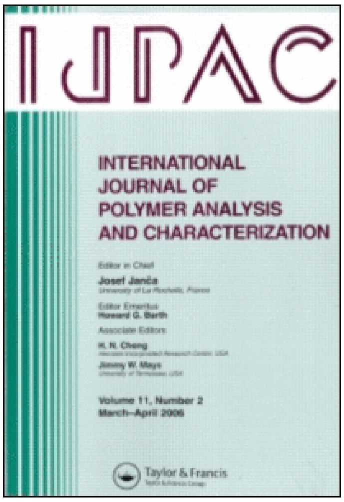


International Journal of Polymer Analysis and Characterization
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713646643

Characterization of Natural Fabric *Sterculia urens*

J. Jayaramudu[a]; B. R. Guduri[b]; A. V. Rajulu[a]
[a] Department of Polymer Science and Technology, Sri Krishnadevaraya University, Anantapur, India [b] Polymers, Ceramics, and Composites, Materials Science and Manufacturing, Council for Scientific and Industrial Research, Pretoria, South Africa

To cite this Article Jayaramudu, J. , Guduri, B. R. and Rajulu, A. V.(2009) 'Characterization of Natural Fabric *Sterculia urens*', International Journal of Polymer Analysis and Characterization, 14: 2, 115 — 125

To link to this Article: DOI: 10.1080/10236660802601415

URL: http://dx.doi.org/10.1080/10236660802601415

International Journal of Polymer Anal. Charact., 14: 115–125, 2009

ISSN: 1023-666X print
DOI: 10.1080/10236660802601415

Characterization of Natural Fabric *Sterculia urens*

J. Jayaramudu,[1] B. R. Guduri,[2] and A. V. Rajulu[1]
[1]Department of Polymer Science and Technology, Sri Krishnadevaraya University, Anantapur, India
[2]Polymers, Ceramics, and Composites, Materials Science and Manufacturing, Council for Scientific and Industrial Research, Pretoria, South Africa

Abstract: The newly identified natural fabric from the tree *Sterculia urens* was characterized by chemical, FT-IR, X-ray and thermogravimetric, tensile test, and microscopic methods. The effect of alkali treatment of the fabric on its properties was also investigated. On alkali treatment, the amorphous hemicellulose was found to be eliminated to a large extent. The improvement of properties of this fabric on alkali treatment was attributed to the lowering of its amorphous hemicellulose content.

Keywords: Crystallinity; FT-IR; Lignocellulose; Morphology; Polarized optical micrograms; Reinforcement; *Sterulia urens* natural fabric; Tensile properties; TGA

INTRODUCTION

As the usage of polymer products in general and composites in particular is increasing day by day, simultaneously, the dangers they pose

Submitted 14 August 2008; accepted 18 September 2008.
This work was supported by University Grants Commission of India sponsored Major Research Project (33-397/2007 SRF).
Correspondence: A. Varada Rajulu, Department of Polymer Science and Technology, Sri Krishnadevaraya University, Anantapur – 515 003 (AP), India. E-mail: arajulu@sify.com

to the environment are also increasing. Most polymer composites have glass fiber as reinforcement. As glass fibers/fabrics are nondegradable, the disposal of the composites containing them as reinforcement is a difficult problem. Moreover, glass fiber is a nonrenewable material. At present, the trend is slowly changing towards using natural fibers as reinforcements. The composites made with natural fiber reinforcements are known as "green composites." In this connection, some green composites were developed by several workers.[1–8] Varada Rajulu et al.[9] studied the effect of alkali treatment on properties of the lignocellulose fabric *Hildegardia populifolia*. In making green composites, it is customary to treat the natural fiber/fabric with alkali and other chemicals to improve its properties. Hill and Abdul Khalil[10] studied the effect of acetylation on the mechanical properties of coir or oil palm fiber-reinforced polyester composites. They reported an increase in interfacial shear strength between the matrix after acetylation treatment of coir. Rout et al.[11] studied the influence of fiber structure modification on the mechanical properties of coir-polyester composites. They reported improved mechanical properties with the modification of the coir-polyester composites. Mohanty et al.[12] modified pineapple leaf fibers by grafting them with polyacrylinitrile to improve their properties. Varada Rajulu et al.[13] coated bamboo fibers with an epoxy/polymethyl methyl methaceylate blend to improve their tensile properties.

