Correlations Between the Physiochemical Characteristics of Plant Fibres and Their Mechanical Properties

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Abstract Lignocellulosic fibres harvested from different plant types exhibit variations in mechanical properties that are associated with their chemical composition and physical features. This diversity indicates that plant fibres could be selected based on their physio-chemical properties for tailored applications such as enhanced vibration damping. In this study, bast, leaf, and mesocarp fibre bundles were investigated to understand correlations between their physiochemical characteristics and their mechanical properties with a particular focus on their vibrational damping ability. Due to the interrelations between the investigated variables such as cellulose content and microfibril angle, a multivariate analysis (principal component analysis) was applied to elucidate trends. The stiffness and strength of the fibre bundles were found to be positively correlated to high cellulose content and low microfibril angle while high toughness was correlated with fibre bundles of high lignin content and high microfibril angle. Conversely, the damping coefficient was found to be positively correlated to fibres containing high level of hemicelluloses, such as those extracted from leafy plants.

Keywords Plant fibers · Chemical composition · Physical characteristics · Mechanical properties

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R. Fangueiro and S. Rana (eds.), *Natural Fibres: Advances in Science and Technology Towards Industrial Applications*, RILEM Bookseries 12,
DOI 10.1007/978-94-017-7515-1_3

Introduction

Lignocellulosic fibres extracted from different parts of the plant including the bast, leaf, or seed may exhibit significant differences in physical and chemical properties. These differences in properties have been tentatively correlated with their biological role in the plant (Newman 2008; Eder and Burgert 2010). High tensile strength and stiffness are associated with fibres containing a high proportion of cellulose such as that found in bast fibres. Conversely, fibres of high cellulose microfibril angle (MFA) are associated with low strength and stiffness but high elongation and toughness (e.g. stipe, mesocarp fibres) (Chakravarty and Hearle 1967; McLaughlin and Tait 1980; Satyanarayana et al. 1982).

However, in terms of vibration damping of plant fibres, limited knowledge is available. The viscous behaviour of European flax fibres has been attributed to the amorphous matrix components of the fibre that include pectin and hemicellulose (Keryvin et al. 2015). Yet, in contrast, Gassan and Bledzki attributed the higher damping capacity of European flax composites compared to jute composites to higher cellulose content in flax fibre indicating that damping could originate from the reinforcing component of the fibres (i.e. the microfibrils) (Gassan and Bledzki 1999).

In wood research, high damping coefficients were observed for specific MFA values of 27° (Reiterer et al. 2001) and 30° (Chauhan et al. 2006). However, in another study, the variation of the damping coefficient with MFA ranging between 15° and 45°, was described with a power law relationship indicating that there was no optimal value of MFA and, the higher the MFA, the higher the damping coefficient. For instance, wood fibre with an MFA of ~45° exhibited a damping coefficient that was 50 % higher compared to an MFA of 15° (Bremaud 2012). Beside MFA, vibration damping in wood was also found to be influenced by its chemical composition and particularly to its hemicelluloses content and their hydration state (Entwistle 2005). Clearly, further work is necessary to fully identify the structural parameters that most influence the damping capacity of plant and wood fibres.

In the present study, it is hypothesised that lignocellulosic fibres of a higher MFA (or a specific value of MFA) will exhibit a maximum in vibration damping capacity indicating that certain fibre types maybe more suited than others in vibration damping applications. However, as indicated previously, the MFA may not be the only parameter of importance and others, such as the moisture content or hemicelluloses content could also influence the damping coefficient. The difficulties with proving such a hypothesis arise due to the number of parameters involved (moisture content, MFA, carbohydrate content etc.) and their interrelations. For example, high cellulose content has been associated in previous research with low MFA and low lignin content (Eder and Burgert 2010). Hence, the different parameters cannot be investigated separately without potentially masking some synergetic or underlaying effects. In this work, a statistical approach is proposed using principal component analysis to further elucidate the mechanisms of damping in plant fibres.

