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Track 3 — Mechanical Engineering, Materials and Local Industrial Processes

HARNESSING THE POTENTIAL OF WATER VAPOUR TO EXTRACT FIBRES FROM PINEAPPLE LEAVES FOR USE IN DYEABLE TEXTILES

Exploiter le potentiel de la vapeur d'eau pour extraire des fibres des feuilles
d'ananas destinées à des applications textiles aptes à la teinture

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1. RÉSUMÉ / ABSTRACT

Résumé

Les feuilles d'ananas (*Ananas comosus*), résidus agricoles abondants, constituent une source prometteuse de fibres lignocellulosiques pour l'industrie textile. Cette étude compare le rouissage à l'eau (14 jours) et un traitement hydrothermique par vapeur d'eau saturée (115 ± 5 °C) pendant 30, 60, 90 et 120 min. Les fibres ont été blanchies au peroxyde d'hydrogène puis teintées par épuisement avec un colorant cuivrique mordancé au sulfate d'aluminium. Les propriétés colorimétriques (CIELAB), mécaniques (traction), thermiques (ATG, méthode de Broido) et la

stabilité colorimétrique sous vieillissement UV ont été évaluées. Une extraction à la vapeur de 60 min offre les meilleures performances mécaniques tout en réduisant fortement le temps d'extraction. La teinture augmente la rigidité des fibres mais diminue légèrement leur stabilité thermique. Les fibres extraites par vapeur présentent également une meilleure stabilité colorimétrique sous UV, confirmant l'intérêt de cette méthode comme alternative rapide, économe en eau et durable au rouissage conventionnel.

Abstract

Pineapple leaves (*Ananas comosus*), which are abundant agricultural residues, represent a promising source of lignocellulosic fibres for the textile industry. This study compares water retting (14 days) with a hydrothermal treatment using saturated steam (115 ± 5 °C) for 30, 60, 90 and 120 minutes. The fibres were bleached with hydrogen peroxide and then dyed by exhaustion with a copper dye mordanted with aluminium sulphate. The colourimetric (CIELAB), mechanical (tensile strength), thermal (TGA, Broido method) properties and colour stability under UV ageing were evaluated. A 60-minute steam treatment offers the best mechanical performance whilst significantly reducing extraction time. Dyeing increases the stiffness of the fibres but slightly reduces their thermal stability. Steam-extracted fibres also exhibit better colour stability under UV exposure, confirming the value of this method as a rapid, water-efficient and sustainable alternative to conventional retting.

Keywords: Pineapple leaf fibres, Water vapour extraction, Hydrothermal treatment, Textile dyeing, Colour stability.

2. INTRODUCTION

In light of the challenges posed by climate change and the depletion of fossil fuels, the development of bio-based, renewable and biodegradable materials is now a priority for industrial sectors, particularly the textile industry. This transition is driven by the need to reduce dependence on synthetic fibres derived from petrochemicals, which are responsible for significant greenhouse gas emissions and microplastic pollution, as well as to limit the environmental impacts associated with intensive cotton cultivation, which is known for its high consumption of water, pesticides and chemical fertilisers [1], [2], [3]. In this context, lignocellulosic fibres derived from agricultural residues appear to be particularly promising alternatives due to their renewable nature, availability, low density, low cost and small carbon footprint [4], [5].

Among these resources, pineapple leaves (*Ananas comosus*) represent a source that remains largely under-utilised [6]. After the fruit has been harvested, several tens of tonnes of leaves are produced per hectare and are generally left on the fields, ploughed under or burnt, resulting in losses of usable biomass as well as greenhouse gas emissions [6], [7], [8]. The fibres extracted from these leaves are characterised by a high cellulose content, generally ranging from 60 to 82 %, low density, and excellent mechanical properties, with tensile strengths that can exceed 600 MPa and a Young's modulus higher than that of many conventional natural fibres such as jute, kenaf or sisal [5], [9], [10]. These characteristics make pineapple leaf fibres particularly attractive for technical textile applications and biocomposites, particularly in tropical pineapple-producing countries such as Cameroon, where their utilisation could contribute to a circular economy based on agricultural residues.

