IAWA Journal, Vol. 27 (2), 2006: 153–172

IDENTIFICATION OF FIBRE COMPONENTS IN PACKAGING GRADE PAPERS

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SUMMARY

Environmental and economic issues have led to a significant increase of recycled paper as the main fibrous component of corrugated board the last years. Qualitative data on the different fibre types are needed for the evaluation of packaging fibre supply sources, which are becoming numerous and heterogeneous. Fifteen different packaging grade papers (7 linerboards and 8 corrugating medium) were selected to represent all the variety of papers available on the Spanish market. The origin of virgin and recycled fibres was identified by their morphological characteristics employing light microscopy and standard fibre analysis techniques. The waste-based papers (Waste based-liners and Fluting), Kraft-liners and Test-liner were highly variable containing 9–18 different wood and non-wood components. Semi-chemical, with 5–13 components, was the less variable grade. Hardwoods were identified as the most important fibre component from a quantitative standpoint. All papers contained in their hardwood mix *Betula*, *Eucalyptus* and *Populus* in significant amounts. *Fagus sylvatica* and *Tilia* were also frequently observed and in some papers were amongst major hardwood components. Prominent softwood components were found to be *Pinus sylvestris*, *P. pinaster*, *P. radiata*, *Picea*, *Larix* and in some papers *Pinus nigra*. The lower presence of a variety of softwood, hardwood and nonwood (mainly grasses) species and genera was due to the paper recycling process.

Key words: Fibre identification, papermaking fibres, recycled fibres, linerboard, corrugating medium, packaging.

INTRODUCTION

The restrictions in availability of forest-based raw materials (FAO 2001) along with favourable environmental policy – *e.g.* EU Packaging and Packaging Waste Directive 94/62/EC (European Commission 1994) – towards alternative sources of raw material has forced packaging industry to shift towards waste paper and other fibre sources such as nonwood and agro-residues (Young 1997). Nowadays, packaging grade papers

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Associate Editor: Susan Anagnost

contain 60–100% recycled fibre and, thus, the effective utilisation of such an alternative raw material is of great environmental and economical importance (Lintu 1990).

Mixed waste, being the most abundant grade of waste paper available (Virtanen & Nilson 1993), varies in composition from source to source and from day to day from a single source. Together with cleanliness of the pulp, the effect of raw materials coming from heterogeneous sources on paper and paper products (carton board and corrugated board) properties is the greatest concern of paper recycling (Groom *et al.* 1992; Abubakr *et al.* 1995; Mckinney 1995; Jahan 2003; Ashley & Hodgson 2003). Besides the different production methodologies (mechanical, chemical and chemical-mechanical pulping) and processing variables (such as beating, refining, flocculation and grammage), the properties of paper and paper products vary due to differences in raw materials (Britt 1971; Bormett *et al.* 1981; Thomas & Kellison 1989; Law *et al.* 1996; Sjostrom & Alen 1999; Drost *et al.* 2004).

Diagnostics assessing the potential quality distribution of fibres from different sources are highly needed to select the appropriate raw material for each end-use (Johansson *et al.* 2002). A first step towards a more economical and effective utilisation of paper and paper products in packaging should be the reliable characterisation of raw pulp materials. Comprehensive characterisation (qualitative classification as to source) of the pulp produced by the waste paper is not a common practice and its potential on paper products quality control has not been fully explored yet.

Most commercial pulps are produced with knowledge as to approximately which species or species groups are included and therefore permit some level of fibre identification (Parham & Gray 1990). This obviously does not apply to waste pulps, which contain a mixture of several wood and nonwood fibre types. Identification of wood species in pulp is a rather complicated work process as all the tissue-distribution patterns have disappeared and attention must be focused on a relatively small number of details found on the fibres or vessel elements. Chemical and mechanical processing greatly diminishes the amount and quality of features useful for microscopic identification of fibre source (Strelis & Kennedy 1967). As a result in most of the cases identification is limited to the genus level or to a choice of similar genera (Panshin & De Zeeuw 1980; Parham & Gray 1990; Ilvessalo-Pfäffli 1995).

Published data on the variety of fibre types used in the production of packaging grade papers could not be located. The primary objective of the present study was to identify the origin of virgin and recycled fibres of different grades that are currently used as linerboard and corrugating medium in corrugated board manufacturing. Data on actual furnish composition will give us an indication of the present situation and are also expected to help the packaging industry evaluate its fibre supply sources, which are becoming numerous and heterogeneous.

MATERIALS AND METHODS

Sample selection for microscopy

For testing, 15 different packaging grade papers (7 linerboards and 8 corrugating medium) were provided by Spanish corrugated board factories. These papers are the

Table 1. Paper characteristics.

Paper ID	Paper grade	Grammage (g/m^2)	Thickness (mm)	Production technology
Linerboards				
KL1	Kraft-liner	228	0.293	Mainly virgin kraft fibres
KL2	Kraft-liner	185	0.258	Mainly virgin kraft fibres
KL3	Kraft-liner	298	0.437	Mainly virgin kraft fibres
TL	Test-liner	124	0.195	1 ply of kraft-liner quality & 1 ply of recycled fibres
WBL1	Waste based-liner	126	0.194	Recycled fibres
WBL2	Waste based-liner	112	0.182	Recycled fibres
WBL3	Waste based-liner	152	0.228	Recycled fibres
Corrugating medium				
SQ1	Semi-chemical	161	0.256	Mainly virgin semi-chemical fibres
SQ2	Semi-chemical	166	0.271	Mainly virgin semi-chemical fibres
SQ3	Semi-chemical	172	0.269	Mainly virgin semi-chemical fibres
SQ4	Semi-chemical	150	0.221	Mainly virgin semi-chemical fibres
SQ5	Semi-chemical	165	0.264	Mainly virgin semi-chemical fibres
F1	Fluting	111	0.189	Heavily recycled fibres
F2	Fluting	107	0.172	Heavily recycled fibres
F3	Fluting	91	0.144	Heavily recycled fibres

most commonly used for the production of corrugated board in Spain and represent all the variety of papers available on the market at the moment. The characteristics of the papers are shown in Table 1.

The corrugated board structural panels are formed from a pair of flat faces called linerboards, which are separated by a periodic fluted core referred to as the corrugating medium (Maltenfort 1996). Linerboards are available in three basic forms (common names): Kraft-liner, Test-liner and Waste based-liner. Kraft-liner is made mainly from virgin softwood and hardwood fibres. Many Kraft-liner grades include some recycled fibres such as clippings from corrugated board manufacture or from old corrugated containers. Test-liner consists mainly of recycled fibres, which comes in many different grades and qualities from corrugated plants, stores, offices and households. The properties of Test-liner are improved by additives and by selection of recycled fibres. In some grades of Test-liner, the one face is of Kraft-liner quality and the other is from selected recycled fibres. Waste based-liner is made from recycled fibres and it has a lower quality than Test-liner. There are two types of corrugating medium: Semi-chemical and Fluting. Semi-chemical is virgin-based medium and is increasingly used for special purposes, such as in humid conditions. The main virgin fibre used to manufacture Semi-chemical corrugating medium is semi-chemical hardwood, which is cheaper than softwood and gives good resistance to forces perpendicular to the flutes. The short hardwood fibres are less flexible than softwood fibres and give greater stiffness to the corrugated structure. Fluting is the multipurpose medium most frequently used. It represents the lowest grade, which has been heavily recycled (EN 643, European Standards 2002).

