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#### INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification <sup>3</sup> :		(11) International Publication Number	r: WO 84/ 03859
B32B 27/04, 27/16, 23/14 B32B 29/06; B29J 5/02	A1	(43) International Publication Date:	11 October 1984 (11.10.84)
C08L 97/02			

(21) International Application Number: PCT/AU84/00050 (81) Designated States:

(22) International Filing Date: 26 March 1984 (26.03.84)

(31) Priority Application Numbers: PF 8651 PF 8653 Publ

(32) Priority Dates: 28 March 1983 (28.03.83)

28 March 1983 (28.03.83)

(33) Priority Country:

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(81) Designated States: AT (European patent), BE (European patent), CH (European patent), DE (European patent), FI, FR (European patent), GB (European patent), JP, SE (European patent), SU, US.

Published

With international search report.

(54) Title: PRODUCTION OF LAMINATES

#### (57) Abstract

In the continuous production of laminates, especially laminated building boards, one or more base sheets impregnated with phenoplast/aminoplast resin mixture in the presence of acid donor curing agents are pressed together at an elevated curing temperature with decorative sheets impregnated with aminoplast resin in the presence of acid donor curing agents.

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## PRODUCTION OF LAMINATES

This invention relates to the manufacture of bonded products particularly of the kind traditionally made by the bonding of cellulosic material using heat cured resins under pressure.

In the art phenoplast resins are used as binding resins for cellulosic materials, such as paper, wood chips, wood fibres and the like. Phenoplast resins are relatively inexpensive resins and are readily available. They provide a degree of resilience and water resistance.

However phenoplast resins are slow in heat curing and this substantially restricts the manufacturing processes in which they are used. The curing is too slow to permit continuous production. Further, the phenoplast resins are cured in alkaline conditions. When laminates of materials bound with phenoplast resins are made with materials bound with aminoplast resins (which are cured in acid conditions) bonding weaknesses may result due to a conflict in curing conditions at the interface.

An object of the present invention is to produce bonded products using phenoplast resins which retain the advantageous properties of the phenoplast resin while minimising the effect of the disadvantageous properties of the type outlined above.

In the manufacture of laminates it is common practice to impregnate papers with low molecular weight polymers such as a low molecular weight phenoplast polymers to provide a base sheet and to impregnate a decorative sheet of paper with a low molecular weight aminoplast resin such as a melamine formaldehyde resin. The impregnated papers are then pressed together and heated to cure the resins and to bond the layers

together. In general a period of say from one to two
hours or so is required in this pressing operation depending
on the number of sheets pressed in one press opening.
It has meant that it is necessary generally to produce



the product in a batch process.

It is one objective of the present invention to provide for the continuous manufacture of laminating materials.

In one common laminating procedure for example there is provided a layer of Kraft quality paper impregnated with a phenol formaldehyde resin or the like. This provides strength to the laminate. This layer is laminated with a decorative grade paper impregnated with an aminoplast resin such as a melamine formaldehyde, urea formaldehyde or other aminoplast resin. Pressing to laminate these materials takes in the order of from one to two hours or so depending on the number of sheets in the press opening which is the time taken for the resin to cure to a sufficient extent. Generally, curing agents are used. Aminoplast resins cure best in acid conditions and acid donor curing agents are used for this purpose. Phenoplast resins and like condensation polymers cure best in alkaline conditions and so acid receptor or alkaline curing agents such as sodium hydroxide or calcium hydroxide and the like are used for this purpose.

It has been found that to cure laminates of papers impregnated with polymers that cure in acid conditions and papers impregnated with polymers that cure in alkaline conditions creates a problem at the interface of the layers. There is a tendency for the layers to peel apart or to suffer other unacceptable defects such as blistering or lack of strength.

