EVALUATION OF THE POTENTIAL OF ALTERNATIVE VEGETABLE MATERIALS FOR PRODUCTION OF PAPER THROUGH KRAFT PROCESSES

JUAN DOMÍNGUEZ ROBLES,[*] EDUARDO ESPINOSA VÍCTOR,[*]
MARIA DEL VALLE PALENZUELA RUÍZ,[**] MARIA EUGENIA EUGENIO MARTÍN,[***]
ALEJANDRO RODRÍGUEZ PASCUAL[*] and ANTONIO ROSAL RAYA[**]

[*]*Department of Inorganic Chemistry and Chemical Engineering, University of Córdoba, 14014 Córdoba, Spain*
[**]*Department of Molecular Biology and Biochemical Engineering, Pablo de Olavide University, 41013 Seville, Spain*
[***]*Laboratory of Cellulose and Paper – INIA, Forest Research Center, 28040 Madrid, Spain*
✉ *Corresponding author: A. Rosal Raya, arosray@upo.es*

Received June 26, 2019

The use of biomass resources different from conventional wood materials fosters the sustainable growth of the paper industrial sector and finds a development path in line with the concept of circular bioeconomy. In this work, six non-wood materials (*Leucaena leucocephala*, tagasaste, rice straw, *Paulownia fortunei*, *Hesperaloe funifera* and empty fruit bunches) were tested and compared to *Eucalyptus globulus* for paper production under Kraft conditions. All the raw materials were chemically characterized to determine holocellulose, cellulose, Klason lignin, ash, hot water solubles, 1% soda solubles and ethanol-benzene extractives. In addition, a biometric test was performed to determine the length and the width of the fibres. The cellulosic pulps obtained from the raw materials were characterized to determine their yield, viscosity, Kappa number and drainage index. As regards the paper sheets made from the cellulosic pulps, they were characterized to determine brightness, stretch and tear index. A comparison of the results suggests that these non-wood species can be used for papermaking, under Kraft operating conditions, when high-brightness paper is not required.

Keywords: cellulose, *Eucalyptus globulus*, *Hesperaloe funifera*, *Leucaena leucocephala*, *Paulownia fortunei*, pulping, rice straw, tagasaste

INTRODUCTION

The use of non-wood raw material by the pulp and paper industry entails an alternative move towards sustainability and offers a viable solution to meet the paper demand within areas characterized by a deficit of forest resources.[1] However, this use is not exempt from difficulties. In an occasional manner, low productivity, combined with transport and storage difficulties, hampers the application of non-conventional fibres. Consequently, the potential use of non-wood raw materials for pulp and paper manufacturing depends on the efficiency of industrial production and also on the geographic location of the crops. Nowadays, many regions of Europe tend to use recycled paper rather than non-wood raw materials, whereas specific Asian and Latin American regions, with insufficient forest resources, strongly lean towards different materials other than conventional cellulose sources, such as *Eucalyptus globulus* and *Pinus pinaster*.[2]

Amongst the alternative sources, we find agricultural and food industry residues, such as rice straw and empty fruit bunches (EFB) from the palm oil industry.[3,4] Other alternative sources are fast-growth and low water-demand plants, such as *Hesperaloe funifera* of the Agavaceae family (cultivated only for ornamental use),[5] *Leucaena leucocephala*, an invasive species of the leguminous family,[6] and *Chamaecytisus proliferus L.F. ssp. palmensis* (tagasaste) and *Paulownia fortunei*, both shrubs being used in agroforestry systems to assure protection against wind and for erosion control, as well as an amendment for soil, to improve its nutrient contents.[7,8] These alternative raw materials have attracted considerable interest in the pulp and paper industry due to their short-growth cycles, as well as high dry biomass contribution (each hectare produces an average of 5-10 tons), easy pulping, excellent fibres for special types of paper, and low lignin content, which reduces the energy consumption and the use of chemicals during pulping.[9]

These advantages make these raw materials not only potential sources of cellulose/hemicellulose for the paper industry, but also, as indicated by various studies, resources to be used for other purposes, such as the production of bioethanol,[10] xylo-oligosaccharides,[11] nanofibres,[12] cardboard,[13] or the generation of electricity and H_2.[14]

There are many studies concerning the use of the alternative raw materials analysed here for pulp manufacturing by means of both traditional and Organosolv processes.[7,8,15-23] However, they mostly tend to optimize specific pulping conditions for each raw material in order to achieve the highest possible yield, without reducing the pulp properties required by the industry. This optimization may hinder the alternative material from being applied on an industrial scale, especially, taking into consideration a certain degree of reluctance to modify *in situ* already standardized processes of production.

