Brazilian waste fibres as reinforcement for cement-based composites ☆

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Abstract

Fibre reinforced cement-based composites were prepared using kraft pulps from sisal and banana waste and from *Eucalyptus grandis* pulp mill residues. The study adapted conventional chemical pulping conditions for the non-wood strands and a slurry vacuum de-watering method for composite preparation followed by air-curing. Plain cement paste and *Pinus radiata* kraft reinforced cement composites were used as reference materials. Mechanical testing showed that optimum performance of the various waste fibre reinforced composites was obtained at a fibre content of around 12% by mass, with flexural strength values of about 20 MPa and fracture toughness values in the range of 1.0–1.5 kJ m^{-2}. Experimental results showed that, of the waste fibres studied, *E. grandis* is the preferred reinforcement for low-cost fibre-cement. © 2000 Elsevier Science Ltd. All rights reserved.

Keywords: Fibre composites; Sisal pulp; Banana pulp; *Eucalyptus grandis* pulp; Residual fibres; Mechanical properties; Physical properties; Low-cost building material

1. Introduction

Wood fibre reinforced cement (WFRC) products obtained by the Hatschek (or wet) process are well known in most developed countries and are commercially used with a high acceptance for building purposes [1].

Tropical countries present significant opportunities for the production of non-wood vegetable fibres [2], especially as they are available from by-products of the main commercial agricultural activities (e.g., cordage and fruit) and from pulp mills. As reported by Agopyan and John [3], the utilisation of natural fibre reinforced cement-based materials (NFRC) prepared with low alkali cements provides an alternative for low cost buildings, since the major concerns about fibre degradation in an alkaline environment are greatly reduced.

In this study, sisal and banana strand fibre residue and *Eucalyptus grandis* kraft pulp-mill waste from Brazilian sources were subjected to various preparatory processes and evaluated as reinforcement for ordinary Portland cement (OPC). OPC was used to enable comparison with the considerable scientific literature that exists regarding the reinforcement of this material with various fibres. The method of production followed the slurry vacuum de-watering process, with a view to the viable use of these materials in civil construction.

The low performance of NFRC composites in earlier studies is mainly associated with the use of chopped strand fibres as reinforcement for ordinary brittle cement matrices produced by conventional dough mixing methods. This is identified as the main reason for the low acceptance of these products by industry. As a consequence, in several developing countries asbestos-cement remains the major composite in use although health hazards are becoming an increasing concern [4].

2. Materials and methods

2.1. Materials and preparation

Three different types of Brazilian fibrous residues were selected and samples brought to the Forest Products Laboratory of CSIRO Forestry and Forest Products, Australia:

- *Sisal (Agave sisalana) field by-product.* This material is readily available (e.g., 30,000 t per annum from a producers' co-operative) and presently of no commercial value. Utilisation of this resource provides a good option as additional income for rural producers. Simple manual cleaning by a rotary sieve provides a suitable starting material.
- *Banana (Musa cavendishii) pseudo-stem fibres.* This by-product has high potential availability (95,000 t per annum, based in São Paulo state, the main producing area), from fruit production. This material has no market value, and only a simple low-cost fibre extraction process is required.
- *Waste Eucalyptus grandis pulp.* This resource accumulates from several kraft and bleaching stages, has low commercial value (US$ 15/t) and is readily available (17,000 t per annum from one pulp industry in the south-east of the country). Disadvantages of this material include short fibre length and high moisture content (about 60% of dry mass).

The sisal and banana strands were subjected to kraft pulping (see Table 1). Each pulp produced was passed through a 0.23 mm Packer screen, vacuum de-watered, pressed, crumbed and stored in a sealed plastic bag under refrigeration. The *E. grandis* pulp was used as received after disintegration in hot water. Beaten New Zealand *Pinus radiata* kraft pulp was adopted as a control as in previous studies [5]. Pulp and fibre properties are summarised in Table 2.

OPC, Adelaide Brighton brand Type GP (Australian Standard AS 3972-1991), was used as the matrix material.

