JOURNAL OF MATERIALS SCIENCE 30 (1995) 162–166

The effect of moisture content on the mechanical properties of a seed shell

L. WILLIAMSON, P. LUCAS
Department of Anatomy, University of Hong Kong, Li Shu Fan Building, 5 Sassoon Road, HK

The mechanical properties of the seed shells of the African mongongo nut, *Schinziophyton rautenenii* (Euphorbiaceae), were measured by compressive C-ring tests in an air-dry condition and also after soaking in distilled water. Young's modulus was found to be about 5 GPa and the fracture strength was 40–50 MPa, for both conditions. However, fracture toughness was affected significantly by moisture content. The critical stress intensity factor, K_{IC}, of air-dried specimens was 27% greater and the work of fracture, R, 69% greater than those of wet specimens. This difference corresponded well with microscopic observations of the complexity of the fracture surface. Viewed either by scanning electron microscopy or confocal microscopy, cracks in the wet shell deviated neatly around individual fibres, while cracks in air-dried shells either crossed individual fibres or ran obliquely across the outer layers of the secondary cell wall leaving a feathered appearance. It is proposed that the increase in toughness of shells which would be obtained from air-drying may help protect embryonic seed tissues from predation by larger animals (e.g. vertebrates such as rodents) after abcission from the parent plant.

1. Introduction

Sclerenchyma is a very thick-walled tissue, with a widespread distribution in plants, that has the sole function of the mechanical protection of living tissue. In the form of fibres, it has an important economic use as the natural fibres of commerce, e.g. jute, hemp and textile fibres [1]. Most nut shells consist purely of sclerenchyma, quite often in the form of a meshwork of randomly oriented fibres. The chemical composition of this tissue appears to be very similar to that of wood [2]. However, nut shells are generally much denser.

The comparison with wood, which is important, is complicated by fibre orientation and by the difficulty of making test specimens. It is also not clear quite how moisture affects the properties of the shells. Oven-dried wood is stiffer and stronger than wet wood [3, 4] but it is claimed that the properties of nut shells are unaffected by moisture content [5].

There are few reports on the mechanical properties of these shells that include much structural information. One such paper, on the shell of the seed of *Macadamia ternifolia* (Proteaceae), which seems to have an approximately random three-dimensional fibre orientation, gave a large range of values for these properties. For a relative density of about 0.95, Young's modulus, E, was 2–6 GPa, fracture strength, σ_F, ranged between 30 and 80 MPa and toughness, G_{IC}, was 0.1–1.0 kJ m^{-2} [5]. The explanation given for these ranges was the experimental C-ring techniques that were employed: a "soft" tensile set-up that gave lower values than the stiffer compressive test. The authors argued that both tensile and compressive C-ring tests were necessary to ascertain uniformity of mechanical properties. However, if sufficient information on the structure of the shell is available, then one test may suffice, particularly if coupled with a theoretical prediction of expected values.

A wider range of tests (tensile, three-point bending and double-cantilever beam) was possible on the shell of another seed, *Mezzettia parviflora* (Annonaceae), which has the same relative density. For the random fibre-oriented part of this shell, average values for E of 7 GPa, σ_F of 67 MPa and G_{IC} of 2 kJ m^{-2} were obtained [6]. These values are comparable to, or slightly greater, than those for the compressive C-ring tests on *Macadamia* shells [5].

Schinziophyton (synonym *Ricinodendron*) *rautenenii* (Euphorbiaceae) is a sub-Saharan African small deciduous tree, the geographical distribution of which has been extensively studied [7, 8]. It produces an oil-rich non-toxic seed (the mongongo nut) that is encased by an irregularly thick woody shell. The shell has numerous channels within it (Fig. 1) which appear to act as "crack-stoppers", producing a double-force peak when the whole seed is compressed to failure [7]. The peak forces that are required to do this are reported as exceeding 4 kN [9]. These are over twice those estimated as necessary for opening *Macadamia* seeds [5].

Mongongo nuts remain viable as they air-dry on the tree, and later on the ground, during the dry season [8]. Any significant change in their properties might influence the ease with which a predator could open them. The nuts are, in fact, a valuable resource for human hunter–gatherers in the sub-Saharan region and are opened with stone tools [8]. African

0022–2461 © *1995 Chapman & Hall*

Figure 1 A circular disc cut from the *Schinziophyton* shell showing the convoluted course of the air-filled channels.

seeds such as this have also been suggested as possibilities for the diet of a fossil hominid called *Australopithecus robustus* which, 1–2 million years ago in the Plio-Pleistocene era, was a closely related competitor of our ancestors. Such seeds may have played a role in our evolution. However, there are reasons to investigate this seed other than anthropological ones. Because the mechanical properties of plant tissues depend on their relative density (i.e. the proportion of tissue volume occupied by cell wall), it would be predicted that high-density seed shells should not only be stiffer than low-density woods but also stronger and tougher [10]. If confirmed, this might indicate an economic niche for the component fibres of seed shells in composites, large quantities of which are produced as waste by the nut industry [2].

