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• **Gaggini, Claudio**
51020 Popiglio (Pistoia) (IT)

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(74) Representative: **Dragotti, Gianfranco et al**
SAIC BREVETTI s.r.l.
Galleria San Babila, 4/D
20122 Milano (IT)

(71) Applicant: **CARTIERA DELL'ANIA SPA**
55055 Ponte all'Ania - Barga (Lucca) (IT)

(72) Inventors:
• **Meucci, Giuliano**
51019 Ponte Buggianese (IT)

(54) **Method for the production of cellulose pulp**

(57) The use of the annual plant known as *Luffa cylindrica* in methods for the production of cellulose pulp for use in papermaking is described.

FIG. 2



EP 0 791 683 A1

Description

The present invention relates to paper production and, in particular, to the production of cellulose pulp for use in papermaking.

Nowadays, paper production is based fundamentally upon the use of plants and trees as the source of fibrous raw materials and upon the processing of recycled paper materials, whereas the use of rags, which for centuries were used as the basic raw material, has now in practice become obsolete.

Fibrous materials means vegetable fibres, that is, the thread-like cells constituting the body of the toughest plant tissues which are destined to form the framework of a future sheet of paper. The criterion by which the suitability of a plant or tree for the production of fibrous material for paper may be judged is very complex and has to take account of both quantitative and qualitative parameters, the most important of which is the amount of cellulose it contains. Moreover, extraction should be easy, the fibres should have suitable technical characteristics and the plant or tree should not have intense coloration or unpleasant odours; amongst the technical characteristics, the tensile index, the tear factor, the bursting index and the freeness are of fundamental importance.

Plants and trees used for the production of paper and paper materials can be divided into three main, large categories:

1. Conifers: these include various types, amongst which the best known and the most used are the genus *pinus* (pine) and the genus *abies* (fir); conifer wood contains approximately 50% cellulose, 30% lignin, 15% hemicellulose, and 5% numerous components such as resin, terpene compounds, fatty acids, etc.; conifers are the trees which are used most and which are most suitable for paper production, providing the best "technical cellulose", which is characterized by fibres several millimetres long which impart considerable mechanical strength to the sheet and have a considerable tendency to form fibrils so that they impart considerable compactness and homogeneity to the future sheet of paper.

2. Broad-leaved trees: until about thirty years ago, these were considered by paper-makers as secondary to conifers since the papers thus produced were not distinguished by good mechanical strength; only recently, as a result of improvements in working techniques, have broad-leaved trees started to be used in the papermaking industry and, currently, they constitute one of the main sources of cellulose.

The broad-leaved trees normally used in the papermaking industry are: poplar, birch, chestnut and beech.

3. Annual plants: annual plants, amongst which straw has mainly been considered in the past (today, straw seems to have been practically abandoned) and which include plants such as, for example, kenaf, bamboo, flax and hemp, generally have a lower cellulose content than the woods of coniferous and broad-leaved trees, in most cases, no more than 35%. Annual plants are actually considered a poor substitute for wood as far as their use in the papermaking industry is concerned; this is due not only to their low cellulose content but, in particular, to problems connected with harvesting difficulties and transportation costs and, moreover, with the fact that the papers produced therefrom are distinguished by particularly poor technical characteristics.

However, continual and incessant usage of plurannual woody plants and trees such as coniferous and broad-leaved trees is causing increasing environmental problems since, although deforestation is carried out rationally and systematically, it now greatly exceeds the spontaneous increase in the sprouting-up and development of trees, which is leading to alteration of the ecosystem not only in the regions affected directly but also in the entire planet. In order to solve this worrying and disturbing problem, counter-measures of various kinds have been adopted; whereas, on the one hand, rational planting is continuously carried out on deforested land and on other, suitably prepared land, on the other hand, efforts are being made by means of suitable initiatives such as, for example, advertising campaigns and the like, to sensitize public opinion towards a usage of paper and of cellulose derivatives more generally, aimed at and limited to the absolutely necessary, and towards an increasing use of "recycled paper" and consequent differentiated waste disposal so as to enable paper to be recycled conveniently and economically. In addition, research is now directed ever more towards the establishment of working techniques which can increase yields and thus reduce wood consumption and towards the finding of alternative sources of cellulose to plurannual plants and trees. However, these factors have to take account of the increasing requirement for "highly technical" papers which anyway makes it practically impossible to use annual plants which, precisely because of their annual or even seasonal cyclicality, could constitute an optimal solution to the problem.