Natural fibre reinforced cement composites with fibre mass fractions ranging from 4% to 12% were prepared in the laboratory by a slurry vacuum de-watering technique. Neat matrix was produced as a control, using the same procedure. In the case of the formulations incorporating 8 and 12% of fibre, matrix materials were added to the appropriate amount of fibre, already dispersed in water, to form a slurry of approximately 20% solids. For formulations containing 4% fibre, slurries of about 30% solids were employed to assist fibre dispersion and minimise separation during de-watering. After stirring for 5 min the slurry was rapidly transferred to an evacuable 125×125 mm^2 casting box and an initial vacuum ($\sim$60 kPa gauge) drawn until the bulk of the excess water was removed and a solid surface formed. The moist pad was tamped flat and vacuum re-applied for 2 min. The pad was then removed from the casting box, transferred to an oiled steel plate and a fine wire mesh placed on top.

In the case of banana and *E. grandis* fibre composites, a total of three pads were prepared in this manner for each formulation, stacked on top of each other and pressed simultaneously at 3.2 MPa for 5 min. In the case of the remaining composites and unreinforced matrix, six pads were prepared to provide sufficient specimens for the determination of flexural properties at two different ages. On completion of press consolidation, the plates and meshes were removed and the pads sealed in a plastic bag to cure in saturated air at room temperature.

After 7 days the pads were removed from the bags and three 125×40 mm^2 flexural test specimens were wet diamond sawn from each pad. Test specimen depth was the thickness of the pad, which was approximately 6 mm. The samples were then allowed to air cure in an environment of 23 ± 2°C and 50 ± 5% relative humidity until tested.

Table 1
Sisal and banana kraft pulping conditions

Parameter	Sisal	Banana
Active alkali (as Na$_2$O) (%)	9	10
Sulphidity (as Na$_2$O) (%)	25	25
Liquor/fibre ratio	5:1	7:1
Temperature (°C)	170	170
Digestion time	∼75 min to temperature 120 min cook	∼85 min to temperature 120 min cook
Total yield (%w/w)	55.4	45.9
Screened yield (%w/w)	45.5	45.3

Table 2
Pulp and fibre properties

Property	Sisal	Banana	*E. grandis*	*P. radiata*
Kappa number[a]	32	45	6.1	17
Canadian standard freeness[b] (ml)	650	222	685	650
Fibre length (length weighted)[c] (mm)	1.65	1.95	0.66	1.72
Fibre width average (μm)	13.5	15.3	10.9	32.4
Aspect ratio	122	127	61	53

[a] Appita P201 m-86.
[b] AS 1301.206s-88.
[c] Kajaani FS-200.

2.2. Test methods

The flexural properties of the materials were measured 28 days after manufacture. In the case of plain matrix, and the sisal and *P. radiata* reinforced composites, these properties were also measured after 42 days.

A three-point bend configuration was employed in the determination of flexural strength (MOR), modulus of elasticity (MOE) and fracture energy properties. The flexural strength and modulus of elasticity were measured as:

$$MOR = 3Pl/2bd^2 \qquad MOE = ml^3/4bd^3,$$

where P is the maximum load carried by the specimen, l the support span, b and d are the specimen breadth and depth, respectively, measured at the nearest undisturbed location to the region of failure, and m is the slope of the load–deflection curve during elastic deformation.

The fracture energy was calculated by integration of the load–deflection curve to the point corresponding to a reduction in load carrying capacity to 50% of the maximum observed. For the purpose of this paper, the fracture toughness was measured as

$$Fracture\ toughness = fracture\ energy/bd.$$

A span of 100 mm and a deflection rate of 0.5 mm min^{-1} were used for all tests on an Instron model 1185 universal testing machine. Test data was digitally recorded and reduced using automatic data collection and processing facilities. Nine flexural specimens were tested for each composite formulation and test condition.

Water absorption, bulk density and void volume values at 28 days were obtained from tested flexural specimens following the procedures specified in ASTM C 948-81. Six specimens were used in the determination of each of these physical properties.