2. Materials and methods

Schinziophyton rautenenii seeds were collected from two sites (Gwaai Forest and Amandundumela Ridge, Zimbabwe) in sub-Saharan Africa by Dr Charles R. Peters in February 1992. These were stored in boxes until testing.

The general method of testing in this study followed the procedures given by Jennings and Macmillan [5]. The shape of the seeds was ellipsoidal and circular sections, of approximately 3 mm in depth, could be cut along the transverse axis from their centres with a diamond circular saw. The sections were drilled out, filed and lightly sanded to make true circular discs of even thickness, t. The effective diameter, d, of the discs was defined as the external diameter minus half the thickness.

The discs were then cut in half to form C-rings [5]. Prior to testing, 50% of these rings were allowed to equilibrate in the laboratory at ambient temperature (21–22 °C) and humidity (49%–51%). These specimens are here termed “air-dry”. The others were immersed for at least 48 h in distilled water and are described as “wet”. Comparison of the strength and toughness of wet and air-dry shell was possible only on different specimens because the tests are obviously destructive. However, for the Young’s modulus, it was possible to test the same specimen firstly, when air-dry and later, when saturated with water. Therefore, in addition to the above tests, eight specimens were tested air-dry to about 33% of the load at which they would likely to have failed (as judged from strength tests). These were then immersed in distilled water for 48 h and tested again.

C-rings were tested in compression at a deformation rate of 5 mm min^{-1} by loading with compression platens on an Instron universal testing machine. To prevent slipping, the outer edges of the rings were lightly sanded [5]. The Young’s modulus, E, was given by [5]

$$E = \frac{3\pi d^3}{4bt^3}\left(\frac{\delta P}{\delta x}\right) \qquad (1)$$

where b is the width of the ring and $\delta P/\delta x$ is the slope of the force–deformation curve. (The ratio d/t for different discs used in these tests varied between 4.0 and 11.0.) The fracture strength, σ_F, was [5]

$$\sigma_F = \frac{P}{bt}\left(\frac{3d}{t} - 1\right) \qquad (2)$$

To obtain K_{IC} values, some rings were notched with a file on their outer surface evenly across the width of the ring at their midpoint. Initially, the notches on some of the specimens, chosen at random, were sharpened with a fresh razor blade but this did not appear to affect the results. In order to get crack propagation at the notch, the notches had to be $\sim 0.33t$. The fracture strength was calculated as in Equation 2 and the critical stress intensity factor, K_{IC}, was given by

$$K_{IC} = \sigma_F(\pi a)^{1/2}\,Y \qquad (3)$$

where Y is a geometrical correction factor. There are no tables available for this correction that apply to a C-ring test. We therefore calculated Y from

$$Y = 0.265(1 - a/t)^4 + \frac{0.857 + 0.265a/t}{(1 - a/t)^{1.5}} \qquad (4)$$

which applies, strictly, to a single-edge notch tensile test [11]. After testing, a random sample of rings were weighed, oven-dried at 105 °C and then re-weighed until constant weight was attained.

The structure of the shells was investigated after testing on a random sample of test specimens. A laser confocal optical imaging system, (MRC-600, Biorad, Hemel Hempstead, UK), connected to a conventional light microscope (LCLM), was able to resolve subsurface structure around the crack by virtue of the natural fluorescence of woody material. Photographs of these images were digitized on a Zeiss Vidas image analysis system. Measurements of the luminal area relative to their cell wall area was made for those fibres in true cross-section and used directly in the determination of relative density of individual fibres, ρ_f.

Specimens were also viewed under a scanning electron microscope (SEM) after air-drying and sputter coating with gold (Cambridge Stereoscan 150, Cambridge, UK). From SEM photographs taken at very low magnifications, the amount of space occupied by the channels could be estimated. If the percentage of space occupied by the channels was c, then the overall density of the shell, ρ^*, corrected for these channels, was calculated as $\rho_f(1 - c)$.

The distributions of all the values for the mechanical properties approximated to normality. Consequently, analyses of variance were used to establish significant differences. However, the C-rings that were tested for their Young's modulus initially when air-dry and, later, when wet, were considered to be paired. These results were therefore analysed by a paired t-test.

