BIO-INNOVATIVE FLAX RETTING

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Abstract: *Flax (Linum usitatissimum L.) is an important commercial crop that supplies both linseed and bast fibres for multiple applications. Retting, which is a microbial process, separates industrially useful bast fibres from non-fibre stem tissues. While several methods (i.e., water- and dew-retting) are used to ret flax, more recently enzymes have been evaluated to replace currently used methods. The enzymes remove the non-cellulosic compounds from the technical fibres, and at the same time are biodegradable and non-toxic, with no dependence on the weather conditions. Therefore, in this paper an enzymatic retting was researched, and its influence to the flax quality. Comparing the properties of enzyme-retted flax fibres the properties of water-retted ones, it can be found that enzymatic retted fibres are coarser, but much stronger. The results have shown that the strongest fibre was achieved with bath having enzyme concentration 0.2 % with similar fineness compared with other bath concentration. Therefore, this concentration can be considered as optimal one. The use of enzymes to extract fibres provides an environmentally friendly method toward developing reliable and sustainable agriculture using bio-based fibres of enhance quality.*

Keywords: *Flax fibres, enzymatic retting, fineness, tenacity*

1. Introduction

The history of flax (*Linum usitatissimum L.*) is long and important and its production goes back to ancient history. It can be grown and harvested within three months under reasonable moisture and relatively cool temperatures. Flax has also been considered as a source of linen, providing high-quality fibres for textiles for thousands of year. Longer fibres are used for spinning into yarn and making textiles. Shorter flax fibres are either spun into yarns, often mixed with cotton or used in many other novel applications including packaging materials, reinforcements for plastics and concrete, asbestos replacement, panel boards, lining materials for vehicles [1-5].

To obtain flax fibres for commercial use, flax steam undergo a process called retting (Fig. 1). Retting is a microbial process to separate the fibre from the non-fibre stem tissues (woody part), and removal of non-cellulosic components, such are pectin, hemicelluloses, lignin, waxes and fats [4-7]. The main idea is to degrade the pectin and other cementing compounds that bind the flax fibres and fibre bundles to other tissues and thereby separated fibres from non-fibre materials. The separated fibres are then cleaned of non-fibre materials by mechanical processing. Insufficient retting, or under-retting, results in poor separation of the non-fibre materials (i.e., shive) from the fibres which reduce fibre yield, processing efficiency, and ultimate fibre quality. On the other hand, over-retting can occur resulting in poor fibre quality.

Retting is of ultimate importance in fibre yield and quality [4-10]. There are two methods used commercially: water- and dew- retting.

In water-retting flax stems are submerged in rivers and lakes, and anaerobic bacteria such as *Clostridium spp.*, colonize the flax stems; produce pectinases and other enzymes to degrade pectin and other matrix compounds, thus freeing fibres from the core tissues [6]. The retting period varies from three days to a week, depending on the temperature of water. It must be carefully judged because under-retting makes separation difficult, and over-retting weakens the fibre. Water-retting results in high quality fibre which is long, strong and fine; therefore excellent for apparel and other textiles. On the other hand, the stench from anaerobic fermentation of the plants, extensive pollution of waterways, high drying costs, and putrid odour of resulting fibres resulted in a move away from anaerobic water-retting to dew-retting. However, the advantage of water- over dew retting is in that it is more controllable and avoids the risk of the crop being spoilt by inclement weather during the weeks that it lies on the ground [4, 8, 11].