Microslide preparation and examination

For each of the papers microscope slides were prepared with fibres as for usual fibre analysis according to ISO 9184-1 (ISO 1990). A representative small quantity (about 0.25 g) from different parts of the papers was torn into small pieces, placed in a small beaker and boiled in water for a few minutes. The softened pieces were rolled with the fingers into small pellets and were shaken vigorously in a large tube with the addition of some water until the pellets were thoroughly disintegrated. The suspension was diluted to a consistency of about 0.05% (wt./vol.) and then 1 ml of the suspension was transferred with a pipette onto clean slides placed on a hot plate. After the slides were completely dried, staining of the fibres to obtain a better resolution of structural details was performed by adding a drop of aqueous safranin and covered with a cover glass. The above procedure produced a count of about 500 fibres per slide.

The microslides were observed under a Nikon Microphot EPI-U2 light microscope equipped with complete optics for bright-field microscopy, cross-hair eyepiece and a 35 mm camera. The entire cover glass area was systematically examined in lines by traversing the slide horizontally. The structural details of fibres were studied to a magnification range of ×100 to ×800. Suspected or identifiable species and genera of fibres were recorded and then, as the analysis progressed, initial observations and conclusions were altered accordingly. At least two slides were examined per paper while in some cases, such as in the search of vessel element information or when fibres were difficult to identify, several slides were required.

Fibre identification

In identifying the components of wood pulps many of the positive morphological features employed in solid wood identification (pore arrangement, parenchyma patterns, ray structures in hardwoods and resin canals, longitudinal parenchyma, earlywood-latewood transition in softwoods) no longer existed and, consequently, attention was focused on the structural characteristics of one or two cell types. Practical limitations on microscopical identification also arose from degradation (cutting and shortening, tearing, fibrillation, etc.) of fibres due to processing as well as from the presence of similar species (*e.g.* species of the same genus that are closely related in anatomical structure) in the pulp mix. These constraints severely limited the identification of individual softwood and hardwood species, which in general was made to genera or subgroups of genera.

The identification of softwoods was performed mainly on the basis of cross-field pitting (pits in the crossings of longitudinal tracheids and the ray cells) of the thin-walled earlywood tracheids (Strelis & Kennedy 1967; Panshin & DeZeeuw 1980). The latewood tracheids have thick walls and narrower lumens, their cross-field pits are fewer, appear reduced in size, crowded and in most of the cases are not distinctive. The type of pits to ray parenchyma, the number of pits per transverse row (cross-field) as well as their arrangement in the cross-field area aids the classification of softwoods into broad groups with similar cross-fields features. In this study the classification proposed by Ilvessalo-Pfäffli (1995) for the most common softwoods used for papermaking was employed and is shown in Table 2. In this classification, besides the cross-field pitting,

Table 2. Grouping of common papermaking softwoods according to Ilvessalo-Pfäffli (1995) based on anatomical features of earlywood tracheids.

Anatomical feature		
Pits to ray parenchyma	Structure of the inner wall of ray tracheids	Softwood groups
Window-like		
2–3 (1–4) per CF, except for *P. strobus*: 1 (1–2)	ray tracheids nondentate	**Strobus group:** *Pinus strobus, P. monticola, P. lambertiana*
1 (1–2) per CF	ray tracheids dentate	**Sylvestris group:** *Pinus sylvestris, P. resinosa*
Pinoid		
2–3 (1–4) per CF	ray tracheids nondentate to shallowly dentate	**Halepensis group:** *Pinus halepensis*
2–5 (1–6) per CF	ray tracheids prominently dentate	**Ponderosa group:** *Pinus ponderosa, P. contorta, P. patula, P. radiata, P. pinaster*
2–5 (1–7) per CF	ray tracheids strongly dentate to reticulate	**Taeda group:** Southern pines (*Pinus taeda, P. echinata, P. elliottii, P. palustris, P. rigida*), *P. caribaea, P. banksiana*
Window-like and Pinoid		
1–2 (3, rarely 4) per CF	ray tracheids mostly dentate	**Kesiya group:** *Pinus kesiya, P. merkusii, P. densiflora, P. nigra*
Piceoid		*Picea, Larix* and *Pseudotsuga*
Cupressoid		*Tsuga* and *Chamaecyparis*
Taxodioid		*Abies, Cryptomeria, Thuja, Taxodium, Sequoia*
Podocarpoid		*Podocarpus spicatus*
Araucaroid		*Araucaria angustifolia*

CF: cross-field

the grouping of pines was made mainly on the basis of the structure of the inner walls of the ray tracheids. Other anatomical features, but only with auxiliary value, used for differentiation were pits to ray tracheids (presence or absence), height of cross-field areas, intertracheid pitting (number of vertical rows), spiral thickenings (presence) and width of earlywood tracheids.

Although hardwoods are more complex in their anatomical structure and have greater diversity of cell types than softwoods, information from the vessel elements was the primary source for their identification. Fibres were excluded due to their similarity in morphology and only the presence of vascular or vasicentric tracheids was of diagnostic value as they occur in only a few species (Strelis & Kennedy 1967; Panshin & De Zeeuw 1980; Ilvessalo-Pfäffli 1995). According to the above, differentiation of hardwood species or genera was based on the features of vessel elements (size and shape, type of perforations, presence of spiral or reticulate thickenings, type of intervessel pitting, size, shape and arrangement of pits to ray parenchyma and presence of pits to vascular or vasicentric tracheids).

Nonwood fibres used for papermaking are derived from selected tissues of various monocotyledonous or decotelydonous plants (Parham & Kaustinen 1974). Due to this wide variety as well as to the difficulties in identifying individual species because of the similar morphology of cellular elements (Strelis & Kennedy 1967), identified components were grouped to grasses (agricultural residues and natural growing plants), bast and leaf fibres and fruit fibres (cotton). Attention was paid to the presence of cells other than fibres such as parenchyma, epidermal, vessel elements and rings from annular vessels, to the general shape of fibres including width and length and to the shape of fibre ends.

Identification of both wood and nonwood fibres of the packaging grade papers was principally made by comparing the fibres and other cell types observed under the microscope with fibre photomicrographs and descriptive information taken from Carpenter & Leney (1952), Strelis & Kennedy (1967), Côté (1980), Parham & Gray (1990) and Ilvessalo-Pfäffli (1995). Direct use of identification keys, such as the one proposed by Panshin & De Zeeuw (1980), was avoided and served only as an additional and confirmative aid in the recognition of fibres. In general, identification keys have proved to be unsatisfactory for commercial pulps, which contain a variety of similar fibre types with disoriented diagnostic features, often distorted from processing (Strelis & Kennedy 1967; Parham & Gray 1990).

