ORIGINAL PAPERS

Mechanical properties of flax fibres treated by alkaline, enzyme and steam-heat

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Abstract In recent years there has been interest in using flax fibres to produce composites because of a number of attributes, including low density, biodegradability and high mechanical properties. It was found that treatment of flax fibres may be required to improve the bond quality with a resin. These treatments also have an impact on the properties of the fibres themselves. The objective of this project was to evaluate the impact of three treatment methods on the mechanical properties of flax fibres. The three treatment methods were alkaline, enzyme and steam-heat. After treatment, flax fibres were tested in tension using a universal test machine. Results showed that tensile strength and Young's modulus of flax fibre can be enhanced significantly by the three treatment methods, compared with untreated flax fibres. Enzyme treatment was shown to be the best approach to improve mechanical properties of flax fibre than alkaline and steam-heat treatment.

Keywords Fibres · Strength · Crack · Heat treatment · Alkaline · Enzyme

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Introduction

Flax fibres (*Linum usitatissimum* L.) have received much attention in recent years due to their high mechanical properties, low density and biodegradability, which make them an excellent alternative to glass fibres in many composite applications. As one kind of environment friendly material, flax fibres were used as reinforcement of polymer matrix composites (Bledzki & Gassan, 1999; Bos et al. 2002; Bhatnagar & Sain, 2005; Arbelaiz et al. 2006a; Arbelaiz et al. 2006b; Kalia et al. 2009), especially in parts used by the automobile and packaging industries where high load-carrying capability is not required (Bledzki & Gassan, 1999; Magurno, 1999; Karus & Ortmann, 2005). Research has shown that mechanical properties of flax fibre composites are limited by relatively poor bond quality between the fibre and the matrix polymer (John & Anandjiwala, 2008; John & Anandjiwala, 2009). Surface modification is generally recommended to improve adhesive bond quality of flax fibres, but these treatment methods have also been found to impact on properties of flax fibres (Joffe et al. 2003).

Much research in recent years has focused on the effects of surface modification on mechanical properties of flax fibre reinforced composites (Andersons et al. 2005; Andersons et al. 2009; Alix et al. 2009). Such surface modification can be achieved by various treatment methods, including those utilizing chemicals, enzyme and heat.

Enzyme has been used to modify properties of natural fibres in recent years (Joffe et al. 2003; Andersons et al. 2005; Akin et al. 2001; Fischer et al. 2006; Pickering et al. 2007). Gulati and Sain (2006) reported that composites made with fungus-treated (*Ophiotoma ulmi*) hemp fibres showed slightly better mechanical properties than composites made with untreated fibres due to improved interfacial adhesion between fibre and unsaturated polyester resin. In addition, fungus-treated fibres showed improved acid-base characteristics and resistance to moisture. Li and Pickering (2008) found that hemp fibre strength was reduced after combined chelator and enzyme treatment. This was attributed to the degradation of cellulose after treatment, as was evidenced by the higher crystallinity index. However, the tensile strength of the composite made with treated hemp fibres was higher than that of the composite made with untreated fibres.

Chemical treatment has also been applied to modify properties of natural fibres (Baley et al. 2006; Gulati & Sain, 2006; Alix et al. 2009). Van de Weyenberg et al. (2003) showed that alkaline treatment gave up to 30% increase in longitudinal properties (both strength and modulus) of flax reinforced epoxy composite, due to the removal of pectins. Baley et al. (2006) showed that the apparent interfacial shear strength of alkaline-treated flax fibres was decreased by 56% compared with untreated sample, due to strong chemical degradation of the flax fibre surface.

In addition to enzyme and chemical treatment, thermal treatment has also been studied. These studies showed that this treatment method also has a significant effect on mechanical properties of natural fibres. Van de Velde and Baetens (2001) found a decrease of 40% – 56% in strain at rupture and a 32% – 36% decrease in peak stress of hackled long flax after heat treatment at 180 ℃ for 2 hours.

Generally speaking, much research has focused on the mechanical properties of flax fibre reinforced composites. Few studies have focused on the mechanical property of pure flax fibres treated by different treatment methods, especially on tensile strength and Young's modulus. As a first step in understanding the influence of fibre treatment method on final product, it is considered important to understand how treatment methods affect mechanical properties of flax fibres.