The objectives of this study were (a) to analyse the chemical, physical and morphological properties of raw materials and pulps from *Leucaena leucocephala*, tagasaste, rice straw, *Paulownia fortunei*, *Hesperaloe funifera* and EFB, which were subjected to pulping under Kraft conditions; (b) to determine the brightness, stretch and tear index of the obtained paper sheets; and (c) to compare the results with those achieved with conventional hardwood fibres from *Eucalyptus globulus*, which had been subjected to pulping under the same process conditions.

EXPERIMENTAL

Raw materials

Seven species of raw materials were selected for this study: *Leucaena leucocephala* and tagasaste were collected from experimental plantations of the research team of the Forest Science Department at University of Huelva (in the southwest of Spain); rice straw was obtained from several farming cooperatives from Valencia (in the east of Spain); *Paulownia fortunei* was supplied by Vicidex Europe and the material was obtained from crops grown in Extremadura (in the southwest of Spain); *Hesperaloe funifera* was kindly supplied by the *Hesperaloe* project research team at the University of Arizona (USA); EFB, a residue of oil palm from Malaysia, was provided by the company Straw Pulping Engineering, S.L., Zaragoza (in the north of Spain); and *Eucalyptus globulus* was received from a paper-industry company Torraspapel Montañanesa Group from the same town.

Raw material characterization

The raw material was conditioned before being used. The material was first air-dried and then homogenized in a single stock (being placed inside polyethylene bags) to avoid variations in the composition and water content. Undesired particles, such as stones or seeds, were discarded. After being cleaned, under room temperature and moisture conditions, the raw material was ground in a mill (Retsch SM 2000) and then stored in polyethylene bags at constant temperature (25 ºC).

For characterization, the raw material was sieved, to obtain a fraction with the size between 0.25-0.40 mm (60 and 40 Tyler meshes). This fraction was stored at room temperature until the experimental analysis. This choice of the fraction size was based on the fact that fractions with a particle size over 0.40 mm are not easily attacked by chemical reagents and fractions with a particle size below 0.25 mm could cause filtration problems.

The raw materials were characterized analysing their content of acid-insoluble (Klason) lignin, α-cellulose, hot water soluble components, 1% NaOH soluble components, ethanol-benzene extractives and ash, which were determined in accordance with Tappi standards: T222om-98, T203om-93, T207om-88, T212om-88, T204om-02 and T-211om-02, respectively.[24] Also, their content of holocellulose was analysed, which was quantified using the method of Wise *et al.*[25] Fibre length and width were biometrically determined under a Visopan projection microscope, with 10X objective for 100X magnification, after microcooking the raw material with 10% of soda at 80 ºC for 1 hour, and subsequently staining the fibres with 1% of safranin.[26-29]

Pulping conditions

The raw materials were cooked in a 15-litre batch reactor, where they were stirred by rotating the vessel *via* a motor connected through a rotary axle to a control unit including the required instruments for measurement and control of pressure and temperature. The vessel was furnished with an outer heating jacket to facilitate the attainment of the working temperature.

Each raw material was ground into pieces of 2-5 cm and washed in the reactor for 10 minutes at 110 ºC.

After washing, the raw material fibres were placed in the reactor along with the reagents of the Kraft process (16% alkalinity and 25% sulfidity, all expressed as Na_2O) and a liquid/solid ratio of 4. The process was performed at 170 ºC (time to reach maximum temperature, 90 min; time at maximum temperature, 40 min). The operating conditions were established based on the results of other authors who worked with these types of raw materials and these conditions are similar to those used on an industrial scale.[30-32]

After pulping, the cooked material was washed to remove residual cooking liquor and fiberized in a disintegrator at 1200 rpm for 30 minutes. The pulp was beaten in a Sprout-Bauer refiner using a disk spacing of 0.1 mm. The fiberized material was passed through a filter with a pore size of 0.16 mm, in order to remove uncooked particles (Sommerville screen model, K134). Finally, the pulp was drained in a centrifuge and allowed to dry to a moisture content of ca. 10% at room temperature.

Pulp properties

The pulp yield was determined gravimetrically in each case. Also, Kappa number, viscosity and drainage index (in a Shopper-Riegler apparatus) of the pulps were determined according to UNE 57034, UNE 57039 and UNE 57025, respectively.[33] Kappa number and viscosity were measured five times and three measurements of drainability were obtained in order to calculate the relative standard deviation.