It has now surprisingly been found and constitutes the subject of the present invention that, the processing of the annual plant commonly known as *Luffa cylindrica* (or *Luffa aegyptiaca*) produces a cellulose pulp with analytical characteristics decidedly better than those of normal annual plants and, in some aspects, even better than those of coniferous and broad-leaved trees, rendering its use in the paper industry particularly advantageous.

Luffa cylindrica is an annual herbaceous plant belonging to the Cucurbitaceae family; *Luffa* is a creeping, climbing, monoecious plant with unisexual flowers and with stems which may reach lengths of 4-8 m; the leaves are petiolate,

simple, alternate, from tri- to hepta-lobate, and 10-15 cm long; the flowers are conspicuous, large (5-10 cm) with a corolla formed by five yellow petals. The fruit is a pepo, dry when mature, cylindrical or only slightly angular, of very variable dimensions (typically 30-60 cm long and 10-20 cm diameter), and straight or curved; when mature, it has a dry and papery epicarp with a fibrous and spongy endocarp owing to the disappearance of the pulp and the persistence solely of the fibro-vascular bundles. A fruit contains on average 200-500 seeds about 10-15 mm long and each weighing about 0.1g; the fibrous reticulum of the mature fruit is composed of cellulose (60%), hemicellulose (29%) and lignin (11%). Luffa is an annual plant with a relatively long cycle; in a mediterranean habitat, from four to five months elapse from germination to the maturation of the fruits, with a first flowering in July and a second flowering in August.

The warmth requirements of Luffa are high: a lethal minimum of 0-2°C, minimum and maximum temperatures for growth of 12-14 and 20-35°C, respectively, with an optimum situated at around 25°C; the water content is 8.12% at 105°C. Luffa is widespread in tropical and subtropical regions where it is generally the subject of domestic horticultural cultivation. The Luffa fruit, dried after removal of the epicarp and the seeds, is commonly sold as a body sponge in shops specializing in natural body-care products, department stores, perfumeries, etc. One of the most interesting mechanical characteristics of Luffa fibres is in fact their so-called "shape memory" owing to which the fruit can be compressed up to 10-20 times and then returns to its original shape and size if immersed in water or subjected to the effect of steam. The Luffa fruit is also used industrially, practically unchanged, for producing thermally-insulating and sound-absorbent panels, packaging and padding materials, and arch-supports for footwear.

Like all fibres of vegetable origin used in papermaking, Luffa has to be subjected to processing with the object of liberating the fibres from the useless and harmful components, that is, to the processing which, in the papermaking field, takes the name of digestion when chemical agents are used or, on the other hand, grinding when the processing is mechanical. A digestion process is described below, by way of a non-limiting example.

The epicarp and the seeds were removed from the fruit which was then dried, after which it was subjected to a soda digestion process normally used for annual plants, with the use of a discontinuous boiler with a capacity of 10 litres, with manual stirring, under the following conditions:

a) Reagents	
NaOH	18%
Concentration of fibre in the boiler	8.5%
N.B. all of the values relate to the dry fibre.	
b) Operative conditions	
Preheating	(20-110°C) 20 min
Impregnation	20 min
Heating	(100-170°C) 30 min
Digestion	300 min
Operating pressure	6 kg/cm ²

Upon completion of the digestion, the liquor was separated for the analysis and the cellulose pulp was washed with running water to eliminate as much of the exhausted liquor as possible. With regard to the purification of the fibre from undigested matter or the like, this was not carried out since the presence of undigested matter or knots was negligible. The fibre yield was 57.82% which itself is an astounding parameter placing Luffa at the same level as a conifer (the fibre yield of a conifer is normally 50-60%, whereas that of an annual plant is 15-20%).

