ARUNDO DONAX L. REED: NEW PERSPECTIVES FOR PULPING AND BLEACHING 1. RAW MATERIAL CHARACTERISATION

Anatoly A. Shatalov, Teresa Quilhó, Helena Pereira

Centro de Estudos Florestais, Universidade Técnica de Lisboa, Instituto Superior de Agronomia
Tapada da Ajuda, 1349-017 Lisboa PORTUGAL
Phone: +351.21 363 46 62 Fax: +351. 21 364 50 00 E-mail: deftec@mail.telepac.pt

ABSTRACT

Arundo donax L. (or giant reed), a plant with annual harvesting period and high biomass productivity, is a promising non-wood fibre crop for pulp and paper production. The presented data of chemical, anatomical and morphological studies of *Arundo donax*, given from the viewpoint of raw material characterization, allow prediction of the potential of this crop for fibre production. The correlation between biometric data of fibres of *Arundo donax* and papermaking properties of pulps produced is discussed.

APPLICATION

The chemical, anatomical and morphological characteristics of *Arundo donax* allow prediction of the potential of this crop for fibre production.

EXECUTIVE SUMMARY

Arundo donax L. (or giant reed) is a perennial rhizomatous plant with annual harvesting period and high biomass productivity. Nowadays an interest in *Arundo donax* as raw material for industrial purposes is increasing. In the present paper *Arundo donax* was characterized as raw material for fibre production, by chemical, anatomical and morphological studies of the main botanically distinct parts of the stem. The lower content of lignin (21.1%) and cellulose (31.1% α-cellulose) and the higher content of the non-structural components (12.1% extractives) were the principal chemical features, which distinguish *Arundo donax* L. from woody materials. Anatomical analysis of *Arundo donax* L. showed heterogeneity in the cellular structure with predominance of ground tissue parenchyma (or parenchyma cells) and lower proportion of vascular tissue (or fibres), as compared with hardwoods (58 vs. 20% and 36 vs. 50%, respectively for fibres and parenchyma). The biometric analysis of *Arundo donax* L. fibres revealed an average length of 1.2 mm, width of 15.8 μm and wall thickness of 5.0 μm, that do not differ greatly from hardwoods.

INTRODUCTION

With increased demand for market pulp and decrease of wood availability, the alternative sources of fibre from annual (or biannual) agricultural crops are of prime interest *(1)*. Among different non-wood plant fibres, the crops from the grass family are more abundant and economically feasible to harvest and to utilize *(2)*. Such non-woody fibers as bamboo, esparto, sugar-cane bagasse and various cereal straws have already an important economic value for the pulp industry, representing about 6% of the world pulp market *(3)*. From an industrial standpoint, the reed *Arundo donax* has a minor importance.

Arundo donax L. (or giant reed), a perennial rhizomatous plant of the family *Gramineae* with a tall erect stem, is the largest member of the genus and one of the largest of the herbaceous grasses *(4)*. Being a native from the Mediterranean region, *Arundo donax* L. has become widely distributed into the subtropical and warm temperate areas *(5)*. It can tolerate a wide variety of ecological conditions with all types of soils (from clays to sands) and grows very fast (30-70 cm. per week) under favourable conditions in well-drained soils with abundant moisture *(6)*. It was reported, that in southern France *Arundo donax* L. produces 20-25 t $year^{-1}$ ha^{-1} dry biomass *(7)* and in northern Italy 32-37 t $year^{-1}$ ha^{-1} *(8)*. The production can be increased up to 100 $t \cdot year^{-1} \cdot ha^{-1}$ under optimal conditions in a warm climate and by using underground irrigation *(9)*.

Nowadays an interest in *Arundo donax* L. as raw material for industrial purposes is increasing. Comprehensive studies to evaluate *Arundo donax* L. as a potential raw material for fibre production were carried out in 1930-1960 *(10,11)*. It was concluded, that pulps can be obtained with satisfactory yield and mechanical properties using conventional processes (soda and Kraft), but the quality of the pulps as well as of the paper sheets was lower, as compared to wood.

With this paper we open a series of publications devoted to the applicability of *Arundo donax* L. reed for modern pulping and bleaching technologies. The present paper reports the results of chemical, anatomical and morphological characterisation of *Arundo donax* L., given from the viewpoint of raw material evaluation for fibre production.