However, the final quality of the fibres depends heavily on the extraction process used. The traditional reference method involves water retting, which consists of immersing the leaves for several days to promote the microbiological breakdown of the pectins responsible for the cohesion between the fibre bundles [6], [8], [11], [12]. Although this technique has the advantage of being simple and inexpensive, it suffers from several major limitations: a relatively long processing time, high water consumption, the production of organic effluents that may pollute aquatic environments, and variability in fibre quality linked to the biological

conditions of the retting process [5], [13]. To address these limitations, several alternative processes have been proposed, including hydrothermal treatments using saturated steam. Under the combined effect of temperature and pressure, these treatments cause partial hydrolysis of pectins and hemicelluloses, as well as limited modification of lignin, which facilitates fibre separation whilst considerably reducing extraction time [14], [15], [16]. Hydro thermiques utilisent peu ou pas de réactifs chimiques, réduisent la production d'eau usées et s'inscrivent davantage dans une démarche de transformation durable de la biomasse lignocellulosique.

Despite the numerous studies devoted to the physical, mechanical, thermal and morphological characterisation of pineapple leaf fibres obtained using various extraction methods [5], [7], [8], [17], their suitability for textile finishing processes remains relatively understudied. In particular, few studies have examined the influence of the extraction process on the colourimetric behaviour of the fibres after bleaching and dyeing, as well as on colour stability under accelerated photochemical ageing. Yet these properties are key criteria for the use of the fibres in the apparel, furnishing and technical textiles sectors.

With this in mind, the present study aims to compare the effects of saturated steam treatment applied for 30, 60, 90 and 120 minutes on the colourimetric, mechanical and thermal properties of pineapple leaf fibres, both before and after dyeing, using fibres obtained by water retting as a reference. The colourfastness to accelerated ultraviolet ageing is also assessed in order to evaluate the durability of the dye. The objective is to identify the optimal treatment conditions that reduce extraction time whilst preserving the fibres' mechanical performance and dyeability, with a view to the sustainable industrial utilisation of pineapple leaf residues.

3. MATERIALS AND METHODS

3.1 From residual pineapple leaf to dyed fibre

Residual pineapple leaves were collected after harvest from the agroecological zone of Loum (Moungo Division, Cameroon) and processed into fibre bundles using two extraction routes (Fig. 1).



Fig. 1. The process of extracting fibre strands by water retting (top) and steam treatment (bottom) of pineapple leaves

The reference route consisted of water retting in fresh water at 25 ± 2 °C for 14 days, followed by manual scraping with a serrated knife, in accordance with Betené et al. [18], [19]. The extracted bundles were rinsed with distilled water, sun-dried for 24 h and vacuum-packed. In parallel, the leaves were subjected to saturated

steam treatment (115 ± 5 °C) for 30, 60, 90 or 120 min after thermal stabilisation, arranged in a single layer on a perforated tray. This hydrothermal treatment induced the partial hydrolysis of pectins [20], hemicelluloses and lignin [21], thereby facilitating fibre bundle release. After scraping, the bundles were washed, air-dried and conditioned at 25 ± 2 °C and $65 \pm 5\%$ RH until constant mass, before being combed to ensure fibre alignment. The bundles were subsequently bleached (Fig. 2) in a solution containing 15 wt% H₂O₂ (material-to-liquor ratio of 1:100 g/mL) at 50 °C for 30 min, rinsed to neutrality and dried. Exhaust dyeing [22] was then carried out in a dye bath containing 6 wt% copper dye and 2 wt% aluminium sulphate (material-to-liquor ratio of 1:40 g/mL) at 90 °C for 30 min under continuous agitation [22]. After cooling, the bundles were rinsed, dried at ambient temperature and coded according to the extraction process (Table 1).

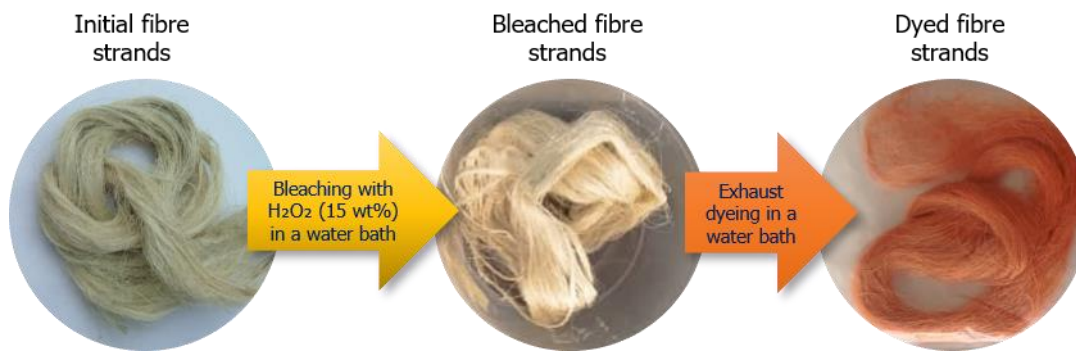


Fig. 2. Dyeing process: from extracted fibre strands to dyed fibres.