In this study, the influence of three treatment methods, namely alkaline, enzyme and steam-heat, on tensile strength and modulus of flax fibres was investigated.

Materials and methods

Materials

The flax fibres (*Linum usitatissimum* L.) used in this study were supplied by Stemergy of Ontario, Canada. Analytical grade sodium hydroxide pellets (97% purity) were used in the alkaline treatment of the fibres. Enzyme laccase (product No. L2157, from *Rhus vernificera*) with enzymatic activity of 120 u/mg was supplied by Sigma-Aldrich of Saint Louis, USA.

Retting and thermal treatment

Three treatment methods were evaluated in this study. They are alkaline, enzyme and steam-heat. Details of the treatment schedules are given below.

Alkaline treatment

Dried flax fibres of 5 g were immersed in 150 mL of sodium hydroxide solution in a flask at 23, 60 and 100 ℃ each for 1 and 2 hours, respectively. The concentrations of sodium hydroxide solution were 3% and 10% wt/v, respectively. A solution to fibre ratio of 30: 1 (mL/g) was maintained for all experiments. Sodium hydroxide solution with a pH value of 14 was prepared by mixing of sodium hydroxide granule with distilled water. After the alkaline treatment the fibres were rinsed with tap water until the pH value of the solution reached 7. Then the fibres were dried in a 60 ℃ oven overnight. There were three replications for each specimen.

Enzyme treatment

Enzymatic treatment was carried out under the following conditions: pH of 3 and 5, temperature of 23, 36 and 50 ℃, and duration of 1 and 2 hours. The enzyme used was laccase with two dosages: 15 and 30 u/g fibre. A solution to fibre ratio of 30:1 (mL/w) was maintained for all experiments. Dried flax fibres of 5 g were treated in each experiment and there were three replications for each specimen. Fresh air was continiously injected into the mixture of flax fibres and enzymatic solution in a flask by plastic tube during the entire treatment process.

Two concentrations of enzymatic solution with enzymatic activity of 15 and 30 u/mL respectively, were prepared with a mixture of enzymatic powder (enzymatic activity is 120 u/mg) and deionized water. The pH value of 3 and 5 of enzymatic solution was adjusted respectively by glacial acetic acid and sodium hydroxide solution.

Steam-heat treatment

Dried flax fibres of 15 g were treated by steam at the following conditions: temperature of 160, 180, 200 and 220 ℃ and duration of 0.5, 1 and 2 hours in an airtight equipment with an atmosphere containing less than 2 percent oxygen. Super-heated steam was used as a heating medium and a shielding gas during the whole treatment process.

Mechanical testing of flax fibres

The treated and untreated fibres were dried and conditioned at room temperature of 23 ℃ and relative humidity of 20% for 3 days before mechanical tests were conducted. The treated and untreated flax fibres were tested in tension using an Instron 5848 Micro Tester with a 50 N capacity load cell at room temperature. In order to avoid pausing the machine to count fibre cracks, the test was carried out at a low loading rate of 0.5 mm/min. Loading was stopped when the single fires were broken totally. There were 30 replications for each specimen.

An optical method was used to measure the diameter of flax fibres. Three apparent diameter measurements were taken at different locations along the fibre as shown in Fig. 1. The same measure process was repeated for each specimen.

Single fibres were manually separated from the flax bundles. Fibre ends were glued onto a paper frame according to the preparation procedure described in ASTM D3379-75 Standard (1989). Between specimen preparation and testing, the specimens were handled only by the paper frame. Fibre length outside the frame (gauge length) was 25 mm. The elongation of the specimen was measured using to cross head movement. Tensile strength and modulus are calculated according to the standard ASTM D3379-75.