In accordance with the present invention the speed of the curing of phenoplast resins can be increased dramatically and acid donor curing agents used when an aminoplast resin

is mixed with the phenoplast resin prior to impregnation. Accordingly, the present invention provides in one aspect a method for the continuous production of laminates including the steps of impregnating one or more continuous base sheet materials with a mixture of a one or more phenoplast resins, one or more aminoplast resins and one or more acid donor curing agents, impregnating one or more continuous decorative sheet materials with one or more aminoplast resin containing one or more acid donor curing agent, pressing said decorative and said base sheet materials together at an elevated curing temperature whereby rapidly to cure said resins and form an integral laminate structure.



By an aminoplast resin as used herein is meant a resin made by the reaction of a compound containing an amide or amino group with an aldehyde. Formaldehyde is the common aldehyde used. Common compounds including an amide or amino groups are the amines such as urea, thiourea, ethylene urea, melamine dicyandiamide, benzoguanamine, aceto-guanamine and the like. Aminoplast resins are used because they are normally light in color and can form relatively clear films thereby displaying the quality of the material in which they are impregnated and to produce materials having a light color appearance. However aminoplast resins are relatively expensive and if used to make the entire product are too brittle.

By the term phenoplast resin as used herein is meant a resin that is the condensation product of a compound containing an hydroxyl group with an aldehyde. Formaldehyde is the common aldehyde used although furfural and other aldehydes may be used as desired. The phenols are the most common hydroxyl containing compounds and include phenol, the cresols, the xylenols, recordinol, napthols and naturally occurring phenols such as tannins and related substances.

Any suitable phenoplast and aminoplast resins can be used in accordance with the present invention. However in most cases phenol formaldehyde will provide a satisfactory phenoplast resin and melamine formaldehyde or urea formaldehyde will provide a satisfactory aminoplast resin.

The phenoplast resin may be prepared in any suitable manner preferably by reacting a phenol with a molar excess of formaldehyde in the presence of catalytic

quantities of alkali.

The aminoplast resin such as melamine formaldehyde may be prepared by reacting melamine and formaldehyde in the presence of an alkali such as sodium hydroxide. A molar excess of formaldehyde is preferably used.

The phenoplast resins are modified by blending in amounts of the aminoplast resin. It is understood that under curing conditions the methylol groups of the phenoplast resin react with the amino or amide groups in the aminoplast resin under acid conditions and that this substantially increases the rate of cure when compared with the rate of cure of a phenoplast resin.

The relative amounts of phenoplast and aminoplast resins may be varied to suit the requirements of the product to be made and the speed at which the process is to be operated. Preferably from about 5 to 70 parts of melamine resin is used for each 100 parts by weight of phenoplast resin more preferably from 20 to 55 parts by weight.

The acid donor curing agents that may be used in accordance with the present invention include acid salts and free acids for example. Preferably the acid donor curing agents are compounds that release acid under curing conditions. For example they may be compounds that react with formaldehyde under curing temperatures to release free acid.

The acid may be weak acid to produce for example a pH of less than 5 and preferably about 3 under the curing conditions. Suitable acid salts include those based on ammonia, amines, substituted amines or other chemical entities which react with formaldehyde in the resin to liberate the acid



component. The salt may be a chloride, sulphate, acetate, formate or sulphonate salt for example. A particularly useful example of an acid curing agent is ammonium chloride. Suitable free acids include benzoic acid, acetic acid and the like.

It will be appreciated that when an acid donor curing agent is used that does not become activated until a curing temperature is reached it is possible to minimize premature cross linking of the aminoplast and phenoplast resins until the curing pressure and temperature is applied. This means that the resin is able to flow satisfactorily and the treated papers have a better storage life.

Thus the present invention provides a superior method for the manufacture of building board products. The curing rate of phenoplast resins may be improved to between 30 to 80 seconds at a temperature of between 110°C and 200°C preferably above 135°C and about 170°C to 190°C and pressure in the order of 10 to 30 kg/sq cm preferably 15 to 20 kg/sq cm. Higher curing pressures may be employed in which case the curing times may be further reduced or alternatively lower temperatures used as may be desired. Acid donor catalysts are used and the resin mixture is compatible with and may be laminated to products containing acid donor catalysed resins. The laminated papers of one aspect of the present invention may include from about 25 to 50% by weight of resin. At lower pressures the resin content may be in the order of 40 to 50% by weight. At higher laminating pressure this may be reduced to about 25%. The impregnated papers may be laminated with other support material as is known in the art to produce commercial decorative laminates.

view of the short curing times, continuous manufacture of laminated products is possible using equipment known in the art. The thickness of the laminates may be in the order of say 0.3 to 2.0 mm for example.