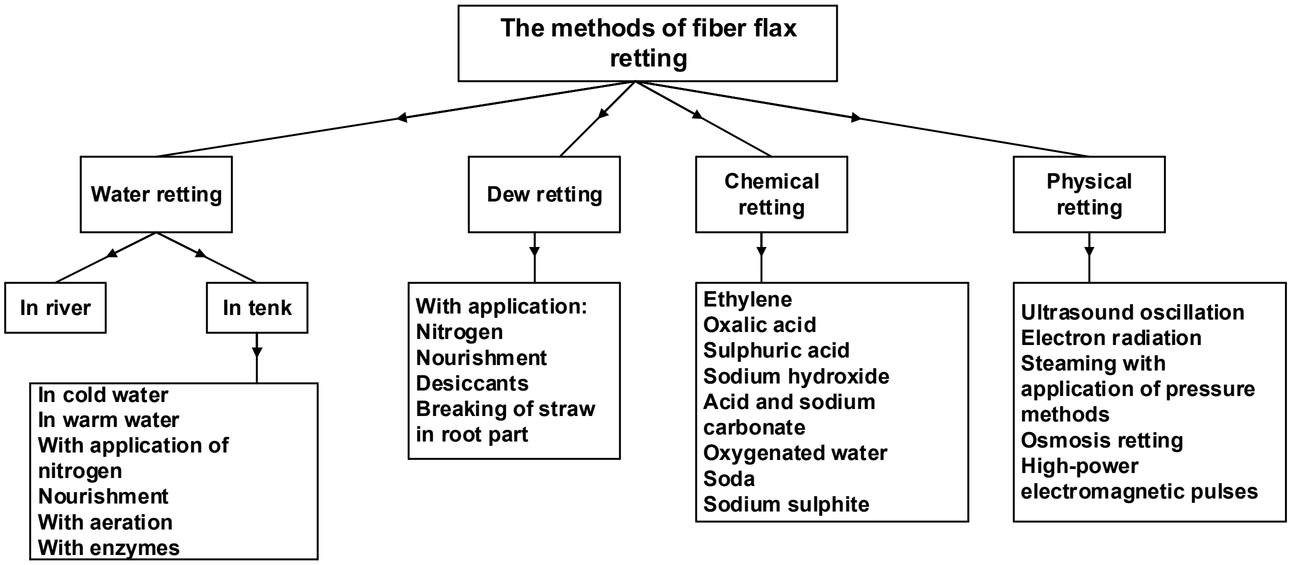

Figure 1. The methods of fibre flax retting

Dew (or field) retting is the method used in western Europe for obtaining textile fibres, and reported to be the oldest one, practiced thousands of years ago by the Egyptians. Dew-retting is the result of colonization and partial plant degradation by plant-degrading, aerobic fungi of flax stems, which are harvested and laid out in swaths in fields. The highest quality linen fibres are produced, but concern exists within this industry about low and inconsistent quality as a result of that the stems on the top of the swath take more moisture from rain than those located on the bottom and dry faster accordingly. To avoid notable difference in retting between stems in top and bottom part of the swath and improve the quality of obtained raw material the swath of straw is turned over – 2 or 3 times, depending primarily on the meteorological conditions during the retting period. Dew retting depends on particular geographical regions that have the appropriate moisture and temperature ranges for retting. The result is coarser and lower quality fibre than if water-retted, and occupation of agricultural fields for several weeks is significant with increase the risk of damage to the crop due to unsuitable weather during the retting period. In areas of proper climate and expertise, commercially dew retting works and has been the method of choice for linen and other flax fibre production. Most of the world's textile flax fibre is produced by field retting [2, 4, 6].

Since the water- and dew-retting are based on the activity of the pectinolytic microorganisms, the enzymes have been introduced as a potential replacement for flax retting by Brown & Sharma in 1983 [6-8]. Due to the structural complexity associated with the flax stem, it was originally suggested that a mixture of enzyme activities (e.g. polygalacturonases, pectin lyase, hemicellulases, and cellulase) are needed flax retting [6-10]. Therefore, few commercially available enzyme mixtures were identified which were capable of retting flax: Flaxzyme (by Novo Nordisk, consisting predominantly of pectinolytic enzyme), Novozyme 465 (Cellulase by Novozymes), Novozyme 249 (Pectinase by Novozymes), Texazyme BFE (pectinase by InoTex), BioPrep 3000L (pectinase by Novo Nordisk), etc. Viscozyme L (Novozymes), an multi-enzyme complex containing a wide range of carbohydrases, including arabanase, cellulase, β-glucanase, hemicellulase, and xylanase, currently is in use for flax retting. In 2000s—ethylenediamine-tetra-acetic acid (EDTA) was introduced in retting process with pectinases as a chelator [8]. Use of Viscozyme and EDTA became the basis to which all other products are compared. The yield and tactile qualities of enzyme-retted fibres are similar to the high-quality water-retted fibres, but with the potential lower fibre strength due to the continued activity of the cellulases in the mixtures.