EXPERIMENTAL

Materials

Arundo donax L. reed was supplied by the Agricultural University of Athens (Plant Production Department) cut in short pieces (*culms*) of 15-20 cm length, free from leaves. To elucidate the impact of morphological structure on pulping behaviour, a part of *Arundo donax* was divided by cutting into nodes and internodes, composed of nodes proper with immediately adjacent short (around 1 cm) transition regions and stalks proper, respectively. For pulping experiments, all raw material was disintegrated manually up to the approximate size of industrial chips: length 1-2 cm; thickness 1.5-4 mm, depending on stem wall thickness; width about 1 cm. For chemical analysis a small portion of chips was ground, first up to match size and then to sawdust, and screened to obtain uniform particle size 40 to 60 mesh.

Methods

Chemical analysis

The extractives content was determined gravimetrically after successive Soxhlet extraction by dichloromethane (6 h), ethanol (16 h) and hot water (16 h). The ash content was determined according to TAPPI T15 os 58 standard *(12)*. The silicates and silica were determined as SiO_2 by the wet ash method according to TAPPI T 245 om-94 standard *(13)*.

Carbohydrates composition was analysed by GC as TMS-derivatives of monosaccharides after Saeman hydrolysis *(14)*.

The lignin content was determined as Klason lignin and acid-soluble lignin by UV-absorbance at 205 nm *(15)*.

The holocellulose was isolated by a modified chlorite method in the presence of sodium acetate buffer at pH 3.2-3.8 *(16)*. The hemicellulose fractions, designated as *fraction A* and *fraction B*, were extracted from holocellulose by aqueous solutions of 5% and 24% potassium hydroxide, respectively, and recovery was made by addition of excess of absolute ethanol *(17)*. The insoluble residue after alkaline extraction of holocellulose was accepted as α*-cellulose.*

The basic specific gravity was determined as the ratio of the oven-dry weight of a reed sample to the weight of water displaced by that sample in its water-swollen state ("green volume").

Anatomical analysis

Five culms were randomly selected and portions of nodes and internodes were fixed in formalin-acetic acid-alcohol mixture (FAA). Transverse sections approximately 17 μm thick were cut with a Reichert sliding microtome, after impregnation with DP 1500 polyethylene glycol, stained with triple staining of chrysodine/acridine red and astra blue and mounted in Euparal. The proportion of tissues was determined by a modification of the "Test Point Method" *(18)*. Light microscopy observations were also made with dissociated elements of nodes and internodes, after staining with astra blue. The material was macerated by Franklin´s method in acetic acid and hydrogen peroxide (1:1) at 60ºC for 48 hours. The parameters measured were length, width and cell wall thickness of 40 whole fibres using a projection microscope and a semi-automatic image analyser. The number of measurements was previously calculated to give an accuracy of 5% at the 0.05 probability level.

RESULTS AND DISCUSSION

General characterisation

Arundo donax has a tall erect fibrous stem with a height from 2 to 8 m. The stem is divided by solid partitions at the *nodes*, with hollow *internodes* varying in length from approximately 10 to 30 cm. Following are some raw-material characteristics of *Arundo donax*:

- Stem diameter, mm	15.0-30.0
- Stem wall thickness, mm	1.5-4.0
- Bulk density of stems, kg/m^3	141.9
- Bulk density of chips, kg/m^3	183.5
- Basic specific gravity of nodes	0.37
- Basic specific gravity of internodes	0.42
- Relative proportion of nodes *vs.* internodes (by weight)	1/6

The values of basic specific gravity obtained for *Arundo donax* are comparable with those for some hardwood species *(19)*, but in general closer to softwoods. The lower basic specific gravity of nodes will contribute to the lower bulk density of *Arundo donax* chips in the digester, where they will correspond to approx. 15% of the digester loading.

Chemical composition

The chemical composition of the nodes and internodes of *Arundo donax* L. is fairly similar (**Table 1**). The only difference was that nodes are higher in hemicelluloses and extractives and lower in lignin and cellulose than internodes, as would be expected from the morphological distinctions. The silica content in nodes and internodes of *Arundo donax* is about equal and not higher in nodes, as reported for straw *(2)*.