The present invention also relates to a method of manufacturing fibreboard, particleboard and the like products. Urea formaldehyde resins and similar resins have been used for the binding of cellulosic material in the manufacture of fibreboard and particleboard. Deficiencies of the resins used include their low water resistance, the resin tends to hydrolize and causes breakdown of the board in high humidity conditions. Also there is a liberation of formaldehyde from the board over a period of time and this causes an odour that in some uses renders the board unsatisfactory.

It has now been found that by use of a resin mix in accordance with the present invention the aforementioned disadvantages can be minimized. According to this aspect the present invention provides a method for the production of building board material including the steps of mixing particulate cellulosic material with a binder resin mixture comprising one or more phenoplast resins,



one or more aminoplast resins and one or more acid donor curing agents and pressing said particulate material at an elevated curing temperature whereby rapidly to cure said resins and bind said particulate cellulosic material. Any of the phenoplast resins above referred to may be used in accordance with this aspect of the present invention. Similarly, any of the aminoplast resins above described may be used in accordance with this aspect of the present invention. In this aspect of the invention it is preferred that an acid donor curing agent be selected of the type which does not have substantial activity at ambient temperatures and pressures but which is activated at the elevated curing temperatures to which the mixture is subjected. The weak acids and acid salts above described are useful in this purpose.

Preferably particles of wood chips or fibres are first coated with resin in an amount of about 7 to 18% by weight preferably 9 to 12% by weight of the total in a batch mix.

They may be fed to a press at a temperature of about 130°C to 200°C. Initial pressure may be between 10 and 60 kg/sq cm. As the mass of particles consolidate pressure between the pressing plattens may be reduced. Steps may be provided to hold the plattens apart at the desired board thickness which may be selected in accordance with the desired end use. Residence time in the press may be less than 10 minutes. The press can be a batch press or a continuous press regulated to allow for an adequate period for curing under pressure.

In accordance with another aspect of the present invention there is provided a new and improved method for

manufacturing decorated boards of bound cellulosic material. In accordance with this aspect of the present invention a building board manufactured in accordance with the present invention may be employed. Alternatively, a building board manufactured in accordance with techniques known in the art may be used. That is, a building board may be manufactured using a urea formaldehyde or like resin. In accordance with this aspect of the present invention there is provided a method for the continuous production of laminates including the steps of impregnating one or more continuous base sheet materials with a mixture of one or more phenoplast resins, one or more aminoplast resins and one or more acid donor curing agents and pressing said base sheet material and a building board material together at an elevated curing temperature. A decorative layer may also be provided if desired. Thus one or more continuous decorative sheet materials may be impregnated with one or more aminoplast resins containing an acid donor curing agent and applied over the base sheet material prior to curing.

In accordance with this aspect of the present invention any suitable phenoplast and aminoplast resins of the type above described may be used. Similarly, any suitable acid donor curing agent may be employed as is known in the art but preferably an agent that is not substantially active under ambient conditions but which is activated under curing conditions to produce free acid.

Preferably a decorative layer is laminated to each side of the building board. The Kraft paper and decorative paper may be impregnated as previously

described with reference to the preparation of paper laminates and pressed to the board at a pressure of say between 10 and 30 kg/sq cm, preferably between 15 and 25 kg/sq cm at a temperature preferably between 150°C and 200°C for a period of generally less than 120 seconds. Those skilled in the art will appreciate that the parameters may be varied to suit various requirements in processing and the products made.

The present invention will be further described with reference to the examples that follow. It should be appreciated that the detail given in these examples is not to be taken as restricting the present invention.

