Selected mechanical properties of sisal aggregates (Agava sisalana)

D. J. CARR[∗](#page-0-0), N. M. CRUTHERS, R. M. LAING Clothing and Textile Sciences, University of Otago, PO Box 56, Dunedin, New Zealand E-mail: d.carr@otago.ac.nz

B. E. NIVEN

Centre for the Application of Statistics and Mathematics, University of Otago, PO Box 56, Dunedin, New Zealand

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The effects of gauge length and test speed on the tenacity and strain of sisal aggregates (Agava sisalana) were determined. Tenacity was generally higher with shorter gauge lengths and slower test speeds. Strain was higher with shorter gauge lengths. However, the effect of test speed on both tenacity and strain was not the same at every gauge length. The mode of fibre failure appeared to differ with the test conditions. Longer specimens and those tested at faster speeds were prone to higher levels of longitudinal splitting. The effects of different ageing regimes on tenacity and strain of sisal aggregates were also determined. Tenacity was affected by elevated temperature and UV; strain by UV and simulated natural ageing.

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1. Introduction

True sisal is obtained from *Agava sisalana*, a monocotyledon plant endemic to Central America [\[1,](#page-4-0) [2\]](#page-4-1). The sclerenchyma ultimate fibres (single cells) of *A. sisalana* are long (1–5 mm long, \sim 20 μ m in 'diameter'), with thick secondary walls, and occur in bundles with ultimate fibre ends overlapping [\[1–](#page-4-0)[3\]](#page-4-2). It is this *sclerenchyma fibre bundle* or *aggregate* that is produced commercially and is often referred to as *the fibre* in studies, in this work the phrase *sisal aggregate* will be used. Sisal aggregates are off-white in colour, between 600–1500 mm in length, and 100–400 μ m in 'diameter' [\[1,](#page-4-0) [2,](#page-4-1) [4\]](#page-4-3), however, it has been reported that the cross-section of sisal aggregates varies with species [\[5\]](#page-4-4). The chemical composition reportedly varies according to age (cellulose 43–88%, lignin ∼9%, hemicellulose $~10\%$ [\[2\]](#page-4-1)).

Commercial interest in sisal has increased since the 1880s, with cultivation predominately in Florida, the Bahamas, and later in Tanzania [\[2,](#page-4-1) [6\]](#page-4-5). During 1998–2000, 0.386 million tonnes of sisal were produced per year, 1.50% of the total natural fibre production [\[7\]](#page-4-6). Interest in natural cellulose fibres, such as sisal, for use in thermoplastic reinforced composites has increased since the late 19[8](#page-4-7)0s [e.g., $8-11$]. The structure and properties of sisal fibres and aggregates has been reported [e.g., [12,](#page-4-9)

[13\]](#page-4-10). Some evidence of changing failure mechanisms with different test speeds has been observed [\[4\]](#page-4-3). Degradation of tensile strength after various treatments has also been reported e.g. after exposure in glacial acetic acid [\[14\]](#page-4-11), elevated temperatures [\[13\]](#page-4-10), and acidic and alkaline solutions [\[15\]](#page-4-12). Because the different test parameters used in these studies varied (gauge lengths, test speeds, environmental conditions), comparisons among published tensile data are difficult. Test data is generally expressed as stress (Pa) (the assumption being the fibre aggregates are circular in cross-section), or as load at failure (N) in published work.

The aim of the current work was to determine the influence of test methods and conditions (gauge length, test speed, and artificial ageing) have on the tenacity and strain of sisal aggregates.

2. Methods

Specimen pre-conditioning (minimum of 24 h) and all measurements were undertaken in environmental conditions of 20 ± 2 °C and $65\pm2\%$ R.H. [\[16\]](#page-4-13). The linear density of each sisal aggregate was measured.

Tensile properties of sisal aggregates were measured using a bench-mounted 4464 Instron fitted with a 100 N load cell and smooth-faced, mechanically operated grips.

[∗]Author to whom all correspondence should be addressed.

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Specimens were pre-loaded (0.82 g clipped to the bottom of the specimen) before the lower jaw was tightened. The load-extension response of the specimen was recorded and the tenacity (N/tex) and strain $(\%)$ calculated. The effect of gauge length (50, 100, 200, 500 mm) and crosshead speed (10, 100, 1000 mm/ min) on the tenacity and strain of sisal aggregates was determined. For each of the 12 gauge length/test speed combinations, blocks of 15 specimens were tested in a randomised order $(n = 30$ for each combination, $n = 24$ blocks). Polyamide monofilament was tested as a reference material before and after each test block to determine if any differences occurred during testing attributable to the machine, environment, or operator.