Table 1 - Sample batch coding

Code	Description
AC	Pineapple leaf fibre extracted by water retting (reference fibre)
30AC	Pineapple leaf fibre extracted after 30 min of saturated steam treatment
60AC	Pineapple leaf fibre extracted after 60 min of saturated steam treatment
90AC	Pineapple leaf fibre extracted after 90 min of saturated steam treatment
120AC	Pineapple leaf fibre extracted after 120 min of saturated steam treatment
AC-D	Pineapple leaf fibre extracted by water retting and dyed (reference dyed fibre)
30AC-D	Pineapple leaf fibre extracted after 30 min of saturated steam treatment and dyed
60AC-D	Pineapple leaf fibre extracted after 60 min of saturated steam treatment and dyed
90AC-D	Pineapple leaf fibre extracted after 90 min of saturated steam treatment and dyed
120AC-D	Pineapple leaf fibre extracted after 120 min of saturated steam treatment and dyed

3.2 Analysis of fibre appearance using colourimetry on extracted and bleached fibre strands

The colourimetric properties of the extracted, bleached and dyed fibre bundles were determined in the CIELAB colour space in accordance with ISO 105-E01 [23]. For each batch, three specimens were prepared by winding the fibre bundles onto a cardboard support to provide a continuous surface and ensure reproducible measurements [22]. The colour coordinates L^* , a^* and b^* were measured using a 3NH colourimeter, model CR1 (8 mm aperture; 0.5 s response time). Three measurements were taken at different locations on each specimen and averaged. The parameters L^* (lightness: 100 = white, 0 = black), a^* (red-green axis) and b^* (yellow-blue axis) were used to calculate the chroma (C^* , Eq. 1), hue angle (h , Eq. 2) and whiteness (WI) indices (Eq. 3). Finally, the total colour difference (ΔE_{ab}^*), determined in accordance with ASTM E313 (Eq. 4), was used to quantify the effects of the extraction process, steam treatment duration and dyeing on the colour evolution of the fibre bundles.

$$C^* = \sqrt{a^{*2} + b^{*2}} \quad (1)$$

$$h = \tan^{-1} \left(\frac{a^*}{b^*} \right) \quad (2)$$

$$WI = L^* - 3b^* \quad (3)$$

$$\Delta E_{ab}^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (4)$$

3.3 Determination of the mechanical properties of extracted and dyed fibre strands

The tensile properties of the extracted and dyed fibre bundles were determined using an LDW-5 universal testing machine equipped with a 100 N load cell. Specimens were prepared in accordance with NF T25-501-2 and mounted on an adhesive paper frame providing a gauge length of $l_0 = 10$ mm (Fig. 2.16a), before being conditioned at 28 °C and 90% RH for 24 h. Each specimen was then clamped between the grips, the supporting frame was cut, and the bundle was subjected to uniaxial tensile loading at a crosshead speed of 2 mm min⁻¹ until failure. The mean diameter of each fibre bundle, measured according to Jeannin et al. [24], was used to estimate the apparent cross-sectional area (A_0) [24], [25]. The nominal stress and strain were calculated as $\sigma = F/A_0$ and $\varepsilon = \delta_T/l_0$, where F is the axial load (in N) and δ_T is the crosshead displacement (in mm). Young's modulus was determined by linear regression over the strain range of 0.2-0.6%. The tensile strength was taken as the maximum stress, with the corresponding strain defined as the strain at failure. Fifteen specimens were tested for each batch.