#### EXAMPLES

## Example 1

A phenolic resin was prefered by mixing the following:

37% formaldehyde

1900 litres

88% phenol

1380 litres

sodium hydroxide

13 kgm

The mixture was heated at a temperature of 85°C for 2 hours, cooled and adjusted to a solids content of 50% by weight with water.

A melamine formaldehyde resin was prepared by mixing the following:



37% formaldehyde 1365 litres sodium hydroxide to a pH of 9 melamine 1300 kgm

The mixture was heated to 95°C. After about 1½ hours the reaction was discontinued, 180 litres of ethyl alcohol added and the solids content adjusted to 50% by weight with water.

100 parts by weight of the phenol formaldehyde resin was mixed with 50 parts by weight of the melamine formaldehyde resin. Ammonium chloride was added in an amount of about 3% by weight, as a 25% solution in water. This resin mixture was used to impregnate a continuous 215 gram Kraft paper in a Vits impregnator to a resin content of 42%.

Melamine formaldehyde and ammonium chloride acid donor curing agent was used to impregnate 80 to 130 gram continuous decorative paper to resin content of 65%.

The papers were brought together in a continuous laminator at a pressure of 15kg/cm<sup>2</sup> and temperature of 185°C. The output rate was adjusted to between 6 to 8 metres per minute to produce a laminate of 0.6 to 0.8 mm.



The product was satisfactory in strength, characteristics and appearance. Stroke cure of the resin was less than 20 seconds.

This example was repeated to determine stroke cure at different curing temperatures. Figure 1 shows the results.

The example was repeated using different amounts of melamine resin and different amounts of hardener. Figure 2 shows the stroke cure rate in seconds for



varying amounts of hardener, and at melamine resin contents of 50 parts by weight, 25 parts by weight, 10 parts by weight and for phenol formaldehyde alone. Curing temperature was 135°C.

Thus, in accordance with the present invention the curing rate of the phenolic resin, normally in the order of about 15 to 30 minutes is reduced to below 80 seconds. Thus, it is possible to laminate in continuous presses. The base layers and decorative layers are compatible. The acid donor curing agent used is not activated until the impregnated paper is heated, avoiding premature curing and ensuring proper penetration of the papers and bonding of the papers.

## Example 2

A 215 gram Kraft paper was impregnated with a resin mixture as in Example I. The impregnated paper was placed on a core of standard particle board.

A decorative paper was impregnated with melamine formaldehyde resin and curing agent as in example I and this was layed over the Kraft paper layer.



The laminate was pressed together at 20 kgm/sq cm for 80 seconds at 170°C. The appearance was very smooth. The experiment was repeated without the use of the Kraft paper layer. It was noted that there were irregularities showing in the surface.

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Each of the samples was subjected to treatment with steam over an extended period. It was observed that in the board not having the Kraft paper layer the surface showed signes of lumpiness due to swelling of the chips



after 15 minutes. The board having the Kraft paper layer did not show these signs for a further 15 minutes of continuous steam treatment.

## Example 3

A particle board was made using processed wood chips. As binder was used 100 parts of the phenol formaldehyde resin of example 1 and 50 parts by weight of melamine formaldehyde resin of example 1. 0.4% by weight of ammonium chloride was added as hardener. The boards were made 18 mm thick, and 9% by weight of resin mix was included in the core and 12% at the surface.

The board was pressed at 150°C for various periods of time and tested for internal bond strength. The boards were boiled in water for ½ hour and the effects observed. The results were compared with similar boards made using standard urea formaldehyde resin.

Board	Curing Time	Bond Strength megapascals	Modulus of Rupture Retention after 1/2 Hour Boiling Treatment
Standard Urea	6 minutes	0.45	<2% strength
Standard Urea	9 minutes	0.48	retention <2% strength
Phenol/melamine	6 minutes	0.43	retention 39% strength
Phenol/melamine	9 minutes	0.52	retention 45% strength
Phenol/melamine	12 minutes	0.54	retention 46% strength retention

Thus by use of the present invention a board with greatly superior water resistance was obtained.