The effect of artificial ageing on tenacity and strain of sisal aggregates was also determined. Specimens were exposed to one of three artificial ageing regimes and tenacity and strain data compared to that for not-aged specimens (*n* $= 30$, gauge length $= 200$ mm, test speed $= 10$ mm/min). The regimes were:

1. Thermal ageing. Specimens were exposed to 90 \pm 0.5 \degree C and 60 \pm 5% R.H. with all light excluded for 102, 204, 306, 408, or 504 h.

2. UV ageing. Specimens were exposed in a UV cabinet under environmental conditions of 65±2% R.H. and 20±2◦C for 132, 267, or 364 h.

3. Simulated natural exposure. Specimens were placed on soil, watered once a day $(250\times10^{-6} \text{ m}^3)$ tap water) and exposed in a glasshouse to 24-hour light (natural and simulated daylight) for 168, 336, 504, or 672 h.

Mean, standard deviation (s.d.) and coefficient of variation (CV) data were calculated. The effects of gauge length, test speed, and ageing regime on the tenacity and strain of sisal aggregates were determined using univariate analysis of variance (ANOVA) [\[17\]](#page-4-14). Differences in the rate of change of tenacity and strain over time attributable to the ageing regime were determined using parallel regression [\[18\]](#page-4-15). Tukey's multiple comparison tests were used to identify where differences among means and slopes occurred

Figure 1 Typical cross-section of sisal aggregate $\times 300$.

at the 5% level of significance [\[19\]](#page-4-16). For all analyses, the assumptions of homogeneity of variance and normality of the residuals were confirmed.

Selected sisal aggregates were examined after failure using a Cambridge 360 scanning electron microscope (SEM). Specimens were mounted on 10 mm aluminium stubs with carbon tape, sputter coated with gold, and observed using an accelerating voltage of 10 kV and a filament current of 2.49 A.

3. Results

No significant difference in tenacity or strain was found for the polyamide monofilament reference material.

The mean linear density of the sisal aggregates was 33 tex $(n = 810, s.d. = 10$ tex). The cross-section of the ultimate fibres in the sisal aggregates was typically irregular in shape (Fig. [1\)](#page-1-0).

The effects of gauge length and test speed on the tenacity and strain of sisal aggregates is summarised in Table [I.](#page-1-1) While the mean data suggested several trends, overlapping standard deviations meant advanced statistical meth-

TABLE I Effect of test conditions on tenacity and ultimate strain of sisal aggregates (*n*=360)

Gauge length (mm)	Test speed (mm/min)	Tenacity			Ultimate strain		
		Mean $(N$ /tex $)$	s.d $(N$ /tex $)$	$CV(\%)$	Mean $(\%)$	s.d. $(\%)$	CV(%)
50	10	0.45	0.13	30.00	5.1	1.1	22.20
	100	0.46	0.12	26.42	5.09	0.92	18.02
	1000	0.46	0.17	36.92	5.4	1.2	22.19
100	10	0.60	0.31	51.43	4.08	0.68	18.80
	100	0.50	0.12	24.90	3.96	0.75	18.93
	1000	0.31	0.12	38.23	3.58	0.98	27.39
200	10	0.39	0.12	29.74	2.78	0.53	19.07
	100	0.35	0.19	53.56	2.73	0.85	31.01
	1000	0.39	0.12	30.69	2.99	0.43	14.50
500	10	0.297	0.082	27.61	2.24	0.46	20.38
	100	0.304	0.086	28.29	2.39	0.57	23.82
	1000	0.30	0.11	37.17	2.22	0.53	24.08

ods were essential to determine the significance of differences in tenacity and strain attributable to gauge length and test speed. Tenacity was higher at short gauge lengths and at slow test speeds ($F_{3,348} = 23.331, p \le 0.001; F_{2,348}$) $= 6.367, p \le 0.005$, but the effect of test speed was not the same at each gauge length ($F_{6,348} = 7.708$, $p \le 0.001$). Strain was higher at short gauge lengths, but was not affected by test speed ($F_{3,348} = 236.336, p \le 0.001; F_{2,348} =$ 0.007, $p \leq NS$). The effect of test speed on strain was not the same at every gauge length ($F_{6,348} = 2.143$, $p \le 0.05$).