Recently, we identified a new uniaxial bark fabric from the tree *Sterculia urens* (Roxb). In order to assign possible applications, it has to be characterized. In the present work, the authors characterized the fabric using chemical, FT-IR, X-ray, thermogravimetric, microscopic, and mechanical testing methods. The authors also studied the effect of alkali treatment of the fabric on its properties. The authors selected this fabric for study because uniaxial natural fabrics are rare in occurrence.

MATERIALS AND METHODS

Extraction of Fabric from the Tree

The tree *Sterculia urens* from which the fabric was extracted belongs to the family Sterculiacea. The average length, breadth, and thickness of the fabric were found to be 300–700 mm, 70–100 mm, and 0.18 mm, respectively. The fabric was washed thoroughly with distilled water and allowed to dry in the sun for about one week. Some quantity of the fabric was treated with 5% aqueous NaOH solution to remove the hemicellulose, lignin, and other greasy materials.

FT-IR Spectral Analysis

One of the samples was cryogenically cooled and powdered. This powder was diluted to 1% using KBr, and pellets were prepared employing a hydraulic press. The Fourier transform-infrared (FT-IR) spectra of the untreated and the alkali-treated samples were recorded in the 4000–500 cm^{-1} region on a Perkin Elmer 16PC FT-IR instrument with 32 scans in each case at a resolution of 4 cm^{-1}.

Chemical Analysis

The *Sterculia urens* fabrics (untreated and alkali-treated) were preconditioned before cellulose extraction took place. The fabrics were washed with distilled water several times and dried in an oven at 80°C for 24 h. Then they were chopped to an approximate length of 5–10 mm. Finally, a de-waxing step was carried out by boiling the fabric in a toluene/ethanol (2:1 volume/volume) mixture in a Soxhlet for 6 h. The de-waxed fabrics were then filtered, washed with ethanol for 30 min, and dried. Subsequently, these were used for cellulose extraction. Initially by treating these fabrics with 0.7 w/v% sodium chlorite Na-ClO_2 the lignin was gradually removed. The remaining holocellulose (α-cellulose + hemicellulose) was then treated with 17.5 w/v% NaOH solution to remove hemicellulose. By this elimination and isolation process,[14,15] the percentage of α-cellulose, hemicellulose, and lignin were determined. In each case, five samples were used and the average values are reported.

Thermogravimetric Analysis

The thermograms of the fabric before and after alkali treatment were recorded on a Perkin Elmer TGA-7 instrument in nitrogen atmosphere at a heating rate of 10°C/min.

X-ray Analysis

The X-ray diffraction (XRD) experiments were performed using a Rigaku Dmax 2500 diffractometer. The system has a rotating anode generator with a copper target and a wide-angle powder goniometer. The generator was operated at 40 kV and 150 mA. All the experiments were performed in the reflection mode at a scan speed of 4°/min in steps of 0.05°.

Morphology

Scanning electron micrographs of the fabric before and after alkali treatment were recorded on a JEOL JSM 820 microscope. The fabric samples were gold coated before recording the micrograms. Polarized optical micrographs were recorded using a Leica DMLP Polarized Optical Microscope.