Components	Nodes	Internodes
Ash	4.77	6.14
- silicates (as SiO_2)	1.31	1.16
Extractives	13.04	11.16
- dichloromethane	0.46	0.37
- ethanol	5.88	4.18
- hot water	6.70	6.61
Lignin	20.92	21.31
- Klason	19.03	19.60
- acid-soluble	1.89	1.71
Holocellulose	61.21	61.41
- α - cellulose	29.18	32.93
- hemicelluloses	32.03	28.48
- fraction A	26.20	23.36
- fraction B	5.83	5.12

Table 1. Chemical composition of Arundo donax L. reed. (% of oven dry material).

As the whole, the chemical analysis of *Arundo donax* reed showed values similar to various other grasses and deviations from soft- and hardwoods. It is evident that *Arundo donax* (with an average 21% lignin and 31% α-cellulose) contains considerably less lignin and cellulose than wood (24-34% and 38-50%, respectively) *(19)*, but closely resemble bagasse (19-24% and 32-44%) and bamboo (21-31% and 26-43%) *(20)*.

The hemicelluloses content of *Arundo donax* (31%) does not differ greatly from hardwoods (20-28%) and grain straw (26-32%) *(19,20)*. The carbohydrate composition (**Table 2**) showed the remarkable predominance of pentosans over hexosans in hemicelluloses (96% and 4% of total hemicelluloses, respectively) and namely the drastic prevailing of xylan, that is typical for all annual plants and closely resembles the hardwoods.

Monosaccharide units	Nodes	Internodes
Arabinose	3.22 (2.83)	2.47 (2.17)
Xylose	35.91 (31.60)	30.92 (27.21)
Mannose	0.16 (0.14)	0.88 (0.79)
Galactose	1.17 (1.05)	0.83 (0.75)
Glucose	59.54 (53.59)	64.91 (58.42)

Note
% of hexosans (glucan, mannan, galactan) = % of hexoses *x* correlation factor (0.90).
% of pentosans (xylan, arabinan) = % of pentoses *x* correlation factor (0.88).

Table 2. Carbohydrate composition of Arundo donax L. reed (% of total monosaccharides and, in parenthesis, % of the corresponding homopolysaccharides).

Other differences from the chemical composition of wood were found in relative high ash (5.5%) and extractives (12.1%) content, also common to grasses *(19)*. Non-wood fibrous plants normally have high content of silica *(20)*, as undesirable constituent for pulping. The silica content of *Arundo donax* comprised about 22.8% of total ash (Table 1). The high content of extractives, particularly of water-soluble substances (6.6%), results from pectins and low-molecular phenolics, that are lost during pulping.

Anatomical analysis

The anatomical structure of *Arundo donax* reed was examined on transverse sections and on macerated samples of nodes and internodes. The light microscopy observation revealed the structural similarity in the anatomy of nodes and internodes with the prevalence of three distinct tissue systems: the epidermal (or cortical) tissue, the fundamental (or ground) tissue and the vascular tissue, composed of fibro-vascular bundles.

The *cortical tissue* contained a layer of thick walled fibre-like cells underlying the epidermis (**Figure 1**, arrow) followed by a layer of thin parenchyma cells. A similar arrangement with a strongly sclerified subepidermal parenchyma was also found for some grasses (bamboo *Sinobambusa tootsik, Zea mays*) *(21)*.