Fibre counting

The centre marking of the eyepiece was located about 2–3 mm from one corner of the cover glass and then the different kind of fibres passing under the cross-hair were counted by traversing the slide in horizontal lines, each 5 mm apart. Counting of fibres was made at a magnification of ×80 while, at points of interest, it was necessary to move the slide vertically and increase the viewing magnification (up to ×800) in order to study the structural details of fibres. The vertical position of the mechanical stage was noted for each particular traverse, to facilitate the return to the original line after such movements. Repeated passing along the same horizontal line were frequently required as to confirm the count of each fibre type. Parts of the same fibre that passed the cross-hair more than once were counted each time. Fibres in a bundle were counted separately as they passed under the cross-hair.

Before actual counting began fibres were classed into softwood, hardwood and nonwood fibre components. Softwood fibres were further classed into an appropriate category from those established beforehand for each paper according to the classification proposed by Ilvessalo-Pfäffli (1995). Softwood fibres that were not possible to be identified (usually latewood fibres) were tallied in the unidentifiable category. Those fibres were then distributed among the identifiable softwood classes by an apportionment based on the final number percentage of each class. The similarity in morphology and structure of hardwood and nonwood fibres precluded the establishment of additional fibre classes for individual species within these groups. Hardwood and nonwood vessel elements were also counted as fibres.

Fibre fragments less than 0.1 mm were ignored as well as parenchyma cells and ray tracheids. Larger fragments of the same fibre type were mentally counted as fractions, so when two or three of them were observed in the same line, they were ultimately converted into whole fibres. Fibres that appeared to have been shortened only slightly (visual estimation) were counted as whole fibres. Unidentifiable softwood fragments were first converted into whole fibres and were then apportioned as mentioned before.

According to ISO 9184-1 (ISO 1990) not less than 600 fibres should be counted in order to achieve an acceptable level of precision for any quantitative fibre analysis. Therefore, at least two slides were counted for each paper.

RESULTS AND DISCUSSION

Knowledge on the geographical source of the sample offers a great advantage in differentiation among similar species (Isenberg 1980, 1981). The origin of virgin and waste pulp (domestic or imported) is of low importance for corrugated board manufacturers as their paper supply is driven mainly by price and availability (Laufenberg 1997). Lack of background information on packaging grade papers used in this study, together with the variety of fibre types incorporated in such products, made fibre identification less specific and resulted in an increase of the number of potential species. Although the geographical distribution of the common papermaking woods (Critchfield & Little 1966) was taken into account in drawing conclusions, it must always be remembered that many cultivated and imported trees are used for papermaking.

The results of this study are summarised in Tables 3 & 4 and in Figures 1–4. Table 3 shows the occurrence of different genera, groups of genera and species of identified softwoods, hardwoods and nonwood fibres while the number of fibres counted for each category is given in Table 4. In Figures 1–4 selected photomicrographs show some of the diagnostic features used for the recognition of the fibre components of papers.

Softwood components

Almost all packaging grade papers were found to contain an abundance of earlywood tracheids belonging to the Sylvestris or Strobus group, the Ponderosa group and to the genera *Larix* or *Picea* (Tables 3 & 4). The Sylvestris and Strobus groups were combined into one group because ray tracheids were rarely located for the proper differentiation between *Pinus strobus* (Strobus group) and the two species of the Sylvestris group (*Pinus sylvestris* and *P. resinosa)* as shown in Table 2. Nondentate ray tracheids (*Pinus strobus*) were only observed in paper KL1 while in papers KL1 and KL2 some earlywood tracheids with the characteristics (narrow window-like pits, tracheids somewhat wider with partly biseriate intertracheid pitting) of *Pinus resinosa* were found. In all the other papers tracheids resembled *Pinus sylvestris* (generally 1 window-like pit per cross-field, uniseriate intertracheid pitting, and dentate ray tracheids when they occurred) (Fig. 1C). Wide earlywood tracheids with 2–3-seriate intertracheid pitting having 2–3 window-like pits per cross-field (*Pinus lambertiana* and *P. monticola*) were not observed. Examination of pines of the Ponderosa group showed that tracheids were mainly *Pinus pinaster* (especially in papers KL1, KL2, KL3, TL, WBL1, WBL3, SQ3, SQ4, F1 and F3) followed by *Pinus radiata* (in papers TL, WBL1, F1 and F3). Apart from the features given in Table 2, *Pinus pinaster* (Fig. 1A) is characterised by

Table 3. Occurrence of different genera, groups of genera and species in packaging grade papers.[1]

Genera, groups of genera and species	Occurrence														
	Linerboards							Corrugating medium							
	KL1	KL2	KL3	TL	WBL1	WBL2	WBL3	SQ1	SQ2	SQ3	SQ4	SQ5	F1	F2	F3
Softwoods															
Sylvestris or Strobus group	+	+	+	++	++	++	++	+	+	+	++	+	++	++	++
Ponderosa group	++	++	++	++	++	+	+		+	+	+	+	+	+	+
Pinus halepensis	+														
Taeda group	+		+	+	+	+	+				+		+		
Pinus nigra			+	+	+	+	+			++			+	+	+
Abies	+		+			+	+				+			+	
Larix or *Picea*	+	+	+	+	+	+	++	++	+	+	+	+	+	+	++
Pseudotsuga menziesii	+		+	+	+	+	+				+		+	+	+
Hardwoods															
Eucalyptus	++	++	+	++	++	++	++		+	++	++	+	++	++	++
Betula	++	++	++	++	++	++	++	++	++	++	++	++	++	++	++
Populus	+	+	++	++	++	++	++	+	++	++	++	+	+	++	++
Fagus sylvatica		+	+	+	++	+	+		++		++		+		+
Tilia	+				+	+	+		++			+			
Acer			+												
Alnus	+		+											+	
Quercus			+												
Castanea sativa								+				+		+	
Liquidambar styraciflua		+			+									+	
Liriodendron tulipifera			+	+	+							+			
Nyssa sylvatica						+							+		
Magnolia acuminata				+						+			+		+
Magnolia grandiflora					+										
Nonwood fibres															
Grasses	+	+	+	+	+	+	+		+	+	+	+	+	+	+
Bast and leaf fibres	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
Fruit fibres (Cotton)				+		+					+		+	+	+

[1]) +: occurrence and ++: frequent occurrence (based on visual estimation of the relative amounts of earlywood tracheids with different morphological characteristics for softwoods, different vessel elements for hardwoods and fibres and associated cells for nonwood components).

[2]) Classification according to morphological characteristics of fibres (earlywood tracheids for softwoods, vessel elements for hardwoods, different cell types for nonwood components).

Table 4. Fibre counts in packaging grade papers.