The mode of failure appeared to vary with the test parameters considered. At smaller gauge lengths (50– 100 mm) and faster speeds (1000 mm/min), longitudinal splitting was observed (Fig. $2a$). At smaller gauge lengths (50–100 mm) and slower speeds (10 mm/min), minimal fibre pullout was evident (Fig. [2b\)](#page-2-0). At test speeds between these two extremes, mixed modes of failure were observed (Fig. [2c\)](#page-2-0). Longitudinal splitting was prominent at longer gauge lengths, irrespective of test speed.

Results from the ageing study are presented in Table [II](#page-3-0) and Figs [3](#page-3-1) and [4.](#page-4-17) Tenacity was significantly lower after thermal ageing $(F_{5,174} = 3.447, p \le 0.001)$, however, exposure at 90 \degree C for 504 h did not affect strain ($F_{5,174}$ = 1.965, $p = NS$). More longitudinal splitting between ultimate fibres was observed in specimens that had been subjected to greater exposure. Lower tenacity and strain were observed as exposure time to UV light increased $(F_{3,116} =$ 39.576, $p \le 0.001$; $F_{3,116} = 24.445$, $p \le 0.001$) with more longitudinal splitting between fibres observed in specimens subjected to longer exposure times. The tenacity of sisal aggregates was not significantly affected by natural degradation ($F_{4,145} = 1.846$, $p = NS$). Strain was lower after greater exposure times ($F_{4,145} = 12.046, p \le 0.001$). No identifiable differences in failure mode were observed between not-aged and naturally aged sisal aggregates.

The ageing regime also affected the rate of change of tenacity and strain for sisal aggregates $(F_{2,444} = 15.88,$ $p \le 0.001$; $F_{2,444} = 9.50$, $p \le 0.001$) (Figs [3](#page-3-1) and [4\)](#page-4-17). For tenacity results from UV ageing differed from those of thermal and simulated natural ageing, and for strain rates for thermal ageing differed from those for UV and simulated natural ageing.

4. Discussion

The tenacity of sisal cells (ultimate fibres) has been reported as 47 gf/tex (0.46 N/tex), and that of sisal filaments (aggregates) as 42 gf/tex (0.41 N/tex) [\[12\]](#page-4-9). These values are comparable to the tenacity measured for sisal aggregates in the current work (0.30–0.46 N/tex). Previous work reported higher tensile strength with shorter gauge lengths for one test speed (15–65 mm, 10 mm/min) [\[4\]](#page-4-3). The current work supports and extends this finding (50–500 mm, 10–1000 mm/min). A strain rate effect was observed for tenacity, i.e. tenacity was generally higher at slow test speeds (10–1000 mm/min). Previous work reported higher tensile strengths with increasing test

Figure 2 Failure of sisal aggregates. (a) Short gauge length, fast test speed 150×. (b) Short gauge length, slow test speed 150×. (c) Mixed mode failure $300 \times$.

speed (1–50 mm/min), and then a lower tensile strength at 500 mm/min [\[4\]](#page-4-3).

Elongation measurements for sisal previously reported include 3.16–8.15% [\[4\]](#page-4-3), 2–3% [\[13\]](#page-4-10), and 3–9% [\[2\]](#page-4-1). These can be compared favourably to the mean values for strain obtained in this study (2.22–5.44%). Differences in

Figure 3 Effect of ageing regimes on tenacity (a: thermal, b: UV, c: natural).

measured strain may reflect differences in processing or in the age of plants from which the aggregates had been extracted. That higher strain was generally measured at shorter gauge lengths and that test speed did not affect strain of sisal aggregates is in agreement with and extends previous work [\[4\]](#page-4-3).

Depolymerisation of cellulose is known to lead to a reduction in tensile properties [\[20\]](#page-4-18), and the results of the present study are consistent with this, however, the three ageing regimes included in this work affected the mechanical properties of sisal aggregates differently. The tenacity of thermally aged sisal aggregates decreased with exposure time, although strain was not affected. Changes in crystallinity probably account for the differences in tensile properties. Cellulose is relatively stable at moderate temperatures over short periods of time, however, breakage of hydrogen bonds, loss of water and crystallinity changes lead to a reduction in mechanical properties [\[20\]](#page-4-18). Heat treatment at 150◦C (for 4 h) reportedly increased the crystallinity of sisal aggregates [\[21\]](#page-4-19).

Sisal aggregates were particularly susceptible to degradation by UV light, both tenacity and strain decreasing with exposure time. Photolysis (caused by UV light) leads to direct rupture of glucosidic bonds, resulting in depolymerisation and loss of tensile properties [\[20\]](#page-4-18), as observed in the current work.