3. Results

Under LCLM, the structure of the shells consisted entirely of small numbers of fibres bundled together and running roughly in parallel but with an orientation that appeared to be random relative to other bundles. The average width of a fibre was about 25 μm. No difference in the inner or outermost parts of the shell, either in fibre dimensions or in structure, could be seen.

None of the characteristics or properties investigated differed significantly with the geographical site at which they were collected. Therefore, results from both collection sites were pooled in the analysis. The physical characteristics of *Schinziophyton* shells are given in Table I (because these characteristics are not always normally distributed, the range is given along with the standard deviation).

The mechanical properties of the shells are given in Table II. Only K_{IC} differed significantly between wet and air-dry states, with air-dry specimens being tougher. The Young's moduli of the eight specimens that were tested both air-dry and then wet were not significantly different ($p > 0.9$). No dependence of the results on d/t could be detected.

There was a clear difference in the appearance of the fracture surfaces in wet and air-dry shell. In wet specimens, crack propagation appeared largely to deviate around fibre bundles such as to avoid crossing the cell lumen (Fig. 2a), causing a clean fracture surface. Thus, if fibres were disposed parallel to the crack direction, straight cracks ran in the middle lamella between the fibres. If, however, the fibres were disposed across the crack path, the crack appeared to deviate around the fibre bundle and then resume running at right-angles to the tensile stress. Despite the drying necessary for viewing these specimens in the SEM, the fracture surfaces remained distinct. The ends of fibres and pits in their walls could be clearly seen (Fig. 2c).

TABLE I Physical characteristics of *Schinziophyton* shells

	n^a	Mean	S.D.	Range
% holes	10	9.5	2.4	6.4–15.3
Cell wall volume/ total cell volume, ρ_f	10	0.941	0.042	0.868–0.98
Moisture content (as % of initial wt):				
air-dry	12	9.2	0.5	8.5–10.1
wet	16	20.5	1.9	18.3–25.3

^{a} n = sample size.

TABLE II Mechanical properties of *Schinziophyton* shells

	n^a	Air-dry	n^a	Wet
E (GPa)	23	4.96(1.73)	24	5.23(2.38)
σ_F (MPa)	15	51.65(16.94)	24	45.66(22.07)
K_{IC} (MPa m$^{1/2}$)	15	2.67(0.78)^b	15	2.11(0.49)^b

Mean (S.D.)
^{a} n = sample size.
^{b} Significant difference at $p < 0.03$.

In contrast, in air-dry specimens viewed by LCLM, there were gaps between the concentric lamellae of the cell wall and the lumen shape was distorted (Fig. 2b). Cracks appeared to "feather" the outer part of the cell wall, often splintering off parts of the outermost layers of the secondary cell wall (Fig. 2d). In every micrograph, at least a few fibres were seen that were fractured across their lumen (there are many seen under the SEM in the upper right of Fig. 2d).

4. Discussion

Seed shell appears to compare very poorly in properties to wood. Ignoring the channels within the *Schinziophyton* shell, the density is higher than all woods (the densest hardwoods being about 0.94 of cell wall density [4]), but it has a low Young's modulus. In *Schinziophyton* shell, a low Young's modulus appears to be a consequence of the random three-dimensional orientation of the fibres. In such a network of short fibres, the fibres are not efficiently oriented to resist loading. Cox [12] developed a type of netting analysis whereby the modulus, E^*, of a structure such as *Schinziophyton* seed shell with a random three-dimensional fibre orientation is given by

$$E^* = (E_s\rho^*)/6 \qquad (5)$$

where E_s is the modulus of the solid material (i.e. cell wall) and ρ^* is the relative density. The elastic modulus of the cell wall of woody material, measured along the long axis of a cell, is about 35 GPa [10]. Substituting $\rho^* = 0.85$ in Equation 5 gives an expected modulus for *Schinziophyton* seed shells of 4.97 GPa, which is more or less identical with our results for air-dry shells and insignificantly smaller than for wet specimens.

