistically significant, particularly between the untreated and pretreated cotton samples (Table S1). These results indicate that THP is highly effective at solubilising COD from polymers to monomers.

Despite the occurrence of COD, VFAs, and intermediates of biogas production during AD, they were absent in this study because of the robust crystalline cellulose framework of cotton, which demonstrates exceptional resistance to the thermal degradation processes necessary for the production of fermentable intermediate compounds during THP. In support of this finding, Strong and Gapes (2012) reported comparable results, demonstrating that Kraft pulp exhibited no detectable VFA formation when subjected to inert HTP conditions, compared with oxidative conditions, where VFAs were only discernible at the highest operating conditions. VFA production is often associated with sCOD levels above 1000 mg/L (Carranza Muñoz et al. 2024; Gracia et al. 2020). Hence, the sCOD values in Fig. 6c after THP are below this level, resulting in the absence of VFA production and most likely, the production of non-fermentable compounds. Therefore, optimising operating conditions is essential to produce VFAs during THP. This necessitates careful optimisation of operational parameters such as reaction time, temperature, and total solids content to maximise biogas production while minimising energy consumption.

Figure 7 illustrates the application of Fourier-transform infrared (FT-IR) spectroscopy to evaluate chemical and structural modifications in cotton fabric under untreated conditions and maximum THP treatment. These analyses facilitate the identification of functional group degradation and physical alterations to fibre morphology, providing insights into the enhanced biodegradability of natural components following pretreatment.

The FT-IR spectra demonstrated a notable reduction in functional group intensity following the THP process, indicating effective solubilisation of the organic substrates.

**Table 4** Two-way ANOVA of the effects of temperature and time on COD solubilisation

| Source of variation | *F* | *p-value* | *F crit* |
|---|---|---|---|
| Temperature (T) | $5.72\times10^{-11}$ | 1.00 | 3.20 |
| Time (*t*) | $2.47\times10^{-13}$ | 1.00 | 3.98 |
| T×*t* | $4.67\times10^{3}$ | $2.14\times10^{-9}$ | 2.85 |
| Residual | 49.84 | – | – |

Specifically, untreated cotton exhibited intense broad peaks corresponding to O−H stretching at 3302 $cm^{-1}$ and C−O stretching at 1313 $cm^{-1}$, representing hydroxyl and ether bonds, respectively. The O−H peak at 3302 $cm^{-1}$, which is commonly associated with the crystalline structure and attributed to the intra- and intermolecular hydrogen bonding of cellulose, showed a significant intensity reduction following THP treatment, indicating successful solubilisation of the cellulosic compounds. The sharp peak observed at 1029 $cm^{-1}$ corresponds to the −C−O−C− asymmetrical bridge vibration, representing the 1,4 β−glycosidic bonds (Binczarski et al. 2022; Chung et al. 2004), which showed that the intensity decreased after THP. Following THP treatment, the C−O bonds within the medium peaks, predominantly observed in the cyclic cellulose structure, were efficiently disrupted due to the combined effects of elevated temperature and extended reaction time. Additionally, the alkane group $CH_2$, which is commonly attached to glucose units, underwent significant degradation during the THP process.

These results confirm that THP effectively promotes the structural breakdown of cotton cellulose, facilitating the solubilisation of organic substrates that microorganisms can readily consume. This enables efficient degradation of the glucose chain, thereby enhancing biogas production during AD.

## Effects of thermal hydrolysis on biogas production during anaerobic digestion

### Biogas yield

Figure 8 shows the cumulative biogas yield and total methane production of untreated and THP-pretreated cotton fibres. Overall, the use of THP-pretreated cotton increased the biogas yield under various operating conditions.

The cumulative biogas production (Fig. 8a) yielded the highest cumulative biogas yield of 93 ± 3.11 mL/g VS under conditions of 140 °C for 60 min, which is considered optimal. In addition, optimal conditions yielded the highest methane yield (17.91 ± 0.37 mL/g VS), as depicted in Fig. 8b. This is because amorphous cellulose undergoes effective hydrolysis, releasing soluble sugars (e.g., glucose) that are converted by anaerobic microbes into intermediates (e.g., acetate, $H_2$, and $CO_2$), which methanogens consume to produce biogas and methane efficiently (Zhou et al. 2021). The disruption of cellulose cell walls under these THP conditions is sufficient to increase biodegradability while avoiding the excessive formation of toxic compounds. Although this study demonstrated a substantial increase in methane yield compared with other substrates detailed in Table 1, the optimal increase (123%) remained lower than that observed for cotton stalks (172%) (Adl et al. 2012), which varied depending on textile grade and agricultural conditions. Compared to THP of pulp and paper sludge which achieved a higher methane increase, cotton waste in this study yielded a lower increase likely due to the cotton's higher TS content inhibiting methanogen access (Goycoechea et al. 2023). This discrepancy can be attributed to differences in material properties, operational parameters, and inoculum type, all of which strongly influence AD performance.

Although the absolute methane yields from cotton remained low due to its highly crystalline structure and extreme C/N ratio, the improvement achieved under optimal THP (140 °C, 60 min) is significant for two reasons. First, methane yield increased by 123% relative to untreated cotton, demonstrating that THP overcomes a major fraction of the hydrolysis limitation. Second, the lag phase decreased to 2.3 days and digestate indicators (pH, FAN, VFAs) remained within non-inhibitory ranges, indicating that the improvement is not achieved at the expense of process stability. Together, these findings establish cotton-specific THP conditions that materially enhance the feasibility of AD as a textile waste valorisation route.

### Inhibition mechanisms

Conversely, under underperforming conditions, the THP of cotton at 160 °C for 60 min produced the lowest biogas yield of 49.94 ± 2.2 mL/g VS and $CH_4$ yield of 7.07 ± 2.2 mL/g VS (Fig. 8b) due to the formation of inhibitory compounds. In contrast, moderate pretreatment at 140 °C for 60 min significantly increased the biodegradability and methane yield, demonstrating the importance of balancing solubilisation with microbial inhibition. At elevated temperatures approaching 160 °C, the lowest methane yield is observed in the 140–165 °C range, which promotes Maillard reactions and leads to the production of recalcitrant compounds such as melanoidins, which negatively affect microbial activity (Mirsoleimani Azizi et al. 2024). In addition, HMF, a degradation product from cellulose, disrupts methanogenic activity by inhibiting key dehydrogenase enzymes and electron transfer systems in *Methanobacteriales*, whereas melanoidins form recalcitrant polymers that resist hydrolysis (Ghasimi et al. 2016; Tan et al. 2021a, b; Wu et al. 2024). Hence, the HMF compound can shift the microbial community toward acidogenesis, suppressing acetoclastic and