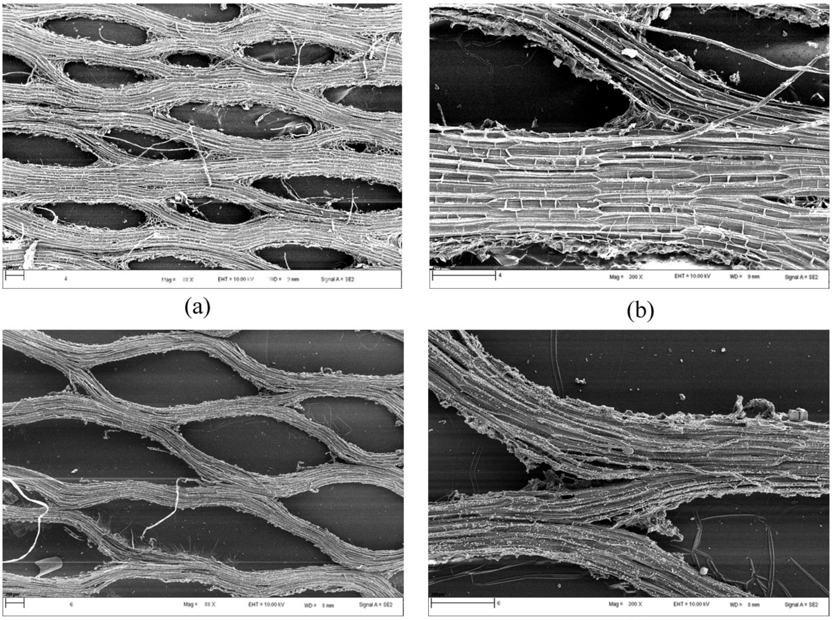
Tensile Properties

Tensile properties such as maximum stress, Young's modulus, and percent elongation at break were determined using an INSTRON 3369 Universal Testing Machine at a crosshead speed of 3 mm/min maintaining a gauge length of 50 mm. In each case, 10 samples were used and the average values are reported.

RESULTS AND DISCUSSION

The tree *Sterculia urens* from which the fabric was extracted belongs to the family of Sterculiacea. Scanning electron micrographs of the untreated and the alkali-treated fabrics are shown in Figure 1(a), (b), (c), and (d) at different magnifications. From these micrographs, it is evident that the fabric is made up of uniaxial roughly parallel fibers. Further, at higher magnification, the void regions present in the fabric are clearly visible. The micrographs also reveal a white layer on the untreated fabric, which may be due to the hemicellulose component. Upon alkali treatment, the white layer content is found to decrease. This may be attributed to the reduction in the hemicellulose content with alkali treatment. Further, the surface of the fabric is found to become rough with alkali treatment.

Polarized optical micrographs of the *Sterculia urens* fabric before and after alkali treatment are presented in Figure 2. The images for the untreated fabric (Figure 2(a) and (b)) are found to be diffuse. This may be due to the presence of the amorphous hemicellulose layer on the surface of the fabric. However, the images in the micrographs of the alkali-treated fabric (Figure 2(c) and (d)) are found to be sharp and bright. This may be due to the elimination of hemicellulose to large extent upon alkali treatment and subsequent increase in the birefringence in the fabric. The optical micrographs corresponding to alkali treatment indicate the formation of micro cracks on the surface of the fabric, as well as a decrease in the thickness of the fabric.



(a) (b) (c) (d)

Figure 1. Scanning electron micrographs of *Sterculia urens* fabric: (a) and (b), untreated and (c) and (d) alkali-treated fabric at different magnifications.

FT-IR spectra of the untreated and the alkali-treated fabric are presented in Figure 3. The band positions and possible assignments are given in Table I. From Figure 3, it can be observed that in the case of untreated fabric, there are well-defined bands at around 3420, 2920, 1630, 1300, and 1030 cm^{-1} in the spectra. Further, there is another band at around 1731 cm^{-1} corresponding to the hemicellulose content. On alkali treatment, the intensity of this band is found to decrease, indicating a reduction in the hemicellulose content. From Table I, it can be seen that the bands around 3420 cm^{-1} and 2920 cm^{-1} correspond to α-cellulose, whereas the remaining bands correspond to lignin. Further, for the alkali-treated fabrics, the intensity of the bands corresponding to α-cellulose increased.

The chemical analysis of the untreated and the alkali-treated *Sterculia urens* fabrics is presented in Table II. From this table, it is evident that on alkali treatment, the percentage of hemicellulose and lignin decreased and, as a result, α-cellulose content increased. This is in conformity with the observations made in the FT-IR analysis.

X-ray diffractograms of the untreated and alkali-treated fabric are shown in Figure 4. From this figure, it is evident that the intensity of

