



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(21) International Application Number: PCT/FI84/00040 (22) International Filing Date: 21 May 1984 (21.05.84) (31) Priority Application Number: 831767 (32) Priority Date: 19 May 1983 (19.05.83) (33) Priority Country: FI (71) Applicant (for all designated States except US): LÄNNEN TEHTAAT OY [FI/FI]; Säskylä, SF-27820 Iso-Vimma (FI). (72) Inventors; and (75) Inventors/Applicants (for US only) : EKLUND, Dan [FI/FI]; Gröndalsvägen 9, SF-02700 Grankulla (FI). ERKKILÄ, Jukka [FI/FI]; INGMAN, Matti [FI/FI]; SF-27820 Iso-Vimma (FI). LASSUS, Anders [FI/FI]; Brahegatan 10 B 27, SF-20110 Åbo 11 (FI). PELTONEN, Kauko [FI/FI]; SF-27820 Iso-Vimma (FI). SARINEN, Kari [FI/FI]; SF-27800 Säskylä (FI).		(74) Agent: RUSKA & CO OY; Kauppiaankatu 7 A, SF-00160 Helsinki 16 (FI). (81) Designated States: AT, AU, BR, DE, FI, GB, JP, NL, NO, SE, SU, US. Published <i>With international search report.</i> <i>With amended claims.</i>
(54) Title: PAPER THAT CONTAINS CHEMICALLY SUBSTITUTED CELLULOSE		
(57) Abstract <p>A paper material has been invented that contains cellulose substituted by means of N-methylol compounds. As N-methylol compounds may be used, e.g., reaction products of urea, glyoxal, and formaldehyde, methylolated melamines, and N-methylol-acrylamide copolymers. The paper is manufactured best by impregnating paper with an aqueous solution or dispersion of a N-methylol compound and by drying it at 130 to 200°C in order to produce condensation. Papers in accordance with the invention have good properties of strength against decomposition and wet strength, which properties can be adjusted readily by varying the degree of substitution or cross-linking of the cellulose. The papers are particularly well suitable, e.g., for the manufacture of plant-growing pots and corrugated fibreboard.</p>		

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Paper that contains chemically substituted cellulose

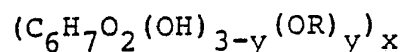
The invention is concerned with paper that contains chemically substituted cellulose. The paper in accordance with the invention has particularly good rot-proof and wet-strength properties. It can be used, e.g., as a raw-material for plant pot systems.

Rot-proof papers are used for a great number of purposes. In agriculture and forestry, they are used, e.g., as production material for plant pot systems.

The present-day production methods are based thereon that varying quantities of PVA fibres are mixed into the paper, by means of which fibres the desired strength against decomposition is obtained for the paper. A drawback of this method is the relatively high cost of the said fibres. It would be preferable to use such a rot-proof paper as is based exclusively on natural fibres.

By adding anti-rot agents to the paper, it is possible to provide a certain, but not sufficient, strength against decomposition. Possible toxic effects of anti-rot agents also restrict their more extensive use.

In the literature, there are several implications to the effect that by modifying the chemical structure of cellulose it is possible to provide prolonged strength against decomposition. For example, "Microbial decomposition of cellulose", Siu, R.G.H., Reinhold Publishing Corporation, New York (1951), gives a theoretical basis for this method. By substituting the hydrogen groups in the hydroxyl groups of cellulose with other groups, compounds of the formula



are obtained, in which formula

R = desired modification,

x = DP; length of the molecule chain (number of glucose units),



y = DS; degree of substitution.

In order to obtain a sufficient strength against decomposition, it is necessary that the O-R linkage is as stable as possible, whereat the hydrolysis, if any, takes place very slowly.

In view of the strength properties of the paper, it is preferable that the reaction conditions are as mild as possible when these derivatives are being prepared.

Now it has been discovered that a paper that contains cellulose fibre chemically substituted with N-methylol compounds has an excellent strength against decomposition and wet strength. In this connection, the expression paper also means paperboard, board, and other, similar products.

Preferable N-methylol compounds that can be concerned are reaction products or urea, glyoxal, and formaldehyde, such as dimethylol-dihydroxy-ethyleneurea (DMDHEU), monomethylol-dihydroxy-ethyleneurea (MMDHEU), dihydroxy-ethyleneurea (DHEU), and acetylene-diureas. Also methylolated melamines and N-methylol-acrylamide-copolymers can be used.