The bio-innovation in cotton scouring using neutral and acid pectinases resulted in environmental friendly and effective process [12]. Enzymes under the trade name Beisol™ (CHT, Germany) were able to remove barrier layers, thus enabling emulsification of the waxes. This resulted in improved hydrophilicity and accessibility of the fibres for bleach active compounds or dyestuffs, soft handle, good whiteness and less weight loss. Additionally, it was unnecessary to perform neutralisation [12, 13] or to use chelating agent. Therefore, in this paper the application of neutral pectate lyase Beisol PRO was research to achieve fineness and maintain fibre strength after the enzyme-retting similar to water-retted flax fibres.

2. Material and methods

2.1. Material

The trial with the fibre flax were carried out at the experimental field of the Faculty of Agriculture of Zagreb (45°49'26" N, 16°02'07" E), on anthropogenized eutric cambisol in 2012. As part of the basic and pre-sowing seedbed preparation, 500 kg/ha of NPK fertilizer (7:20:30) was applied while 100 kg/ha of calcium ammonium nitrate (27 %) was added with topdressing when the plants were 10 cm high. Sowing density was 2 500 germinable seeds/m^2. The main trial plot size was 10 m^2 (10 rows x 0.1 m row spacing x 10 m length).

Water-retting. Flax stems were subjected to biological maceration for 72 hours in hard hot water (32 ºC) under controlled conditions. They were dried with a stream of warm air and weighed. A scutching machine was used to separate straw (woody matter) from fibre.

Bio-innovative retting. Pectin splitting enzyme Beisol PRO (CHT/Bezema), applicable for discontinuous, semi-continuous and continuous processes, was used for the bio-innovative flax retting. It is capable product for individual application as well as for integration within a process of enzymatic desizing or enzymatic scouring of cotton. It does not require the presence of a sequestering agent, is efficient within a wide temperature range from 20 to 98 °C, and pH of 6.0-9.0 as well. In this research it was applied by exhaustion method in Polycolor turbomat (Mathis) in three concentrations: 0.1 %, 0.2 % and 0.5 % owf (over weight of fibre), in buffered solution at 55 °C for 60 min. The Fluka buffer solutions were used for varying pH: Buffer solution pH 5.0 (citric acid/ sodium hydroxide solution), pH 7.00 (potassium dihydrogen phosphate/ sodium hydroxide); and pH 9.0 (borax/ hydrochloric acid).

2.3. Methods

The influence of enzymatic retting on the flax fibre quality was researched. After the retting, flax fibres were characterized by the following textile-technological properties: fineness and tenacity determined on tensile testers Vibroscop and Vibrodyn 400. Fineness was determined according to ISO 1973:1995 *Textile fibres - Determination of linear density - Gravimetric method and vibroscope method.* Tenacity was determined according to ISO 5079:1995 *Textile fibres - Determination of breaking force and elongation at break of individual fibres.* The standards and regulations were adapted to testing technical flax fibres. The cogged steal clamps were placed on the standard clamps, and the selected testing speed was 3 mm/min. The selected gauge length of the sample is 5 mm to ensure that all elementary fibres are included in the tested sample during fineness and tenacity testing. Due to the non-homogeneity of flax fibres, the number of measurements was increased (n=100) and determined according to statistical indications of the degree of reliability. Measurements of fibres of tested properties were performed on conditioned samples.

3. Results and Discussion

The evaluation of the quality of flax fibres is given on the basis of the assessment of external properties, fineness and tenacity of fibres after enzyme-retting, and compared to water-retted fibres. At least 100 measurements of the fibre properties were performed. The statistical indicators: arithmetic mean, $\overline{x}$, minimum value, x_{min}, maximum value, x_{max}, standard deviation, s, coefficient of variation, V and practical margin fault P_{gg} were calculated. The results of fineness with statistical indicators are collected in Table 1 and compared to water-retting in Figure 2.

Table 1. The results of flax fibres fineness measurement after retting with statistical indicators

Bath	$\overline{x}$ [dtex]	x_{min} [dtex]	x_{max} [dtex]	s [dtex]	V [%]	p_{gg} [%]
Water-retted	37.81	33.32	98.45	10.46	27.67	5.42
0.1 %, pH 7	43.49	29.76	102.60	15.84	24.95	4.89
0.1 %, pH 5	48.00	23.29	97.68	13.33	27.78	5.44
0.1 %, pH 9	51.78	25.16	90.28	15.22	29.39	5.76
0.2 %, pH 7	42.65	20.79	131.00	16.31	38.24	7.49
0.2 %, pH 5	40.98	20.12	72.22	11.11	27.10	5.31
0.2 %, pH 9	38.52	20.14	65.73	12.02	31.20	6.12
0.5 %, pH 7	49.07	24.13	84.85	12.80	26.08	5.11
0.5 %, pH 5	50.43	21.54	91.77	15.23	15.10	4.19
0.5 %, pH 9	51.46	24.39	86.88	14.65	28.47	5.58