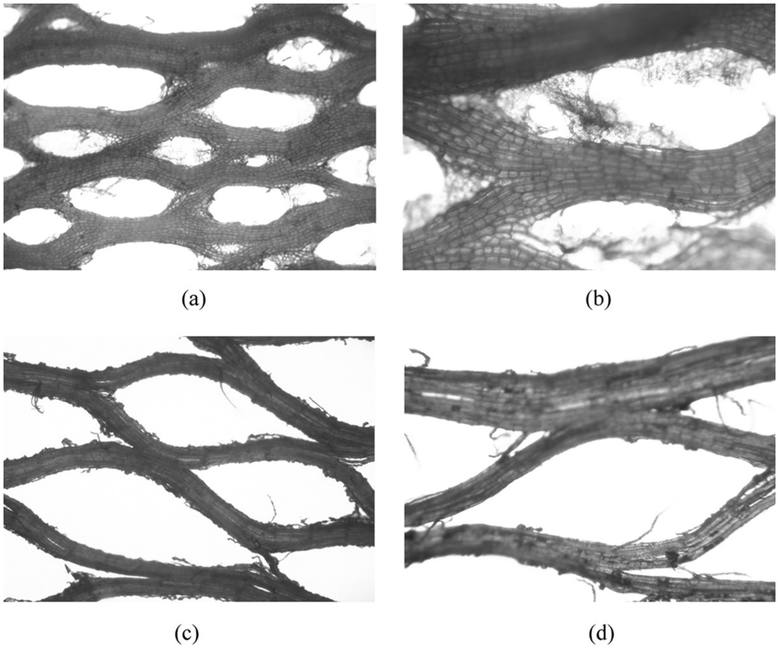


Figure 2. Polarized optical micrographs of *Sterculia urens* fabric: (a) and (b) untreated and (c) and (d) alkali-treated fabric at different magnifications.

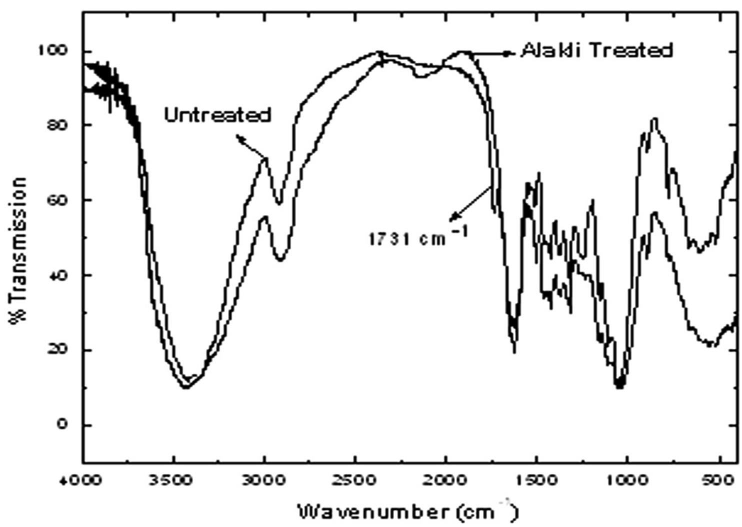


Figure 3. FT-IR spectra of untreated and alkali-treated *Sterculia urens* fabric.

Table I. Peak positions and assignments of chemical groups in untreated and alkali-treated *Sterculia urens* fabrics

Wave number (cm^{-1})		
Untreated	Alkali treated	Assignments
3421	3438	O-H stretching of α-cellulose
2912	2913	Alkyl CH stretching
1731	—	C=O stretching of hemicellulose
1623	1631	Absorbed water
1432	1432	CH_2 symmetric bending
1324	1336	CH bending (deformation)
1051	1034	Symmetric C-OH stretching of lignin

the peaks of alkali-treated fabric is higher than that of the untreated fabric. This indicates an increase in crystallinity of the fabric by alkali treatment. The elimination of amorphous hemicellulose by alkali treatment may be responsible for the increase in crystallinity. This is in conformity with the observations made by the FT-IR spectra and chemical and polarized microscopic analyses.

Primary thermograms of the untreated and alkali-treated fabrics are presented in Figure 5. Using these thermograms, the initial degradation temperature, the final degradation temperature, and the inflection point (where the degradation rate is maximum) were calculated and are presented in Table III. Using the Doyle[16] method, the refractoriness (T^*) and the thermal stability index-IPDT were determined. These values are also presented in Table III. From the table it is clearly evident that the initial degradation temperature of the alkali-treated fabric is slightly higher than that of the untreated fabric. A similar observation was also made for the inflection point and final degradation temperatures. The decrease in amorphous hemicellulose and lignin with alkali treatment may be the reason for this observation. Further, these results indicate

Table II. Chemical analysis of untreated and alkali-treated *Sterculia urens* fabrics

Component	Untreated (%)	Alkali treated (%)
α-Cellulose	62.9	81.5
Hemicellulose	24.3	7.5
Lignin	12.0	10.8