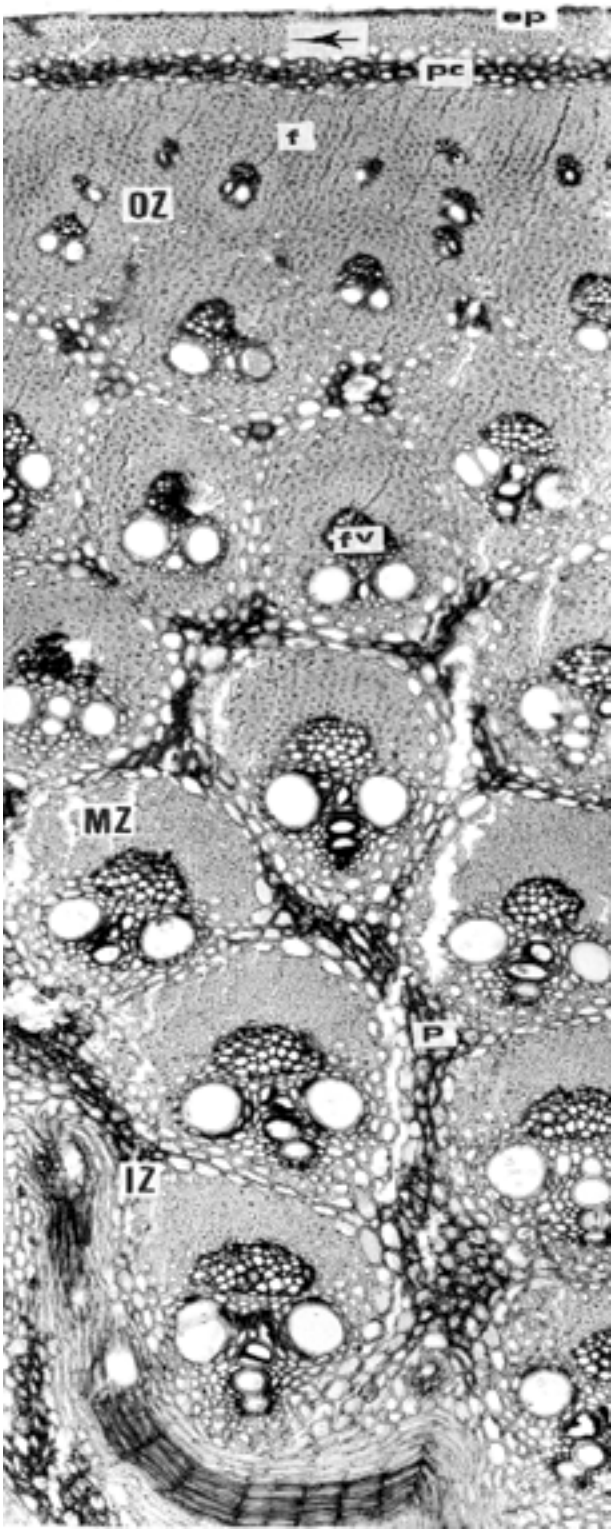


Figure 1. Transverse section of the internode of Arundo donax culm (100x): outermost zone (OZ), middle zone (MZ) and inner zone (IZ); epidermis (ep) followed by a layer of strongly sclerified parenchyma cells (arrow) and a layer of typical cortical parenchyma (pc); fibres (f), parenchyma (p), fibro-vascular bundle (fv).

The *ground tissue parenchyma* was in general homogeneous in wall thickness except for minor inclusions of thick walled elements in the outermost zone of the culms. The shape of the parenchyma cells varied over a wide range from a "*barrel*" to a "*rod*" type (**Figure 2**, ***a*** and ***b***, respectively). The percentage of tissues (**Table 3**) did not show any essential distinctions in parenchyma cells content between nodes and internodes (60% and 56%, respectively). The average content of parenchyma in *Arundo donax* (58%) resembles wheat straw (68%) and cornstalks (50%) *(20)* and differs significantly from wood (7% and 20% for soft- and hardwoods, respectively *(19)*).

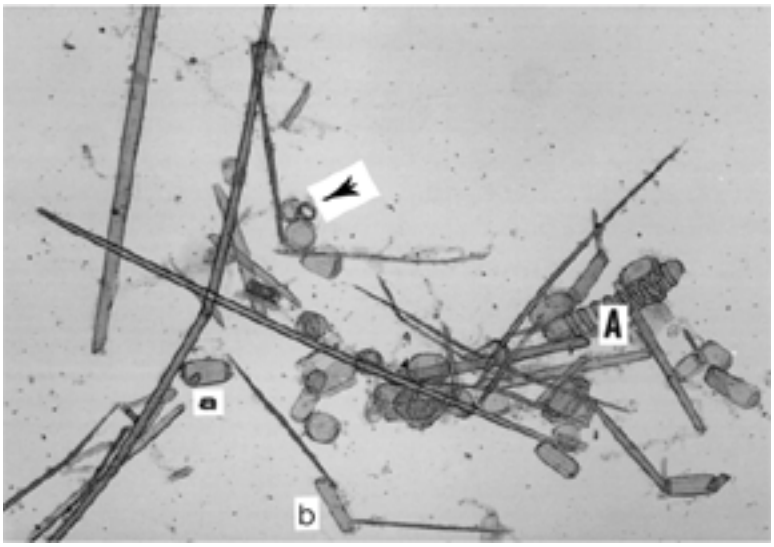


Figure 2. Macerated sample of the node (100x): "barrel"-like parenchyma cell (a), "rod"-like parenchyma cell (b), annular vessel (A) and ring of the annular vessel (arrow).