3.4 Analysis of the thermal degradation of extracted and dyed fibre strands

The mass loss and thermal degradation kinetics of the extracted and dyed fibre bundles were characterised using a TGA Q50 thermogravimetric analyser. Approximately 12 mg of each sample was placed in an aluminium crucible and heated from 25 to 650 °C at a rate of 10 °C/min under a nitrogen atmosphere (10 mL/min) to ensure inert conditions. The activation energy (E_a) was estimated using the Broido method [26], according to Equation (5):

$$\ln \left[\ln \left(\frac{1}{\alpha} \right) \right] = - \left(\frac{E_a}{R} \right) \left[\left(\frac{1}{T} \right) + K \right] \quad (5)$$

where $\alpha = m_t/m_0$ is the normalised mass, with m_t denoting the sample mass at time t and m_0 the initial mass. R is the universal gas constant (8.32×10^{-3} kJ/mol.K), T is the absolute temperature in Kelvin, and K is a constant. The activation energy E_a was determined from the linear relationship between $\ln[\ln(1/\alpha)]$ and $1/T$, with the slope corresponding to $-E_a/R$. This method provides a robust and reproducible estimation of the kinetic parameters.

3.5 Experiments on the ageing of dyed fibre strands under ultraviolet radiation

As lignocellulosic materials are particularly susceptible to photodegradation (Fig. 3a), the colour stability of the dyed fibre bundles was assessed by accelerated UV photoageing according to a protocol adapted from Jiang et al. [23]. For each condition, three specimens were prepared by uniformly winding 2 g of fibre bundles onto an aluminium plate to ensure secure positioning and homogeneous irradiation. The tests were carried out in a closed chamber (Fig. 3b) equipped with a 16 W UV lamp (model, manufacturer; 50/60 Hz; wavenumber 280 nm), positioned horizontally 25 cm above the specimens to provide a vertical radiant flux simulating incident solar radiation [22]. The initial colour coordinates (L^* , a^* , b^* , C^* and h) were used as the reference state and subsequently measured after 70, 360, 1440, 3420, 7200 and 10,800 min of exposure. The data, processed using Microsoft Excel, were used to calculate the mean ΔE^* values relative to the initial state, as well as the colour differences between fibre bundles extracted by steam treatment and water retting, in order to evaluate the effect of the extraction process on their colour stability.

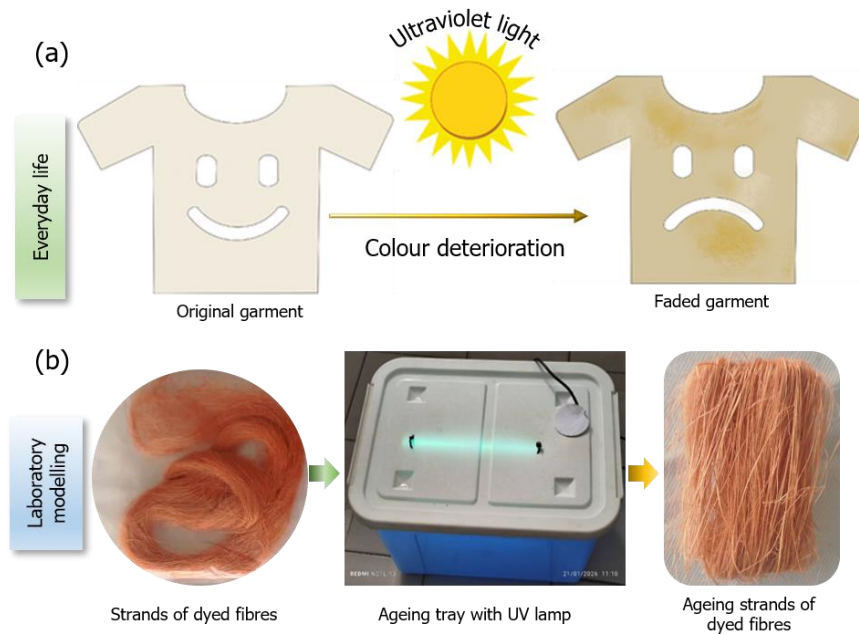


Fig. 3. Illustration of (a) the degradation of clothing under UV radiation under real-world conditions (adapted from Jiang et al. [23]) and (b) its experimental evaluation in the laboratory at the level of dyed fibre strands.