Fibre category	Fibre counts														
	Linerboards							Corrugating medium							
	KL1	KL2	KL3	TL	WBL1	WBL2	WBL3	SQ1	SQ2	SQ3	SQ4	SQ5	F1	F2	F3
Softwoods															
Sylvestris or Strobus group	48	31	23	100	33	49	40	11	18	3	71	21	99	112	52
Ponderosa group	113[1]	159	197	63	28	12	29	–	6	38	28	2	16	16	18
Taeda group	13	–	27	7	9	3	7	–	–	–	3	–	3	–	–
Pinus nigra	–	–	31	7	4	6	18	–	–	67	–	–	28	4	5
Abies	11	–	23	–	–	9	11	–	–	–	7	–	–	12	–
Larix or *Picea*	10	7	20	14	14	25	47	35	12	24	20	5	33	28	36
Pseudotsuga menziesii	3	–	8	3	4	3	3	–	–	–	1	–	2	1	2
Sub–total	198	197	329	194	92	107	155	46	36	132	130	28	181	173	113
Hardwoods	415	409	281	421	491	508	499	635	552	419	475	601	410	440	462
Nonwood fibres	31	35	20	33	46	53	37	5	26	54	53	6	41	40	32
Total fibre count	644	641	630	648	629	668	691	686	614	605	658	635	632	653	607

[1]) and *Pinus halepensis*.

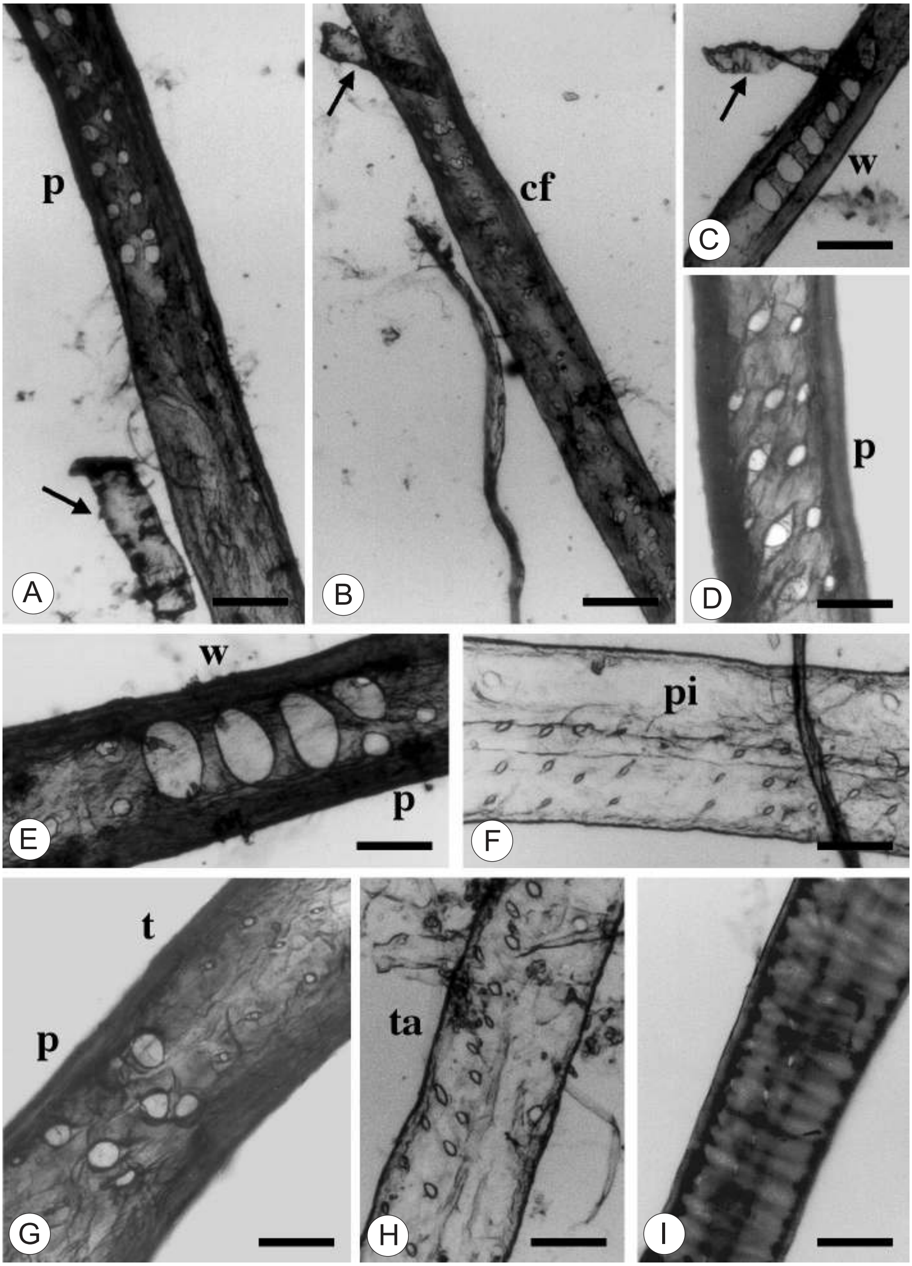
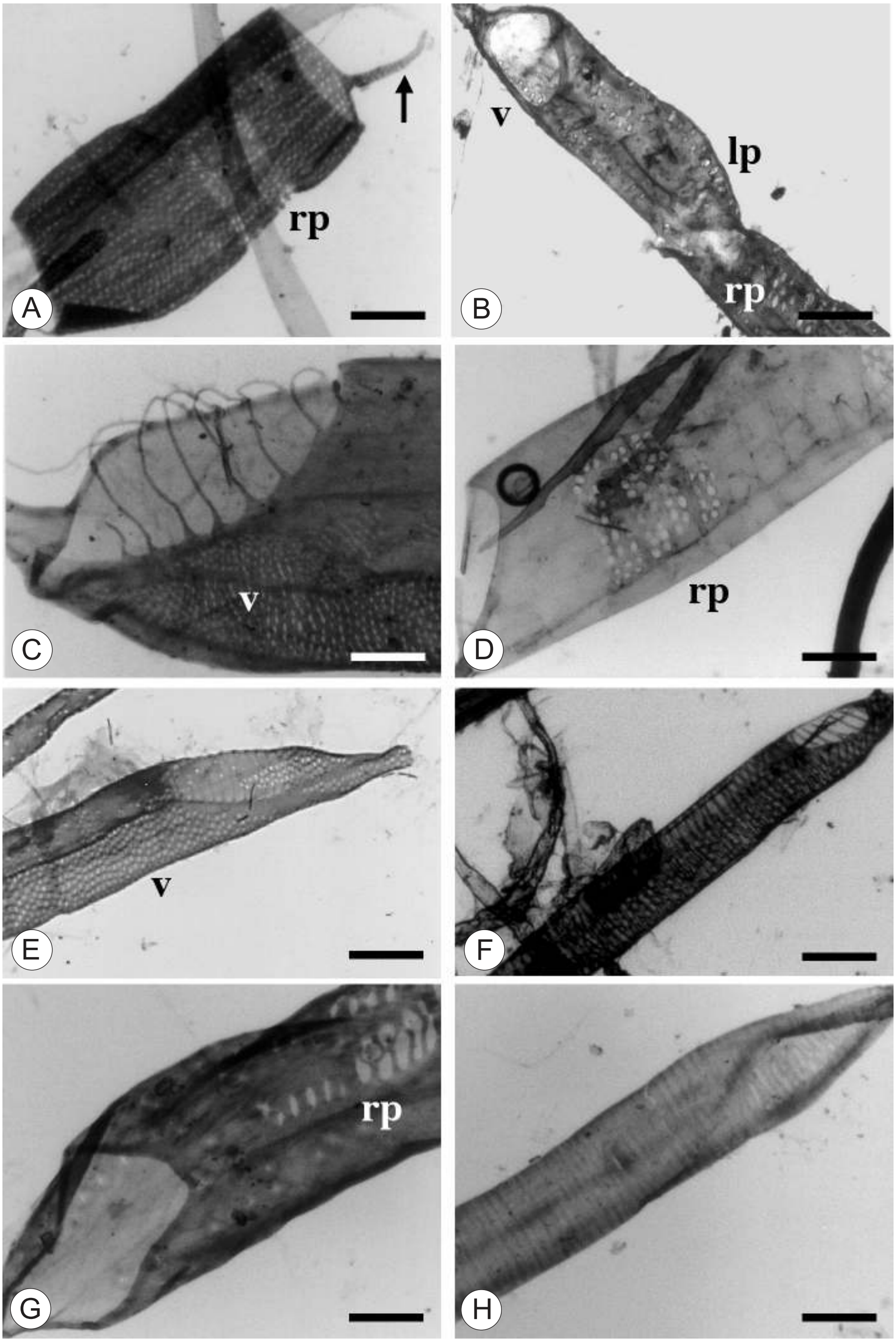