Sample	Internode			Node		
	Parenchyma	Fibre	Vascular tissue	Parenchyma	Fibre	Vascular tissue
1	66.9	28.9	4.2	52.7	41.5	5.8
2	67.7	26.2	6.1	54.5	37.6	7.9
3	48.5	45.4	6.1	54.5	39.4	6.1
4	53.0	39.5	7.5	60.1	34.7	5.2
5	63.3	29.7	7.0	57.3	35.5	7.2
Average	59.8	33.9	6.2	55.8	37.8	6.4
St.dv.	8.7	8.3	1.3	2.9	2.8	1.1

Table 3. Percentage of tissues in the internodes and nodes of 5 culms of Arundo donax L. reed (in area % of total cross-sectional area).

The stem of *Arundo donax* is solid at the nodes through the retaining of fragments of the fundamental tissue (the *pith*), which was degraded during the growth of the stem. This residual ground tissue, enclosed inside the hollow cylinder of the stem, forms a *nodal diaphragm* located at the node level and consisted mainly of parenchyma cells. Taking into account that the results

of the microscopic observations (Table 3) refer only to the stem wall material, thereby excepting the nodal diaphragm, the real content of the parenchyma tissue in the nodes will be higher than in the internodes.

The *fibro-vascular bundles* have a scattered arrangement and consisted of pitted vessels of xylem (V) forming metaxylem, tracheids (T) of metaxylem, annular vessels (A) forming protoxylem and sieve tubes (ST) and companion cells (C) forming protophloem (**Figure 3, 4**). The non-functional protophloem was also present (Fig. 3, arrow). The annular vessels, unable to accompany the extension of the adjacent cells, are stretched and sometimes completely destroyed, with separate rings (Fig. 2, arrow). The average percentage of vascular tissue in *Arundo donax* was 5.0% (4.6% in the internodes and 5.3% in the nodes), that differs from wood (30% for hardwoods *(19)*).

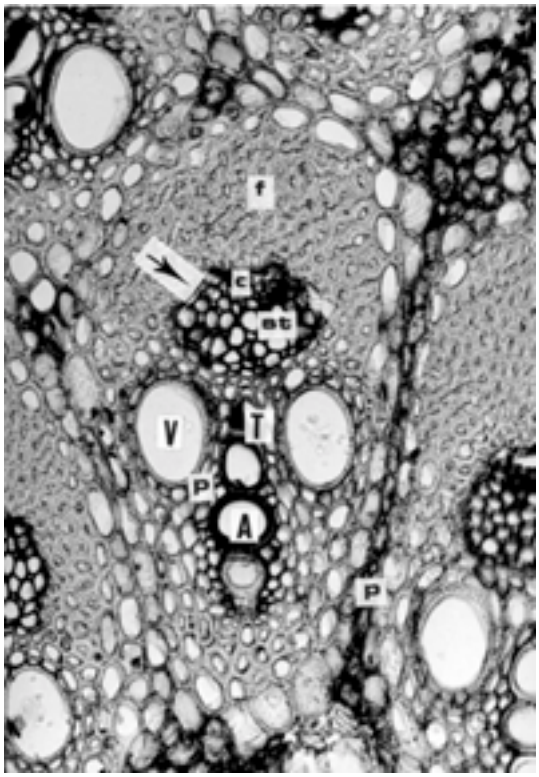


Figure 3. Transverse section of a fibro-vascular bundle (400x): vessel (V) and tracheid (T) of metaxylem; annular vessel (A) of protoxylem; non-functional protophloem (arrow), sieve tubes (st) and companion cells (c) of protophloem; fibres (f); parenchyma (p).