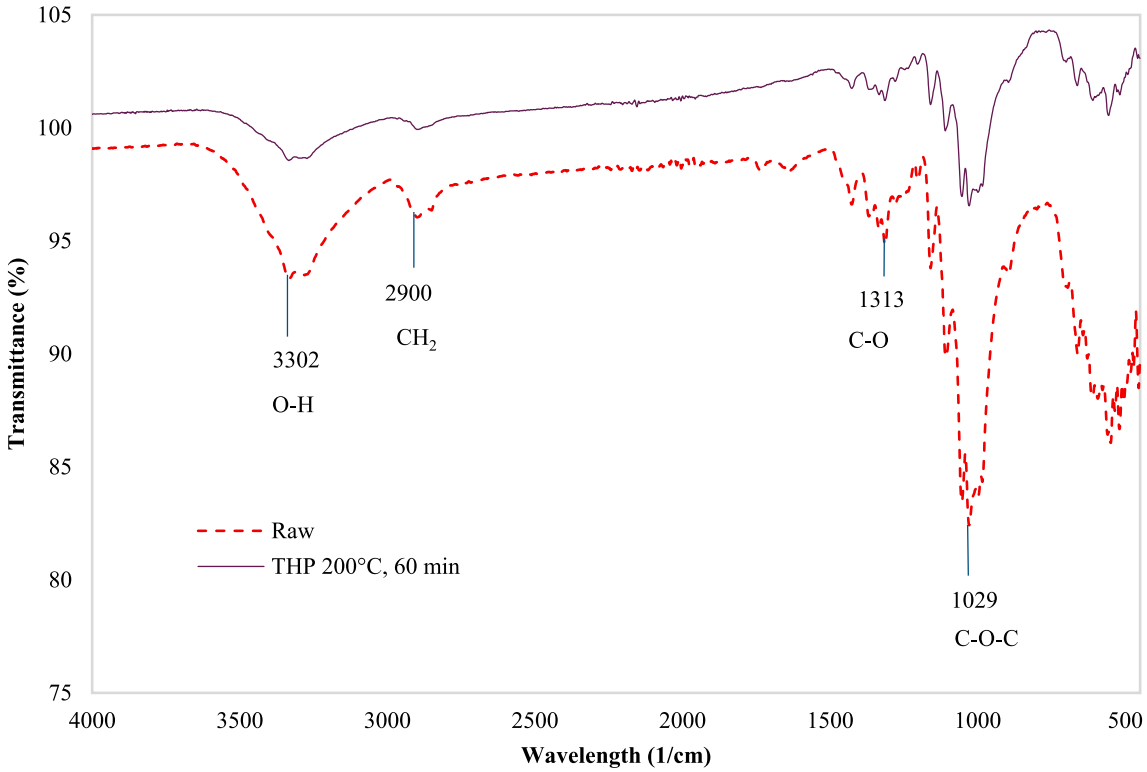



**Fig. 7** FT-IR spectra of raw cotton and the maximum THP conditions (200 °C and 60 min)

hydrogenotrophic methanogenesis and reducing methane yields despite increased COD solubilisation. These results suggest that the optimal THP conditions must balance the benefits of solubilisation with the potential risk of microbial inhibition.

Nevertheless, when the temperature increased to 180 °C, biogas and methane production increased due to the degradation of inhibitory compounds during rehydration under acidic conditions, which led to the formation of simple carboxylic acids (Árvai et al. 2024). Although the formation of carboxylic acids is less inhibitory than that of HMF is, their presence can still negatively affect methanogenic activity and overall biogas yields (Kumar et al. 2014). Therefore, the maximum THP (200 °C and 60 min) yielded less methane (16.35 ± 8.67 mL/g VS) than did the optimal conditions (140 °C and 60 min). Although additional inhibitors are generated under these severe thermal conditions, fermentable organic compounds outweigh their adverse effects, enabling microorganisms to operate efficiently during acidogenesis and acetogenesis (Kumar et al. 2014; R Wang et al. 2021). Moreover, temperatures above 180 °C promote extensive breakdown of carbohydrate complexes, facilitating the conversion of crystalline cellulose into amorphous forms. This enhanced COD solubilisation (Fig. 5d) improved microorganism biodegradation. Despite increased biogas production, THP remains an energy-intensive technology with substantial energy requirements. For instance, Fattahi et al. (2025) report that integrating THP with anaerobic digestion of municipal sludge reduces digester volumes, enhances organic matter degradation efficiency, and increases biogas energy content by 32%, yet results in a negative energy balance due to high steam generation requirements. The energy input was 23,276 kWh/d, while recovered biogas energy content was 7495 kWh/d, resulting in an energy balance of − 15,781 kWh/d. Nevertheless, a comprehensive energy balance analysis is valuable for evaluating the net energy gain (biogas yield vs. pretreatment energy input) of textile waste in practical applications.

From a techno-economic perspective, THP requires substantial capital investment, ranging from US$2–4 million for pilot-scale projects, with reported costs typically between US$54–85 per ton of biomass or organic waste treated (Preethi and Banu 2023). Despite substantial initial capital requirements, hydrothermal exhibits comparatively low operating and maintenance costs per ton, particularly when benchmarked against chemically intensive or energy-demanding pretreatment alternatives, owing to reliance on water as the processing medium (Mihajlovic et al. 2025; Tharmarajah et al. 2024a). A techno-economic analysis conducted by Terrell et al. (2021) reported that THP-AD for pelagic Sargassum resulted in positive cash flow, leading to a positive operating profit margin, net present value, and