Characterization of paper sheets

Paper sheets were obtained on an Enjo-F39.71 sheet former according to Tappi-T205sp-95.[24] The paper obtained was characterized for brightness, stretch and tear index in accordance with the following applicable standard methods: UNE 57062, UNE 57054 and UNE 57033, respectively.[33]

RESULTS AND DISCUSSION

Raw material. Chemical composition

The chemical composition of the materials has a considerable impact on the pulp yield and fibre properties.[34] Table 1 presents the results of chemical characterization (the results are on dry matter basis) and biometric analysis of raw materials.

The contents of ash of *Leucaena leucocephala* (1.3%), tagasaste (1.0%) and *Paulownia fortunei* (0.1%) were similar to the contents of *E. globulus* found in this study (the same order of magnitude) and to the results reported in several previous works for other wood species used in the paper industry.[19,35] However, rice straw (12.8%), *Hesperaloe funifera* (7.2%) and EFB (5.3%) presented significantly higher ash contents; although, with these percentages of inorganic matter, the effects of abrasion are usually not significant.[36]

On the other hand, the percentage of extractable components with regard to the total content was between 45-70% for the alternative raw materials (sum of hot water solubles, soda solubles and ethanol-benzene extractables). These results were also higher than the percentage of extractable compounds in the total content for *E. globulus* (16.4%), and compared to the results provided by other authors who worked with other species of *Eucalyptus*, as well as with pine and acacia.[37-39] The content of 1% NaOH solubles was higher than the rest of extractable compounds. The values of alternative materials were between 27-50% and, in the case of *E. globulus*, it was 12.4%. The presence of these compounds could cause problems, as they adhere to industrial machinery and reduce the quality of the pulp.[8,16] In general, pulp mass yield decreases with higher extractable contents.

Among other factors, ash, lignin and total extractable contents influence the optical properties of paper sheets. In general terms, the higher the content of these compounds the lower the brightness of the sheet. The contents of lignin in *Leucaena leucocephala*, tagasaste, *Paulownia fortunei* and EFB were similar to those in *E. globulus* (around 20%). However, the contents of lignin in rice straw and *Hesperaloe funifera* were significantly lower (around 10%). Lignin is a hydrophobic constituent, which is the reason why a high proportion of pulp would inhibit water absorption, making the refining difficult. Also, the lignin content can influence the reaction time of delignification in the digester or the reagent concentration. This fact has an impact on the economics of the paper production process and an environmental impact.[34,36]

The contents of holocellulose (76.5%) and cellulose (52.3%) of *Hesperaloe funifera* were similar to those of *E. globulus*; by contrast, in the remainder of materials, these contents were lower (on average 68.6% and 41.3%, respectively). In any case, the values obtained for these parameters in the alternative materials studied were similar to the results revealed by other studies for hardwood and softwood commonly used in the wood fibre industry.[35,39-43]

Table 1
Chemical and biometric characteristics of alternative raw materials compared with those of *E. globulus*

	Ash (%)	Hot water sol. (%)	1%soda sol. (%)	ET-BE ext. (%)	LI (%)	HO (%)	α-CE (%)	CE/LIr atio	FL (mm)	FW (µm)	FL/FW ratio
Leucaena leucocephala	1.3	12.9	30.9	1.8	20.3	68.6	39.3	1.9	0.7	29.0	24.1
Tagasaste	1.0	9.8	32.8	2.7	17.2	73.4	39.7	2.3	0.9	20.2	44.6
Rice straw	12.8	18.8	49.5	1.5	10.1	64.0	39.5	3.9	1.3	7.4	175.7
Paulownia fortunei	0.1	12.1	27.5	6.4	17.8	69.7	40.9	2.3	0.9	35.3	25.5
Hesperaloe funifera	7.2	17.9	36.2	2.2	7.24	76.5	52.3	7.2	4.2	7.2	583.3
EFB	5.3	14.1	32.0	0.8	17.0	67.3	47.1	2.8	1.7	14.0	121.4
Eucalyptus globulus	0.6	2.8	12.4	1.2	20.0	80.5	52.8	2.6	1.0	13.0	76.9

Mean values are presented (n=3). Standard deviations of the three replicates with respect to the means were always less than 10%. All percentages are on dry matter basis

Solubles (sol.), ethanol-benzene extractables (ET-BE ext.), lignin (LI), holocellulose (HO), α-cellulose (α-CE), hemicellulose (HE), fibre length (FL), fibre width (FW)