The strain of sisal aggregates which had been exposed to simulated natural ageing decreased with increasing time, although tenacity was not affected. Simulated natural ageing combined several potential degradation mechanisms and their interactions, e.g., pH of the soil, elevated temperature, water, UV and sunlight. Glucosidic bond scission did not apparently occur to a level that significantly affected tenacity. Reduction in strain might be attributable to the greater moisture content of the aggregates leading to plasticisation of the hemi-cellulose/lignin matrix.

The rate of deterioration of tenacity was greater when sisal aggregates were exposed to UV than thermal or natural ageing. For strain, the rate of degradation was greater

Figure 4 Effect of ageing regimes on strain (a: thermal, b: UV, c: natural).

for UV and simulated natural ageing than thermal ageing. The different response between laboratory environments (thermal and UV) and a simulated natural environment demonstrated the need for careful consideration in choosing a degradation environment to match the objective of a study.

5. Conclusions

There are two conclusions from this work:

1. The test conditions investigated (gauge length, strain rate, ageing environment) affected tenacity, strain, and apparent mode of failure of sisal aggregates. This finding has implications for the manufacture of items containing sisal aggregates e.g. composites, non-wovens.

2. Care is required in selecting ageing environments for degradation studies to ensure that the conditions match the objective. This work on sisal aggregates suggested that for tenacity thermal ageing more closely matched simulated natural ageing whilst for strain exposure to UV more closely simulated natural ageing.

Acknowledegments

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References

- 1. J. E. FORD, *Textiles* **17** (1988) 72.
- 2. Y. L I, V. W. MAI and L. Y E, *Compos. Sci. Technol.* **60** (2000) 2037.
- 3. M.-L. E. FLORIAN, D. P. KRONKRIGHT and R. E. NORTON, in "The Conservation of Artifacts Made from Plant Materials" (The Getty Conservation Institute, 1992).
- 4. P. S. MUKHERJEE and K. G. SATYANARAYANA, *J. Mater. Sci.* **19** (1984) 3925.
- 5. F. J. NUTMAN, *Emp. J. Experim. Agri.* **V** (1937) 75.
- 6. W. BA L LY, *CIBA Rev.* **99** (1953) 3542.
- 7. I. A. SCHENEK, *Int. Text. Bul.* **2** (2002) 8.
- 8. P. V. JOSEPH, G. MATHEW, K. JOSEPH, G. GROEN-INCKX and T. ^S ., *Compos. Part A-App. S* **34** (2003) 275.
- 9. P. V. JOSEPH, S. JOSEPH, S. THOMAS, C. K. S. PILLAI, V. S. PRASAD, G. GROENINCKX and M. S A R K I S S OVA, *ibid.* **34** (2003) 253.
- 10. C. PAVITHRAN, P. S. MUKHERJEE, M. BRAHMAKU-MAR and A. D. DAMODARAN, *J. Mater. Sci.* **26** (1991) 455.
- 11. G. V. VARGHESE, B. KURIAKOSE and S. THOMAS, *J. Appl. Polym. Sci.* **53** (1994) 1051.
- 12. A. C. CHAKRAVARTY and J. W. S. HEARLE, *Text. Res. J.* (1967) 651.
- 13. N. CHAND and ^S . A. R. HASHMI, *J. Mater. Sci.* **28** (1993) 6724.
- 14. N. CHAND, S. VERMA and A. C. KHAZANCHI, *J. Mater. Sci. Lett.* **8** (1989) 1307.
- 15. T. PA D M AVAT H I and ^S . V. NAIDU, *Ind. J. Fib. Text Res.* **23** (1998) 128.
- 16. British Standards Institution, 1992. BSEN 20139: 1992 Textiles— Standard Atmosphere for Conditioning and Testing, London, British Standards Institution.
- 17. P. R. KINNEAR and C. D. GRAY, in "SPSS for Windows Made Easy" (Lawrence Earlbaum Associates Publishers, Hove, UK, 1995).
- 18. J. H. ZAR, in "Biostatistical Analysis" (Prentice-Hall, NJ, USA, 1984).
- 19. J. W. TUKEY, in "Exploratory Data Analysis" (Addison-Wesley, Reading, 1977).
- 20. T. P. NEVELL and S. H. ZERONIAN, in "Cellulose Chemistry" and its Applications" (Ellis Horwood Limited, Chichester, 1985).
- 21. P. YANG and S. KOKOT, *J. Appl. Polym. Sci.* 60 (1996) 1137.

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