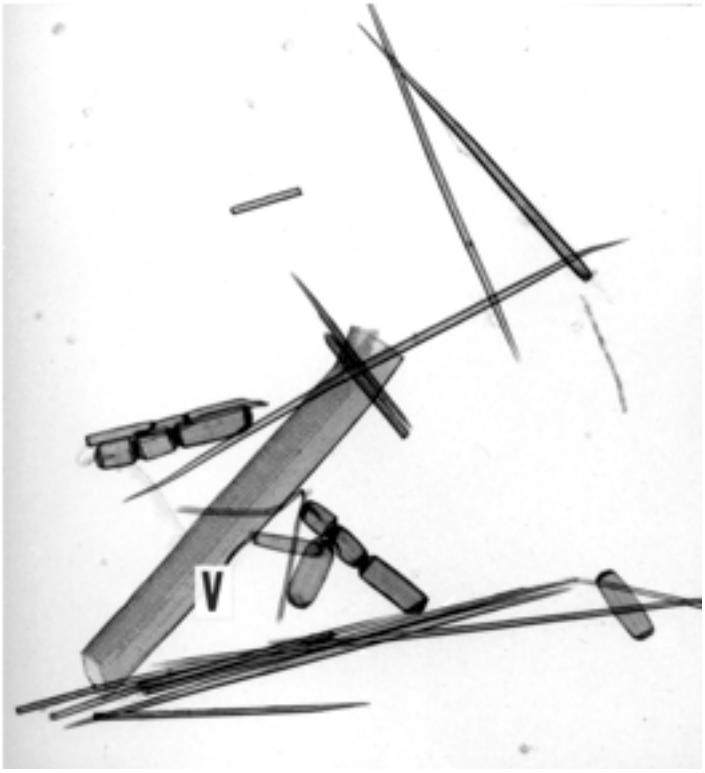


Figure 4. Pitted vessel (V) of metaxylem tissue in the node (macerated sample, 100x).

The microscopic observations demonstrated some decrease in fibre content from the outermost to the innermost zone (OZ and IN, Fig. 1) of the culms, with evident heterogeneity in the fibre wall thickness. The fibres are especially in abundance and with thicker walls in the outermost zone near the epidermis as well as near the vascular tissues (protophloem). No essential differences in the fibre content between nodes and internodes was found (38% and 34%, respectively). In general, the proportion of fibres was much lower as compared to wood (93% and 50% for soft- and hardwoods, respectively *(19)*) and resembles bagasse (40%) *(20)*.

Fibre morphology

The biometric analysis of fibres (**Table 4**), performed on macerated samples, did not reveal any statistical significant differences in fibre length between nodes and internodes (1.20 mm and 1.16 mm, respectively). In contrast, the fibre diameter and wall thickness were fairly different (16.9 μm and 5.3 μm for nodes; 14.6 μm and 4.6 μm for internodes).

As a whole the fibre dimensions of Arundo donax reed do not vary considerably from those of other grasses. The average fibre length of Arundo donax is approx. 1.2 mm, that is fairly close to hardwoods (0.7-1.3 mm for Eucalyptus globulus L.) and resembles esparto (approx. 1.5 mm), wheat straw (approx. 1.5 mm) and bagasse (1-1.5 mm) (20). Fibre width of Arundo donax (approx. 15.8 μm) is lower as compared to wood (approx. 32.0-43.0 μm and 18.0-30.0 μm for soft- and hardwoods, respectively (19)), but comparable with some eucalypts (13.0-19.0 μm (19)) and resembles wheat straw (15.0 μm), bamboo (15.0 μm) and cornstalks (18.0 μm) (20). The fibre wall thickness of Arundo donax (approx. 5.0 μm) does not differ greatly from woods (2-8 μm) (19).

Sample	Internode			Node		
	Length (mm)	Width (μm)	Wall thickness (μm)	Length (mm)	Width (μm)	Wall thickness (μm)
1	**1.172** 0.735-1.900	**16.2** 26.4-9.9	**5.0** 2.5-9.0	**1.520** 0.681-3.301	**18.5** 11.7-23.9	**5.8** 2.9-9.8
2	**1.299** 0.602-2.533	**15.0** 22.9-8.3	**5.0** 2.5-7.5	**1.312** 0.663-2.828	**19.0** 11.0-31.1	**5.7** 3.2-9.6
3	**1.163** 0.537-2.837	**14.3** 20.5-8.6	**4.6** 0.8-7.7	**1.090** 0.601-2.380	**16.3** 9.4-27.1	**5.0** 2.7-8.3
4	**1.210** 2.801-0.644	**14.0** 19.1-8.6	**4.2** 2.1-6.4	**1.014** 0.451-2.347	**15.8** 10.4-22.0	**5.2** 2.4-8.7
5	**0.940** 0.513-1.281	**13.7** 21.7-9.4	**4.4** 2.6-7.7	**1.022** 0.432-1.391	**14.7** 7.9-20.1	**4.8** 2.9-7.6
Average	1.157	14.6	4.6	1.197	16.9	5.3
St.dv.	0.133	0.99	0.36	0.214	1.83	0.44