In this study, the cellulose/lignin ratios of *Leucaena leucocephala*, tagasaste and *Paulownia fortunei* were slightly lower than the ratio determined for *E. globulus*. As for rice straw and EFB, these were slightly higher, and in the case of *Hesperaloe funifera*, significantly higher (about three times on average). For all papermaking processes, a high ratio of cellulose/lignin is preferred. High cellulose content, generally, produces paper with stable brightness, high tensile strength and good resistance to deformation.[8]

Table 1 also shows the results of fibre morphology determinations. The observations made in this investigation indicated that there are remarkable differences in the fibre length of *Hesperaloe funifera*, compared to the other species analysed. All the values were similar (around 1 μm), except for *Hesperaloe funifera*, which had a fibre length four times higher than the average of the others. Thus, the fibre length correlates with the mechanical properties of paper, which, to a certain extent, tend to improve if the fibre length increases.[35] Consequently, the morphology of the *Hesperaloe funifera* could be the most appropriate one for manufacturing paper sheets with good physical-mechanical properties.

Last but not the least, we determined the width of each fibre type. This parameter is related to fibre flexibility. It can be expected that paper manufactured from thin fibre is dense and well-formed.[8] The results show that the fibre width values of *Leucaena leucocephala*, tagasaste and *Paulownia fortunei* were higher, those of EFB were similar and those of rice straw and *Hesperaloe funifera* were lower than those of *Eucalyptus globulus*.

In paper manufacturing, the ratio of fibre length to fibre width (slenderness) is of interest. A high value of this ratio provides better handsheet formation and a well-bonded paper.[8] In this study, this ratio was lower in the non-wood materials than in *E. globulus*; with the exception of EFB, rice straw and *Hesperaloe funifera*, whose ratios were higher, about 1.5, 2.0 and 7 times, respectively. In any case, all the fibres presented relatively good slenderness ratios, resulting in fibre flexibility, suitable for fibre bonding.[16]

Properties of cellulose pulps

The yield, viscosity, Kappa number and drainage index for the pulps of alternative species and *E. globulus* are presented in Table 2. As can be seen, the yields for non-wood species were lower than that obtained for *E. globulus* (51.2%) in this study, and even lower than the one provided by Alaejos *et al*. (2006)[44] in his work on Kraft processes applied to softwoods, such as the elm (50.5%). This may be due to the high extractable content that was noted (see Table 1). *Leucaena leucocephala* presented the lowest yield (26.4%); *Hesperaloe funifera* presented the highest yield (46.2%) and the percentage yields for the other species were about 41.0%. The yields were lower than the results obtained for these types of raw materials with an Organosolv process,[4,18-20,45] although similar to the ones obtained by Kraft processes with other alternative resources, such as banana agricultural waste[46] and vine shoots,[47] and even the ones determined in other wood species of *Eucalyptus* (*citriodora*, *tereticornis*).[48]

The viscosity of the alternative raw materials was similar to the values of viscosity determined for *E. globulus* (832 $cm^3 \cdot g^{-1}$), with the exception of *Leucaena leucocephala* and tagasaste, which were significantly different, 462 $cm^3 \cdot g^{-1}$ and 1198 $cm^3 \cdot g^{-1}$, respectively. With the exception of tagasaste, all the pulps from the alternative raw materials studied presented higher values than the ones provided by Vargas *et al*. (2012) for wheat straw (536.0 $cm^3 \cdot g^{-1}$) used in a Specel® process. A pulp with relatively low initial viscosity would be problematic in bleaching industrial processes, since they cause further degradation of the cellulose in the fibre and, consequently, the viscosity would be further reduced.[36]

All the alternative pulps presented Kappa numbers higher than that of *E. globulus* (between 42.2-122.6 vs 28.0), with the exception of rice straw and *Hesperaloe funifera*, whose Kappa numbers were lower (11.3 and 22.3, respectively). However, the lignin content of *E. globulus* was similar to the content of *Leucaena leucocephala* and higher than those of other alternative species. These results can be explained by the fact that (1) lignin could be condensed and, therefore, its extraction becomes difficult; and (2) it can cause interference because of the presence of hexenuronic acids formed during the Kraft process.[49]

Finally, Table 2 presents the ºSR values obtained for each pulp. Pulp refining can help improve the final properties of paper sheets.[19] All the ºSR values of the pulps obtained from the alternative species were similar to that of *E. globulus* (12.3), except that of *Hesperaloe funifera*, which was higher (19.8), although of the same order; which allows for the comparison of the physical properties of the different sheets made from each pulp.