The toughness is much more complex to try to predict because of the orientation of fibres. However, in wet shells, the crack path is almost entirely between cells, something which is predicted by Gibson and Ashby for woods of very high ρ^* [10]. For our fracture toughness tests on *Schinziophyton* shells, ρ^* should be considered to be 0.941 (Table I) and not 0.85 because the holes in the shell were not relevant to K_{IC} measurement. However, a relationship between K_{IC} and relative density for woods [10] suggests a fracture

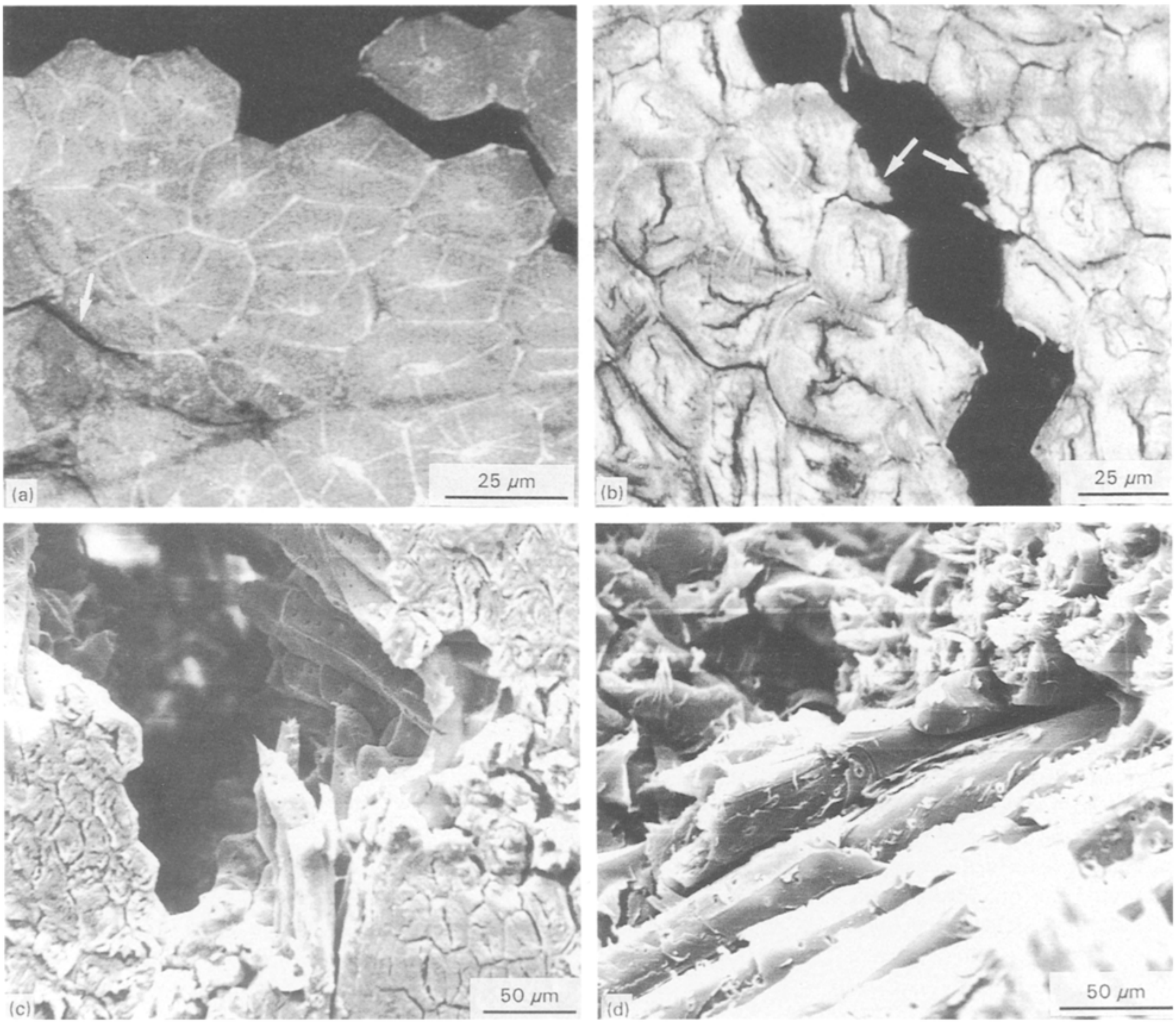

Figure 2 Fractured surfaces of *Schinziophyton* shell. (a, c) Wet specimens; (b, d) air-dry specimens. (a, b) LCLM micrographs; (c, d) SEM. See text for explanation.

toughness along the grain of 1.64 MPa $m^{1/2}$ for a wood of $\rho^* = 0.941$. This is about one standard deviation below our K_{IC} value for wet shells. The extra fracture toughness of air-dry shells must be attributable to greater damage to the cell wall but we cannot model this without knowing the fracture toughness of the cell wall.

The difference in toughness between wet and air-dry shells is amplified in an approximate calculation of the work of fracture, R (which we take here to be identifiable with the critical strain energy release rate, G_{IC}). Assuming

$$R = \frac{K_{IC}^2}{E} \tag{6}$$

then for wet shell, $R = 0.851$ kJ m^{-2}, while for air-dry shell, $R = 1.437$ kJ m^{-2}, which is 69% greater. However, the latter is again low compared to "across the grain" fracture in wood [13] and tends to substantiate a general opinion that nutshells are rather brittle.

