MINISTRY OF AGRICULTURE FISHERIES AND FOOD

RESEARCH AND DEVELOPMENT FINAL PROJECT REPORT

PROPERTIES OF FIBRE EXTRACTED FROM UNRETTED HEMP AND FIBRE PERFORMANCE IN COMPOSITES

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Section 4 : Executive summary

Background

Retting of hemp stems in field is a part of the normal practice to allow separation of the fibre from the woody core. If fibre of useful quality could be extracted from unretted hemp, the problems caused by the need to ret the stems could be avoided. These problems include microbiological contamination of the fibre, variability of fibre properties, and insecurity of fibre supply owing to poor weather during field retting. They limit the competitiveness and range of application of the fibre. A prototype apparatus to extract fibre from unretted hemp had been developed at Silsoe Research Institute in a previous project but the properties of the fibre and its performance in higher value applications such as composites had not been established. The research reported here aimed to establish the properties and performance in composites of fibre extracted from unretted hemp stems, so as to open the way to the exploitation of this UK-grown crop.

Objectives

1. Determine physical and mechanical properties of fibre bundles extracted from unretted hemp and compare with bundles from the same hemp material but retted.
2. Characterise the adhesion between an appropriate matrix material and the retted and the unretted fibre.
3. Make small panels of composites from the promising combinations of materials identified in objective 2, and determine their performance.
4. Investigate the properties of early harvested hemp for comparison with hemp harvested at the mature stage.

Method

Hemp at normal maturity from the 1997 harvest was used for the initial work. Stems of hemp from commercial trial plots were collected from 1999 harvest at intervals of one week and dried without retting. This gave two samples younger than the normal harvest maturity, one at normal maturity and one a week post mature. For the retting treatment, stems were submerged in water previously used for retting, at 25°C for 5d at which time they were judged correctly retted. Fibre was extracted using the modified decorticator. Normal procedure was to pass the stems through the machine to produce tangled fibre, but an alternative method which we shall call ‘pinning’ was also used, in which the stems were put part-way into the machine and then withdrawn, to fibrillate the worked end of the stems and thus produce well-aligned fibre. Fibre was characterised by measuring dimensions, stiffness and strength of individual strands, and fineness by air permeability. Fibre was cut into lengths of 60 mm, so single pieces could not reach from one end of the test piece to the other. Composite panels were made by adding epoxy adhesive to this aligned fibre, randomly arranged lengthwise in a mould. Test pieces were cut from the panels, strain gauges were attached and stiffness and strength measured by tensile tests. Fibre volume fraction was measured after testing by measuring the area ratio of fibre to resin by image analysis on a polished cross-section.

Results

- Separation of fibre from unretted hemp stems in quantities of a few kg was straightforward, both from dry stems stored for eighteen months and from stems harvested and dried shortly before processing.
- Unretted stems yielded 30% of their mass in the fibre fraction and 59% shiv compared with 31% fibre and 65% shiv for retted stems. The remainder was lost as dust and small particles.
- Fibre bundles extracted by decortication from unretted and from retted hemp were not significantly different in strength or stiffness. Fibre extracted by pinning was coarser than decorticated and gave higher strength and stiffness. Fibre from retted stems was finer than from unretted, as would be expected.
- Composite samples from unretted and from retted mature fibre produced by decortication gave equally good tensile stiffness, typically 5.5 GPa, and strength, typically 50 MPa. Composites from fibre produced by pinning gave better stiffness, typically 7.5 GPa, and strength, typically 85 MPa, (see Figs 1 & 2).
- Composite stiffness reduced with stem maturity but showed similar strength.
- Breakdown of the interfaces between the epidermis and the fibre bundles appears to have an effect on composite properties by reducing the stress transfer from the resin to the fibre bundles.

Conclusions

1. Fibre can be extracted from unretted hemp stems and used to make composite materials that are as stiff and strong as those made with retted fibre. The percentage yield of fibre was found to be similar for unretted stems as for retted stems.
2. Further improvement in composite properties from unretted fibre could be made by removal of more of the waxy epidermis from the stems during fibre extraction.