It has been noticed that all of the above compounds form stable linkages with cellulose. When cyclic N-methylol compounds are used, it is advantageous that a carbon atom placed as a neighbouring atom of a nitrogen atom has a nitrogen or oxygen atom as its neighbouring atom. This is the case, e.g., in the case of dimethylol-dihydroxy-ethyleneurea and of methylolated melamine.

The paper in accordance with the invention is preferably prepared by surface-treating conventional paper. When high temperatures or, in particular, a long reaction time is used, it is recommended to use surface treatment taking place in a unit separate from the paper machine. In principle, it is also possible to add the agents as early as in the size



press of the paper machine and to perform mere condensing in a separate unit.

In order to produce the reaction, an elevated temperature and evaporation of the water generated in the reaction are required. N-methylol compounds additionally require a catalyst in order to react with cellulose. Latent or potential acids can function as a catalyst. A typical catalyst is $MgCl_2$; an appropriate catalyst concentration is, e.g., 20 per cent by weight as calculated from the reactive compound. The temperature may be, e.g., between $130^{\circ}C$ and $200^{\circ}C$, preferably between $140^{\circ}C$ and $180^{\circ}C$. The condensation time required depends on the temperature. If a strongly acid catalyst and a high temperature are used, a very short condensation time can be obtained.

By varying the concentrations of reactive agent in the solution or dispersion, it is, in a simple way, possible to adjust the quantity of reactive agent absorbed to the paper and thereby to affect the level of the strength against decomposition of the paper. An appropriate dosage quantity is, depending on the desired strength against decomposition, 1 to 10 % of N-methylol compound, calculated from the dry solids content of the paper.

By using difunctional or polyfunctional reagents, it is possible to produce cross-linkages, which, besides the strength against decomposition, also give the paper an excellent wet strength.

The raw-materials of the papers in accordance with the invention are of relatively low cost, the papers can be manufactured under quite mild conditions, and, since only reagents soluble or dispersed in water are used in the production, the production is also easy from the point of view of work hygiene.

The papers in accordance with the invention have good strength against decomposition and good wet strength, and the degree of these properties can also be



adjusted easily by varying the degree of substitution or cross-linkage of the cellulose. The N-methylol compounds also increase the dimensional stability and rigidity of the paper products, which is advantageous, e.g., in the production and use of corrugated fibreboard.

The papers can be used in principle for all applications in which good strength against decomposition or good wet strength is required. As examples should be mentioned packaging materials, such as sacks, corrugated fibreboards, boxes and barrels, paper strings, and in particular plant pots and plant-growing bases.

When paper sacks, strings, barrels, and other products are being produced, it is possible either to treat the paper out of which the product concerned will be manufactured, or to shape the said product ready and to treat the complete product. The treatment is, however, always the same. The paper is impregnated with a solution or dispersion containing a N-methylol compound, whereupon the drying and the condensation that requires heat take place. As an intermediate mode of production may be considered a production in which the paper is impregnated, dried, the product concerned is shaped, and the complete product is treated with heat.

It is essential in the production of all of these products that the degree of sizing of the paper is low, that rosin sizes are avoided, and that the paper is, if possible, made of conifer cellulose. In this way, no high concentrations of N-methylol compounds are required in order to provide sufficient strength against decomposition. On the other hand, brittleness of paper and deterioration of other paper-technical properties are avoided.

Since hardwood cellulose contains plenty of carboxyl groups, during its modification ester bonds are also formed, which are not equally stable as ether bonds are. When portions containing hardwood pulp are prepared into conifer-pulp-based paper, a paper is obtained

which decomposes by forming holes, whereas the skeleton of the paper, yet, still remains solid. This can be taken advantage of in plant pot systems.

Such a paper can be manufactured, e.g., by
5 into bleached pine sulfate pulp, under acid conditions, mixing rosin-sized reject that contains 50 to 100 per cent by weight of birch fibre as gently pulped. Out of the pulp prepared in this way, a paper is obtained that contains flocs of hardwood fibres in a matrix of conifer
10 fibres, provided that it is taken care that the flocs cannot be disintegrated in pumps or agitators.

A paper which decomposes by forming holes can also be prepared by applying substance containing carboxyl groups on certain spots of the paper. On these
15 spots N-methylol compound will react only with this "protective substance" and cellulose will remain unsubstituted. A suitable substance is e.g. carboxymethyl cellulose (CMC). It is suitable to use an aqueous solution of low viscosity in order to get the absorption
20 of the substance sufficient. A suitable CMC concentration is e.g. 7 %.

The substance containing carboxyl groups can be added into the paper e.g. with a screen cylinder. After the aqueous solution has been added the water is
25 evaporated by drying. Thus the substance adheres to the fibres and spreading of the substance outside of the spots is avoided.