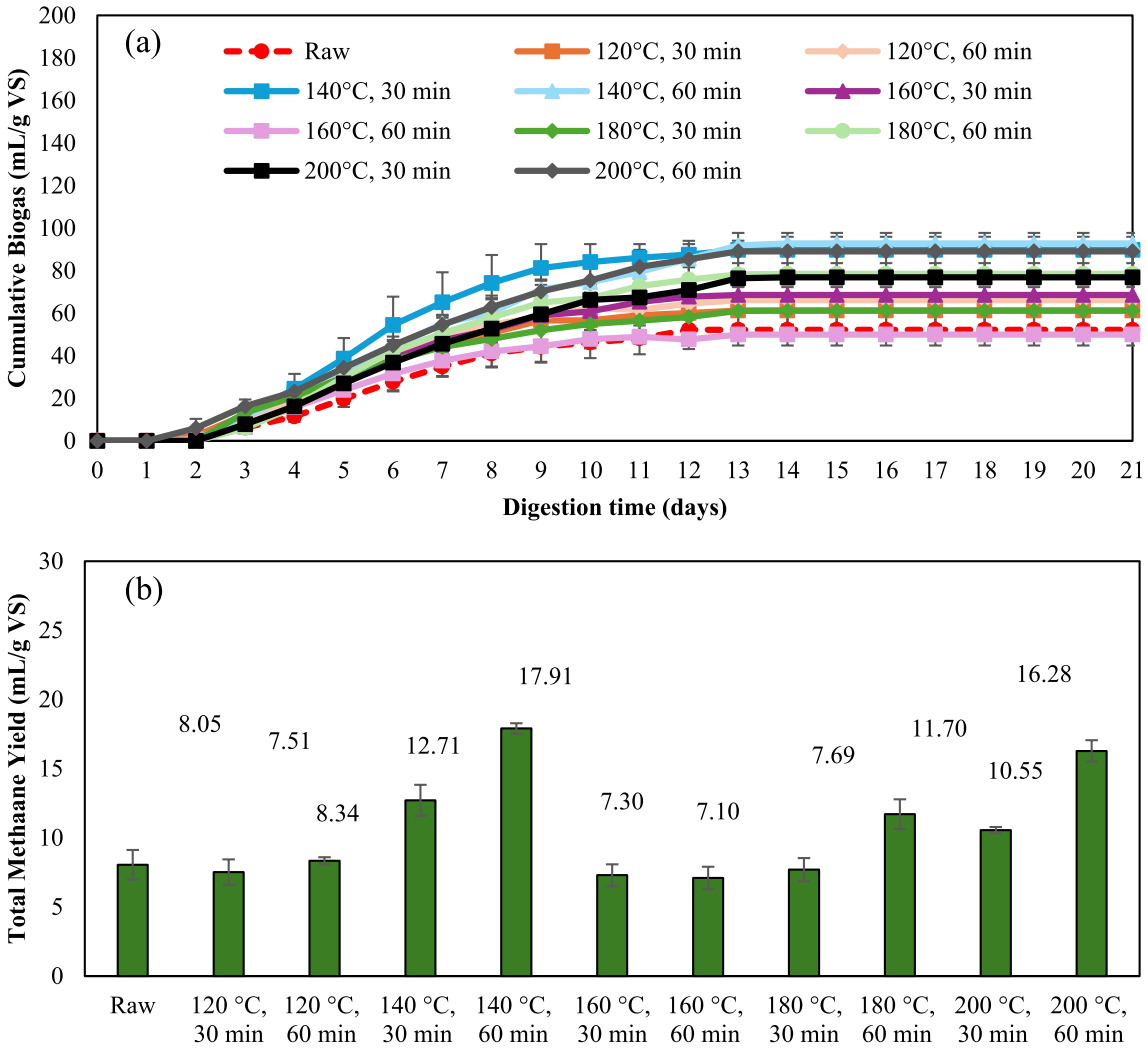



**Fig. 8** Cumulative **a** daily and **b** total methane yields over a 21-day digestion period of cotton

rate of interest. Electricity and soil fertiliser are sold 50% each to local and exported markets. Notably, the economic feasibility of applying THP, integrated with AD, to cotton textile waste should be evaluated by comparing the capital costs, additional energy requirements, and operational costs of pretreatment with electricity and fertiliser production, and by considering the reduction in cotton textile waste disposal costs.

Statistical analysis via two-way ANOVA revealed a significant interaction effect between THP temperature and time on biogas yield ($p = 2.1 \times 10^{-4}$), indicating that changes in solubilisation are attributed to THP (Table 5). To further explore this relationship, the Tukey HSD test confirmed that most pairwise comparisons between THP temperature and time were statistically significant for the operating conditions of 140 °C for 30 min, 140 °C for 60 min, 180 °C for 60 min and 200 °C for 60 min (Table S2), which indicates a significant increase in biogas content under these conditions.

On the basis of the microbial community and metabolic effects of cotton hydrolysis, simple sugars such as glucose from cotton cellulose stimulate the activity of hydrolytic and fermentative bacteria, Clostridium sensu stricto, which rapidly converts these substrates into volatile fatty acids (VFAs), hydrogen and carbon dioxide via acidogenesis (Ali et al. 2021; Jin et al. 2022).VFAs, particularly acetic acid, further convert acetate through acetogenesis by syntrophic bacteria, support the growth of acetoclastic methanogens, primarily Methanothrix, and facilitate efficient methanogenesis (J Jiang et al. 2020; Jin et al. 2022). This thereby enhances overall methane production from THP cotton at 140 °C due to the presence of glucose, a highly valuable compound that yields the highest methane production. At 180 °C and above, the presence of formic acid and levulinic acid, which are less inhibitory to methanogens, causes an increase in methane. In contrast, the presence of toxic intermediates, such as HMF, disrupts the syntrophic balance

between bacteria and archaea, causing a decrease in methane production at 160 °C. These compounds inhibit the enzymatic activity of fermentative and methanogenic organisms, leading to acidification, suppressed methane formation, and reduced digestion efficiency. For future studies, a comprehensive microbial analysis on microbial community dynamics via 16S rRNA sequencing would be beneficial for understanding acetogenic–methanogenic interactions, substrate utilisation pathways, and process stability during THP cotton textile waste digestion.

### Biogas composition

The cotton substrates were analysed for their biogas composition (Fig. 9), including methane ($CH_4$), carbon dioxide ($CO_2$), and hydrogen sulphide ($H_2S$) contents. A two-way ANOVA was conducted to evaluate the effects of temperature and time during THP on methane content, revealing a statistically significant increase ($p = 5.27 \times 10\text{^} - 4$), as shown in Table 6. However, Tukey's HSD pairwise comparisons revealed no significant differences among the treatment conditions, particularly between the untreated and THP-treated samples (Table S3). This finding indicates that untreated cotton has an inherent capacity to produce substantial methane fractions, despite the observed 20% increase at 140 °C for 30 min, identified as the optimal pretreatment condition. The reduction in methane composition beyond this optimal point can be attributed to the formation of inhibitory compounds, which offset the beneficial effects of THP.