Table 4. Biometric data of the fibres in the internodes and nodes of 5 culms of Arundo donax reed (mean value for each sample (in bold) and minimal and maximal value).

The physical properties of a sheet of paper (i.e., tearing, burst and tensile strengths) are closely related to morphological properties of the pulp fibres *(22)*, with cell wall thickness, density, fibre length and diameter as the most important parameters influencing pulp strength and surface properties *(23)*. A number of different ratios of fibre dimensions has been proposed for quantitative characterisation of relationships between fibre and pulp properties *(24)*. The tear resistance was found to be positively correlated with the *felting coefficient* (fibre length/fibre diameter) and with the *coefficient of flexibility* (lumen diameter/fibre diameter). Burst and tensile strength are negatively correlated with cell wall thickness, e.g. with the *Runkel ratio* (twice cell wall thickness/lumen diameter) *(25)*. It is obvious that the maximum pulp strength properties can be obtained from fibre material with thin cell walls and a large fibre length to fibre diameter ratio.

In **Table 5** the calculated ratios of *Arundo donax* fibre dimensions are presented for rough estimation of papermaking properties. The comparison with wheat straw and bamboo, which have fibre and parenchyma content similar to *Arundo donax (21)*, promises to expect the production of *Arundo donax* pulps with quite high tear resistance (the felting coefficient for *Arundo donax* is higher than for wheat straw and comparable with bamboo). The burst and tensile resistances of the *Arundo donax* pulps promise to be higher than for bamboo pulps, but lower than for wheat straw pulps (the Runkel ratio for *Arundo donax* is lower than for bamboo, but higher than for wheat straw). Thereby this analysis allows to predict the quite high papermaking properties of *Arundo donax* pulps, which can be compared with pulp properties of such leaders of the non-woody pulp production as wheat straw and bamboo. The essential

difference in fibre and parenchyma content between *Arundo donax* and wood does not allow to compare correctly their respective pulp properties using only fibre biometric data. However, if only the fibre fraction is considered, without the weakening impact of parenchyma, a high tear resistance of *Arundo donax* fibres, comparable with hardwood, may be predicted.

Ratios		*Arundo donax* L.		Wheat straw *(26)*	*Bambusa Arundinaria (27)*
		internode	node		
Runkel ratio	$\frac{2e}{l}$	1.70	1.68	1.14	2.67
Felting coefficient	$\frac{L}{d}$	79	71	67	79
Coefficient of flexibility	$\frac{l}{d}$	0.37	0.37	0.46	0.27

Note e - wall thickness, l - lumen diameter, L - fibre length, d - fibre diameter.

Table 5. Biometric ratios of fibres dimensions.

CONCLUSIONS

Arundo donax L. reed has some negative aspects as raw material for pulp and papermaking: heterogeneity in the cellular structure with predominance of parenchyma; lower proportion of fibres and higher content of extractives, as compared with wood; high ash and especially silica content; low basic specific gravity.

Inspite of the mentioned drawbacks, *Arundo donax* as raw material for pulp production has some obvious advantages over woody plants. First of all it has an annual harvesting period, a high biomass productivity and a posibility to tolerate a wide variety of ecological conditions. The lower content of lignin in *Arundo donax*, as compared to wood, allows to use simple pulping chemical systems with reduced chemical charge, but keeping a high rate of delignification. The higher content of water-soluble substances indicates the high degree of accessibility of *Arundo donax* cell-wall to pulping liquor, assuming more gentle time-temperature cooking conditions, as compared to wood. Finally, the biometric data of fibres of *Arundo donax* reed are close to those of hardwoods, indicating production of pulps with quite high papermaking properties, if cooked under appropriate conditions.

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