Section 5 : Scientific report

Introduction

Retting is controlled degradation of plant stems to allow the fibre to be separated from the woody core. Carried out to the appropriate degree it gives fibre with little woody material attached. The only economic method currently available is to expose the stems to weathering in field for 3-6 weeks after cutting during which micro-organisms grow on the stem and digest elements of it. This retting process is a problem in several respects for growers and potential users. These are the arguments for avoiding retting:-

- The absence of micro-organisms from the fibre and the core particles of unretted fibre make both more attractive to potential users because of the reduced health risk.
- Retting is a serious problem for growers. Retting is a source of variability in the product, and variability is an aspect of quality where natural fibres have problems competing with synthetics.
- The crop may not dry adequately in the often poor weather at the end of the season, well demonstrated in 1998 harvest, so that spoilage of the fibre may occur in field or in store. This adds costs and supply uncertainties.
- The need to ret for 6-8 weeks holds up cultivation and hence reduces returns from the following crop, costs which must be borne by the fibre crop.

Extraction of fibre from unretted stems has been achieved previously but only at the expense of yield of fibre being severely reduced compared with retted stems, and mechanical damage being increased, resulting in shorter fibres with weaknesses owing to damage. Hammer milling is very crude and gives fibre only suitable for pulping (1). Stems have been artificially dried to <5% and subjected to multiple passes in a system of rollers. This has been shown to be effective for flax by Univ of Bonn Institut fur Landtechnik (Personal communication) but is expensive, causes too much fibre damage to be acceptable and some loss of yield. Other approaches include steam explosion and ultrasonic treatments (1, 2, 3), which both separate the material into its component fibre cells and hence destroy the structure of fibre bundles that the plant has provided. Silsoe Research Institute have made an advance, not yet published, by showing that their decorticator system is able to separate hemp fibre from unretted stems.

Aim and objectives

The aim of the study was to determine how well the properties of composite materials made with fibre from unretted hemp compare with properties of retted fibre composites.

The objectives were

1. Determine physical and mechanical properties of fibre bundles extracted from unretted hemp and compare with bundles from the same hemp material but retted.
2. Characterise the adhesion between an appropriate matrix material and the retted and the unretted fibre.
3. Make small panels of composites from the promising combinations of materials identified in objective 2, and determine their performance.
4. Investigate the properties of early harvested hemp for comparison with hemp harvested at the mature stage.

Materials and methods

Hemp stems

Hemp stems grown in 1997 in Essex had been cut from a commercial crop of Felina 34 on 15 September using a Busatis double reciprocating mower. They were left in a swathe to field dry for 5 days under ideal drying conditions and then bundled to finish drying and stored under cover in a well-ventilated building. Stems from 1999 harvest again from Essex were cut by secateurs. 200 stems were cut at each of four different harvest dates, from trial plots, and laid out to dry under cover. The harvests were taken at weekly intervals over a 4 week period with the third harvest coinciding with the recommended stage for the commencement of the commercial harvest. The harvest dates were 11, 17, 23 and 31 August 1999.

Retting

Some stems were tank retted for 5 days in a controlled environment; a vat of continuously circulated liquor kept at a temperature of 25°C and inoculated using water that had been used previously for retting.

Fibre extraction

Fibre was removed from the stems using a decortication machine that has been developed at SRI (4). Two removal methods were used. One was the standard treatment where the fibre passed though the machine in the normal way. For the other treatment stems were fed part way into the machine and then reversed back out. This results in the ends of the stems being crushed and the fibre separated, however a significant proportion of the fibre remains attached to the stem. This fibre is therefore better aligned.

Mechanical testing of fibre bundles

The mechanical properties of fibre bundles from the different treatments was investigated in the following way. Bundles of fibre of varying thickness and at least 10 cm long were selected from the different batches of fibre, 10 from each batch. A range of fibres from the thickest to the thinnest were chosen from each batch. These were glued for support into cardboard windows that were clamped into the tensile test machine and the side bars cut. The fibre was tested in tension and the maximum force before failure was recorded. The specimens were then cut out of the windows and weighed using a balance accurate to 1/100 000 g. Specimens were compared using the ratio of force to mass. The tensile stiffness of unretted strips of fibre-containing tissue, removed by hand from the stems of 1999 harvest, was also investigated. Strips from plants harvested at earliest and latest dates were compared, 10 strips from 2 stems for each harvest date. Strips of tissue were peeled from the outside of the stems. The angle between peel and stem was kept as small as possible to avoid damage to the tissue. Strips were then mechanically tested as described above, except that load deflection curves were recorded, along with the width and thickness of the tissue. From this data, stress-strain curves were constructed and the tensile stiffness was determined.