## Example 4

Similar tests were conducted using wood fiber chips impregnated with 100 parts of the phenol formaldehyde resin of example 1 and 50 parts by weight of the melamine formaldehyde resin of example 1. 11% by weight of the resin mixture was used. The boards were pressed at 170°C for varying periods and boiled in water for 2 hours.

Resin	Pressing Time	Boiling Water	
Standard Urea (11%)	6 minutes	Disintegrates	in ½ hour
Standard Urea (11%)	6.5 minutes	Disintegrates	in ½ hour
Standard Urea (11%)	7 minutes	Disintegrates	in ½ hour
Phenol/Melamine	6 minutes	115% swelling	
Phenol/Melamine	6.5 minutes	93% swelling	
Phenol/Melamine	7 minutes	75% swelling	
Phenol/Melamine	8 minutes	88% swelling	
Phenol/Melamine	9 minutes	90% swelling	•

Thus optimum treatment time for the phenol melamine formaldehyde resins was 7 minutes. The phenol/melamine blends were markedly superior to the standard urea resins in imparting water resistance.

### Example 5

The phenol/melamine formaldehyde resin mix of example 4 was used to bind wood chips. 1½% of saturated ammonium chloride was used as curing agent. The product was 16 mm thick. The amount of resin in the core was 9% and in the surface layers 12%. The mixture was pressed at 155°C for various periods of time and compared

with similar product bound with urea formaldehyde.

PROPERTY	6 MINS	YCLE TIM 9 MINS	E_ 12 MINS	UREA FORMALDEHYDE
Density (kg/m³)	680	690	690	700
Strength (MPa)	18.9	21.6	22.9	20
Strength % Retention after 3 minutes steam	20			
treatment	38	45	46	Disintegrates
% Swell (2 hours)	2.2	2.5	1.3	Disintegrates
% Absorption (2 hours)	13.8	10.4	10.1	18
% Moisture	9.2	8.7	8.1	7.4

## Example 6

Example 1 was repeated using urea formaldehyde resin in place of melamine formaldehyde resin. Stroke cure was measured for mixtures containing 100 parts of phenol formaldehyde resin and 50 parts of urea formaldehyde resin, 25 parts, 10 parts and 0 parts urea formaldehyde resin. Curing temperature was 135°C and various quantities of 25% ammonium chloride curing agent were used. The results are set out in figure 3. These show that urea formaldehyde is a satisfactory resin for use as an aminoplast to rapidly cure phenol formaldehyde resins in acid conditions.



It will be appreciated that many modifications, variations and additons may be made to the above described methods, constructions and arrangements without departing from the ambit of the present invention.



The claims defining the invention are as follows:

1. A method for the continuous production of laminates including the steps of:

impregnating one or more base sheet materials with a mixture of a one or more phenoplast resins, one or more aminoplast resins and one or more acid donor curing agents,

impregnating one or more continuous decorative sheet materials with one or more aminoplast resin containing one or more acid donor curing agents,

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pressing said decorative and said base sheet materials together at an elevated curing temperature whereby rapidly to cure said resins and form an integral laminate structure.

2. A method for the production of laminated building board including the steps of impregnating one or more continuous base sheet materials with a mixture of one or more phenoplast resins, one or more aminoplast resins and one or more acid donor curing agents, and

pressing said base sheet material and a formed building board at an elevated curing temperature whereby rapidly to cure said resins to bind said base sheet material to said building board.

- 3. A method as claimed in claim 2 including the step of impregnating one or more continuous decorative sheet materials with one or more aminoplast resins containing one or more acid donor curing agents, and applying said decorative sheet over said base sheet, prior to said pressing.
- 4. A method for the production of building board including the steps of:

mixing particulate cellulosic material with a



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binder resin mixture comprising one or more phenoplast resins, one or more aminoplast resins and one or more acid donor curing agents and pressing said particulate material at an elevated curing temperature whereby rapidly to cure said resins and bind said particulate cellulosic material.