Fig. 1. Identification of softwoods in packaging grade papers based on the cross-field pitting of earlywood tracheids. – A: Oval pinoid pits (p) in horizontal rows and a ray tracheid with prominently dentate inner walls (arrow) of *Pinus pinaster* (paper KL3). – B: *Pinus halepensis* with small oval pinoid pits in horizontal rows forming a high cross-field area (cf) and shallowly dentate inner walls (arrow) of a ray tracheid (paper KL1). – C: *Pinus sylvestris* in paper F1

the oval pinoid pits, uniform in size, arranged in horizontal or diagonal rows while *Pinus radiata* (Fig. 1D) shows a diagonal arrangement and pointed ends of the pinoid pits (Ilvessalo-Pfäffli 1995). A few tracheids of the rest of the pines comprising the Ponderosa group (*Pinus ponderosa*, *P. contor*ta and *P. patula*), having their pinoid pits in irregular groups or rows, were evidently observed only in paper KL2. These species do not show well-discernible distinguishing features (Ilvessalo-Pfäffli 1995), so a decision included uncertainties. It should be noted that identification of pines within the Ponderosa group was not always possible as it was often difficult to find intact pinoid cross-field pits. As shown in Table 4, tracheids of *Larix* or *Picea* (Fig. 1F) with piceoid ray parenchyma pits (Table 2) were numerous in Semi-chemical (except for SQ5) and in waste-based papers (Waste based-liner and Fluting). *Larix* and *Picea* are not distinguishable from one another in mixed pulps (Strelis & Kennedy 1967; Parham & Gray 1990), a rule that was also verified in this study. Nevertheless, in a few cases some features helped to separate the two genera. Narrow earlywood tracheids of *Picea* with uniseriate intertracheid pitting were clearly observed in papers KL1, KL3, F1 and F2 while wider earlywood tracheids of *Larix* profusely pitted with frequent occurrence of high-cross field areas and biseriate intertracheid pitting were apparent in papers KL2, TL, WBL2, WBL3 and F3 (Table 3).

In paper KL1 (see Table 3) a small number of tracheids having the characteristics of *Pinus halepensis*, 2–3 small oval pinoid pits in horizontal rows forming high cross-field areas and nondentate to shallowly dentate inner walls of ray tracheids (Ilvessalo-Pfäffli 1995), were detected (Fig. 1B). Tracheids of the Taeda group (Fig. 1G) were found mostly in linerboards but in small amounts (Table 4) and usually broken. Although the differentiation between the Taeda and Ponderosa groups, according to Ilvessalo-Pfäffli (1995), is based on the structure of the inner walls of the ray tracheids (see Table 2), in the present study this practice proved ineffective due to the fact that ray tracheids were rarely located (except for paper KL3). Tracheids within the Taeda group were identified by being in general wider (Koch 1972) and showing 2–3-seriate intertracheid pit-ting (mostly uniseriate within the Ponderosa group). Also, those tracheids had more pinoid pits in regular groups while the pinoid pits within the Ponderosa group tend to be in irregular groups or in rows (Ilvessalo-Pfäffli 1995). Identification of individual species was not possible due to the inadequate diagnostic features. Nevertheless, all the evidences suggested a southern pine. This was also supported by the exclusion of *Pinus rigida* and *P. banksiana* (their tracheids are narrower with uniseriate intertracheid pitting).

←
with window-like ray parenchyma pits (w) and dentate inner walls (arrow) of a ray tracheid. – D: *Pinus radiata* in paper TL characterised by the diagonal arrangement and pointed ends of the pinoid pits (p). – E: Combination of window-like (w) and pinoid (p) ray parenchyma pits in a cross-field area of *Pinus nigra* (paper WBL3). – F: Cross-field area of *Picea* or *Larix* composed of piceoid (pi) pits (paper SQ4). – G: Wide earlywood tracheid of the Taeda group (paper KL3) with 2–3-seriate intertracheid pitting (t) and a group of pinoid pits (p). – H: Portion of a cross-field area of *Abies* showing taxodioid ray parenchyma pits (ta) in paper SQ4. – I: Part of an earlywood tracheid of *Pseudotsuga* showing spiral thickenings (paper F2). — Scale bars = 40 μm for A; 50 μm for B & C; 25 μm for D–I.


rp
A
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H

In some papers (especially in SQ3, F1, KL3 and WBL3) tracheids of the Kesiya group (Table 2) were identified by both window-like and pinoid cross-field pits. Geographical distribution of pines that belong to the Kesiya group (Critchfield & Little 1966) but, more significantly, comparison of their diagnostic features according to Ilvessalo-Pfäffli (1995) revealed that in all cases the species was *Pinus nigra* (Fig. 1E). Cross-field areas containing only window-like ray parenchyma pits were common, making the differentiation from *Pinus sylvestris* difficult in papers where both species were present. The fact that tracheids of *Pinus nigra* may be wider with locally biseriate pitting (Ilvessalo-Pfäffli 1995) helped the differentiation in some cases.