In retting process flax fibre needs to be separated from the non-fibre stem tissues by the removal of non-cellulosic components; such are pectin, hemicelluloses, lignin, waxes and fats. The pectin on cellulose fibres is a complex mix of different substances out of pectin, mainly polygalacturonic acid, the rhamno-galacturonan, which is bound at the rest of the rhamnosyl residue irregularity with arabinan, galactan, and many other compounds, mostly polysaccharides, cellulose, protein etc. including Ca, Mg and Fe which forms an interlaced net structure difficult to solve in water [4, 12]. It is important to degrade the pectin net structure to emulsify the fats and waxes beneath it. A complete degradation is unnecessary because the fragments of the complex structure are soluble in water and can be washed off.

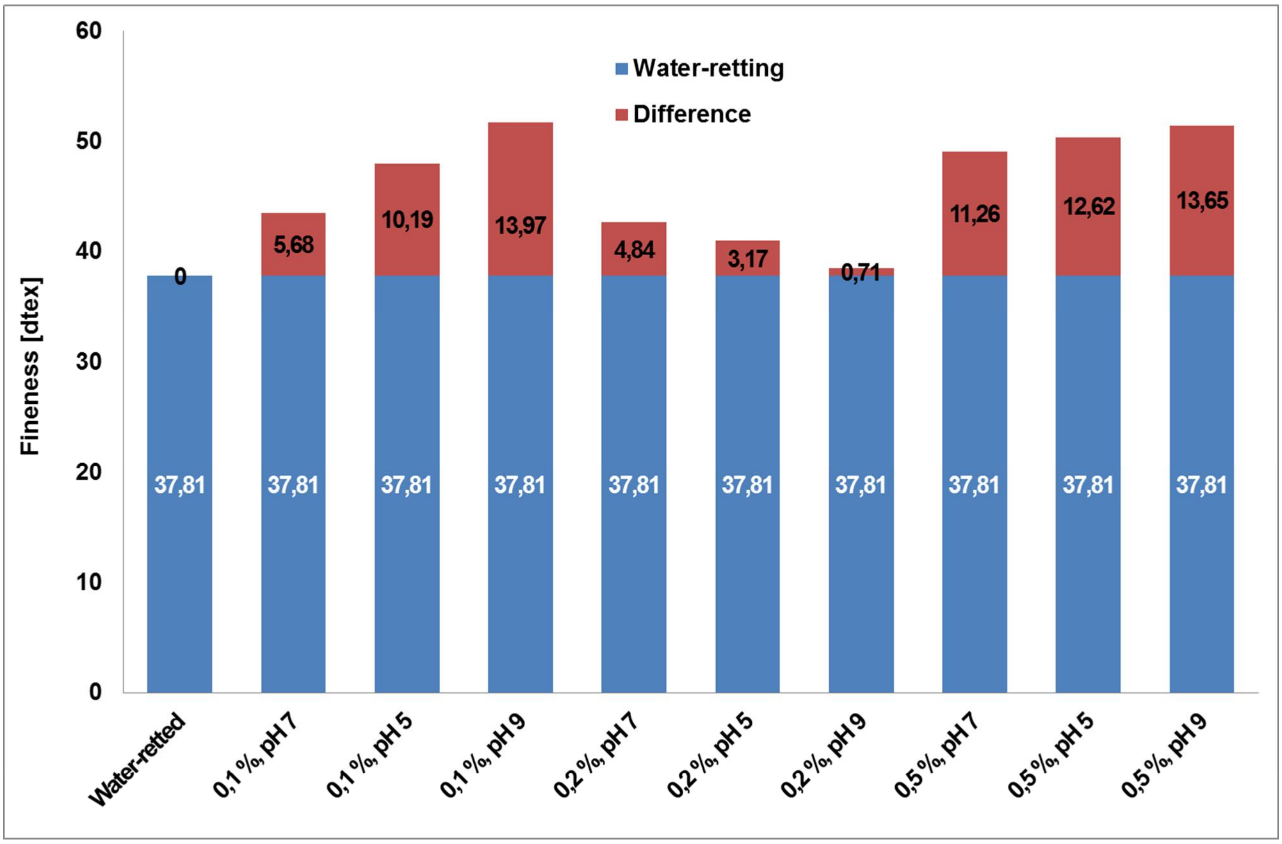

Figure 2. The fineness of flax fibres after enzymatic retting compared to water-retting