Results and discussions

Alkaline treatment

The results of tensile strength (σ_t) and Young's modulus (E) of untreated and treated flax fibres are shown in Table 1. Alkaline treatment reduces diameter of flax

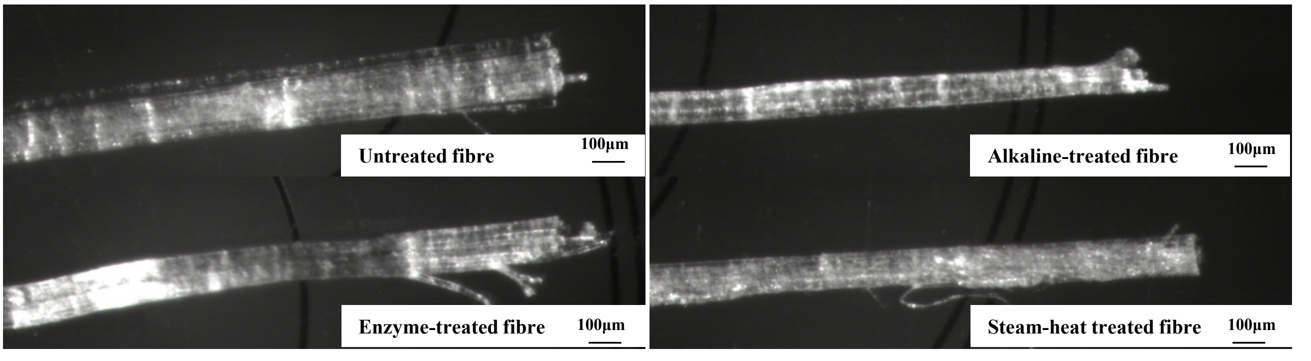

Fig. 1 Diameter measurement of untreated and treated flax fibres observed under optical microscopy

fibres by about 50% on average. Treatment temperature plays an important role during alkaline treatment of flax fibres. Group Al result shows that room temperature alkaline treatment is not effective because both σ_t and E were reduced slightly at even low concentration of chemical (3% NaOH). Increasing the temperature of the treatment to 60℃ at the same concentration improved tensile strength by 19% and 13% at 1 h and 2 h treatment duration respectively. The Young's modulus was also increased by 38% after treatment for 1 h, but decreased slightly after 2 h of treatment (group A3). Overall, with alkaline treatment method, for NaOH concentration higher than 3% and treatment temperature of greater than 60℃, degradation of flax fibres occurred, as was evident by the reduced σ_t and E values in groups A4, A5 and A6. It is apparent from Fig. 2 that the treatment temperature of 60 ℃ is optimum for improving the mechanical strength of flax fibres compared with 23 and 100 ℃. Under the conditions studied, the optimum alkaline treatment schedule was found to be 3% NaOH, 60 ℃ and 1 hour, which improved σ_t and E by 19% and 38% respectively.

Enzyme treatment

Enzyme treatment produced a reduction in fibre diameter of about 6%. The mechanical property results are illustrated in Fig. 3. A general conclusion that can be made from Fig. 3 is that no benefit can be obtained by increasing temperature of treatment above 36 ℃. The results also suggest that there is no significant difference between pH3 and pH5 results. For the eight groups (E5-E12) that were treated at 50 ℃, Young's modulus values are consistently better with the 1 h treatment time than 2 h, irrespectively of pH and dosage. Tensile strength results do not show a clear trend with treatment time. The best result was obtained with the treatment schedule of 23 ℃, 2 h, pH5 and 30 u/g, which produced a 53% and 28% increase in tensile strength and Young's modulus respectively.

Steam-heat treatment

Steam-heat treatment reduced diameter of fibres by about 7% on average. Temperature plays an important role in steam-heat treatment to modify mechanical properties of flax fibres. In this study, four treatment temperatures: 160, 180, 200 and 220 ℃, were evaluated. The results are summarized in Fig. 4. It can be concluded that treatment temperature above 200℃ should be avoided because both tensile strength and Young's modulus were reduced at 220 ℃. Within the temperature range of 160–220 ℃, the optimum treatment duration is 1 hour within the range of parameters studied, the most optimum treatment schedule is 200 ℃ and 1 hour, which provided an improvement of 31% and 50% respectively in tensile strength and Young's modulus.