Genera with minor importance were *Abies* (except for paper KL3) and *Pseudotsuga* (Table 4). Earlywood tracheids of *Abies* (Fig. 1H) with 1–2-seriate intertracheid pitting were observed, generally in small numbers, mainly in linerboards. *Abies* could be confused with the other genera having taxodioid pits (Table 2), especially *Thuja* and *Taxodium*, but the tracheids of the latter are wider (Isenberg 1980) with 2–3-seriate intertracheid pitting walls that tend to be profusely pitted (Ilvessalo-Pfäffli 1995). In papers KL3 and WBL3 the presence of high cross-field areas was an indication of *Abies alba*. *Pseudotsuga* was seldom found (2–3 tracheids per slide, usually broken) mainly in the waste based papers (Waste based-liner and Fluting) and could be visibly identified by the occurrence of spiral thickenings in the earlywood tracheids (Fig. 1I).

Hardwood components

Except for kraft-liner KL3, in all packaging grade papers hardwoods were found to be the main fibre component (Table 4). Particularly, some semi-chemical grades such as SQ1, SQ2 and SQ5 were almost entirely composed of hardwoods. The most important hardwood components were *Eucalyptus*, *Betula* and *Populus* as their vessel elements were plentiful in all packaging papers (Table 3). *Eucalyptus* (Fig. 2A) was apparently identified by the medium to large, oblong, profusely pitted vessel elements with thread-like tails and characteristic ray parenchyma pitting (pits in horizontal rows or groups, relatively large, mostly simple, oval to rounded, with a tend to elongate vertically). The presence of vasicentric tracheids and fibre tracheids gave additional

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Fig. 2. Identification of hardwoods in packaging grade papers based on the features of vessels elements. – A: Large, profusely pitted vessel element of *Eucalyptus* in paper F3 with tread-like tail (arrow) and pits to ray parenchyma (rp) in horizontal rows. – B: Vessel element of *Fagus sylvatica* in paper SQ2 showing intervessel pits (v) and pits to ray (rp) and longitudinal parenchyma (lp). – C: Very small intervessel pits (v) and scalariform perforation on a vessel element of *Betula* (paper SQ1). – D: Vessel element of *Populus* in paper SQ2 with simple perforation and pits to ray parenchyma (rp) in horizontal groups of 2–3 rows. – E: Crowded intervessel pits (v) with oval outlines and scalariform perforation on a vessel element of *Alnus* in paper KL1 (note the difference from C where the pits of *Betula* are smaller, more crowded and confluent). – F: Linear vessel element of *Tilia* showing prominent spiral thickenings (paper SQ2). – G: Portion of a vessel element of *Magnolia acuminata* in paper F3 showing simple perforation and large, oval to elongated, pits to ray parenchyma (rp). – H: Combination of scalariform perforation and spiral thickenings in a vessel element of *Magnolia grandiflora* (paper WBL1). — Scale bars = 100 μm for A; 50 μm for B, D & F; 25 μm for C, E, G & H.

evidence. *Eucalyptus* species are not distinguished from one another in pulp but the three major species used for pulp production are *Eucalyptus globolus*, *E. camaldulensis* and *E. grandis* (Wagenführ & Scheiber 1974). Together with *Eucalyptus*, the fairly large linear vessel elements (up to about 1 mm) of *Betula* (Fig. 2C), with scalariform perforations and very small, crowded, alternately arranged intervessel pits, were the most plentiful (than vessel elements of the rest genera) in all papers (Table 3). Vascular tracheids, with closed ends, of *Betula* were occasionally present. The two European species of the genus, *Betula verrucosa* and *B. pubescens*, are important pulpwoods (Hora 1981) but cannot be distinguished in pulp (Ilvessalo-Pfäffli 1995). In papers KL1, KL3 and F2 *Betula* could be confused with *Alnus* (Fig. 2E) that was present in small amounts in the hardwood mix. Vessel elements of *Alnus* and *Betula* appeared similar in low magnification and closer examination was necessary to reveal that the pits of *Betula* are smaller, more crowded and often confluent (Parham & Gray 1990). *Alnus* could be either *Alnus glutinosa* or *A. incana*, the two European species used for pulping (Wagenführ & Scheiber 1974). Vessel elements of *Populus* (Fig. 2D) were also found in all papers in small or large amounts. *Populus* was easily recognised by the medium-long to long linear vessel elements with simple perforations and characteristic homocellular ray parenchyma pitting (pits were relatively large, oval to oval-angular, simple and occurred in horizontal groups of usually 2 or 3 rows). Intervessel pits, alternate and crowded, were also visible. *Populus* comprises many species and hybrid forms (Panshin & DeZeeuw 1980), which are not distinguished from one another in pulp (Parham & Gray 1990).

Vessel elements of *Fagus sylvatica* (Fig. 2B), the only species of the genus *Fagus* that grows in Europe (Luukkanen 1981), were found in most of the papers and were sometimes (SQ2 and SQ4) amongst major hardwood components. Simple perforations and scattering pitting characterise the medium-long vessel elements of *Fagus sylvatica* (Parham & Gray 1990; Ilvessalo-Pfäffli 1995). Narrow vessel elements with scalariform perforations were also noted. *Tilia* was also frequently observed in the papers (frequent occurrence in SQ2 and in all Waste based-liners) by having small linear vessel elements with very prominent spiral thickenings (Fig. 2F) and small ray parenchyma pits. The spirals of *Tilia* are more prominent and widely spaced compared to those of *Acer* (Ilvessalo-Pfäffli 1995), which were observed in paper KL3. The genus *Tilia* comprises about 35 species, with *Tilia cordata* and *T. platyphylla* being the most common in Europe (Grosser 1977).

The rest of the hardwood species was infrequently encountered in the papers (1–2 vessel elements per slide), with the exception of *Magnolia acuminata* in paper F3 and *Liriodendron tulipifera* in papers WBL2 and F1 (Table 3). Most of the species are indigenous to America and Asia (Fowells 1965; Little 1979) and their rare presence should be attributed to the recycling process. *Liquidambar styraciflua*, *Lyriodendron tulipifera* and *Nyssa sylvatica* are often pulped together (Isenberg 1981) and characterised by the large linear vessel elements with scalariform perforations. Their differentiation (Fig. 3A-C) was based on diagnostics by Ilvessalo-Pfäffli (1995), which included the size of vessel elements (max length 1.7 mm in the two first, 1.0 mm in the third), the number of bars (20–55, 15–25 and 2–10, respectively), intervessel pitting (opposite, scalariform