4. RÉSULTS AND DISCUSSION

4.1. Effect of steam exposure time and bleaching on fibre colour

As shown in Fig. 4, increasing the duration of exposure to the vapour (30 - 120 min) gradually reduces L^* and WI , which fall below the reference values after 60 min, whilst a^* and b^* increase but remain below the ACB reference values at 30 and 60 min respectively. The colour difference remains moderate ($\Delta E^* \approx 4 - 5$), except at 120 min ($\Delta E^* \approx 6$). The correlation between WI and the crystallinity index highlights the better preservation of fibres treated for 30 minutes, confirming that prolonged hydrothermal treatments promote the formation of chromophores and a loss of whiteness [15], [27].

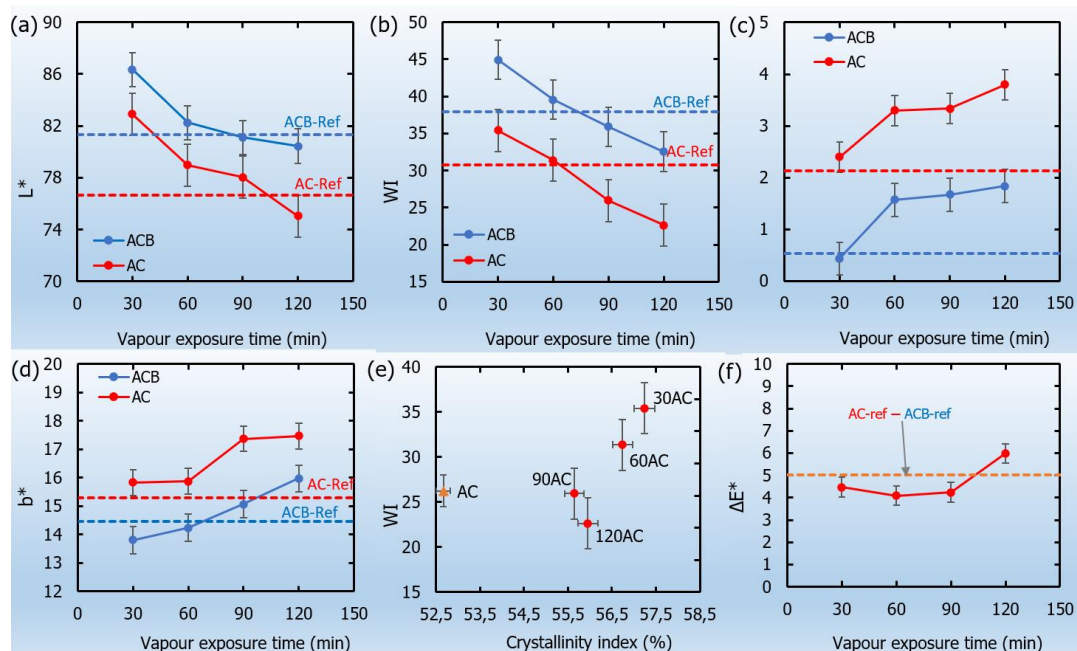


Fig. 4. Changes in colour measurements: (a) L^* , (c) a^* , (d) b^* , and (b) the whiteness index (WI) as a function of steam exposure time. (e) Correlation between the whiteness index and the crystallinity index, and (f) changes in colour after bleaching as a function of steam exposure time.

4.2 Effect of dyeing and steam exposure time on mechanical properties

Table 2 shows that hydrothermal treatment progressively improves the mechanical properties of the fibres up to 60 minutes, at which point the tensile strength ($510,8 \pm 173$ MPa) and Young's modulus ($21,1 \pm 9,4$ GPa) reach their maximum values. This improvement is attributed to the removal of amorphous constituents (pectins, hemicelluloses) and to a better organisation of the cellulose microfibrils, whilst the decrease observed at 90 and 120 minutes reflects degradation of the cell wall due to prolonged hydrolysis [5], [28]. After dyeing, the strength is generally maintained, with a peak also at 60 minutes ($512,4 \pm 341$ MPa), whilst the increase in Young's modulus to $24,8 \pm 12$ GPa suggests a stiffening effect of alum mordanting, without any notable change in elongation at break [8], [29].