Composite forming

A low viscosity epoxy system, which incorporates a slow hardener, was used to make composites (S.P. Systems Ampreg 20). The resin and hardener were thoroughly mixed in a ratio of 4 parts resin to 1 part hardener. The decorticated fibre was orientated by hand combing repeatedly in a single direction. The stripped fibre was already well aligned. The aligned fibres were then cut into 6 cm lengths and arranged into moulds in an overlapping configuration. Epoxy resin was poured over the fibre and left for 10 minutes to soak into the fibre mat. Trapped air was gently squeezed out of the mat using a wide flat-ended metal probe to reduce possible damage. The fibre and resin was then left for 30 minutes to allow air bubbles to escape from the surface of the resin. Finally a steel top was placed on the mould. The volume fraction of fibre in the composite was controlled by adding known weights of fibre and resin. The composite was left to cure for 48 hours before being removed from the mould. Post curing was continued for a further hour in an oven at 60°C.

Composite testing

The cured composites were cut into dumbbell shaped specimens, fitted with strain gauges and stretched in a Davenport Nene DN10 testing machine. Tensile stiffness and strength were recorded. Transverse sections of untested composites were cut using a fine toothed band saw and then finely polished using 40 :m grit sandpaper. The sections were mounted under a light microscope with a camera, which was connected to a PC. The interface between the fibres and the resin was examined visually and the volume fraction of fibre was accurately measured using an image analysis package.

Results

Yield

The mean mass of the fibre and shiv fractions for two samples of retted and two of unretted is shown. Each samples was approximately 4 kg of stems. The waste fraction is calculated, and represents the fine particles and dust. The yield of fibre was not significantly different. More waste and less shiv were produced from the unretted stems.

% Fibre (s.d.)	% Shiv	% Waste	
Retted stems	31.2 (0.6)	64.7	4.1
Unretted stems	30.5 (0.9)	58.9	10.6

Fibre bundle properties

After decortication, unretted fibre is coarser than retted fibre and consists largely of thin strips of fibre tissue, each containing several bundles. Fibre from retted hemp comprises a greater proportion of individual bundles. Neither treatment separates the bundles into individual cells to any significant extent.

The fibre bundles from the different treatments had the same values of force/weight, except for fibre bundles from the unretted pinned treatment which had significantly higher values (ANOVA with 5 % significance level). Variation within each treatment was large.

Unretted pinned	Unretted decorticated		Retted pinned	Retted decorticated	
Mean N/g	5310	3768		3343	3681
s.d.	1230	1396		1507	1835

The stiffness of the unretted, hand extracted fibre tissue did not change significantly with harvest time as shown below.

1st harvest	4th harvest		
Mean stiffness (GPa)		21.2	23.2
s.d.		2.9	3.9

The results of fibre fineness for the different treatments and harvest dates are shown below. The standard decortication produced finer fibre than the pinning method and retting resulted in finer fibre than that from unretted tissue. Fineness decreased slightly from first to last harvest.

Sample			Mean airflow	CV	fineness
[l/min] [%]	[dtex]				
Number	Treatment	Extraction method			
1	retted	Standard decortication	14.88	5.95	16.6
2	retted	Pinned	17.90	5.6	19.2
3	unretted	Standard decortication	19.75	5.87	20.8
4	unretted	Pinned	24.03	6.95	24.5
5	unretted	Pinned 1st harvest	21.15	6.56	22.0
6	unretted	Pinned 2nd harvest	21.63	3.77	22.5
7	unretted	Pinned 3rd harvest	24.42	8.88	24.9
8	unretted	Pinned 4th harvest	25.30	7.39	25.6

Composite properties

The unretted fibre is coarser than the retted fibre and is composed largely of narrow strips of fibre tissue with some cuticle still adhering to the outside. These strips of tissue contain several fibre bundles. Retted fibre on the other hand is composed largely of individual fibre bundles, with some individual fibre cells and there is very little cuticle left.