Comparison of the three treatment methods

All three treatment methods evaluated in this study can improve the tensile strength and Young's modulus of

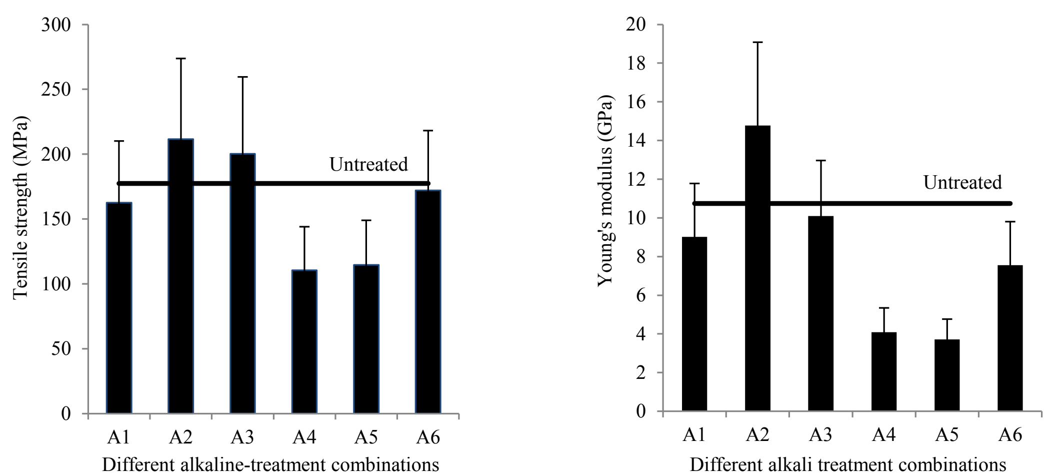

Fig. 2 Mechanical strength of alkaline-treated flax fibres - comparison among different treatment combinations

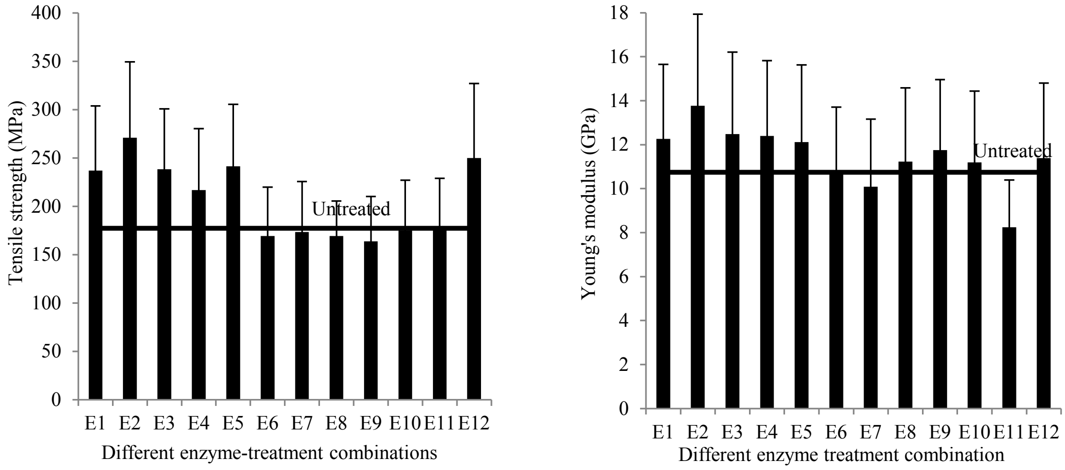

Fig. 3 Mechanical properties of enzyme-treated flax fibres - comparison among different treatment combinations