To assess biogas quality from the perspective of methanogenic activity efficiency, the methane-to-carbon dioxide ($CH_4$:$CO_2$) ratio was used as an indicator. A high $CH_4$:$CO_2$ ratio in the range of 1.2–2.3 signifies efficient conversion of volatile fatty acids (VFAs) to acetate and subsequent methane production. The results demonstrated that all the THP conditions achieved ratios exceeding 3, with the optimal conditions of 140 °C for 60 min yielding the highest $CH_4$:$CO_2$ ratio of 5.28, indicating the most efficient acetate-to-methane conversion. Additionally, THP-treated cotton showed a notable advantage for AD, producing undetectable $H_2S$ and resulting in higher-quality biogas with reduced corrosion potential and diminished cleaning requirements for commercial-scale applications (Thompson, Ramin, et al. 2021). Therefore, integrating THP with existing AD infrastructure offers a viable pathway for industrial-scale treatment of cotton textile waste, primarily because of the elevated methane purity achievable for energy recovery applications.

**Table 5** Two-way ANOVA of the effects of temperature and time on biogas yield

| Source of variation | *F* | *p-value* | *F crit* |
|---|---|---|---|
| Temperature (T) | $1.31 \times 10^{-11}$ | 1.00 | 3.20 |
| Time (*t*) | $4.31 \times 10^{-13}$ | 1.00 | 3.98 |
| T × *t* | $4.76 \times 10^{3}$ | $2.1 \times 10^{-4}$ | 2.85 |
| Residual | 469.65 | – | – |

### Gompertz model

The modified Gompertz model was applied to evaluate methane production kinetics for raw and optimal THPs of cotton (140 °C for 60 min). The simulated maximum biogas production (*P*) showed a strong correlation with the actual biogas yield (*B*), with an $R^2$ of 0.99 (Table 7), indicating a robust model fit for kinetic modelling (Chicco et al. 2021).

In addition, compared with raw cotton, THP treatment significantly increased all the kinetic parameters. The measured *B* increased from 52.74 ± 7.38 to 92.92 ± 3.11 mL/g VS, whereas the maximum $R_{max}$ improved from 8.44 to 11.71 mL/g VS/d, which reflects that THP enhances the rapid degradation of cotton. In addition, the lag phase ($\lambda$) of THP cotton decreased to 2.3 days, indicating faster microbial adaptation to pretreated substrates, which improved the methane yield by approximately 2.5 times. A previous study, Lee et al. (2023) confirmed that hydrothermal pretreatment increases the temperature from 160 to 200 °C, and reduces the $\lambda$, which also improves the methane yield from 0.047 to 0.112 ($Nm^3$ $CH_4$/kg VS). Most notably, the biodegradability of THP increased twofold compared with that of the untreated sample, confirming that THP is effective for microbial bioconversion to methane. These results demonstrate that moderate THP conditions significantly increase cotton biodegradability while maintaining stable methane production kinetics, thereby improving process efficiency.

In an efficiently operated anaerobic digestion system, methane conversion efficiency typically ranges from 50 to 70% of the feedstock's TBMP (Meegoda et al. 2018). Although the $B_D$ value of the THP cotton sample was greater than that of the untreated sample, the value (4.51%) did not remain within the efficient range of methane conversion. Further studies can be conducted on co-digestion with nitrogen-rich textile substrates (e.g., wool) to support sustainable textile waste management. Wool is a protein-rich (nitrogenous) textile waste (Kabir et al. 2013) and co-digesting it with cotton could lower the overall C/N ratio toward an optimal range. Alternatively, an oxidative pretreatment, such as an advanced thermal hydrolysis process (ATHP), to increase the biodegradability of the cotton substrate.

## Effect of thermal hydrolysis on solid reduction during anaerobic digestion

Figure 10 presents the removal efficiencies of total solids (TS) and volatile solids (VS) for the untreated and THP

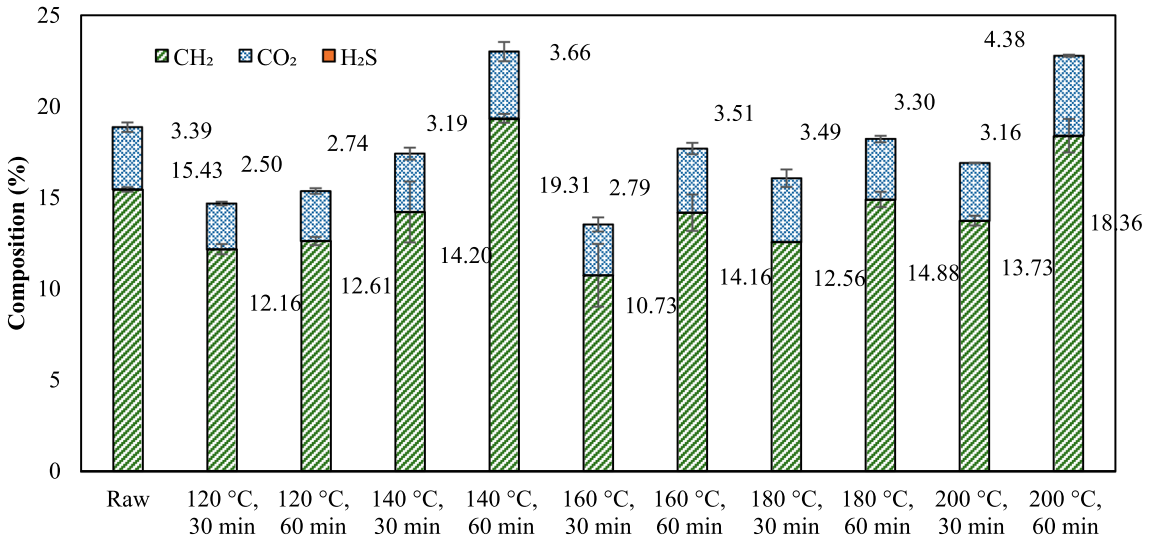



**Fig. 9** Cotton textile AD biogas composition under different thermal hydrolysis conditions

**Table 6** Two-way ANOVA of the effects of temperature and time on methane composition

| Source of variation | *F* | *p-value* | *F crit* |
|---|---|---|---|
| Temperature (T) | $5.34\times10^{-13}$ | 1.00 | 3.20 |
| Time (*t*) | $1.83\times10^{-13}$ | 1.00 | 3.98 |
| T×*t* | 133.34 | $5.27\times10^{-4}$ | 2.85 |
| Residual | 16.05 | – | – |

cotton substrates with inoculum during anaerobic digestion (AD). TS and VS were measured on whole slurries (with no phase separation) on day 0 (after mixing with inoculum) and day 21. The TS and VS reductions in the inoculum were 7.67 ± 1.13% and 39.85 ± 5.52%, respectively. Both parameters decreased substantially throughout the AD process, demonstrating similar degradation trends across all experimental conditions.