Property data was subjected to one-way analysis of variance to determine the statistical significance of observed differences in means at the 95% confidence level ($\alpha = 0.05$).

3. Results and discussion

The results of the mechanical and physical tests, with single standard deviations of sample means indicated, are shown in Tables 3 and 4 for all the fabricated materials. For easier comparison between the mechanical performance of the various composites, some results are presented in Figs. 1–4. For the sake of clarity, some data

Table 3
Properties of refined kraft *P. radiata* (PR) and kraft sisal (S) reinforced cements at 28 and 42 days

Fibre-content (%w/w)	Flexural modulus (GPa)		Flexural strength (MPa)		Fracture toughness (kJ m^{-2})		Water absorption (%w/w)	Density (g cm^{-3})	Permeable void volume (%v/v)
	28 days	42 days	28 days	42 days	28 days	42 days			
0	23.5 ± 4.6	23.9 ± 4.3	11.8 ± 3.7	12.9 ± 2.5	0.04 ± 0.01	0.04 ± 0.01	10.7 ± 0.5	2.18 ± 0.03	23.4 ± 0.8
PR-4	13.8 ± 1.4	15.3 ± 0.7	19.2 ± 1.9	19.9 ± 1.7	0.64 ± 0.09	0.60 ± 0.10	18.5 ± 0.5	1.69 ± 0.02	31.1 ± 0.5
PR-8	10.3 ± 0.8	10.7 ± 0.8	23.5 ± 0.8	24.3 ± 1.4	1.32 ± 0.11	1.39 ± 0.23	22.3 ± 0.5	1.54 ± 0.02	34.3 ± 0.5
PR-12	8.21 ± 0.69	8.51 ± 0.53	25.0 ± 2.1	26.0 ± 1.5	1.93 ± 0.42	2.12 ± 0.12	24.4 ± 0.6	1.46 ± 0.03	35.6 ± 0.3
S-4	14.5 ± 1.9	15.2 ± 1.1	16.5 ± 0.6	16.5 ± 1.3	0.39 ± 0.06	0.36 ± 0.05	17.9 ± 0.3	1.70 ± 0.01	30.5 ± 0.5
S-8	10.9 ± 1.13	11.4 ± 1.13	21.5 ± 1.6	20.7 ± 1.6	0.92 ± 0.13	0.86 ± 0.16	19.9 ± 0.7	1.54 ± 0.02	30.7 ± 0.8
S-12	7.54 ± 0.42	7.73 ± 0.41	20.3 ± 1.4	19.4 ± 2.0	1.41 ± 0.20	1.29 ± 0.16	23.6 ± 1.1	1.41 ± 0.02	33.2 ± 1.2

Table 4
Properties of kraft banana (B) and waste kraft *E. grandis* (EG) reinforced cements at 28 days