From the results presented in Table 1 and in Figure 2 it can be seen that the enzymatic retting has influenced to the flax fineness. All the fibres are coarser than water retted ones regardless of applied concentration of enzymes. Considering the applied concentrations, the concentration of 0.2 % owf resulted in the finest fibres, similar to water-retted ones (less than 10 % of difference in fineness); whilst the concentration of 0.5 % owf resulted in the coarser fibres (more than 29 %) The pH of the bath has influence to the fineness as well. From Figure 2 can be seen that the application of enzymes in the neutral bath (pH 7) results with finer fibres, except in bath with enzyme concentration 0.2 % owf. This was expected since this enzyme Beisol PRO is pectinase with optimal activity in neutral medium. The coarsest fibres were the ones obtained from enzymatic retted with 0.1 % and 0.5 % in the bath of pH 9. These fibres are 36 % coarser than water-retted one. These results indicate lower the enzyme activity in alkaline medium, less fibre impurities are removed and therefore fibres are coarsest.

The results of flax fibres tenacity measurement after water and enzymatic retting are shown in Table 2 with statistical indicators and compared to water retting in Figure 3.

From the statistical indicators of variability shown in Table 2 it can be seen a relatively high variation coefficient (ranging from 24 % to 38 %) as the consequence of a high fineness variability of flax fibres. However, it is evident that the enzymatic retting of flax results in better tenacity of fibres than water retting (80.35 cN/tex). The reason for that can be that the coarser fibres are stronger. The only exception is the retting process in the bath containing 0.5 % of enzyme at pH 9 (71.68 cN/tex) which resulted in the coarsest fibre as well.

Table 2. The results of flax fibres tenacity measurement after retting with statistical indicators

Bath	$\overline{X}$ [cN/tex]	x_{min} [cN/tex]	x_{max} [cN/tex]	s [cN/tex]	V [%]	p_{gg} [%]
Water-retted	80.35	38.33	155.23	23.81	29.63	5.81
0.1 %, pH 7	92.69	36.26	147.92	26.04	28.09	5.51
0.1 %, pH 5	92.41	41.58	92.41	30.02	32.48	6.37
0.1 %, pH 9	81.34	23.16	146.53	25.25	31.04	6.08
0.2 %, pH 7	106.32	28.18	191.05	34.22	32.19	6.31
0.2 %, pH 5	109.30	35.20	197.32	36.12	33.05	6.48
0.2 %, pH 9	119.00	61.27	196.52	38.17	32.08	6.29
0.5 %, pH 7	87.33	34.46	144.49	25.97	29.53	5.79
0.5 %, pH 5	80.77	35.37	148.56	24.18	29.93	5.87
0.5 %, pH 9	71.61	22.48	125.77	25.10	17.53	4.86