Experimental Procedures

Materials

Fibres were purchased and/or harvested from garden centre plants in Rotorua, New Zealand (38.1378° South, 176.2514° East). European flax (*Linum usitatissimum*) and ramie fibres (*Boehmeria nivea*) were purchased as slivers from Tongling Worldbest Linen and Ramie Textile Co Ltd. The flax fibres were retted, scutched and hackled into textile grade slivers. No additional information was provided concerning the origins or the processing of the ramie fibres. Jute fabric (*Corchorus*) was purchased from a domestic supplier (Spotlight), while sisal twine (*Agave sisalana*) was obtained from Donaghys Ltd. and loose mesocarp coir fibres (*Cocos nucifera*) from Horticom (using two suppliers and labelled coir 1 and 2). Fibre bundles of jute were obtained from the weft and warp directions of a plain weave fabric, while the sisal fibres were extracted from the twine. All of the fibre bundles were straightened in water to remove any waviness caused by the weave or twine, dried and equilibrated at room temperature. Windmill palm fibres (*Trachycarpus fortunei*) were gathered from the sheath of the leaves of a palm tree growing in Rotorua. Harakeke fibre bundles (*Phormium tenax*) were collected from a private garden in Rotorua and were hand-extracted from mature leaves harvested from the top, middle and bottom of leaves in July 2013. All of the fibre specimens were weighed using a Mettler AT261 Delta range scale (Metler Toledo, Switzerland) with five decimal precision to calculate the linear mass in tex after conditioning (>48 h at 23 °C and 50 % relative humidity). The equilibrium moisture content (EMC) of the fibres was also measured by weighing following conditioning at 23 °C and 50 % RH for a period of 1 week, and then weighing specimens once a constant weight was achieved following drying at 105 °C.

Chemical Composition of Fibres

The chemical composition of the fibres was carried out by hydrolysis and anion exchange liquid chromatography on a Dionex ICS 3000 (Thermo Fisher Scientific Inc., Waltham, MA, USA). 2 g of fibres were pre-ground to 40 mesh through a Wiley mill (Thomas Scientific Inc., Swedesboro, NJ, USA). Ground samples (including routine *Pinus radiata* for quality control) were extracted with dicholoromethane in a Soxhlet apparatus (Tecator Soxtec System Model HT 1043) using a boiling time of 1 h and a rinsing time of 1 h. The total lignin content was determined in duplicate as the sum of Klason plus acid-soluble lignins following Tappi standard T222 om-88 1988 and Tappi standard UM-250 1991. In the cases of the windmill palm, the analysis was scaled down to analyse 0.25 g fibre sample due to the limited amount available. Monomeric sugars in the filtrates from Klason lignin determinations were analysed in duplicate by ion chromatography (Petterson 1991).

The results were expressed as anhydrosugar units. Lignin and carbohydrate results were validated when the duplicates were within 2 % of each other and sample total sugar and lignin values within the range 90–100 %.

Measurement of the Microfibril Angle

Wide angle X-ray scattering (WAXS) patterns were collected on the SAXS/WAXS beamline at the Australian Synchrotron (Melbourne, Victoria). The radiation wavelength was 0.8266 Å. Diffraction patterns were collected with a two-dimensional MAR-165 CCD detector (Rayonix LLC, Evanston, IL, USA), located 120.714 mm from the specimen. The beam size was approximately 150 μm in diameter and the detector pixel size was 79 × 79 μm. 15 scans of 1 s exposure at different positions were recorded and average values were taken using 5 replicate specimens. The data was processed using the Australian Synchrotron software Scatterbrain Version 1.235. The windmill palm fibres were not characterised by X-ray diffraction as they were not available at the time of the experiments.

Tensile Properties of Fibres

Tensile testing was performed on a RSA-G2 DMTA instrument (TA instruments, New Castle, DE, USA) using a 35 N load cell. The gauge length was 10 mm for all tests. 20–30 fibre bundles per type of fibre were tested to obtain average values. Quasi-static tests were carried out using a crosshead speed of 2 mm s^{-1}. Load-displacement curves were recorded, providing values of tenacity in Newton/tex and strain to failure (ε_f). The stiffness was determined from the initial slope of the load-displacement curve (between strains of 0–0.1 %). The toughness was derived from the tenacity-strain curves by numerical integration of the area under the curve. The damping coefficient (tan δ) was determined during dynamic tensile tests at a temperature of 23 °C and 50 % R.H. Consecutive frequency sweeps from 0.1 to 100 Hz were performed at 0.05 % strain along with a preload of $\sim$10 % of the failure load ensuring that the test was carried out in the linear visco-elastic range. Tan δ was measured at a frequency of 1 Hz and averaged over the 10th and 20th cycles to avoid potential damping artefacts caused by the handling of the fibres during clamping.