| Fibre-content (%w/w)	Flexural modulus (GPa)	Flexural strength (MPa)	Fracture toughness (kJ m^{-2})	Water absorption (%w/w)	Density (g cm^{-3})	Permeable void volume (%v/v)
B-4	13.1 ± 1.5	15.5 ± 1.3	0.21 ± 0.03	16.5 ± 0.2	1.71 ± 0.02	28.2 ± 0.3
B-8	8.85 ± 0.81	19.5 ± 1.4	0.53 ± 0.08	18.4 ± 0.4	1.58 ± 0.02	29.0 ± 0.7
B-12	7.04 ± 1.22	20.1 ± 2.5	1.01 ± 0.15	21.4 ± 0.9	1.50 ± 0.04	32.1 ± 0.8
EG-4	15.3 ± 0.9	15.6 ± 0.8	0.29 ± 0.04	16.8 ± 0.8	1.78 ± 0.03	29.8 ± 0.8
EG-8	11.4 ± 0.9	21.4 ± 0.9	0.82 ± 0.11	20.7 ± 0.7	1.60 ± 0.02	33.3 ± 0.6
EG-12	8.04 ± 1.06	22.2 ± 1.3	1.50 ± 0.18	24.8 ± 0.8	1.47 ± 0.02	36.5 ± 0.6
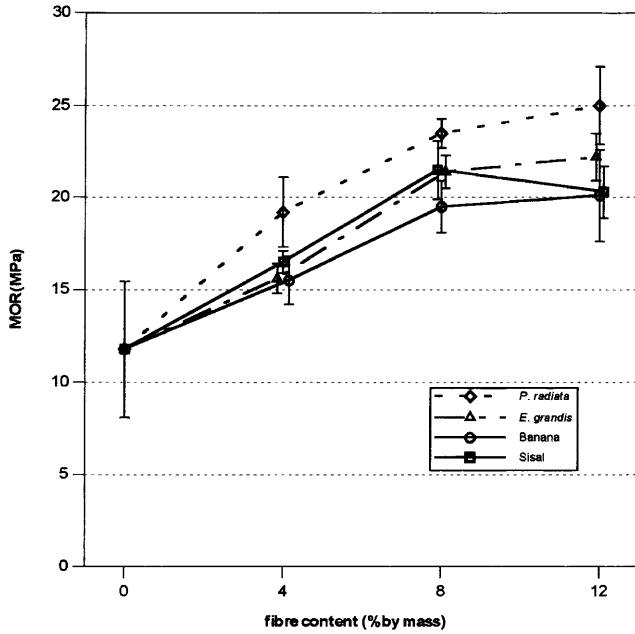
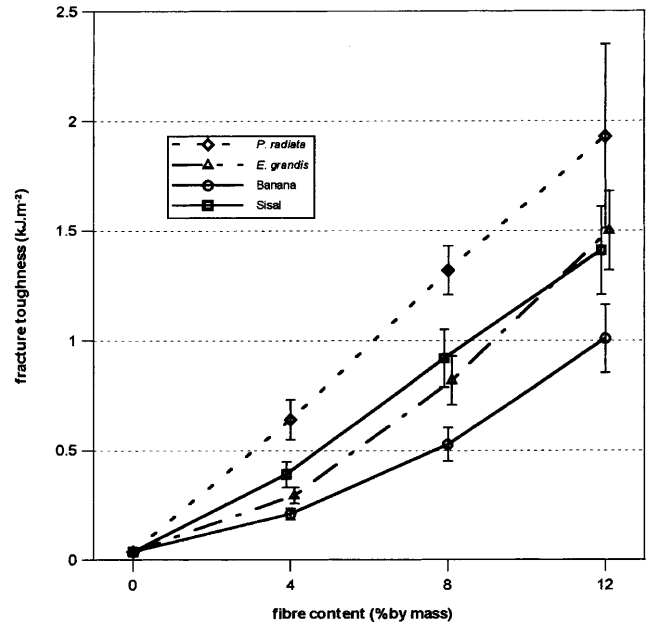Fig. 1. Variation of MOR with fibre content at 28 days.