Fig 1a, b (see attached) show the stiffness and strength of composites made with fibre from a previous years harvest. Analysis of variance was used to compare the different types of fibre and fibre removal methods. There were no significant differences in either stiffness or strength between composites made from retted fibre and composites made from unretted fibre. However there were significant differences between the two decortication treatments ($P<0.05$). The standard decortication treatment results in composites with lower stiffness and strength than composites made with fibre from the pinning-decortication method. Photographs of the fibre after decortication and re-alignment show that there are significant differences in fibre orientation, between the two types of fibre from the different extraction procedures. In the pinned-decorticated fibre, all the fibres are closely aligned. In the standard decorticated fibre, the larger fibre bundles are well aligned but the smaller bundles and individual fibres are twisted and bent out of alignment. These differences are preserved in the final composites.

Fig 2a, b (see attached) show the stiffness and strength of composites made with fibre from this years harvest. The stiffness of the composites decreases with age from the first harvest at 93 days after drilling to the last harvest at 113 days after drilling. This trend is significant (Regression analysis, $R^2 = 0.5$, ANOVA $P = 0.001$). The strength data is more variable and there were no significant trends with age of harvest. Photographs of cross sections of composite show clear differences between first and last harvest. In early harvest composites the tissue strips appear to be intact. However in later harvest composites gaps appear between the bundles and the epidermis. These gaps are not filled by the resin.

Discussion

Fibre properties

Effect of extraction method. The unretted, pinned fibre was stronger and coarser than that produced by the other treatments because the treatment left the fibre bundles within the tissue strips relatively undamaged and well stuck together. Standard decortication treatment was more vigorous, so that the fibre-containing tissue became more finely divided and more damaged.

Effect of retting. Retting reduced strength of bundles by weakening the interfaces between the fibres within the bundles. Retted fibre extraction by either method produced more separation of the fibres, thus increasing fineness.

Effect of harvest date. Harvest date did not significantly affect the stiffness of hand-extracted, fibre-containing tissue showing that an observed reduction in composite stiffness was not due to a change in stiffness of the fibre. The fineness of fibre decreased slightly with harvest date. This could be the result of increasing lignification of the junctions between fibres with age.

Composite properties

Effect of retting. Absence of retting does not significantly change the reinforcing capabilities of hemp fibre. This is because in unretted tissue the fibre cells and bundles are well stuck together and strips of tissue act as effective fibres. Retting breaks down the interfaces between bundles, however the decortication process then separates these bundles. Thus in retted decorticated tissue the effective fibres are bundles and individual cells, rather than strips of tissue. However it is possible that the unretted fibre tissue may degrade over a time scale of months or years, after it has been embedded in resin. This could seriously affect the useful life of the composites and requires further investigation.

Effect of harvest date. Harvest date of hemp stems has a significant effect on the stiffness of composites that can be manufactured from the fibre. Early harvesting seems to be beneficial, resulting in stiffer composites. This is because the fibre bundles in the tissue are well stuck to the epidermis and tissue strips work as a single large fibre. As stems mature the interfaces between fibre bundles and the epidermis begin to break down, but not enough to enable the decortication process to efficiently remove all the epidermis. Significant amounts of epidermis remain loosely attached to the underlying tissue. Resin cannot penetrate into the gaps between the epidermis and fibre tissue and therefore stress is not transferred to the bundles very effectively. Thus the larger strips of tissue, containing a number of bundles provide less effective reinforcement. However enough stress is still transferred to the bundles to cause them to break at composite failure (which may be initiated by failure in the resin matrix). Thus the harvest date of the fibre does not have a significant effect on the tensile strength of the composites. Previous work had indicated that the adhesion between the stem cuticle and resin may be poor. However this work indicates that it is poor adhesion between the epidermis and fibre tissue that can cause weakening of the composites. This problem would be particularly severe in retted tissue if the cuticle was not removed during the processing. This is one advantage of the decortication extraction in that quite a lot of the cuticle is removed in unretted tissue and almost all the cuticle in retted tissue. The disadvantage of course is that the fibre is damaged by the process. The breakdown between the epidermis and fibre tissue appears to begin even before the stems have been harvested and may well continue after the fibre has been embedded in resin.

Effect of fibre extraction method. There was a significant difference in the properties of composites made using fibre from standard decortication and fibre from pinning-decortication. The standard decortication produced composites that were less stiff and weaker. This can be explained by the reduced alignment of fibre from the standard decortication. The finer fibres become tangled into small knots and tangles, which are very difficult to straighten. The pinning process on the other hand does not tangle the fibres and therefore stiffer, stronger composites can be made.