Properties of paper sheets

Table 3 shows the brightness, stretch and tear index results for the paper sheets. In general, a comparison of the results for the handsheets made from the alternative species and *E. globulus* revealed that the brightness percentages for the alternative species were lower than those for the wood species. In contrast, in almost all the cases, stretch and tear index were higher for the alternative species than for *E. globulus*.

The brightness percentage for *E. globulus* was 27.2%, compared to the percentages of rice straw and *Hesperaloe funifera* (24.3% and 20.0%, respectively), which were the highest ones among the non-wood species. Precisely, the lignin contents and Kappa numbers for rice straw and *Hesperaloe funifera* were lower than those for the other species. Anyhow, the brightness percentages were lower than the results obtained for these species subjected to pulping with organic solvents (Ethanolamine, Diethylenglycol, Diethanolamine, Ethyleneglycol).[19,45,50]

Table 2
Properties of alternative biomass pulps compared with those of *E. globulus* pulp

Raw material	Yield (%)	Viscosity ($cm^3 \cdot g^{-1}$)	Kappa number	Drainage index (ºSR)
Leucaena leucocephala	26.4	462.3	122.6	13.2
Tagasaste	41.4	1198.1	79.5	11.6
Rice straw	38.0	890.2	11.3	13.6
Paulownia fortunei	42.4	944.1	154.0	12.4
Hesperaloe funifera	46.2	855.4	22.3	19.8
EFB	43.2	814.3	42.2	13.3
Eucalyptus globulus	51.5	832.1	28.0	12.7

Mean values are presented: viscosity (n=5), Kappa number (n=5) and drainage index (n=3). The relative standard deviations of the replicates in each test with respect to the means were always ≤10%. All percentages are on dry pulp basis

Table 3
Properties of paper sheets made from alternative biomass pulp compared with those of *E. globulus* handsheets

Raw material	Brightness (%)	Stretch index (%)	Tear index ($mN \cdot m^2 \cdot g^{-1}$)
Leucaena leucocephala	14.0	1.1	2.5
Tagasaste	16.5	1.0	2.8
Rice straw	24.3	1.1	2.4
Paulownia fortunei	15.4	0.9	2.4
Hesperaloe funifera	20.0	1.7	5.8
EFB	17.2	1.3	2.9
Eucalyptus globulus	27.2	0.7	2.4

Mean values are presented. In each test, two measurements were made on ten handsheets. The results are on dry matter basis. The standard deviations of replicates in each test with respect to the means were always less than 10%

Stretch and tear index are related to fibre morphological characteristics and intrinsic resistance. The greater the degree of refining, the stronger the influence wielded on the resistance.[51] Scott and Abbot (1995)[46] indicated that the morphological characteristics of the fibres that influence the properties of resistance of paper are fibre length (FL), fibre width (FW), and their ratio (FL/FW). It is known that long and slender fibres, and consequently high ratios, improve mechanical properties.[53] Gurnagul *et al.*[54] indicated that chemical composition affects fibre resistance. It is known that fibre resistance decreases with cellulose degradation for some types of Kraft pulp. In this study, fibre length, the FL/FW ratio, and holocellulose and α-cellulose contents for

Hesperaloe funifera (4.2 mm, 583.3, 76.5%, 52.3%, respectively) were higher. These results can explain that the stretch index (1.7%) and tear index (5.8 $mNm^2 \cdot g^{-1}$) for these species were higher. Stretch and tear indexes for the remainder of the studied non-wood materials were between 0.9-1.3% and 2.4-2.9$mNm^2 \cdot g^{-1}$, respectively. These results can be explained by slight variations of the FL/FW ratios and of cellulose and lignin contents. In the case of *Eucalyptus globulus*, the FL/FW ratio was between the values indicated and holocellulose, cellulose and lignin contents were higher; nevertheless, the stretch index (0.70%) and tear index (2.40 $mNm^2 \cdot g^{-1}$) were low. This could be caused by deeper cellulose degradation than in the case of the other species.

In any case, the results of physical properties achieved for the handsheets made from the alternative species Kraft pulps were better than those obtained for paper sheets made by an ecological process[17,18,21,22,55,56] and those made by the Specel® process.[36]

CONCLUSION

The results of this study revealed that the chemical composition and morphology of the fibres of *Leucaena leucocephala*, *Paulownia fortune* and tagasaste were similar to the ones of *Eucalyptus globulus*, whereas in the case of rice straw, EFB and *Hesperaloe funifera*, these properties were better than those of *Eucalyptus globulus*.