As shown in Fig. 10a, the TS removal efficiency increased from 62% for untreated cotton to 65% at 140 °C for 60 min, indicating enhanced hydrolytic activity under moderate thermal pretreatment. A marked decrease in TS removal was observed at 160 °C for 30 min (58%), indicating that the formation of recalcitrant compounds adversely affected biogas production efficiency. TS removal gradually improved at 180 °C, indicating that thermal degradation of inhibitory compounds, such as HMF, was generated during pretreatment. The maximum TS removal was achieved at 140 °C for 60 min, attributed to optimal solubilisation of organic substrates within the cotton textile substrate.

The VS removal efficiency (Fig. 10b) followed a similar trend to that of TS removal, which was consistent with the conversion of organic matter to biogas during methanogenic processes. The highest VS removal of 90% was achieved at 140 °C for 60 min, representing a substantial improvement compared with 16% for untreated cotton. Thus, the maximum VS removal was achieved at 140 °C for 60 min, and this pretreatment condition corresponded to the highest biogas yield because the release of soluble organic compounds was sufficient to maximise it.

Figure 11 presents the chemical oxygen demand (COD) removal efficiencies before and after anaerobic digestion for both untreated and THP cotton substrates with inoculum. The COD reduction of the inoculum alone was 83.85 ± 1.46%. The COD removal pattern exhibited similarities to those observed for TS and VS removal, with variations in the magnitude of removal efficiency. Notably, the COD removal trend was closely correlated with biogas performance and methane production (Fig. 8), indicating a direct relationship between organic matter conversion and methane generation.

The maximum COD removal of 26.1% was achieved at 140 °C for 60 min, representing a substantial improvement compared with 10% for untreated cotton. This significant enhancement demonstrates the effective conversion of solubilised organic substrates to biogas under optimal pretreatment

**Table 7** Kinetic analysis modified by the Gompertz model for raw and optimal THP conditions for cotton

| Operating condition | *B* (mL/g VS) | *P* (mL/g VS) | $R_{max}$ (mL/g VS/d) | *Λ* (day) | $R^2$ | $B_D$ of $CH_4$ (%) |
|---|---|---|---|---|---|---|
| Raw cotton | 52.74 ± 7.38 | 52.70 | 8.44 | 2.65 | 0.9988 | 2.03 |
| THP cotton (140 °C and 60 min) | 92.92 ± 3.11 | 94.74 | 11.71 | 2.3 | 0.9969 | 4.51 |

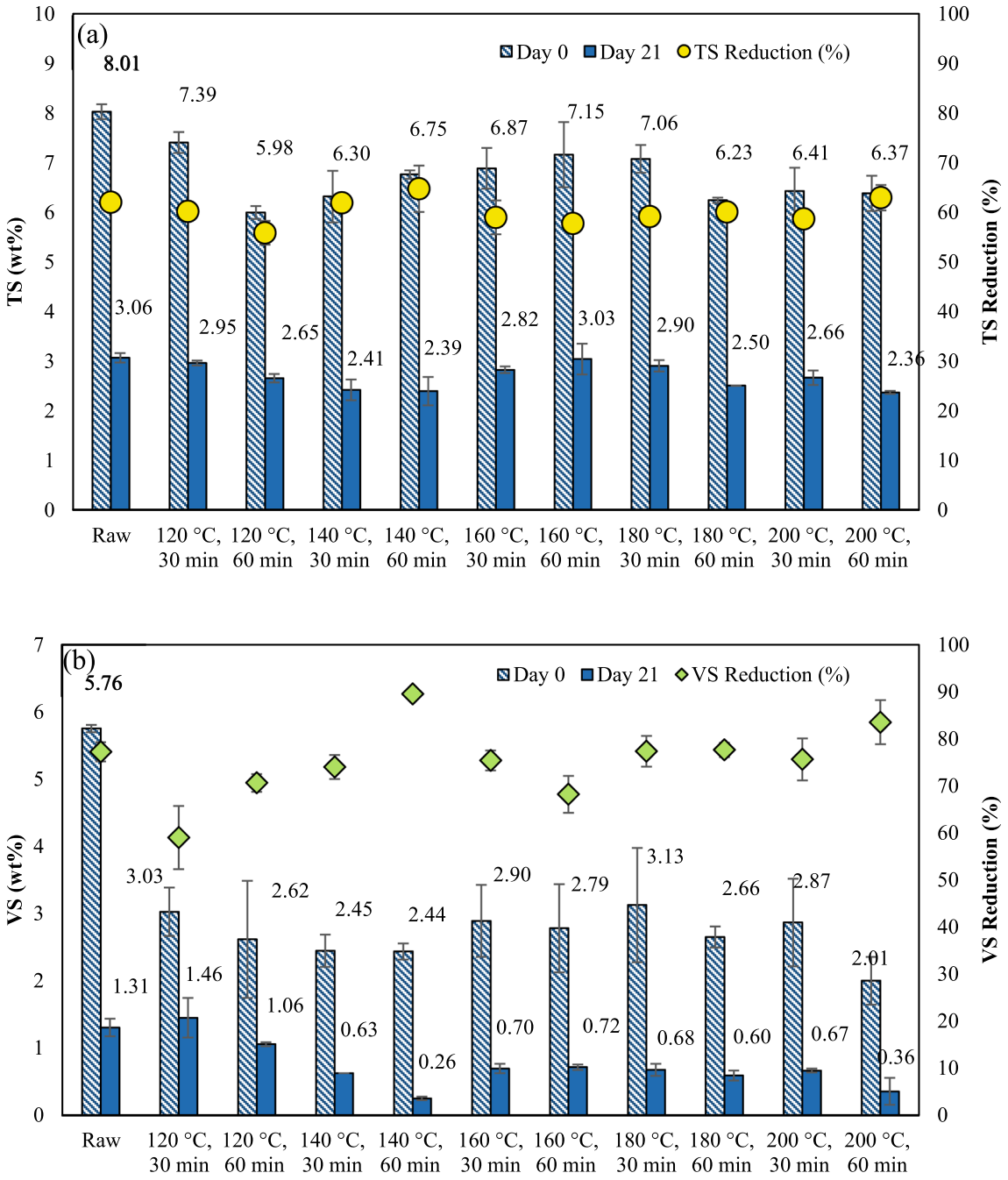



**Fig. 10** Effect of THP cotton with inoculum on **a** total solids and **b** volatile solids before (day 0) and after (day 21) anaerobic digestion

conditions. The strong correlation between COD removal and biogas yield confirms that the organic compounds quantified via COD analysis serve as the primary carbon source for methanogenic processes. A decrease in COD removal was observed at 160 °C, likely due to the formation of melanoidin compounds, which inhibited microbial activity and hindered effective COD degradation. Several studies have examined the correlation between COD removal and biogas production. For example, Thompson et al. (2021a, b) reported that hydrothermal pretreatment of Sargassum seaweed and food waste yielded the highest methane, resulting in the highest COD removal (97%). Another notable example is the study by Gong et al. (2019), which found that hydrothermal pretreatment of sewage sludge at different solid–liquid ratios substantially enhanced biogas production and chemical oxygen demand (COD) removal. Under optimal conditions, they reported a significant increase in the cumulative methane yield (425.57 mL/g VS) and recorded final methane contents of

64–66%, which were closely correlated with increased COD solubilisation during THP. The results demonstrate that THP under moderate conditions (140 °C, 60 min) provides an optimal balance between substrate solubilisation and preservation of biodegradable organic compounds, leading to increased COD removal and increased AD performance.