In the screen cylinder method the size and amount of the spots are more easily regulated than in
30 the floc method.

In the manufacture of rot-proof corrugated fibreboard it is preferable that both the corrugating



medium (fluting) and the facing are treated with a N-methylol compound before corrugating and joining of the layers together. In this way a sufficient impregnation is also obtained for the middle layer of the corrugated fibreboard. The glue for corrugated fibreboard must be waterproof. Such a glue is, for example, starch paste to which synthetic resin, such as urea-formaldehyde or resorcinol-formaldehyde, has been added. The condensation of the N-methylol compound takes suitably place after corrugation.

As raw-materials of corrugated fibreboard, it is possible to use the facing and fluting board qualities available at present. It is, however, important that the sizing degree of the facing is as low as possible, which facilitates the impregnation. There is also a risk that side reactions occur between carboxyl-containing resin sizes and the N-methylol compound. The present-day corrugating medium is often made of semi-chemical hardwood pulp. Such a quality, however, contains an abundance of carboxyl groups, because of which it is preferable that, e.g., corrugating medium made of conifer (or perhaps wastepaper) fibres is used.

The strength of the papers in accordance with the invention was tested by fermenting them for three days in a solution containing

	2.5 l	buffer solution
	25 g	"Meicelase" powder (cellulase enzyme)
	0.7 g	"Triton" (antifoam agent)
	31 g	citric acid
30	40 g	$\text{Na}_2\text{HPO}_4 + 2\text{H}_2\text{O}$

With papers impregnated with DMDHEU, prolonged decomposition tests were also performed (Example 2). The samples were placed into boxes which were filled with soil, pH 4.8, conductivity 4.7, and con-



centrations of nutrient agents (mg/l): Ca 830, K 117, F 20, Mg 283, and N 10. The boxes were kept in a "Potma" incubator at 30°C and in a relative humidity of 75 %.

5 The tensile strengths were determined in accordance with the SCAN standards by means of an "Alwetron" apparatus.

Example 1 MMDHEU-impregnated paper

10 The paper was impregnated with solutions of different concentrations containing monomethylol-dihydroxyethyleneurea (urea : glyoxal : formaldehyde = 1:1:1), wherein as catalyst was used $MgCl_2 \times 6 H_2O$ (20 % of the weight of the resin), and the paper was
15 dried at about 150°C for 10 minutes. Out of the paper, the increase in weight on impregnation as well as the tensile strength as wet and as dry before and after the fermentation were measured.



Paper	Increase in weight (%) on im- pregnation	(N) Tensile strength dry		(N) Tensile strength wet	
		before ferm.	after ferm.	before ferm.	after ferm.
Bleached pine sulfate SR25	28.65	111.4	84.0	52.7	60.3
"	11.80	106.5	80.4	77.3	44.8
"	4.92	97.6	92.8	39.0	30.1
Unbleached birch sulfate SR25	34.48	79.7	74.4	58.2	43.5
"	14.85	86.9	65.8	63.2	21.9
"	6.15	77.9	53.3	55.2	9.0

Example 2 Paper impregnated with DMDHEU

Papers were impregnated with solutions containing 2 to 8 % of dimethylol-di-hydroxyethyleneurea (MgCl₂ as catalyst) for 10 s at 25°C and dried for 10 min at 160°C.

Paper	Increase in weight (%) on impregnation	Tensile strength (N) dry		Tensile strength (N) wet	
		before ferm.	after ferm.	before ferm.	after ferm.
Bleached pine sulfate SR 25	4.98	95	73	70	40
"	2.03	94	70	62	38
"	0.65	90	13	36	1.0
Unbleached pine sulfate SR 25	2.97	87	85	75	65
"	0.72	105	63	62	12
Bleached birch sulfate SR 25	5.29	86	56	49	25
"	2.31	69	30	43	7
Unbleached birch sulfate SR 25	6.19	78	62	63	18
"	2.44	96	16	43	0.5
Unbleached spruce sulfite SR 30	3.58	93	66	64	56
"	0.66	89	76	50	36
"	0.39	94	88	61	42



Example 2 continued

Paper	Increase in weight (%) on impregnation	Dry tensile strength (N) after decomposition		
		14 days	21 days	35 days
Bleached pine sulfate SR25	10.60	59	48	51
Unbleached pine sulfate SR25	6.49	64	25	13
Bleached birch sulfate SR25	10.57	13	4	0
Unbleached birch sulfate SR25	5.74	6	3	0
Unbleached spruce sulfite SR30	7.02	78	27	18
Bleached pine sulfite SR50	9.87	66	50	32



Example 3 Paper impregnated with urea-glyoxal resin (DHEU)

Impregnated with urea-glyoxal resin (1:1), dried for 10 min. at about 150°C.