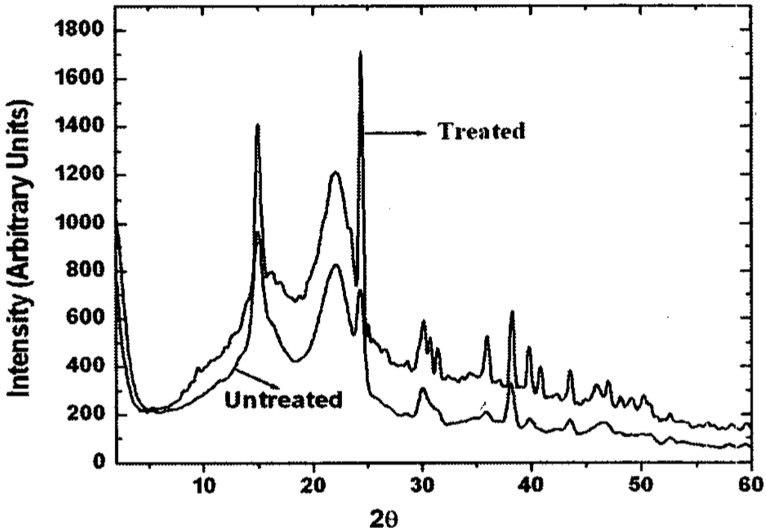


Figure 4. X-ray diffractograms of untreated and alkali-treated *Sterculia urens* fabric.

that alkali-treated *Sterculia urens* fabrics are favorable for reinforcement materials for composites, even with thermoplastic matrices whose processing temperature is below 240°C.

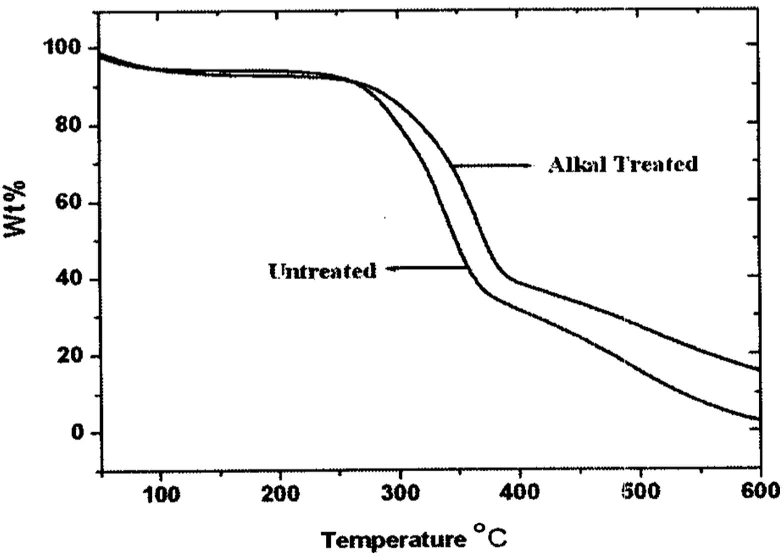


Figure 5. Thermograms of untreated and alkali-treated *Sterculia urens* fabric.

Table III. Thermal degradation parameters of untreated and alkali-treated *Sterculia urens* fabric

Degradation parameter (°C)	Untreated	Alkali treated
Initial degradation temperature	226	239
Final degradation temperature	394	401
Inflection point	341	361
IPDT	141	236
Refractoriness (T*)	164	316

Table IV. Tensile properties of untreated and alkali-treated *Sterculia urens* fabrics

Parameter	Untreated	Alkali treated
Maximum stress (MPa)	10.03	18.92
Young's modulus (MPa)	640.70	2018.67
% Elongation at break	2.00	2.47

The tensile parameters of the untreated and alkali-treated *Sterculia urens* fabric are presented in Table IV. From this table, it is evident that the maximum stress, the modulus, and elongation at break of the alkali-treated fabric increased on alkali treatment. This may be due to the increase in crystallinity due to the elimination of amorphous hemicellulose to a larger extent with alkali treatment. Further, due to its higher modulus values, renewable nature, and biodegradability, the uniaxial natural fabric *Sterculia urens* can be considered as reinforcement in the preparation of green compositions. The unique uniaxial nature of this fabric can be exploited to control the mechanical properties of the composites by properly orienting it towards the stress direction.