A simplified beating curve was then performed on the crude cellulose pulp in order to assess the behaviour of the fibre and the curves of the physical-mechanical characteristics upon beating. Moreover, a typical bleaching process was carried out on a portion of beaten pulp in two successive stages which, with regard to the reagents used, are represented below:

1) First stage

Sodium hypochlorite:	6%
NaOH:	1%
Residual hypochlorite:	0
Working temperature:	45-50°C
Duration:	60 min

2) Second stage

Hydrogen peroxide (oxidizing agent):	3%
NaOH (oxidation regulator):	3%
Sodium silicate (oxidation regulator):	0.5%
Working temperature:	65-70°C
Duration:	180 min

N.B.: the quantities relate to the weight of dry cellulose.

Chemical, physical and mechanical determinations were carried out on the crude, beaten and bleached cellulose pulp by means of the apparatus listed below:

Apparatus used	Analytical parameter
FRANK dynamometer	breaking index
KORPUT burst meter	bursting index
ELMENDORF tear tester	tear factor
ELREPHO white	brightness
RIETH hollander	beating curve
TONIOLO sheet form	analysis slips

The methods used were the ATICELCA methods and, where these were lacking, the SCAN methods were used. The results obtained are given in Table 1 and are compared with those relating to a common annual plant (in this particular case, sorghum):

TABLE 1

	Crude Luffa		Bleached Luffa	Crude Annual		Bleached Annual	
Degree of beating (°S.R.)	18 untr.*	37	37	34 untr.	45	34 untr	45
Tensile index (kNm/kg)	44.1	56.0	64.3	54.65	68.2	48.8	65.5
Bursting index (MN/KG)	4.84	5.05	5.06	3.38	3.77	2.60	3.86
Tear factor (Nm ² /kg)	12.12	10.7	10.2	5.2	4.2	-	3.60
Porosity (m ³ /100cc)	3	16	15	80	540	62	780
Ash %	2.4	-	-	4-6	-	-	-
Brightness G.E.	48	40.5	71.5	45.5	-	73	-

*untr. = untreated cellulose pulp, not beaten.

As can be seen from the data relating to the degree of beating, the freeness of the pulp produced from *Luffa* is unexpectedly better than that of the best annual plants and similar to that of a broad-leaved tree. Similarly, the bursting index and the tear factor are clearly better than those of the most common annual plants, whereas the silica content is a great deal lower, as can be seen from the different percentages of ash; only with regard to the tensile index can *Luffa* be considered equivalent to a good annual plant.

The data given above are also confirmed by the images of Figures 1, 2, 3 and 4 which represent, respectively, the fibres (enlarged one hundred times) of crude *Luffa cylindrica*, of beaten *Luffa cylindrica*, of a coniferous-broad-leaved mixture (fir-beech), and of an annual plant (sorgum), from which the similarity between the fibres of *Luffa cylindrica* and those of the coniferous-broad-leaved mixture is clear.

The results relating to the use of *Luffa* are also particularly advantageous with regard to the ecological balance of the process, as can be seen from the data given below:

Ecological Balance	
1) Exhausted liquor	
C.O.D.:	20400 ppm
C.O.D. (kg per ton of pulp):	10.5
Ratio C.O.D./organic matter:	435
2) Bleach	
C.O.D. (kg per ton of bleached pulp - 2-stages):	60.2

In fact, the C.O.D. was in practice less than 50% of that normally necessary as far as annual plants are concerned, with a consequent reduction in disposal costs and, in particular, in possible ecological damage.

On the basis of these considerations, *Luffa cylindrica* is clearly particularly suitable as a starting material for methods for the production of cellulose pulp for use in papermaking; these methods need not be limited solely to digestion processes with soda but may also include all methods in which a vegetable starting material is reduced to fibres or fibre fragments.

Claims

- Method for the production of cellulose pulp for use in papermaking, of the type in which a vegetable material is processed so as to be reduced to fibres or fibre fragments, characterized in that the vegetable material is *Luffa cylindrica*.
- Method according to Claim 1, in which the processing is a digestion process, that is, processing with solutions of chemical and semi-chemical reagents suitable for dissolving most of the lignin contained in the vegetable starting material.
- Method according to Claim 1, in which the processing is carried out by purely mechanical means such as to reduce the vegetable starting material to a mass of individual fibres and fibre fragments.
- Method according to Claim 2, in which the process is digestion with soda.
- Method according to Claim 2, in which the process is digestion with sulphate.
- Method according to Claim 2, in which the process is digestion with sulphite.
- Method according to Claim 2, in which the process is digestion with bisulphite.
- Method according to Claim 2, in which the process is chloro-soda digestion.
- Cellulose pulp for use in papermaking, characterized in that it is produced from *Luffa cylindrica*.