Conclusions

1. Fibre can be extracted from unretted hemp stems by standard decortication and by a partial process we have called pinning. The percentage yield of fibre from stems was 31% for both unretted and for retted.
2. Unretted fibre made composite materials that were as stiff and strong as those made with retted fibre.
3. Increased maturity of hemp at harvest did not affect composite strength, and reduced stiffness by ~ 10 % per week.
4. Further improvement in composite properties from unretted fibre could be made by removal of more of the epidermal layer from the stems during fibre extraction.

References

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Fig 1 a. Stiffness of composites made from different fibre samples

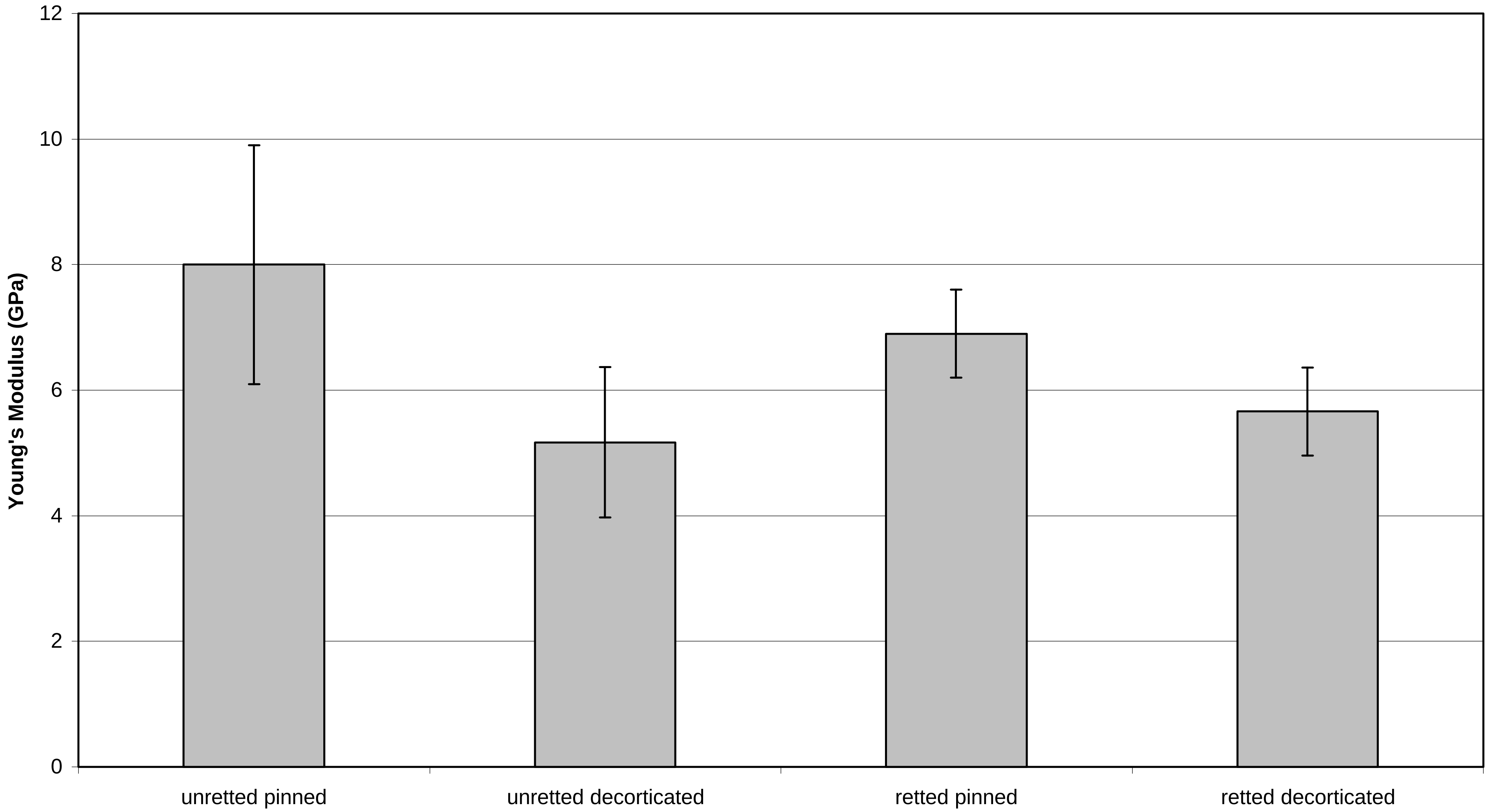

Fig 1b. Strength of composites made from different fibre samples

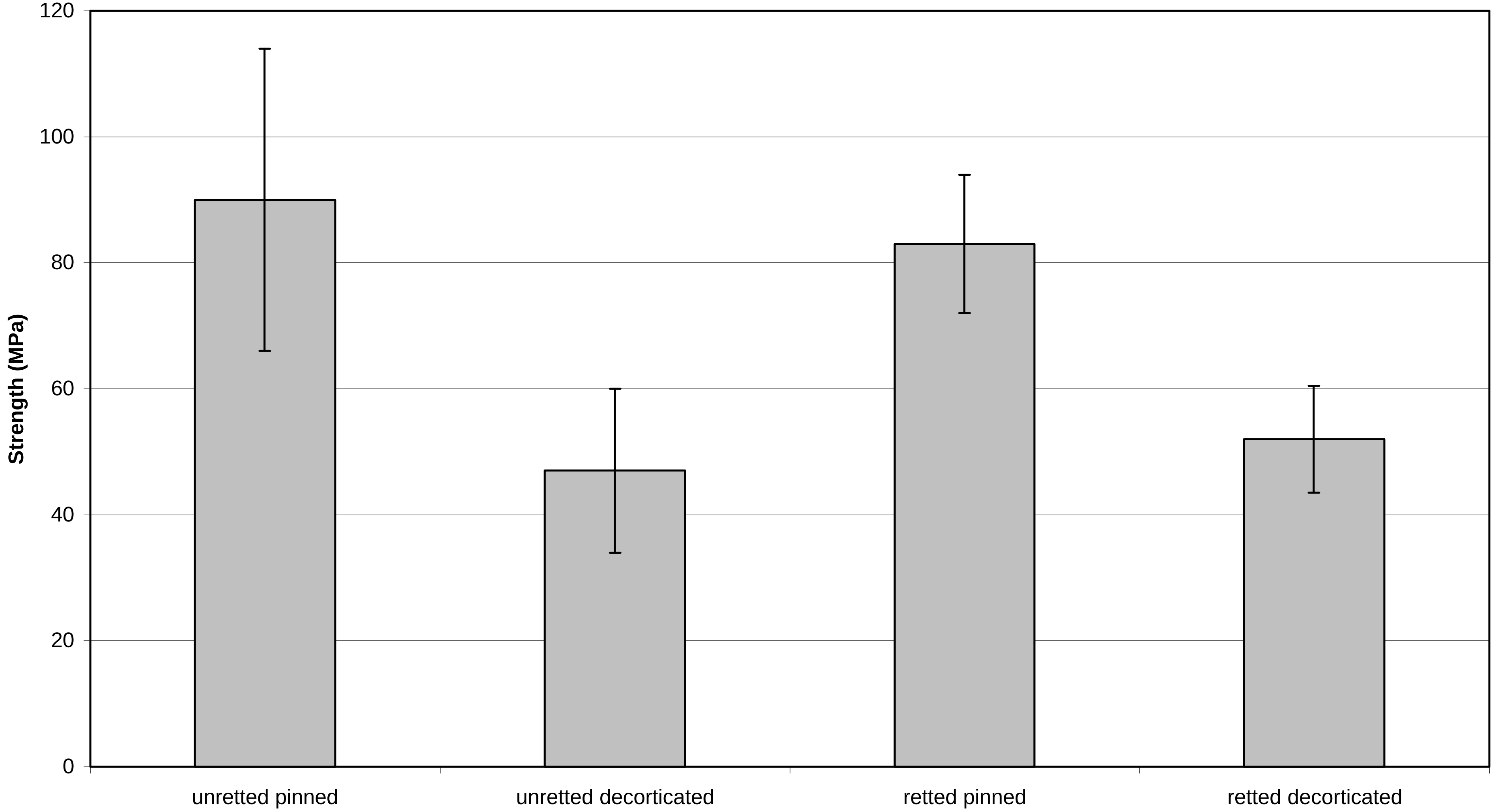

Fig 2a. Stiffness of composites with harvest time of plants (from drilling)