With regard to the analysis of the physical properties, all the handsheets made from alternative vegetable materials presented stretch and tear index values higher than the ones obtained for the handsheets prepared from *Eucalyptus globulus* Kraft pulp (more than double in the case of *Hesperaloe funifera*). Nevertheless, the brightness index was lower in all the cases (a difference between approximately 3 and 13 percentage points).

These results justify the industrial use of this type of alternative biomass species with Kraft processes for the production of cellulosic pulp and paper grades that do not require high brightness.

ACKNOWLEDGEMENT: The authors would like to acknowledge the Ministry of Economy and Competitiveness of Spain (Project CTQ2016-78729-R), the University of Córdoba (RNM 2323) and the University Pablo de Olavide of Seville (PPI 1401). Finally, we would like to thank MatchBetter Translations, namely Carmen Torrella, for translating and reviewing our paper.

Conflict of interest statement: All the authors and the responsible authorities as well have agreed on and authorized the publication of this manuscript upon the final revised version. There is no conflict of interest.

REFERENCES

[1] E. D. Sitz and D. S. Bajwa, *Ind. Crop. Prod.*, **75**, 200 (2015), https://doi.org/10.1016/j.indcrop.2015.05.006

[2] R. Miranda, E. Bobu, H. Grossmann, B. Stawicki and A. Blanco, *Cellulose Chem. Technol.*, **44**, 419 (2010), http://www.cellulosechemtechnol.ro/pdf/CCT10(2010)/P.419-430.pdf

[3] L. Jiménez, L. Serrano, A. Rodríguez and R. Sánchez, *Bioresour. Technol.*, **100**, 1262 (2009), https://doi.org/10.1016/j.biortech.2008.08.013

[4] M. Nuruddin, A. Chowdhury, S. A. Haque, M. Rahman, S. F. Farhad *et al.*, *Cellulose Chem. Technol.*, **45**, 347 (2011), http://www.cellulosechemtechnol.ro/pdf/CCT45,5-6(2011)/p.347-354.pdf

[5] A. Rosal, C. Valls, A. Ferrer and A. Rodríguez, *Cellulose Chem. Technol.*, **46**, 105 (2012), http://www.cellulosechemtechnol.ro/pdf/CCT1-2(2012)/p.105-114.pdf

[6] A. Pérez, PhD Thesis, University of Córdoba, Spain, 2006, http://hdl.handle.net/10396/11656

[7] M. J. Unkovich, J. S. Pate, E. C. Lefroy and D. J. Arthur, *Aust. J. Plant Physiol.*, **27**, 921 (2000), https://doi.org/10.1071/PP99201

[8] A. Ashori and A. Nourbakhsh, *Eur. J. Wood Prod.*, **67**, 323 (2009), https://doi.org/10.1007/s00107-009-0326-0

[9] R. W. Hurter and F. A. Riccio, in *Procs. NANonwood Fiber Symposium*, Atlanta, 1998, pp. 1-11, http://citeseerx.ist.psu.edu/viewdoc/download;jsessionid=8C79B2893AEB10FEF23C28FCAA8FC895?doi=10.1.1.476.8647&rep=rep1&type=pdf

[10] J. Domínguez-Robles, R. Sánchez, E. Espinosa, D. Savy, P. Mazzei *et al.*, *Int. J. Mol. Sci.*, **11**, 327 (2017), https://doi.org/10.3390/ijms18020327

[11] Y. Zhang, G. Yu, B. Li, X. Mu, H. Pen *et al.*, *Carbohyd. Polym.*, **141**, 238 (2016), http://dx.doi.org/10.1016/j.carbpol.2016.01.022

[12] E. Robles, I. Urruzola, J. Labidi and L. Serrano, *Ind. Crop. Prod.*, **71**, 44 (2015), https://doi.org/10.1016/j.indcrop.2015.03.075

[13] P. Jeetah, N. Golaup and K. Buddynauth, *J. Environ. Eng.*, **3**, 52 (2015), https://doi.org/10.1016/j.jece.2014.11.013