FIG. 1

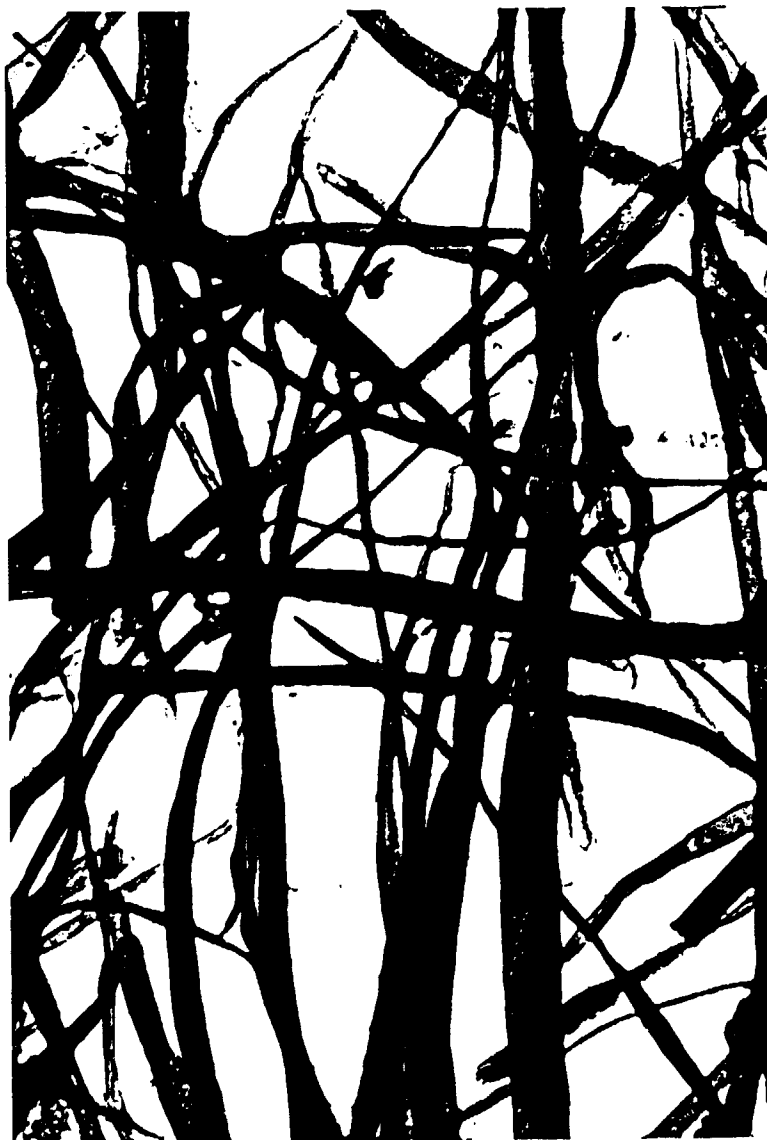


FIG. 2



FIG. 3



FIG. 4





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EUROPEAN SEARCH REPORT

Application Number
EP 97 20 0443

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
A	US 4 243 446 A (MATHEY JEAN M) 6 January 1981 * the whole document *	1	D21C5/00
A	HAMILTON, F. BENGT, L.; KOCUREK, M.J.: "Pulp and paper manufacture. Third edition. Vol. 3: Secondary fibers and non-wood pulping." 1983, JOINT TEXTBOOK COMMITTEE OF THE PAPER INDUSTRY TAPPI CPPA, ATLANTA US XP002031212 * page 4 - page 13 *	1-9	
			TECHNICAL FIELDS SEARCHED (Int.Cl.6)
			D21C
The present search report has been drawn up for all claims			
Place of search		Date of completion of the search	Examiner
THE HAGUE		20 May 1997	Bernardo Noriega,F
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			

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