Paper	Increase in weight (%) on impregnation	Tensile strength (N) dry after ferm.		Tensile strength (N) wet after ferm.	
		before ferm.	after ferm.	before ferm.	after ferm.
Bleached pine sulfate SR 25	19.17	87	59	56	3.6
"	8.40	96	41	62	2.5
"	3.47	103	17	59	0.7
Unbleached birch sulfate SR 25	25.32	103	12	37	-

Example 4 Paper impregnated with N-methylol-acrylamide

Impregnated with a solution containing water and latex at the ratio 1:1, the said latex containing 5 % methylol-acrylamide and 95 % vinylacetate, dried at 130°C for about 9 min.

Paper	Increase in weight (%) on impregnation	Tensile strength (N) dry after ferm.		Tensile strength (N) wet after ferm.	
		before ferm.	after ferm.	before ferm.	after ferm.
Bleached pine sulfate SR 25	41.7	67	66	27	28
Unbleached birch sulfate SR 25	50.5	74	58	28	19

Example 5 Paper impregnated with methylolated melamine

The paper was impregnated for 20 s with a solution that contained 80 g/l of methylolated melamine ("Kaurit M-70", Basf) and 8 g/l of $MgCl_2 \cdot 6 H_2O$, and it was condensed for about 10 min. at 160 to 170°C.

Paper	Increase in weight (%) on impregnation	Tensile strength (N)		Tensile strength (N)	
		before ferm.	after ferm.	before ferm.	after ferm.
Bleached pine sulfate SR 25	6.95	76	56	58	13
Unbleached pine sulfate SR 25	6.75	94	58	76	42
Unbleached birch sulfate SR 25	6.87	72	17	49	4.0
Unbleached spruce sulfite SR 30	8.26	95	48	71	14

Example 6 Bleached pine sulfate paper impregnated with mono-, di-, tri- or tetramethylol-acetylene-diurea

The resin was prepared as follows: a mixture of urea, glyoxal, and formaldehyde (2:1:1-4) was diluted to a solution of 0.5 M with water, the pH was adjusted to 8 to 9, and the mixture was allowed to stand for about 5 h.

Paper was impregnated with the solution and dried at about 150°C for 10 min. The catalyst was MgCl₂ · 6H₂O as a quantity of 20 % from the weight of the resin.

15



Example 6 continued

Impregnation agent	Increase in weight (%) on impregnation	Tensile strength (N) dry after ferm.		Tensile strength (N) wet after ferm.	
		before ferm.	after ferm.	before ferm.	after ferm.
Monomethylol-acetylene-diurea	11.78	96	52	51	1.8
Dimethylol-acetylene-diurea	11.40	102	53	58	2.1
Trimethylol-acetylene-diurea	12.45	97	59	59	13
Tetramethylol-acetylene-diurea	13.08	113	67	64	23
Untreated	-	85	-	1.6	



WHAT IS CLAIMED IS:

1. Paper, characterized in that it contains cellulose fibre substituted by means of a N-methylol compound.
2. Paper as claimed in claim 1, characterized in that the N-methylol compound is a reaction product of urea, glyoxal, and formaldehyde.
3. Paper as claimed in claim 2, characterized in that the N-methylol compound is dimethylol-dihydroxy-ethyleneurea.
4. Paper as claimed in claim 3, characterized in that the content of dimethylol-dihydroxy-ethyleneurea is 1 to 3 % of the weight of the paper.
5. Paper as claimed in claim 2, characterized in that the N-methylol compound is mono-, di-, tri-, or tetramethylol-acetylenediurea.
6. Paper as claimed in claim 1, characterized in that the N-methylol compound is a reaction product of melamine and formaldehyde.
7. Paper as claimed in claim 1, characterized in that the N-methylol compound is a copolymer of N-methylol-acrylamide.
8. Paper as claimed in any of claims 1 to 7, characterized in that the cellulose is conifer cellulose.
9. Paper as claimed in any of claims 1 to 8, characterized in that the paper contains spots with unsubstituted cellulose.
10. Process for the preparation paper, characterized in that unsubstituted cellulose fibre is treated with an aqueous solution or dispersion of a N-methylol compound and dried at 130 to 200°C in any phase of the process.



11. Process for the substitution of cellulose in a paper, characterized in that the paper is treated with an aqueous solution or dispersion of a N-methylol compound and dried at 130 to 200°C.