Principal Component Analysis

Principal component analysis (PCA) was performed on the average values of 10 variables using a correlation matrix. The variables included the cellulose content,

lignin content, hemicellulose content, MFA, tenacity, stiffness, strain, toughness, damping and water content at 50 % R.H. The PCA was interpreted using R version 3.1.0 (R Development Core Team), and R studio version 0.98.945 (RStudio, Inc.).

Results and Discussion

Carbohydrate and Lignin Content of the Plant Fibres

Cellulose, lignin and hemicellulose were the main components of the chemical composition of the fibres (Table 1). "Other" components included waxes, minerals and pectin. Only chemicals that were common to all of the fibres were taken into account in the analysis, although it must be noted that pectin is a significant component of European flax and as such may influence damping behaviour. The chemical composition of the fibres was similar to previous work, highlighting the significant variation in chemical composition across different natural fibres (Table 1) (Müssig et al. 2010).

Differences in the cellulose to lignin ratio were observed for the harakeke fibres sampled at three different locations in the leaf (Table 2). Harakeke fibres extracted from the bottom of the leaf were richer in lignin compared to that of the top part of the leaf. Richter et al. made similar observations in the lignin distribution through

Table 1 Chemical composition of plant fibres (wt%)

	Cellulose	Hemicelluloses	Lignin (Klason)	Others
Pinus radiata	44.1	19.8	27.6	7.9
European flax	72.6	8.7	3.2	14.7
Ramie	96.0	0.5	0.4	3.1
Jute	46.2	12.5	15.4	23.3
Harakeke	49.2	21.2	6.8	18.7
Sisal	45.3	20.4	15.1	15.5
Windmill palm	36.2	17.8	35.6	18.6
Coir 1	32.9	25.2	35.0	5.5
Coir 2	34.1	20.9	33.6	9.6

Table 2 Chemical composition of Harakeke fibres as a function of the location within the leaf (wt%)

	Cellulose	Hemicelluloses	Lignin (Klason)	Others
Top	59.0	22.0	5.1	10.0
Middle	57.5	22.4	5.7	10.1
Bottom	55.6	22.6	7.6	9.5

harakeke leaf using Raman spectroscopy. It was hypothesised that increased lignin at the base of the harakeke plant provides the additional required stiffness in this part of the leaf (Richter et al. 2011). In contrast, little variation in the hemicellulose content was observed across different locations in the harakeke leaf (Table 2).

Measurement of the Plant Fibres Microfibril Angle

The MFA determined by X-ray diffraction were consistent with values given in the literature with the exception of the ramie fibres which was lower than previously reported (Table 3) (Eder and Burgert 2010). Trends in the MFA were clearly observed with bast fibres having an MFA of ~4–10°, while that of leaf fibres was ~10–20° and that of mesocarp/stipe fibres was >20°. The MFA value of 39.5° for windmill fibres was used from literature (Zhai et al. 2013).

Tensile Properties of Plant Fibres

The stiffnesses of the fibres from European flax, ramie and jute were found to be of the same order of magnitude (Table 4). European flax fibres exhibited the highest ultimate tensile strength, while coir fibres had the highest strain to failure and toughness as expected from previous research (Müssig et al. 2010). In terms of intermediate properties, harakeke fibres exhibited the highest combined performance in terms of the ultimate tensile strength, strain to failure and stiffness.

The tensile properties of harakeke fibres extracted from the tip and mid sections of the leaf were similar, while the fibres extracted from the lower section were stiffer although not as strong or as tough as the fibres from the other sections (Table 5). These findings are also supported by the work of Richter et al. that reported a higher lignin content within the lower part of the harakeke leaf (Richter

Table 3 Experimental and literature values of the MFA of the various plant fibres studied in this work

Plant species	Fibre type	MFA (Exp.)	MFA (literature)	Reference
European flax	Bast	4.5 (0.1)	4.4	Müller et al. (1998)
Ramie	Bast	4.2 (0.1)	7–8	Bledzki and Gassan (1999)
Jute	Bast	6.6 (0.1)	8	Bledzki and Gassan (1999)
Harakeke	Leaf	11.4 (0.3)	16	Richter et al. (2011)
Sisal	Leaf	15.2 (0.2)	20	Pavithran et al. (1987)
Windmill palm	Leaf/Stipe	N/A	37.8	Zhai et al. (2013)
Coir 1	Mesocarp	46.8 (0.8)	45	Martinschitz et al. (2008)
Coir 2	Mesocarp	N/A	45	Martinschitz et al. (2008)