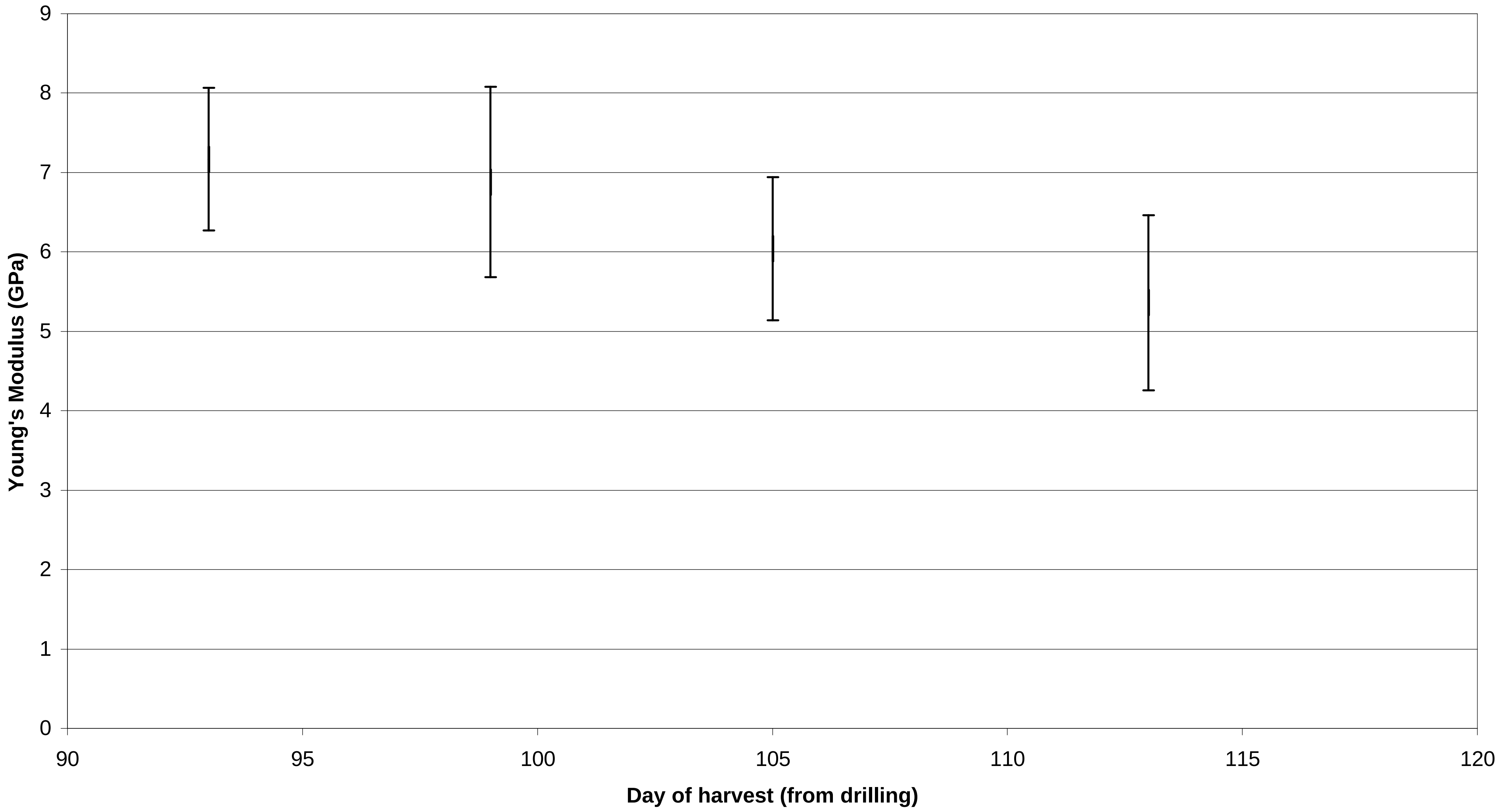

Fig 2b. Strength of composites with harvest time of plants (days from drilling)