Our results contrast with those of Jennings and Macmillan [4] who found no difference in toughness between wet and dry shells in *Macadamia ternifolia* (their dry shells contained only 2% moisture content on a wet weight basis) but the variation in their results is great. If we are correct, that there is an increase in toughness in woody tissues with a reduction in moisture content then it might have rather general implications. The toughening mechanism in wood itself ("tensile" buckling and helical fracture) may be much less well-expressed in trees, when wood is saturated, than in timber. Dried wood is slightly stiffer and stronger than wet wood [3] but we can find no clear information on the effect on toughness.

A seed contains the embryo – a potential member of the next generation of a species – and its food store. The latter is an attraction for seed predators. Most seeds are heavily protected from predation by chemical or mechanical means. Many seeds, though not normally those species from tropical rainforests, can survive a considerable reduction in their moisture content and reduce their metabolic activity to a very low (dormant) level. After abcission from other tissues, these seeds can lie on the ground or in the soil as a "seed bank" [14]. If the effects on toughness that we have demonstrated were to be general, then the outer protective layer of such seeds, nearly always a thick-walled dead tissue (a "palisade" fence-like tissue if not truly fibrous [15]), would act more efficiently after air-drying. This would be expected to reduce the rate of seed loss to predators, particularly from those predators large enough for crack propagation between cells to be a factor. These would include many mammals, typically rodents which specialize on dry fruits, but also peccaries and some human groups. The effectiveness of the fruit pod (a dry type of pericarp which protects orthodox seeds in, for example, legumes) may also be enhanced by drying. Some seed predators, particularly primates like New and Old World monkeys and also apes, tend to eat unripe seeds [16, 17]. The need to attack the seed while the toughness of the seed coat or shell is still low may play some part in this decision. Germination of a seed follows a period of water uptake. An increase in water content would decrease the toughness of protective tissues. However, this reduction in toughness is probably necessary if germination is dependent on turgor pressure to open the shell [6]. Mechanical protection is obviously ruled out at this stage.

Acknowledgements

We thank Brian Darvell, Dental Materials Science, for access to the testing machine, Tony Yuen, Dental Materials Science, and P. Y. Cheng, Anatomy, for help in making specimens. Jason Tam, Zoology, and Amy Wong, Electron Microscope Unit, for help with LCLM and SEM, respectively; all are University of Hong Kong staff. Grants from the CRCG and RGC (Hong Kong) helped support this study.

References

1. K. ESAU, "Anatomy of seed plants" (Wiley, New York, 1977) p. 71.
2. C. M. PRESTON and B. G. SAYER, *J. Agric. Food Chem.* **40** (1992) 206.
3. J. M. DINWOODIE, "Timber, its structure, properties and utilisation" (Macmillan, London, 1981) p. 197.
4. K. NIKLAS, "Plant Biomechanics" (University of Chicago Press, Chicago, IL, 1992) p. 303.
5. J. S. JENNINGS and N. H. MACMILLAN, *J. Mater. Sci.* **21** (1986) 1517.
6. P. W. LUCAS, T. K. LOWREY, B. P. PEREIRA, V. SARAFIS and W. KUHN, *Func. Ecol.* **5** (1991) 545.
7. C. R. PETERS, *Am. J. Phys. Anthrop.* **73** (1987) 333.
8. *Idem, Econ. Bot.* **41** (1987) 494.
9. *Idem, Int. J. Primatol.* **14** (1993) 315.
10. L. J. GIBSON and M. F. ASHBY, "Cellular solids" (Pergamon, Oxford, 1988) p. 278.
11. H. TADA, P. C. PARIS and G. R. IRWIN, "The stress analysis of cracks handbook" (revised) (Del Corp., PA, 1978) p. 2.10.
12. H. L. COX, *Br. J. Appl. Phys.* **3** (1952) 72.
13. G. JERONIMIDIS. *Proc. R. Soc.* **B208** (1980) 447.
14. T. C. WHITMORE, "Tropical Rain Forests of the Far East", 2nd Edn (Oxford University Press, Oxford, 1984) p. 75.
15. E. J. H. CORNER, "The Seeds of Dicotyledons", Vol. 1 (Cambridge University Press, Cambridge, 1976).
16. W. G. KINZEY and M. A. NORCONK, *Int. J. Primatol.* **14** (1993) 207.
17. M. LEIGHTON, *ibid.* **14** (1993) 257.

Received 12 October 1993
and accepted 16 June 1994