Table 2 - Mechanical and thermal properties of the extracted fibres, depending on the extraction process and dyeing

Fibre strands	Mechanical properties			Thermal properties		
	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)	Activation energy (kJ/mol)	T _{ini} (°C)	T _{max} (°C)
AC	343,3 ±254,2	9,4 ±6,1	3,7 ±0,9	68,0	227,1	358,9
30AC	362,5 ±140	15,0 ±6	2,6 ±0,5	65,5	221,2	360,4
60AC	510,8 ±173	21,1 ±9,4	2,5 ±0,5	65,7	247,4	364,8
90AC	427,4 ±141	19,5 ±8,1	2,4 ±0,5	62,6	239,5	359,6
120AC	345,5 ±148	15,1 ±5,4	3,0 ±1,4	62,4	226,8	358,8
AC-D	354,9 ±230	17,3 ±9,7	2,5 ±0,6	38,7	171,2	256,0
30AC-D	322,4 ±180	19,0 ±7,8	2,8 ±1,1	44,1	170,7	256,9
60AC-D	512,4 ±341	22,6 ±12,1	2,9 ±0,4	47,0	174,5	259,3
90AC-D	495,2 ±218	23,4 ±9	2,8 ±0,6	39,2	170,1	254,6
120AC-D	473,2 ±300	24,8 ±12	2,7 ±0,6	48,4	171,2	253,2

4.3 Effect of dyeing and duration of exposure to steam on the thermal degradation of fibre strands

Fig. 3 and Table 2 show that steam treatment improves the thermal stability of the fibres up to 60 minutes, with an increase in T_{ini} (from 227,1 to 247,4 °C) and T_{max} (from 358,9 to 364,8 °C), due to the partial removal of amorphous compounds. This trend, consistent with the observed mechanical optimum, reflects improved structural organisation of the fibres [30]. Beyond this point, the decrease in these parameters and in the activation energy (from 68,0 to 62,4 kJ/mol) indicates a gradual degradation of the cellulose and lignocellulosic constituents [31]. Dyeing significantly reduces E_a, T_{ini} and T_{max} (T_{ini} ≈ 170 - 175 °C), probably due to the catalytic action of the dye and the mordant on the degradation of cellulose [5], [32], [33].