CONCLUSIONS

The newly identified uniaxial natural fabric *Sterculia urens* was characterized by chemical, FT-IR, X-ray, microscopic, thermogravimetric, and mechanical testing methods. The FT-IR and chemical analyses indicated lowering of hemicellulose and lignin content with alkali treatment. The XRD and optical microscopic analyses revealed an increase in crystallinity with alkali treatment. The thermal stability and tensile properties of this fabric increased with alkali treatment. Due to its higher modulus, this natural fabric can be put to use as reinforcement in green composites.

REFERENCES

[1] George, J., S. S. Bhagawan, and S. Thomas. (1998). Improved interactions in chemically modified pineapple leaf fiber reinforced polyethylene composites. *Compos. Interfaces*. **5**, 201–223.

[2] Bledzki, A. K., S. Reihmane, and J. Gasan. (1996). Properties and modification methods for vegetable fibers for natural fiber composites. *J. Appl. Polym. Sci*. **59**, 1329–1336.

[3] Amada, S., T. Munekata, Y. Nagese, Y. Ichikawa, A. Kirigai, and Y. Zhifei. (1996). The mechanical structure of bamboos in view point of functionally gradient and composite materials. *J. Compos. Mater*. **30**, 800–819.

[4] Varada Rajulu, A., X. H. Li, L. Ganga Devi, G. Babu Rao, and Y. Z. Meng. (2003). Tensile properties of ligno-cellulose fabrics coated with styrenated polyester resin. *Cellulose*. **10**, 179–183.

[5] Mannan, K. H. M., and M. D. S. Munir. (2000). Characterization of jute fibers treated with soap-glycerol micelles. *J. Appl. Polym. Sci*. **77**, 852–856.

[6] Gañán, P., J. Cruz, S. Gabizu, A. Arbelaiz, and I. Mondragon. (2004). Stem and bunch banana fibers from cultivation wastes—Effect of treatments of on physico- chemical behavior. *J. Appl. Polym. Sci*. **94**, 1489–1495.

[7] d'Almedia, J. R. M., R. C. M. P. Aquino, and S. N. Monteiro. (2006). Tensile mechanical properties, morphological aspects and chemical characterization of piassava (Attaleafunifera) fibers. *Composites Part A*. **37**, 1473–1479.

[8] Mohanty, S., and S. K. Nayak. (2006). Mechanical and rheological characterization of treated jute-HD composites with a different morphology. *J. Reinf. Plast. Compos*. **25**, 1419–1439.

[9] Varada Rajulu, A., Y. Z. Meng, X. H. Li, G. Babu Rao, L. Ganga Devi, K. Mohana Raju, and R. Ramakrishna Reddy. (2003). Effect of alkali treatment on properties of the lignocellulose fabric Hildegardia populifolia. *J. Appl. Polym. Sci*. **90**, 1604–1608.

[10] Hill, C. A. S., and H. P. S. Abdul Khalil. (2000). Effect of fiber treatments on mechanical properties of coir or oil palm fiber reinforced polyester composites. *J. Appl. Polym. Sci*. **78**, 1685–1697.

[11] Rout, J., M. Misra, S. S. Tripathy, S. K. Nayak, and A. K. Mohanty. (2001). The influence of fiber surface modification on the mechanical properties of coir-polyester composites. *Polym. Compos*. **22**, 468–476.

[12] Mohanty, A. K., P. C. Tripathy, M. Misra, S. Parija, and S. Sahoo. (2001). Studies on the effect of chemical modification of pine apple leaf fibre by graft copolymerization of acrylonitrile. *J. Appl. Polym. Sci*. **77**, 3035–3043.

[13] Varada Rajulu, A., G. Babu Rao, and R. Lakshminarayana Reddy. (2000). Chemical resistance and tensile properties of epoxy/polymethylmethyl methcrylate blend coated bamboo fibres. *Indian J. Fibre Text. Res*. **25**, 295–297.

[14] Chattopadhyay, H., and P. B. Sarkar. (1946). A new method for the estimation of cellulose. *Proc. Natl. Inst. Sci. India*. **12**, 23–46.

[15] Sarkar, P. B., A. K. Mazumdar, and K. B. Pal. (1948). The hemicelluloses of jute fibre. *J Text. Inst*. **39**, 44–58.

[16] Doyle, C. D. (1985). Thermogravimetric analysis. In *Encyclopedia of Polymer Science and Engineering*, ed. J. J. Kroschwitz, Vol. 14, no. 40, 1–41. New York: Wiley Interscience.