[14] D. Yan, X. Yang and W. Yuan, *J. Power Sources*, **289**, 26 (2015), https://doi.org/10.1016/j.jpowsour.2015.04.164
[15] R. Joedodibroto, in *45th Annual General Conference*, Melbourne, 1991, https://pascal-francis.inist.fr/vibad/index.php?action=getRecordDetail&idt=5516387
[16] S. Caparrós, M. J. Díaz, J. Ariza, F. López and L. Jiménez, *Bioresour. Technol.*, **99**, 741 (2008), https://doi.org/10.1016/j.biortech.2007.01.028
[17] J. Labidi, A. Tejado, A. García and L. Jiménez, *Bioresour. Technol.*, **99**, 7270 (2008), https://doi.org/10.1016/j.biortech.2007.12.052
[18] M. M. García, F. López, A. Alfaro, J. Ariza and R. Tapias, *Bioresour. Technol.*, **99**, 3451 (2008), https://doi.org/10.1016/j.biortech.2007.08.004
[19] L. Jiménez, A. Rodríguez, A. Pérez, A. Moral and L. Serrano, *Ind. Crop. Prod.*, **28**, 11 (2008), https://doi.org/10.1016/j.indcrop.2007.12.005
[20] F. López, M. M. García, R. Yáñez, R. Tapias, M. Fernández *et al.*, *Bioresour. Technol.*, **99**, 4846 (2008), https://doi.org/10.1016/j.biortech.2007.09.048
[21] R. Sánchez, A. Rodríguez, A. Requejo, A. García and L. Jiménez, *Cellulose Chem. Technol.*, **44**, 327 (2010), http://www.cellulosechemtechnol.ro/pdf/CCT9(2010)/p.327-334.pdf
[22] A. Ferrer, A. Rosal, C. Valls, B. Roncero and A. Rodríguez, *BioResources*, **6**, 1298 (2011), https://bioresources.cnr.ncsu.edu/
[23] W. J. J. Huijgen, G. Telysheva, A. Arshanitsa, R. J. A. Gosselink and P. J. Wild, *Ind. Crop. Prod.*, **59**, 85 (2014), https://doi.org/10.1016/j.indcrop.2014.05.003
[24] TAPPI, in "Official Test Methods", Technical Association of the Pulp and Paper Industry, 1997, https://www.tappi.org/publications-standards/standards-methods/
[25] L. E. Wise, M. Murphy and A. D'Addieco, *Paper Trade J.*, **122**, 35 (1946), https://digitalcommons.usu.edu/aspen_bib/6698/
[26] W. R. W. Daud, K. N. Law and J. L. Valade, *Cellulose Chem. Technol.*, **32**, 133 (1998), http://www.cellulosechemtechnol.ro/
[27] A. Rodríguez, R. Sánchez, M. E. Eugenio, R. Yañez and L. Jiménez, *Cellulose Chem. Technol.*, **44**, 239 (2010), http://www.cellulosechemtechnol.ro/pdf/CCT44,7-8(2010)/P.239-248.pdf
[28] A. Rodríguez, R. Sánchez, A. Requejo and A. Ferrer, *J. Cleaner Prod.*, **18**, 1084 (2010), https://doi.org/10.1016/j.jclepro.2010.03.011
[29] R. Sánchez, A. Rodríguez, A. Requejo, A. Ferrer and E. Navarro, *Bioresour. Technol.*, **101**, 7032 (2010), https://doi.org/10.1016/j.biortech.2010.04.008
[30] A. Requejo, A. Rodríguez, Z. González, F. Vargas and L. Jiménez, *BioResources*, **7**, 3142 (2012), https://bioresources.cnr.ncsu.edu/
[31] A. Rodríguez, A. Moral, L. Serrano, J. Labidi and L. Jiménez, *Bioresour. Technol.*, **99**, 2881 (2008), https://doi.org/10.1016/j.biortech.2007.06.003
[32] R. Martín-Sampedro, M. E. Eugenio, J. A. Moreno, E. Revilla and J. C. Villar, *Bioresour. Technol.*, **153**, 236 (2014), https://doi.org/10.1016/j.biortech.2013.11.088
[33] "UNE Standards", Instituto Nacional de Racionalización del Trabajo, Spain, 1989, http://www.worldcat.org/identities/lccn-n50062277/
[34] F. Potücek and M. Milichovsky, *Cellulose Chem. Technol.*, **45**, 23 (2011), http://www.cellulosechemtechnol.ro/pdf/CCT1-2(2011)/p.23-28.pdf
[35] L. Alonso, "Análisis químico de maderas de diferentes especies forestales" [Chemical Analysis of Woods of Different Forest Species] (in Spanish), Instituto Nacional de Investigaciones Agrarias, Spain, 1976, https://www.worldcat.org/title/analisis-quimico-de-maderas-de-diferentes-especies-forestales/oclc/1085785387?referer=di&ht=edition
[36] F. Vargas, Z. González, R. Sánchez and L. Jiménez, *BioResources*, **7**, 4161 (2012), https://bioresources.cnr.ncsu.edu/
[37] J. A. García, in "Fibras papeleras" [Paper Fibers] (in Spanish), Universidad Politécnica de Cataluña, Spain, 2007, https://dialnet.unirioja.es/servlet/libro?codigo=302218
[38] S. K. Gulsoy and F. Ozturk, *Mad. Cie. Tecn.*, **17**, 875 (2015), https://doi.org/10.4067/S0718-221X2015005000076
[39] N. P. Marinho, U. Klock, E. C. Lengowski, G. I. Bolzon de Muñiz and E. H. Z. Zamarian, *Flor. Amb.*, **24**, e00099214 (2017), http://dx.doi.org/10.1590/2179-8087.099214
[40] J. C. Parajó, J. L. Alonso, D. Vázquez and V. Santos, *Holzforschung*, **47**, 188 (1993), https://doi.org/10.1515/hfsg.1993.47.3.188
[41] H. Eroglu, "Fiberboard Industry," Karadeniz Technical University, Turkey, 1998, p. 212, http://www.ktu.edu.tr/en
[42] G. Garrote, M. E. Eugenio, M. J. Díaz, J. Ariza and F. López, *Bioresour. Technol.*, **88**, 61 (2003), https://doi.org/10.1016/S0960-8524(02)00256-0
[43] J. A. Honorato, F. Apolinar and G. Colotl, *Rev. Mex. Cie. For.*, **7**, 47 (2016), https://cienciasforestales.inifap.gob.mx/
[44] J. Alaejos, F. López, M. E. Eugenio and R. Tapias, *Bioresour. Technol.*, **97**, 2110 (2006), https://doi.org/10.1016/j.biortech.2005.09.021
[45] A. Rodríguez, L. Serrano, A. Moral, A. Pérez and L. Jiménez, *Bioresour. Technol.*, **99**, 1743 (2008), https://doi.org/10.1016/j.biortech.2007.03.050
[46] N. Cordeiro, M. N. Belgacem, I. C. Torres and J. C. V. P. Moura, *Ind. Crop. Prod.*, **19**, 147 (2004), https://doi.org/10.1016/j.indcrop.2003.09.001
[47] L. Jiménez, V. Angulo, E. Ramos, M. J. de la Torre and J. L. Ferrer, *Ind. Crop. Prod.*, **23**, 122 (2006), https://doi.org/10.1016/j.indcrop.2005.05.001