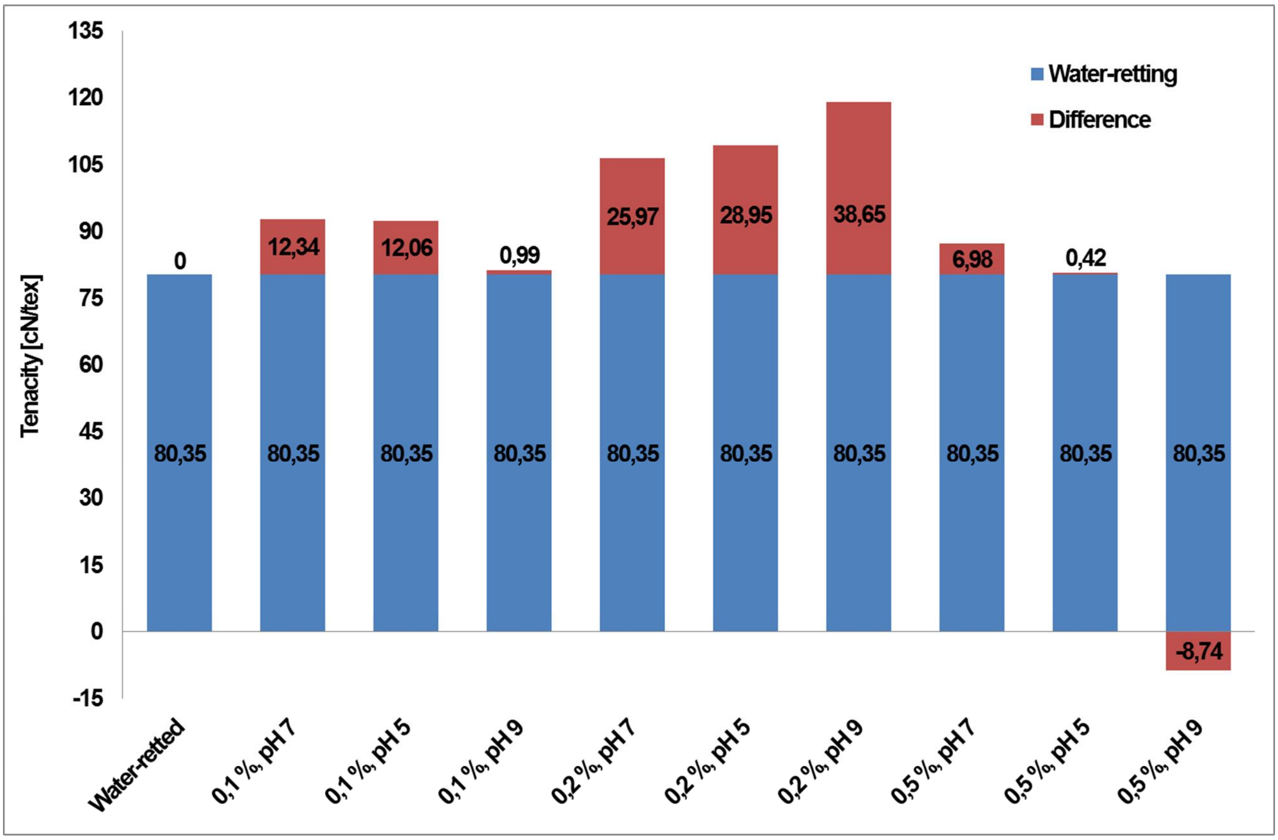

Figure 3. The tenacity of flax fibres after enzymatic retting compared to water-retting

The tenacity in alkali medium is similar to water-retted flax fibres (0.1 % owf), or lower (0.5 % owf) suggesting higher cleaning of wax and fats in alkali. Once again it is observed that the concentration of 0.2 % owf leads to the best results. The tenacity is the highest if 0.2 % owf of enzyme applied regardless of pH in the bath (improved more than 32 %). These fibres are the strongest one what is in accordance with results of the fibre fineness. If the fineness and tenacity results for enzymatic flax retting are both considered and compared to water retting result, it can be seen that the fibres of similar fineness (0.2 % owf) are stronger.

4. Conclusion

Fibre fineness and tenacity are the most important processing properties and determine the quality and suitability of flax fibres as a textile raw material for yarn and fabric manufacturing. Therefore, in this paper the influence of the bio-innovative flax retting to the fibre quality was investigated.

Comparing the properties of enzyme-retted flax fibres the properties of water-retted ones, it can be found that enzymatic retted fibres are coarser, but much stronger. Enzyme-retted flax fibres in the bath having 0.2 % owf Beisol PRO have the similar fineness and significantly higher strength (32-48 % stronger). Therefore, this concentration can be considered as optimal one. Higher and lower concentration result in coarser and weaker fibres, especially in alkaline medium.

The advantages of this enzyme application for flax retting over water-retting are:

1. time savings of 4ï 5 days (retting was done in 1 h),
2. no dependence on the weather conditions,
3. increased yield and fibre consistency,
4. stronger fibres,
5. environmental-friendly process.

The use of enzymes to extract fibres provides an environmentally friendly method toward developing reliable and sustainable agriculture using bio-based fibres of enhance quality. It is also evident that further investigation is needed. Therefore, a long-term objective is to develop bio-innovated flax retting to remove the non-cellulosic compounds from the technical fibres, and at the same time are biodegradable and non-toxic, with no dependence on the weather conditions.

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