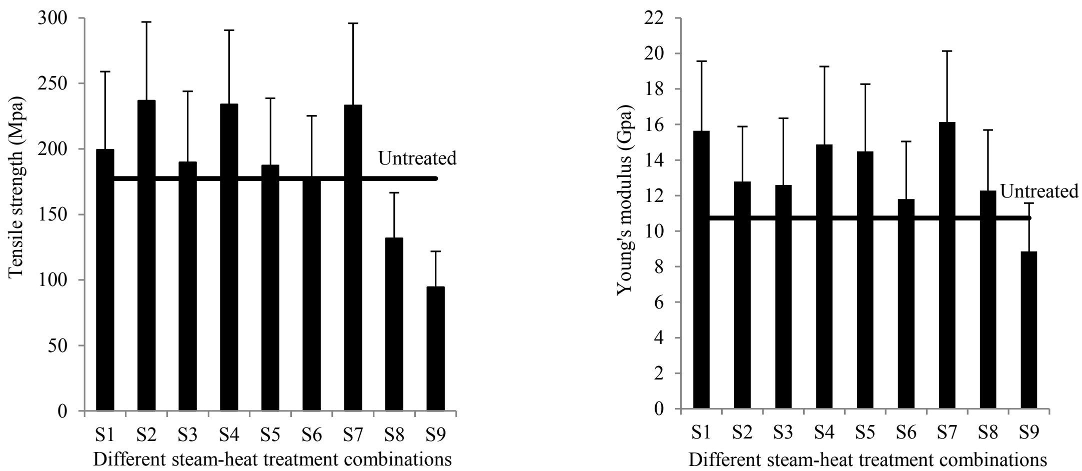

Fig. 4 Mechanical strength of steam-heat treated flax fibres comparison among difference treatment combinations

flax fibres significantly. Compared with untreated flax fibres, under the most optimum condition in each case, the three treatments of alkaline, enzyme and steam-heat improved tensile strength of flax fibres by 19%, 53% and 34% respectively. Correspondingly, Young's modulus was increased by 38%, 28% and 50% respectively. However, these treatment methods have also been found to decrease tensile strength and Young's modulus with certain treatment schedules as shown in Table 1. Based on these results, both enzyme and steam-heat treatment provided the better improvement in mechanical properties. However, steam-heat treatment is energy-intensive and most likely more costly. Therefore, enzyme treatment appears the most promising for fibre treatment purposes.

The tensile strength and Young's modulus determined in this study for flax fibres appear to be lower than those reported in the literature. Baley (2002) summarized relevant studies and reported that the tensile strength of flax fibres was in the range of 600-2000 MPa while Young's modulus was between 12 and 85 GPa. Dejong et al. (1999) explained that the relatively high tensile strength and Young's modulus of elementary flax fibres is imparted by the cellulose microfibrils embedded in the secondary cell wall with a low microfibrillar angle. However, like most natural materials, flax fibres have a high variability of mechanical properties (Bledzki & Gassan, 1999). This is because mechanical properties of natural fibres are affected by not only the growth conditions of the plants, but also subsequent harvesting and

Table 1 Mean values and standard deviation of tensile strength and Young's modulus of flax fibres by difference treatments

Methods	Group	Treatment parameters	Diameter (μm)	Tensile strength (MPa)	Young's modulus (GPa)
Untreated			90.56 ± 21.00	177.34 ± 49.00	10.74 ± 3.12
Alkaline treatment	A1	23 ℃, 2 h, 3%	88.20 ± 18.00	162.52 ± 48.00	9.01 ± 2.77
	A2	60 ℃, 1 h, 3%	83.71 ± 22.00	211.64 ± 62.00	14.77 ± 4.32
	A3	60 ℃, 2 h, 3%	87.00 ± 14.00	200.30 ± 59.00	10.09 ± 2.88
	A4	60 ℃, 1 h, 10%	84.17 ± 15.00	110.49 ± 34.00	4.09 ± 1.26
	A5	60 ℃, 2 h, 10%	85.55 ± 13.00	114.65 ± 34.00	3.72 ± 1.05
	A6	100 ℃, 2 h, 3%	80.00 ± 20.00	172.11 ± 46.00	7.55 ± 2.25
Enzyme treatment	E1	23 ℃, 1 h, pH3, 15 u/g	77.56 ± 20.00	237.06 ± 67.00	12.26 ± 3.39
	E2	23 ℃, 2 h, pH5, 30 u/g	74.42 ± 18.00	271.08 ± 78.00	13.77 ± 4.16
	E3	36 ℃, 2 h, pH3, 30 u/g	89.84 ± 24.00	238.32 ± 62.00	12.48 ± 3.74
	E4	36 ℃, 2 h, pH5, 30 u/g	79.22 ± 17.00	216.97 ± 63.00	12.40 ± 3.42
	E5	50 ℃,1 h, pH3, 15 u/g	81.71 ± 17.00	241.44 ± 64.00	12.11 ± 3.51
	E6	50 ℃, 1 h, pH5, 15 u/g	96.95 ± 24.00	169.33 ± 51.00	10.68 ± 3.03
	E7	50 ℃, 2 h, pH3, 15 u/g	87.17 ± 20.00	173.58 ± 52.00	10.09 ± 3.08
	E8	50 ℃, 2 h, pH5, 15 u/g	79.65 ± 18.00	169.42 ± 36.00	11.24 ± 3.35
	E9	50 ℃, 1 h, pH3, 30 u/g	82.41 ± 20.00	163.72 ± 46.00	11.75 ± 3.21
	E19	50 ℃, 1 h, pH5, 30 u/g	80.45 ± 18.00	175.10 ± 52.00	11.19 ± 3.25
	E11	50 ℃, 2 h, pH3, 30 u/g	88.92 ± 21.00	178.35 ± 51.00	8.25 ± 2.15
	E12	50 ℃, 2 h, pH5, 30 u/g	80.88 ± 18.00	250.05 ± 77.00	11.39 ± 3.42
Steam-heat treatment	S1	160 ℃, 0.5 h	90.05 ± 28.00	199.39 ± 60.00	15.65 ± 3.91
	S2	160 ℃, 2 h	89.71 ± 26.00	236.90 ± 60.00	12.79 ± 3.09
	S3	180 ℃, 0.5 h	87.30 ± 26.00	189.85 ± 54.00	12.60 ± 3.74
	S4	180 ℃, 1 h	78.20 ± 22.00	234.06 ± 57.00	14.88 ± 4.39
	S5	180 ℃, 2 h	74.89 ± 18.00	187.45 ± 51.00	14.49 ± 3.77
	S6	200 ℃, 0.5 h	82.31 ± 21.00	176.35 ± 49.00	11.80 ± 3.24
	S7	200 ℃, 1 h	75.94 ± 22.00	233.18 ± 63.00	16.13 ± 3.99
	S8	200 ℃, 2 h	76.44 ± 15.00	131.74 ± 35.00	12.28 ± 3.40
	S9	220 ℃, 2 h	85.21 ± 22.00	94.47 ± 27.00	8.86 ± 2.71