## Effects of thermal hydrolysis on inhibitors during anaerobic digestion

Inhibitor formation is common in anaerobic digestion systems. Primary inhibitors include pH, TAN, and VFAs. These inhibitors are detrimental to methanogenic species, reducing or preventing biogas production. This study evaluated changes in pH, ammonia nitrogen content, and VFA concentration to assess inhibition during anaerobic digestion of THP cotton textile waste.

Figure 12 shows the pH values of the untreated and THP cotton substrates with inoculum before and after anaerobic digestion. The initial (day 0) and final (day 21) TANs of the inoculum were 1026 and 1507.5 mg/L, respectively. A consistent pH increase was observed across all the experimental conditions (Fig. 12a). HTP effectively maintained the optimal digester pH within the range of 6.8–7.2, thereby supporting stable long-term methanogenesis (Thompson et al. 2020). pH stability represents a significant operational advantage, as it minimises the risk of acidification while ensuring consistent biogas production performance throughout the digestion period. As shown in Fig. 12b, the observed decrease in TAN directly correlates with the increase in pH, driven by interconnected biochemical processes during anaerobic digestion. The low ammonia nitrogen in cotton supports efficient microbial assimilation, reducing ammonia nitrogen. pH-TAN relationship is fundamentally attributed to the chemical equilibrium between ammonium ions and free ammonia, where higher pH conditions favour the formation of free ammonia ($NH_3$) following the completion of anaerobic digestion (Y Jiang et al. 2019). Consequently, the simultaneous increase in both parameters reflects the successful conversion of organic nitrogen compounds and the establishment of favourable conditions for sustained methanogenic activity. Elevated ammonia concentrations can potentially inhibit methanogenic bacteria (e.g., *Methanosaeta spp*.) when FAN exceeds the toxicity threshold of 80 mg/L (Thompson et al. 2021a, b). Free ammonia nitrogen (FAN) is the primary inhibitor that causes methanogenic toxicity in AD processes.

Table 8 presents the FAN concentrations in the digestate on days 0 and 21 of AD. Overall, the cotton THP substrate concentrations are below the threshold limit on day 0 and remain modest due to the poor nitrogen content, which prevents methanogen toxicity and ensures stable biogas production. On day 21, the FAN for the untreated and HTP-treated substrates increased. The increase in FAN was due to the influence of the inoculum, but it remained below 80 mg/L, which is beneficial for AD stability. The optimal conditions at 140 °C for 60 min yielded the lowest concentration (23.8 mg/L) on day 21, which was correlated with the maximum biogas production (Fig. 8). Conversely, 160 °C for 30 min produced the greatest amount of free ammonia (41.3 mg/L), corresponding to the lowest biogas yield and highest pH (7.64) of the digestate (day 21) due to FAN formation, which inhibits microbial activity. High

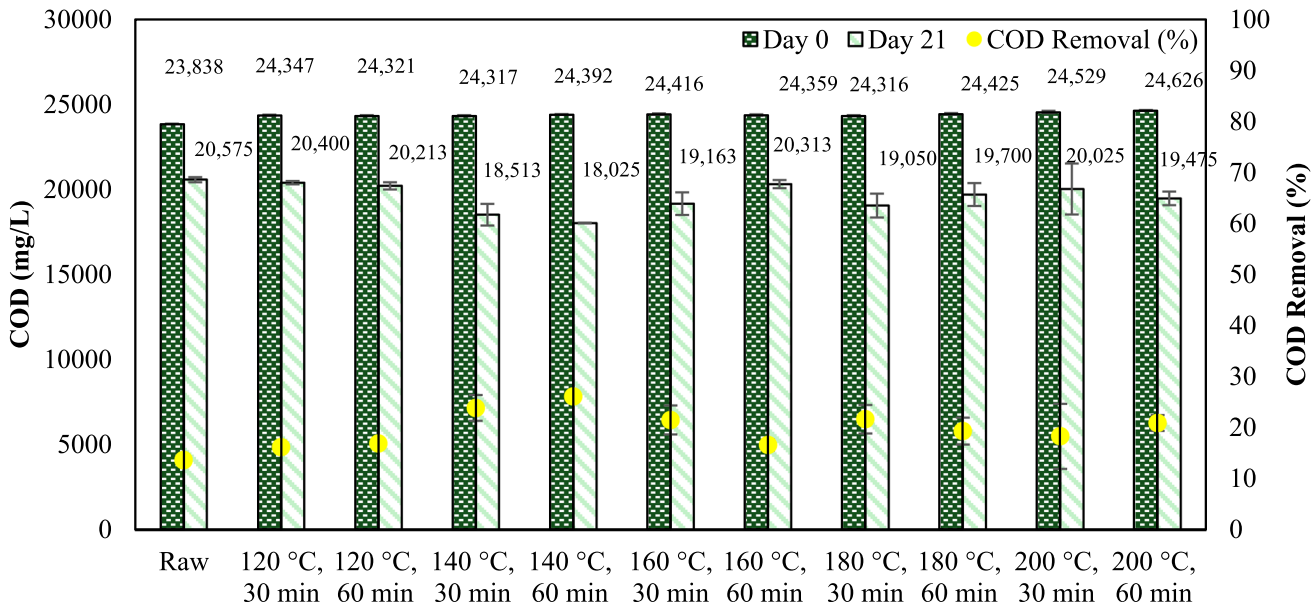



**Fig. 11** Effect of THP cotton with inoculum on total chemical oxygen demand reduction before (day 0) and after (day 21) anaerobic digestion