12. Process for substitution cellulose in a paper, characterized in that substance containing carboxyl groups is added to the paper on certain spots, and the paper is treated with an aqueous solution or dispersion of a N-methylol compound and dried at 130 to 200°C.

13. Process for the preparation of paper products, characterized in that paper containing cellulose substituted with a N-methylol compound is used as the paper.



AMENDED CLAIMS

(received by the International Bureau on 17 October 1984 (17.10.84))

1. (amended) Paper, c h a r a c t e r i z e d in that it contains conifer cellulose fibre substituted by means of
5 a N-methylol compound which is a reaction product of urea, glyoxal, and formaldehyde.
2. (cancelled)
3. (amended) Paper as claimed in claim 1, c h a r a c -
t e r i z e d in that the N-methylol compound is dimethylol-
10 dihydroxy-ethyleneurea.
4. Paper as claimed in claim 3, c h a r a c t e r i z e d in that the content of dimethylol-dihydroxy-ethyleneurea is 1 to 3 % of the weight of the paper.
5. (cancelled)
- 15 6. (cancelled)
7. (cancelled)
8. (cancelled)
9. Paper is claimed in any of claims 1 to 8, c h a r a c -
t e r i z e d in that the paper contains spots with unsubstituted
20 cellulose.
10. (amended) Process for the preparation paper, c h a r a c t e r i z e d in that unsubstituted conifer cellulose fibre is treated with an aqueous solution or dispersion of a N-methylol compound which is a reaction product of urea,
25 glyoxal, and formaldehyde, and dried at 130 to 200°C in any phase of the process.
11. (amended) Process for the substitution of cellulose in a paper, c h a r a c t e r i z e d in that the paper is treated with an aqueous solution or dispersion of a N-methylol
30 compound which is reaction product of urea, glyoxal, and formaldehyde and dried at 130 to 200°C
12. (amended) Process for substitution cellulose in a paper, c h a r a c t e r i z e d in that substance containing carboxyl groups is added to the paper on certain spots, and
35 the paper is treated with an aqueous solution or dispersion of a N-methylol compound and which is a reaction product of



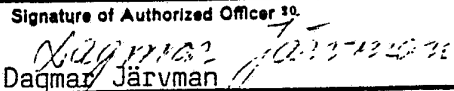
urea, glyoxal, and formaldehyde, and dried at 130 to 200°C.

13. (amended) Process for the preparation of paper products, characterized in that paper containing cellulose substituted with a N-methylol compound which is a reaction product of urea, glyoxal, and formaldehyde is used as the paper.



INTERNATIONAL SEARCH REPORT

International Application No PCT/FI84/00040

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ² According to International Patent Classification (IPC) or to both National Classification and IPC ³ D 21 H 5/14				
II. FIELDS SEARCHED				
Minimum Documentation Searched ⁴				
Classification System	Classification Symbols			
IPC 3	D 21 H 1/40, 5/14			
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁵				
SE, NO, DK, FI classes as above				
III. DOCUMENTS CONSIDERED TO BE RELEVANT ¹⁴				
Category ⁶	Citation of Document, ¹⁵ with indication, where appropriate, of the relevant passages ¹⁷	Relevant to Claim No. ¹⁸		
X	GB, A, 1 156 792 (GEORGE WILLIAM LEWIS) 2 July 1969	1, 6, 10		
X	US, A, 3 773 612 (ROBERT P AVIS) 20 November 1973 & FR, 2213957 DE, 2402488 BE, 809797 GB, 1455381 CA, 1026490	1, 2, 7		
X	US, A, 3 858 273 (HERMAN B GOLDSTEIN JOHN T OUDERKIRK) 7 January 1975	1, 3, 4		
<table style="width: 100%; border: none;"> <tr> <td style="width: 50%; border: none; vertical-align: top;"> <ul style="list-style-type: none"> • Special categories of cited documents: ¹⁶ "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier document but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed </td> <td style="width: 50%; border: none; vertical-align: top;"> <ul style="list-style-type: none"> "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art. "&" document member of the same patent family </td> </tr> </table>			<ul style="list-style-type: none"> • Special categories of cited documents: ¹⁶ "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier document but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed 	<ul style="list-style-type: none"> "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art. "&" document member of the same patent family
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IV. CERTIFICATION				
Date of the Actual Completion of the International Search ¹	Date of Mailing of this International Search Report ⁸			
1984-08-27	1984 -08- 2 9			
International Searching Authority ¹	Signature of Authorized Officer ¹⁰			
Swedish Patent Office	 Dagmar Järvmán			