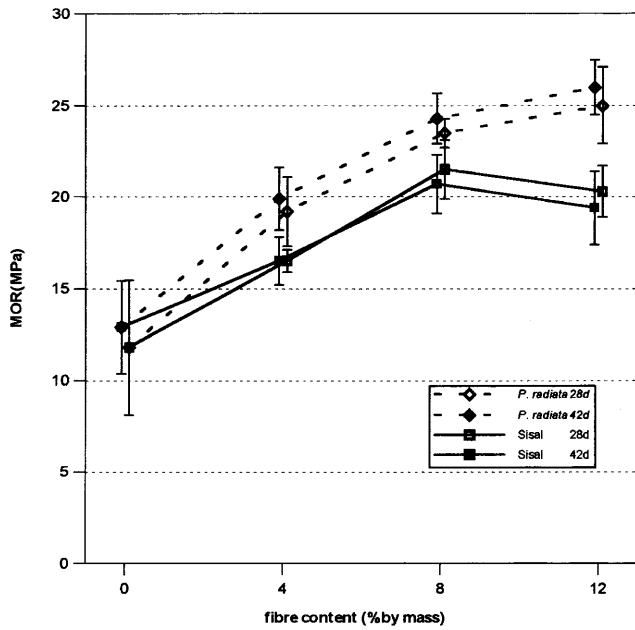Fig. 3. Variation of fracture toughness with fibre content at 28 days.
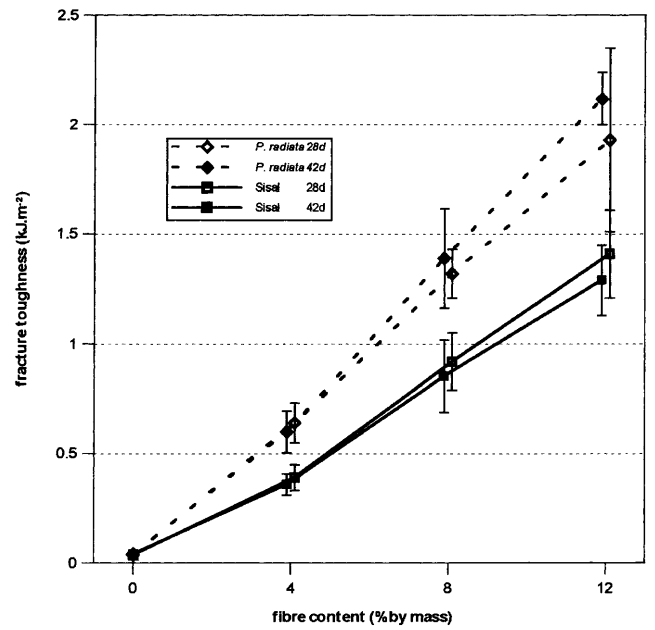

Fig. 2. Variation of MOR with age.

Fig. 4. Variation of fracture toughness with age.

points are shown slightly offset from their true positions along the horizontal axis.

3.1. Flexural strength and modulus

At a content of 8%, all of the fibres studied provided a considerable increase in MOR (at least 65%) relative to that of the unreinforced matrix at 28 days (see Tables 3 and 4 and Fig. 1). Strengths observed at a fibre content

of 12% were not significantly different from those at 8%, however, except in the case of sisal fibre, there was some tendency toward improvement. For a given fibre content, the only significant difference in waste NFRC composite strengths observed was that for 8% banana fibre, which was lower. Despite its low aspect ratio (see Table 2), *E. grandis* fibre performed relatively well, producing composite strengths similar to those achieved with sisal at fibre contents of 4% and 8%. At a content of

12%, *E. grandis* fibre provided the highest measured waste NFRC strength. The apparent decrease in strength of the sisal fibre composite at this content could be associated with poor distribution of the longer fibres within the matrix and thus less load bearing capability. Analogous results were previously achieved in studies carried out with sisal pulp reinforced cement mortars [6]. The strengths of *P. radiata* reinforced composites exceeded those of the corresponding waste fibre composites and may be attributed to both the superior fibre and the beating process, which produces fibrillation and consequently improved fibre–matrix bonding.

Mechanical properties of banana fibre reinforced composites were very similar to those presented by Zhu et al. [7], whose pulping procedures were used as a basis for this study. The mediocre mechanical performance of both banana and sisal composites could be associated with the impure nature of the fibres. Despite the low yield obtained (Table 1), sisal pulp contained non-fibrous impurities after Packer screening. The banana pulp possessed a strong smell and dark colour, which suggested extractives remained in the pulp.

Fig. 2 depicts the flexural strengths of sisal and *P. radiata* reinforced composites at two different ages. The results of the tests at 28 and 42 days for both composites and the unreinforced matrix indicate that the short span of time between the tests was insufficient to show any real variation with time. As the age effect was not sufficiently clear at 42 days, further long-term durability tests are now in progress.