[48] P. Khristova, O. Kordsachia, R. Patt and S. Dafaalla, *Bioresour. Technol.*, **97**, 535 (2006), https://doi.org/10.1016/j.biortech.2005.04.006
[49] A. Alfaro, A. Pérez, J. C. García, F. López, M. A. M. Zamudio *et al.*, *Cellulose Chem. Technol.*, **43**, 295 (2009), http://www.cellulosechemtechnol.ro/pdf/CCT7-8-2009/p.295-306.pdf
[50] A. Alfaro, A. Rivera, A. Pérez, R. Yánez, J. C. García *et al.*, *Bioresour. Technol.*, **100**, 440 (2009), https://doi.org/10.1016/j.biortech.2008.05.003
[51] R. H. Reeves, in "Paper Machine Operations", edited by B. A. Thorp, Tappi Press, Atlanta, 1991, https://www.tappi.org/
[52] W. E. Scoot and J. C. Abbot, "The Fundamental Aspects of Paper Properties", Tappi Press, 1995, Chapter 8, https://www.tappi.org/
[53] T. E. Amidon, *Tappi J.*, **64**, 123 (1981), https://www.tappi.org/
[54] N. Gurnagul, D. H. Page and M. G. Paice, *Nord. Pulp Pap. Res. J.*, **7**, 152 (1992), https://doi.org/10.3183/npprj-1992-07-03-p152-154
[55] F. López, A. Alfaro, M. M. García, M. J. Díaz, A. M. Calero *et al.*, *J. Chem. Eng. Res. Des.*, **82**, 1029 (2004), https://doi.org/10.1205/0263876041580730
[56] L. Jiménez, A. Rodríguez, J. L. Ferrer, A. Pérez and V. Angulo, *Afinidad*, **62**, 100 (2005), https://www.raco.cat/index.php/afinidad/search/search