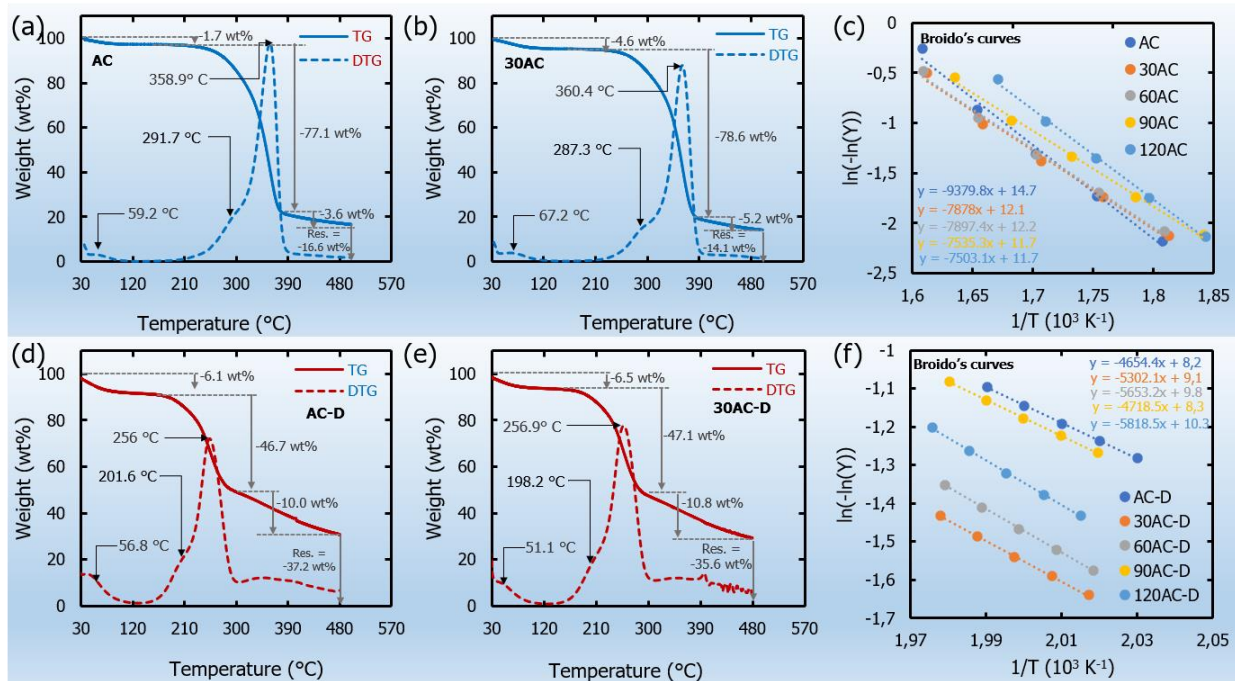


Fig. 5. Thermogravimetric behaviour (TG/DTG) of the fibres: (a) AC, (b) 30AC, (d) AC-D and (e) 30AC-D, as well as the Broido curves for the raw fibres (c) and the bleached fibres (f).

4.4 Colour change caused by photochemical ageing

Fig. 6 shows that UV ageing causes a gradual increase in ΔE^* , with a more pronounced change during the first 1440 minutes (24 h) of exposure, a period associated with the rapid photooxidation of residual lignin [34], after which the values stabilise (Table 2), reflecting a slowing down of the photooxidation process. The dyed strands resulting from the steam treatment exhibit a $\Delta E^*(D)$ lower than that of the rust-treated control after 60 minutes of steam treatment, indicating better colour stability, consistent with their reduced lignin content [30], [35]. The $\Delta E^*(D - Init)$ confirms the cumulative effect of dyeing and ageing, whilst the $\Delta E^*(D - B)$ remains moderate, showing that bleaching limits the extent of subsequent colour change [13], [36].

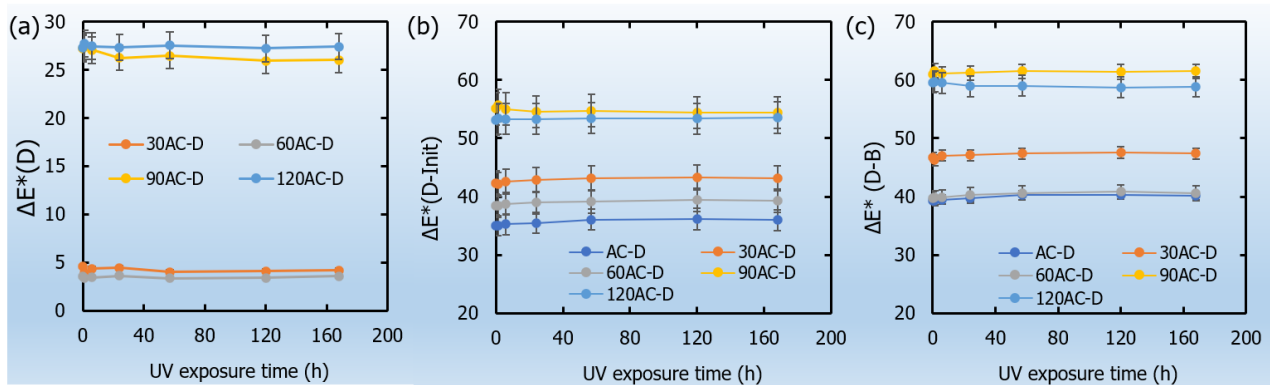


Fig. 6. Variation in the overall colour differences used in this study: (a) $\Delta E^*(D)$, the colour difference between the steam-extracted dyed fibre strands and the reference strands (dyed and extracted by retting) prior to ageing; (b) $\Delta E^*(D-Init)$, the colour difference between the aged dyed strands and the initial undyed and unaged strands; (c) $\Delta E^*(D-B)$, the colour difference between the aged dyed strands and the corresponding bleached, undyed and unaged strands.

5. CONCLUSION

This study compared conventional water retting with a saturated steam hydrothermal treatment for the extraction of fibres from pineapple leaves for use in textile dyeing. A 60-minute steam treatment proved to be

optimal, offering the best mechanical properties (tensile strength and Young's modulus) whilst significantly reducing the extraction time compared with the 14 days required for retting. Dyeing with copper dye mordanted with alum remains compatible with these fibres, despite a slight reduction in their thermal stability and activation energy. UV ageing tests show better colour stability in the steam-treated fibres. These results confirm the potential of this technique as a rapid, water-efficient and sustainable alternative for the industrial utilisation of pineapple leaf residues, particularly in tropical contexts.

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