processing of the plants for extracting the fibres (Bos et al. 2002; Van de Velde & Baetens, 2001). For example a number of researchers found that retting method affects mechanical properties of the produced fibres (Alix et al. 2001; Sharma et al. 1999; Morrison et al. 2000).

Fibre characteristics are also known to affect mechanical properties. Fibre characteristics are in turn dependent on the location in the stem (Charlet et al. 2007). Bledzki and Gassan (1999) found that tensile strength of flax fibres depends on the fibre length. The presence (Baley, 2004) and amount of kink bands (Kamat et al. 2002) in a fibre are shown to affect its strength.

Fibre diameter variation is not the primary mechanism determining the strength distribution of the elementary flax fibres (Spārniņš & Andersons, 2009). However, it was reported that the Young's modulus decreases with fibre diameter (Baley, 2002). This trend was not observed in this study because each Young's modulus was obtained under unique treatment combination.

The test method, including specimen preparation procedure, can also affect the results of tension testing of natural fibres. As yet, there has not been a universally acceptable test method to measure strength and stiffness properties of natural fibres. In most studies, flax fibres were generally assembled into bundles of 10 to 40 fibres rather than tested as a single fibre, with the longitudinal and the transverse dimensions distributed over the range of 4–77 mm and 5–76 μm, respectively (Baley, 2002). Method of measuring dimensions of the fibre bundles also differed between studies. Attachment of the fibres to the test machine also varied between studies.

Overall, while the mechanical properties reported above are lower than those in the literature, this discrepancy should not alter the conclusions from this study regarding suitable treatment method for improving mechanical properties of flax fibres because the obtained results were compared with untreated fibres which were tested using the same test procedure.

Conclusions

The effects of alkaline, enzyme, steam-heat treatment on mechanical properties of flax fibres were investigated in this study. Tensile strength and Young's modulus of flax fibres can be increased by these treatment methods by the appropriate selection of treatment schedule. It should be noted that reduction in mechanical properties is possible with some of the treatment schedule. The maximum increase in tensile strength and Young's modulus was found to be 53% and 28% respectively by enzyme treatment. Enzyme treatment achieves the best improvement in mechanical properties of flax fibres, compared with alkaline and steam-heat treatment, and it will be selected for future research work to treat flax fibres.

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