The elastic modulus in bending decreased as the fibre content increased. Thus the elastic modulus for the unreinforced cement paste was approximately 24 GPa and steadily fell to the range of 7.0–8.2 GPa no matter which fibre was present. Age appeared to improve the stiffness of both composites and plain matrix, although generally not at a significant level over the period studied. This behaviour can probably be associated with compressive strength increase [8].

The high standard deviation associated with the elastic modulus of the unreinforced matrix is a consequence of the heterogeneous cracking arising from the stresses generated by shrinkage during drying.

3.2. Fracture toughness

This property was significantly enhanced by fibre inclusion; at 12% fibre loading fracture toughness values exceeded 1.0 kJ m^{-2}, a 25-fold increase in energy absorption over the measured value of the brittle matrix material. These results also represent an improvement over those previously obtained by Savastano and Agopyan [9], in whose study chopped sisal strand fibre reinforced OPC displayed fracture toughness values of 0.5 kJ m^{-2}. A dough mixing process was used in that study for composite preparation.

Fig. 3 depicts the change in fracture toughness values with change in fibre loading for the various NFRC composites studied.

Fracture toughness is often correlated with the length of a reinforcing fibre. As the composite material is subjected to a load the stress is transferred from the matrix to the fibre. Debonding can take place at the interface and the fibre may then be pulled out through the matrix, generating considerable frictional energy losses which contribute to fracture toughness [1]. In the case of the banana pulp, low fibre strength could have resulted in not only the low MOR observed but also in the fracture of fibres before pull-out could take place and hence in the significantly lower fracture toughness values obtained. The low freeness of the banana pulp (Table 2) indicates an undesirably high incidence of fines (as high as 16% in the study carried out by Zhu et al. [6]). This behaviour is being further studied by the use of scanning electron microscopy and will be reported in a later paper.

Over the range of fibre contents studied, the performance of the *P. radiata* reference composites was significantly better than that of the waste fibre composites and is again associated with good fibre–matrix bonding as described in previous work [5].

No significant changes in fracture toughness values of *P. radiata* and sisal fibre reinforced composites were observed over the time frame studied (Fig. 4).

3.3. Density and water absorption

Density, water absorption and porosity are all interrelated physical properties. The data in Tables 3 and 4 show that as the fibre content is increased, significant decreases in density and increases in water absorption occur, in keeping with analogous studies [10,11].

After an initial drop corresponding to the addition of 4% fibre, density decreased at a relatively constant rate with increased fibre loading, giving rise to an overall decrease of approximately 30–35% at a fibre content of 12%. This characteristic is useful when considering lightweight construction materials, however the water absorption had more than doubled at 12% fibre content.

4. Conclusions

Sisal and banana fibrous wastes collected in tropical Brazilian agricultural fields possess potential for the production of chemical pulps suitable for cement reinforcement. Both these alternative pulps and *E. grandis* kraft pulp waste were suitable for composite manufacture

by a slurry vacuum de-watering method similar to the process used in commercial production.

Compared with similar materials reinforced with chopped strand vegetable fibres, vegetable pulp reinforced cements present superior mechanical performance. The incorporation of 12% by weight of Brazilian waste fibre pulps in cement produced composites with MOR values of about 20 MPa and fracture toughnesses in the range of 1.0–1.5 kJ m^{-2}. The physical properties are in accordance with similar studies of other natural fibres. Although the strength and toughness properties of the waste fibre reinforced composites studied were inferior to those of composites incorporating the preferred *P. radiata* fibre, they are sufficient for the use of these materials in low-cost housing construction. It would be expected that further property improvements could be achieved through optimisation of the processing variables and fibre refining.

Finally, some favourable characteristics of *E. grandis* waste can be summarised:

- Non-commercial fibrous waste available in large quantities close to Brazil's largest urban areas.
- Since the fibres are already in pulp form, low processing energy requirements (hot water disintegration only).
- Easily dispersed in cementitious matrices, even at relatively high fibre contents.
- Acceptable performance as reinforcement in cement-based composite materials for low-cost housing applications.

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