Values in parentheses represent 1 standard deviation

Table 4 Tensile properties of the selected plant fibres

	Ultimate stress (N/tex)	Ultimate strain (%)	E-modulus (N/tex)	Toughness (N/tex)
European flax	0.52 (0.08)	1.89 (0.21)	20.8 (3.7)	0.0051 (0.0014)
Ramie	0.31 (0.07)	1.84 (0.16)	26.8 (4.4)	0.0057 (0.0008)
Jute	0.39 (0.09)	1.53 (0.21)	24.6 (5.6)	0.0032 (0.0009)
Harakeke	0.48 (0.07)	2.77 (0.31)	17.2 (2.5)	0.0095 (0.0024)
Sisal	0.40 (0.05)	2.65 (0.30)	18.7 (3.0)	0.0058 (0.0012)
Windmill palm	0.12 (0.01)	17.94 (2.71)	4.0 (0.2)	0.0154 (0.0029)
Coir 1	0.12 (0.02)	24.44 (4.21)	3.8 (0.6)	0.0210 (0.0051)
Coir 2	0.14 (0.02)	38.50 (5.48)	3.1 (0.3)	0.0365 (0.0069)

The value in parentheses is the 95 % confidence interval

Table 5 Tensile properties of the harakeke fibres extracted from the tip, middle, and bottom of the leaves

	Ultimate stress (N/tex)	Ultimate strain (%)	E-modulus (N/tex)	Toughness (N/tex)
Top	0.49 (0.06)	3.66 (0.24)	17.0 (2.7)	0.0099 (0.0014)
Middle	0.48 (0.10)	3.47 (0.38)	17.2 (2.5)	0.0095 (0.0024)
Bottom	0.37 (0.10)	3.02 (0.32)	18.7 (2.0)	0.0071 (0.0015)

The value in parentheses is the 95 % confidence interval

et al. 2011). From a biological perspective, the lower part of the leaves requires greater stiffness that supports the plant structure above. Conversely, the top of the leaves requires greater flexibility to reduce mechanical stresses at the base of the plant (e.g. during windy weather) and also maximise the orientation of the leaves surface towards sunlight.

During tensile loading, the force acting on the cellulose microfibrils is a function of the MFA. The larger the MFA, the less the cellulose microfibrils contribute to the stiffness at the beginning of a tensile test explaining the low stiffness of mesocarp fibres compared to bast fibres (Table 4).

In the case of the bast fibres, the microfibrils realignment was minimal due to their initial low MFA (≤10°). Hence, the stress-strain deformation was quasi-linear. In the case of leaf fibres the MFA was ranging between 10° and 20°. The initial deformation was elastic, similarly to bast fibres but then gradually deviated from linear behaviour indicating irreversible plastic deformation.

When the MFA was greater than 20°, the deformation was bi-phasic and the transition was clearly indicated by a yield point. The initial elastic region of the curve was matrix-dominated. Past the yield point, the load gradually transferred to the cellulose microfibrils exhibiting a second linear behaviour with a steeper slope than the beginning of the test, caused by the realignment of the cellulose microfibrils (Martinschitz et al. 2008).

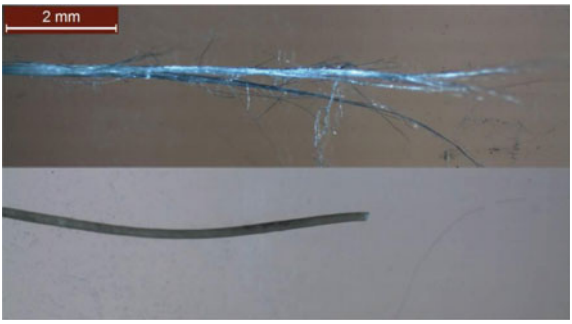

Fig. 1 Tensile fracture of coir fibre (*bottom*) and harakeke fibre (*top*) indicating a cohesive in-plane fracture of the coir fibre and defibrillation of the harakeke fibre

In terms of fracture, coir and windmill fibres failed abruptly after extensive plastic deformation. Coir fibre had a smooth fracture surface after tensile testing where all of the cells within a single fibre fractured on the same plane (Fig. 1). In contrast, the cells within the harakeke, sisal and European flax fibres failed independently of each other, causing the fibres to slide past each other and leading to extensive defibrillation (Fig. 1).