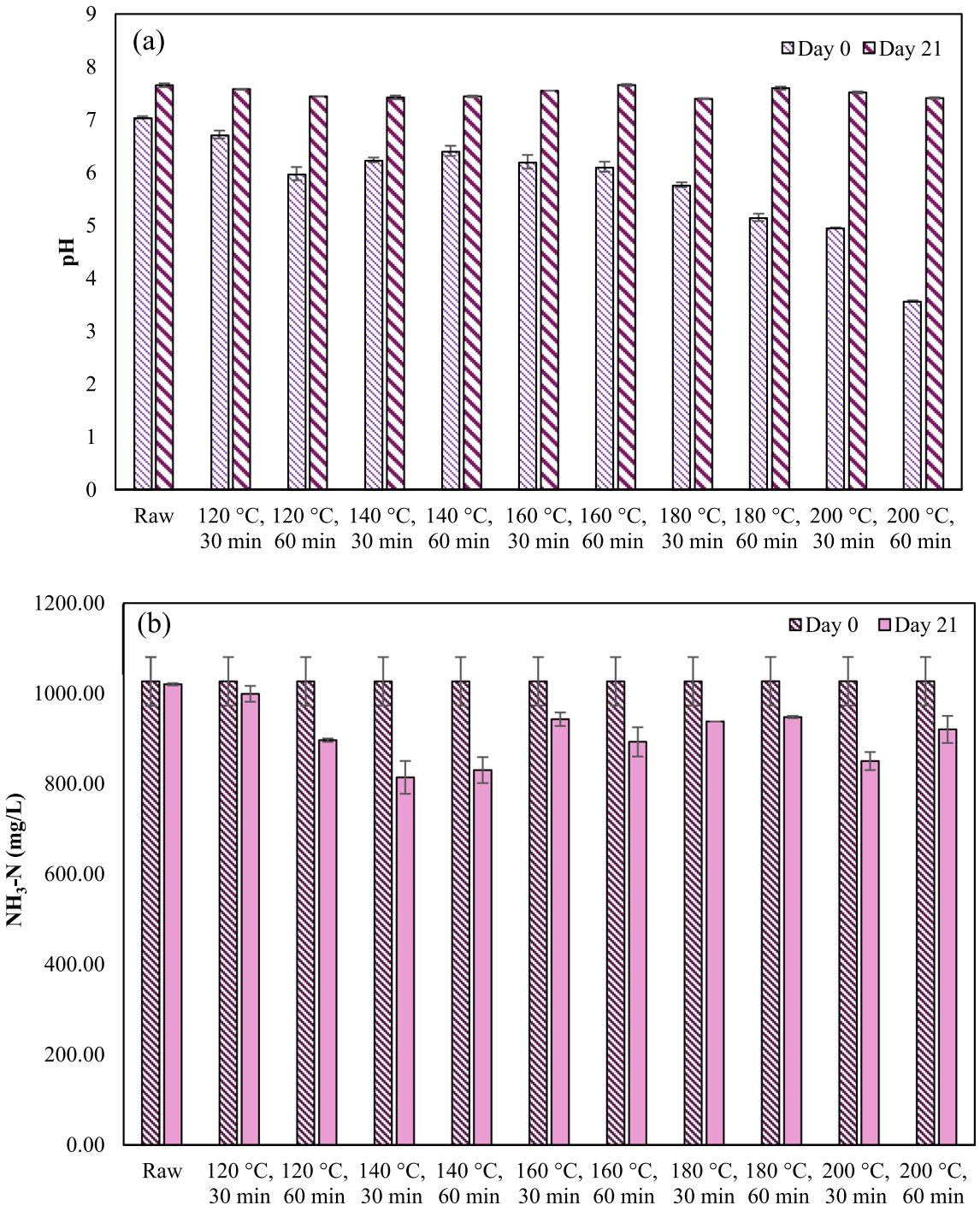



**Fig. 12** Effect of thermal hydrolysis of cotton with inoculum on **a** pH and **b** total ammonia nitrogen (TAN) before (day 0) and after (day 21) anaerobic digestion

concentrations of FAN affect AD through methanogen toxicity, resulting in system failure and VFA accumulation (Yenigün & Demirel 2013). However, compared with untreated substrates, THP cotton substrates produced lower FAN concentrations, providing significant advantages for maintaining a stable AD system, which keeps the FAN of digestate below the threshold limit.

Volatile fatty acids (VFAs) constitute an additional inhibitory factor in anaerobic digestion, with a critical threshold of 15,000 mg/L established for maintaining stable and efficient biogas production (Harirchi et al. 2022; Nleya et al. 2025a). When VFA levels exceed this threshold, acidification occurs, resulting in detrimental effects on methanogenic bacterial populations (Meegoda et al. 2018; Nleya et al. 2025a). In the present

study (Fig. 13), the VFA profiles before AD demonstrated that on day 0, only the inoculum contributed to the composition, which was limited to acetic acid (25.74 ± 2 mg/L) and propanoic acid (14 ± 1.66 mg/L), as no VFAs were produced during the THP process. However, post-AD analysis (day 21) revealed that VFAs were detected across all conditions due to the efficient breakdown of acetogenic microbes on the THP cotton substrates, with only acetic acid being detected in the digestate samples.

Acetic acid, which serves as a critical precursor for methanogenesis and is particularly essential for acetoclastic methanogens, constituted the dominant component of the VFA profile across all analysed samples. Throughout the progression of the anaerobic digestion process, specialised microbial consortia undergo adaptive changes to increase their metabolic efficiency. Specifically, acetogenic bacteria and methanogenic archaea demonstrate an increased capacity to utilise acetic acid as the primary substrate for methane biosynthesis. This temporal adaptation reflects the establishment of syntrophic relationships between acetogenic and methanogenic populations, ultimately optimising the conversion of organic matter to biogas through the acetoclastic methanogenic pathway. This metabolic specialisation is key to the overall performance and stability of anaerobic digestion systems. The efficient utilisation of acetic acid directly correlates with increased methane yields and process optimisation, highlighting the importance of maintaining favourable conditions for acetoclastic methanogenesis in biogas production systems (Bonk et al. 2018; Kim et al. 2003; Nleya et al. 2025b).

This study identified the optimal THP conditions as 140 °C for 60 min, resulting in the lowest VFA concentration of 8.06 mg/L. This treatment resulted in the lowest VFA content, facilitating efficient conversion to biogas in terms of yield and composition, as shown in Fig. 8. The results demonstrated a significant reduction in VFA following AD, particularly in the THP of the cotton samples, which was conducive to increased biogas production. The substantial decrease in VFA concentrations can be attributed to the improved substrate biodegradability during THP. The THP process accelerates hydrolysis and solubilisation, increasing the accessibility of VFAs to methanogenic microorganisms and thereby promoting more efficient biogas conversion pathways.