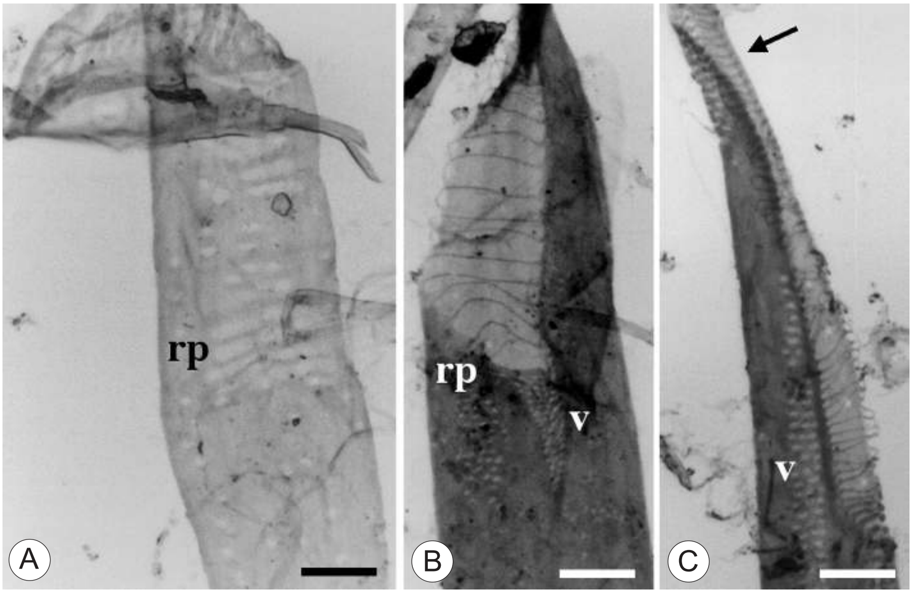

Fig. 3. Differentiation between *Liquidambar styraciflua*, *Lyriodendron tulipifera* and *Nyssa sylvatica*. – A: End and central portion of a vessel element of *Lyriodendron tulipifera* in paper F1 with scalariform perforation and large, oval to elongated pits to ray parenchyma (rp). – B: Vessel element of *Nyssa sylvatica* (paper WBL2) showing scalariform perforation, small pits to ray parenchyma (rp) in groups and opposite intervessel pitting (v). – C: End portion of a long vessel element of *Liquidambar styraciflua* in paper KL3 showing scalariform perforation, scalariform intervessel pitting (v) with 1–3 pits per transverse row and faint spiral thickenings (arrow) on the tip of the vessel element tail. — Scale bars = 25 μm.

to opposite and opposite, respectively) and the size of pits to ray parenchyma (small, medium and large, respectively). *Magnolia acuminata* was recognised by the scalariform intervessel pitting and simple perforations of linear vessel elements (Fig. 2G) while *Magnolia grandiflora* was identified only in paper WBL1 by the combination of scalariform perforations, spiral thickenings and scalariform intervessel pitting (Fig. 2H). Ring-porous hardwoods were represented sporadically in the papers by *Castanea sativa* (papers SQ2, F1 and F3) and *Quercus* (only in paper TL). In both cases, large and wide earlywood vessel elements were found, which appeared similar in low magnification, and only a systematic examination allowed their differentiation. The pits (oval) to ray parenchyma of *Castanea sativa* tend to be elongated horizontally, those of *Quercus* vertically (Ilvessalo-Pfäffli 1995), a feature that proved the most reliable.

Nonwood fibre components

Nonwood fibres were found less frequently than softwoods and hardwoods (Table 4) but comprised a significant fibre component in most of the packaging grade papers. Papers with high numbers were the waste-based (liners and flutings) and the semi-

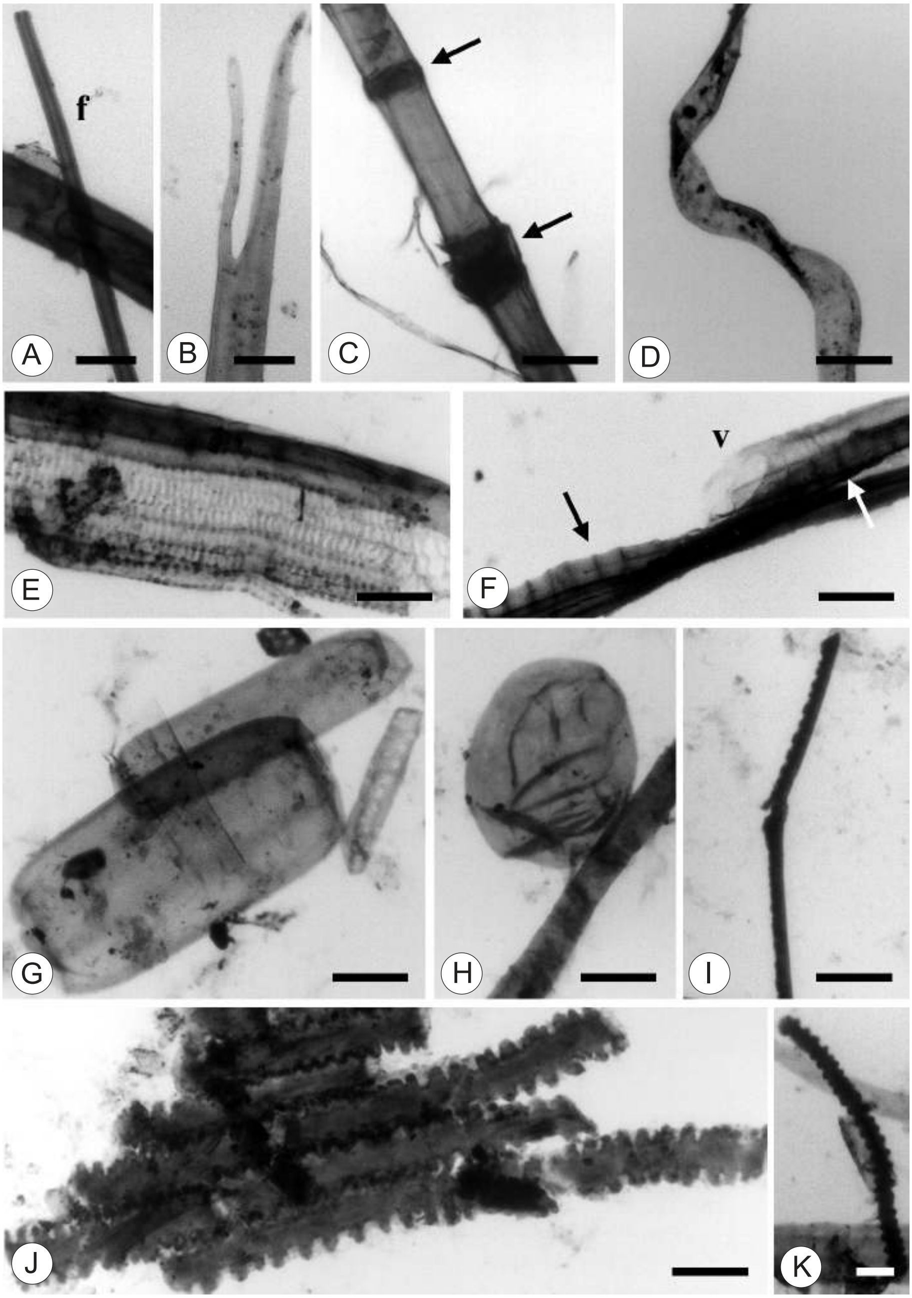

Fig. 4. Different types of cells of nonwood fibre components observed in packaging grade papers. – A: Typical narrow, thick-walled grass fibre (f) with blunt end (paper KL2). – B: Forked end of a wide, thin-walled grass fibre (paper WBL3). – C: Dislocations (arrows) in a bast fibre (paper WBL2). – D: Portion of a ribbon-like, twisted fibre of cotton (paper SQ4). – E: Pitted

chemicals SQ3, SQ4. Only in two of the papers examined (SQ1 and SQ5) nonwood fibres were found to be a trivial component. Obviously, nonwood fibres were not used directly in the production of the papers but entered the manufacturing process through recycling.