Damping Coefficient of Plant Fibres

The range of damping coefficient was measured from 0.0125 to 0.0198 (Table 6). Berthelot et al. reported damping coefficients of 0.012 for glass fibres, 0.0062 for carbon fibres and 0.0165 for aramid fibres measured by acoustic method. Their results indicated that plant fibres have a significant advantage in damping applications compared to glass and carbon fibres (Berthelot and Sefrani 2007). Differences in the damping coefficient between the fibre types were observed;

Table 6 Damping coefficient for selected plant fibres

	Damping coefficient	Moisture content (wt%)
European flax	0.0140 (0.0018)	10.6
Ramie	0.0142 (0.0020)	10.2
Jute	0.0128 (0.0010)	9.4
Harakeke	0.0176 (0.0027)	10.2
Sisal	0.0198 (0.0023)	9.5
Windmill palm	0.0194 (0.0018)	10.2
Coir 1	0.0180 (0.0012)	9.6
Coir 2	0.0165 (0.0026)	8.1

The value in parentheses is the 95 % confidence interval

Table 7 Damping coefficient of harakeke fibres extracted from the tip, middle, and bottom of the leaves

	Damping coefficient	Moisture content (wt%)
Top	0.0167 (0.0018)	10.1
Middle	0.0188 (0.0012)	10.2
Bottom	0.0187 (0.0026)	10.1

The value in parentheses is the 95 % confidence interval

however, no obvious trend emerged with MFA, moisture content or location in the leaf (Table 7). As mentioned previously, the damping coefficient was believed to be influenced by a combination of several variables (e.g. chemical composition, MFA). Hence, the influence of these variables on damping were investigated using multivariate analysis.

Determination of the Correlation Between Variables Using Principal Component Analysis

The principal components analysis was limited to the first two components as they accounted for 89 % of the variance. A bi-plot of the data in the space of the first two principal components was produced with the fibres' scores and the variables (loadings) (Fig. 2). The analysis could highlight 3 clusters of variables:

- Group 1: MFA, lignin, strain and toughness
- Group 2: cellulose, stiffness, water content and tenacity
- Group 3: hemicellulose and damping

Group 1 and 2 were oppositely related. This observation indicated that when the variables from Group 1 (MFA, lignin, strain, toughness) were high, the variables from Group 2 were low (Fig. 2). The fibre toughness was strongly correlated with high strain, high MFA and high lignin content (e.g. windmill palm and coir fibre). During a tensile test, the cellulose microfibrils are known to realign towards the fibre axis, enabling extensive plastic deformation (Martinschitz et al. 2008). Hence, while bast fibre such as European flax exhibited a tenacity 4 times higher than coir fibres, their toughness was up to 7 times lower (Le Guen 2014). Intermediate toughness was found for fibres with MFA between 10° and 20°, for which the deformation curves indicated a bi-phasic behaviour and the absence of a distinctive yield point (Le Guen 2014). The microfibrils could reorient in the axis direction, causing enough shear stresses in the hemicellulose/lignin matrix to provoke a gradual transition from elastic to plastic deformation as exhibited by harakeke and sisal fibres. In contrast to studies on wood, the damping coefficient did not correlate with the MFA (Chauhan et al. 2006). The second cluster of variables indicated correlation between cellulose content, stiffness, tenacity and water content (Fig. 2). A high tenacity was correlated with a high cellulose content (i.e. low lignin content) similar to previous studies (Eder and Burgert 2010). The larger is the MFA, the