**Table 8** Free ammonia nitrogen of the digestate before (day 0) and after (day 21) anaerobic digestion

| Operating conditions | Free ammonia nitrogen ($NH_3$-N), mg/L | |
|---|---|---|
| | Day 0 | Day 21 |
| Raw + Inoculum | 17.24 ± 3.21 | 46.83 ± 4.39 |
| 120 °C, 30 min | 5.88 ± 1.3 | 38.72 ± 1.00 |
| 120 °C, 60 min | 1.08 ± 0.25 | 25.71 ± 0.75 |
| 140 °C, 30 min | 1.93 ± 0.04 | 24.43 ± 1.14 |
| 140 °C, 60 min | 2.92 ± 0.77 | 23.82 ± 0.37 |
| 160 °C, 30 min | 1.81 ± 0.44 | 34.17 ± 1.18 |
| 160 °C, 60 min | 1.47 ± 0.39 | 41.3 ± 1.81 |
| 180 °C, 30 min | 0.65 ± 0.1 | 24.32 ± 0.59 |
| 180 °C, 60 min | 0.16 ± 0.03 | 24.6 ± 1.14 |
| 200 °C, 30 min | 0.1 ± 0 | 28.81 ± 0.26 |
| 200 °C, 60 min | 0 ± 0 | 24.98 ± 1.03 |

## Conclusion

Thermal hydrolysis pretreatment materially improved the anaerobic digestibility of cotton textile waste, increasing methane yield by 123% and doubling biodegradability under optimal conditions (140 °C, 60 min), while maintaining non-inhibitory digestate chemistry.. The maximum THP condition (200 °C, 60 min) effectively disrupted the cellulose crystalline structure, reducing the crystallinity from 81 to 69%, thereby increasing COD solubilisation to 51% and facilitating efficient microbial degradation pathways for efficient methane conversion. Specifically, the optimal THP conditions were established at 140 °C for 60 min, resulting in a remarkable increase in biogas production from 52 mL/g VS in untreated cotton to 93 mL/g VS. Correspondingly, kinetic modelling demonstrated that optimal THP conditions improved biodegradability from 2.3% to 4.51% and decreased the lag phase to 2.3 days during AD. Optimal THP conditions maintained stable pH levels, maintained ammonia nitrogen and VFAs below inhibitory thresholds, and produced high-quality biogas with $CH_4$:$CO_2$ ratios exceeding 2.3, which is most beneficial for efficient methane conversion. Conversely, elevated temperatures at 160 °C promoted the formation of inhibitory cellulose degradation products, leading to reduced biogas yields despite achieving a maximum COD solubilisation of 51% and subsequent accumulation of recalcitrant compounds.

This research provides foundational insights into the behaviour of cotton textile waste under THP conditions and establishes performance metrics for enhanced biogas production. The proposed THP-AD process for cotton textile waste offers an environmentally sustainable approach by converting recalcitrant waste into renewable energy, thereby reducing landfill disposal and associated greenhouse gas emissions. Nevertheless, several critical investigations require attention to overcome existing limitations. Overall, these findings establish cotton-specific THP operating conditions and provide a clear basis for evaluating THP–AD as a scalable, lower-emissions route for diverting cotton textile waste from landfill. Future work should focus on optimising THP parameters such as the textile size, solid content, operating conditions, and energy recovery, to improve cotton AD conversion efficiency. Future studies should employ GC–MS or HPLC analysis to identify

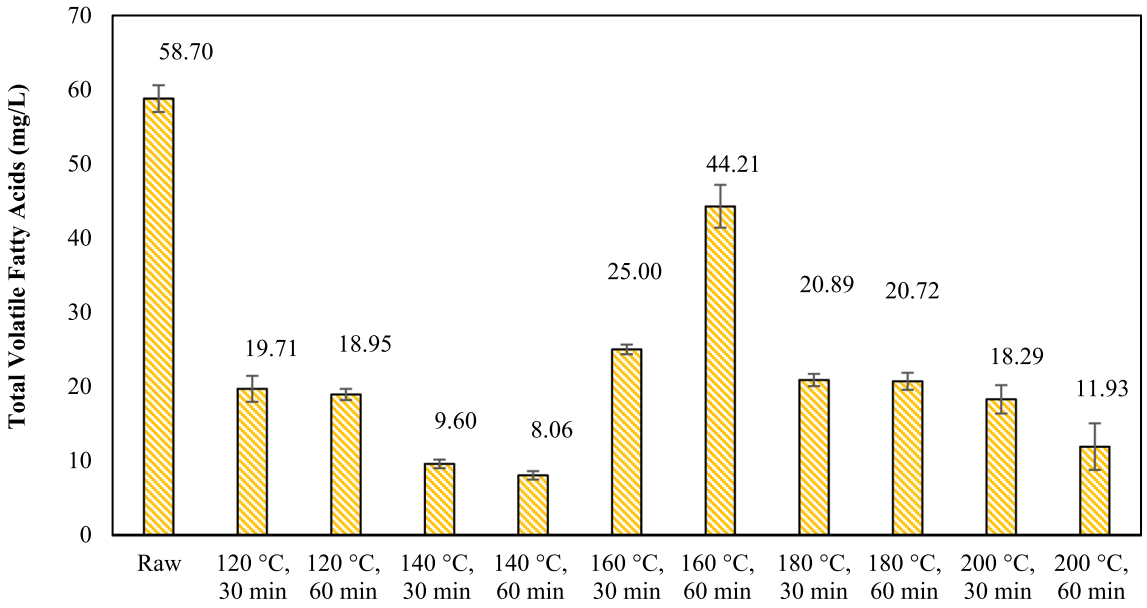



**Fig. 13** Changes in VFAs of THP cotton on day 21 anaerobic digestion

specific toxic compounds (e.g., furfural, HMF) formed at ≥ 160 °C, which would help explain the observed drop in methane yield. Moreover, future research should be extended to THP cotton co-digested with wool textiles or other nitrogen-rich substrates to validate the proposed C/N ratio. Additionally, a comparison of the energy inputs for the THP and the energy recovered from the AD plant is necessary to strengthen the sustainability argument. The economic feasibility of THP integrated with existing AD for textile waste management should be investigated in a subsequent study. Furthermore, studies should investigate the use of ATHP in cotton, which facilitates oxidative reactions that could increase cotton biodegradability by efficiently breaking down its structure. The implications of the suggested aspects may contribute to more sustainable textile waste management practices.

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## Declarations

**Conflict of interest** The authors declare that they have no conflicts of interest.

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