The leading group of nonwood fibres from a quantity standpoint were grasses. Different types of cells of grasses (fibres, vessel elements, parenchyma cells and cells of epidermis) were numerous in the waste-based papers (Flutings and Waste based-liners) as well as in some Semi-chemicals (SQ3 and SQ4) and rare in papers SQ2 and SQ3. The great variation in size and shape of cells showed that in most of the papers more than one grass was present. Kraft-liners and Test-liner appeared less complex and all the features observed were typical of a cereal straw (large sac-like parenchyma cells, large regularly shaped epidermal cells) in paper KL2, of reed (*Phragmites communis*) (large pitted vessel elements, relative large mostly oblong parenchyma cells, similar to epidermal cells of cereal straws but smaller) in papers KL1 and KL3, or of their combination (paper TL). Identification of individual species in the rest of the papers (except for SQ1, SQ2 and SQ5, where the rarity of diagnostic features precluded any attempt) was a rather complex task because of the similarity in appearance of the cellular elements. The typical narrow, thick-walled grass fibres with pointed or blunt ends (Fig. 4A) and the wide, thin-walled fibres with varying shapes of fibre ends (Fig. 4B) that were observed are of restricted diagnostic value (Ilvessalo-Pfäffli 1995). Very narrow fibres found in papers WBL2, WBL3, SQ4, F1 and F3 are characteristic of sabai (*Eulaliopsis binata*), rice (*Oryza sativa*), albatrine (*Lygeum spartum*) and esparto (*Stipa tenacissima*) (Ezpeleta & Simon 1971). The large pitted vessel elements that were observed in papers WBL2, F1, F2 and F3 resembled those of sugar cane (*Saccharum officinarum*) and reed, while the small ones in paper SQ3 and SQ4 occur in albatrine and esparto. Different combinations of pitted vessel elements (Fig. 4E), parenchyma and epidermal cells with varying shapes and sizes were also observed, often in the same paper. A conclusion about the species based on these cell types could be made only in a few cases. The large, thin-walled, sac-like parenchyma cells in papers WBL2, SQ3 and F1 (Fig. 4G, H) are characteristic of cereal straws, corn (*Zea mays*) and sugar cane (Strelis & Kennedy 1967). Small, rectangular, thick-walled parenchyma cells in paper F3 gave an indication of bamboo (*Dendrocalamus strictus*). The characteristic regularly shaped long epidermal cells of cereal straws (Strelis & Kennedy 1967) were noted in papers WBL2, F1 and F3 (Fig. 4K). Also regularly shaped epidermal cells, smaller than those of cereal straws, esparto and reed, were found in almost all papers (Fig. 4J). In paper KL1, the characteristic epidermal cells of rice were detected (Fig. 4I).

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vessel element of a grass in paper F3. – F: Bast fibres with dislocations (arrows) accompanied by a pitted vessel element (v) in paper F3. – G: Thin-walled grass parenchyma cells of various sizes with folded ends in paper WBL2. – H: Rounded grass parenchyma cell in paper F1. – I: Narrow epidermal cell of rice with conical protuberances (paper F3). – J: Regularly shaped epidermal cells of a grass (WBL2). – K: Large, regularly shaped long epidermal cell of a cereal straw (paper F1). – Scale bars = 40 μm for A, D, F, H & I; 20 μm for B; 25 μm for C, E & K; 50 μm for G & J.

In all papers, especially in waste-based (Flutings and Waste based-liners) and in some Semi-chemicals (papers SQ2, SQ3 and SQ4), a very small number of bast fibres was found (Fig. 4C & F). The presence of a bast fibre species (flax, hemp, kenaf, jute, ramie, *etc*.) was revealed mainly by the typical thick-walled bast fibres and less by other associated cells. Recognition was possible due to the structural features (general shape, surface markings, shape of lumen, shape of fibre ends, irregularities in fibre walls) of the fibres and the most reliable amongst them proved to be the varying shape of fibre ends (pointed, blunt, forked, *etc*.). The limited availability of diagnostic features did not allow species identification. Leaf fibres (abaca, sisal, *etc*.), similar in appearance to bast fibres but stiffer and fairly smooth, were grouped together with bast fibres and were also encountered sporadically in the papers. The fruit fibre found in some of the papers (especially in the Flutings) was cotton. Not more than 2–3 cotton fibres (linters) were observed per slide and were easily recognised by being smooth-looking, ribbon-like and twisted (Fig. 4D).

CONCLUSIONS

The waste-based papers were highly variable containing up to 18 different wood and nonwood components in the case of Waste based-liners and up to 16 in the case of Flutings. Kraft-liners and Test-liner also exhibited significant variability comprising 9–16 components. Semi-chemical was the less variable grade, having 5–13 components. In most of the papers the number of hardwood and nonwood components (3–11) was much higher than the number of softwood components (2–7) (Table 3).

Fibres of the Sylvestris or Strobus group (mainly *Pinus sylvestris*), Ponderosa group (mainly *Pinus pinaster* followed by *P. radiata*) and of genera *Larix* or *Picea* were found in abundance in almost all packaging grade papers. *Pinus nigra* as well as pines of the Taeda group (mainly southern pines) were present in small amounts in some papers. Genera with minor importance were *Abies* and *Pseudotsuga*.

Hardwoods were identified as the major fibre component in all papers, except one kraft-liner grade. All papers contained *Betula, Eucalyptus* and *Populus* (with this order in most of the papers) in their hardwood mix. *Fagus sylvatica* was found in most of the papers and in some of them it was amongst the major hardwood components. *Tilia* was also frequently observed . The rare presence of *Alnus, Castanea sativa, Quercus, Liquidambar styraciflua, Lyriodendron tulipifera, Nyssa sylvatica, Magnolia acuminata* and *M. grandiflora* (with the exception of *Magnolia acuminata* and *Liriodendron tulipifera* in some waste based papers) was attributed to the recycling process.

Nonwood fibres, mainly grasses, were found in all packaging grades (less frequently than softwoods and hardwoods) as a result of the recycling process. In most of the papers more than one grass was present but identification of individual species was a rather ambitious task. In all papers a very small number of bast and leaf fibres was found. In addition, in some papers cotton linters were located but in insignificant numbers.

Packaging paper manufacturing integrates a continuously increasing variety of species, many of them being out of their natural distribution, a phenomenon which besides the recycling process is due to globalisation and international trade of pulp, paper and wood.

ACKNOWLEDGEMENTS

This study has been carried out within the framework of a EU Marie Curie Postdoctoral Fellowship titled "Characterization of packaging from paper and paper products through the study of fibre composition" (contract number HPMD-CT-2000-00043, project coordinated by Prof. Dr. José-Vicente Oliver).

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