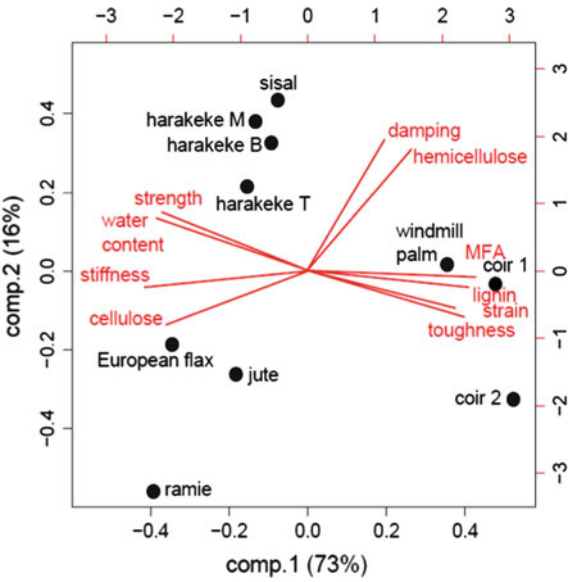

Fig. 2 Bi-plot of the variables (loading and scores) using principal component 1 and 2

lesser is the cellulose microfibrils' contribution to the stiffness at the beginning of the test explaining the low stiffness of mesocarp fibres compared to bast fibres. Water content was not associated with the damping coefficient. This observation indicated that the source of damping was not directly linked to the motion of the water molecules but the plasticising of the hemicelluloses (Salmen 2004). However, no correlation was found between hemicellulose and water content. Jarvis hypothesised that the cellulose chains at the surface of a microfibril are loosely packed compared to the cellulose within the crystal, enabling more hydrogen bonding between the cellulose and the hemicellulose or water (Jarvis 2005). It was also believed that a small amount of hemicellulose was present between the cellulose microfibrils enabling access for a limited amount of water (Jarvis 2005). These observations are in line with the correlation between water and cellulose content. The last cluster of variables included the damping coefficient and hemicellulose content (Fig. 2). Fibres that contain a higher proportion of hemicelluloses (e.g. harakeke, sisal) exhibited a higher damping coefficient. In wood research, energy losses are explained by the hemicellulose molecular hydrogen bonding structure, especially, in the presence of water. Vibrational energy loss in wood was reported to increase almost linearly with the hydration of hemicellulose (Entwistle 2005). Bucur reported that the damping coefficient typically increased from 5.5×10^{-3} to 18.5×10^{-3} as the moisture content increased from 5 to 35 % (Bucur 2006). The hydration of hemicelluloses increases the energy dissipation by the (i) motion of water molecules and (ii) hydrogen bonding shifts between hydrated hemicellulose and cellulose (Bremaud et al. 2010; Ebrahimzadeh and Bertilsson 1998; Newman 2005). The first mechanism relies on the water molecules capacity to move freely due to their small size and the second mechanism on the

plasticisation of the hemicelluloses by water (Entwistle 2005). It was shown previously that there was no direct correlation between damping and water content; hence, the second damping mechanism is favoured. Hydrated hemicellulose molecules do not have the same mobility due to their size, molecular structure and conformation (Åkerholm and Salmén 2001; Newman 2005). However, no obvious trend emerged with the different types of hemicelluloses in this study (results not presented here) (Le Guen 2014).

Conclusions

Selected plant fibres were characterised for their mechanical properties (strength, stiffness, strain, toughness and damping), physical property (morphology and MFA) and chemical composition (hemicellulose, cellulose and lignin content). Correlations between variables were analysed by PCA. The highest mechanical properties in terms of stiffness and strength were observed for bast fibres, while leaf and mesocarp fibres exhibited the higher toughness.

In terms of energy absorption properties, the toughness of the technical fibres increased with increasing MFA and was correlated with fibres containing high level of lignin and low levels of cellulose. The damping coefficient was only related to the hemicellulose content. The literature states that damping increases with moisture due to plasticisation of the hemicelluloses. However, the present findings indicate clearly that the amount of absorbed water was only correlated with cellulose content. Harakeke and sisal fibre exhibited intermediate mechanical properties to bast and mesocarp fibres (e.g. tenacity, toughness) and the highest damping coefficient. Hence, natural fibres that contain a higher proportion of hemicelluloses would be preferable for maximising vibration damping applications if used in a hydrated-like state.

Acknowledgements The authors thank the Biopolymer Network Ltd for funding under the New Zealand Ministry of Business, Innovation and Employment contract number BPLY0801, Mrs. Sunita Jeram for carrying out the chemical analysis and Dr. Nigel Kirby for his help during the X-ray diffraction experiments.

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