- 5. A method as claimed in any preceding claim, wherein said phenoplast resin is selected from the group consisting of the reaction product of formaldehyde and one or more of: phenol, recordinol, resol, xylenol and napthol.
- 6. A method as claimed in any preceding claim, wherein said aminoplast resins which may be the same or different is selected from the group consisting of reaction product of formaldehyde and one or more of melamine, urea, thiourea, ethylene urea, dicyandiamide, benzoguanamine and aceto-quanamine.
- 7. A method as claimed in any preceding claim wherein said acid donor curing agent is a curing agent activatable to release free acid at said elevated curing temperature.
- 8. A method as claimed in claim 7 wherein said curing agent is an acid salt that reacts with formaldehyde at said elevated curing temperature to release free acid.
  - 9. A method as claimed in claim 8, wherein said curing agent is an acid salt of ammonia, an amine or a substituted amine.
  - 10. A method as claimed in any one of claims 1 to 6 wherein said curing agent is a weak acid.
  - 11. A method as claimed in any one of claims 1 to 6, wherein said curing agent is selected from the group consisting of: ammonium chloride, sulphate, acetate,



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formate or sulphonate, or similar salts of triethanolamine, diethanolamine, monoethanolamine, morpholine, guanidine or melamine; benzoic acid or acetic acid.

- 12. A method as claimed in any preceding claim wherein the curing is effected at a pH of less than 5.
- 13. A method as claimed in claim 12 wherein the curing is effected at a pH of about 3.
- 14. A method as claimed in claim 1, wherein said decorative sheet material and/or said base sheet material is a continuous paper sheet.
  - 15. A method as claimed in claim 14, wherein said continuous paper sheet is a Kraft paper sheet.
  - 16. A method as claimed in claim 14 or claim 15 wherein said curing temperature is between 135 and 200°C.
  - 17. A method as claimed in claim 16, wherein said curing temperature is between 170 and 190°C.
  - 18. A method as claimed in any one of claims 14 to 17 wherein the pressure at curing is between 10 and 30 kg/sq cm.
- 20 19. A method as claimed in claim 18 wherein the pressure at curing is between 15 and 20 kg/sq cm.
  - 20. A method as claimed in any one of claims 14 to 19 wherein from 20 to 55% by weight of resin is impregnated in said material.
  - 21. A method as claimed in any one of claims 14 to 20 wherein the thickness of the laminate is from 0.3 to 2.0 mm.
  - 22. A method as claimed in claim 2 or claim 3 and any claim appended thereto wherein continuous base sheets and optionally decorative sheets are laminated to one or

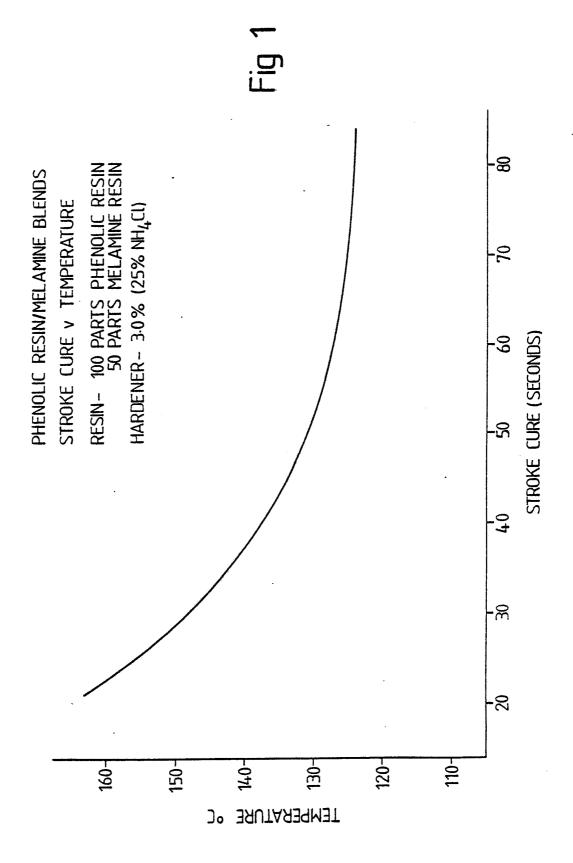


both faces of said building board.

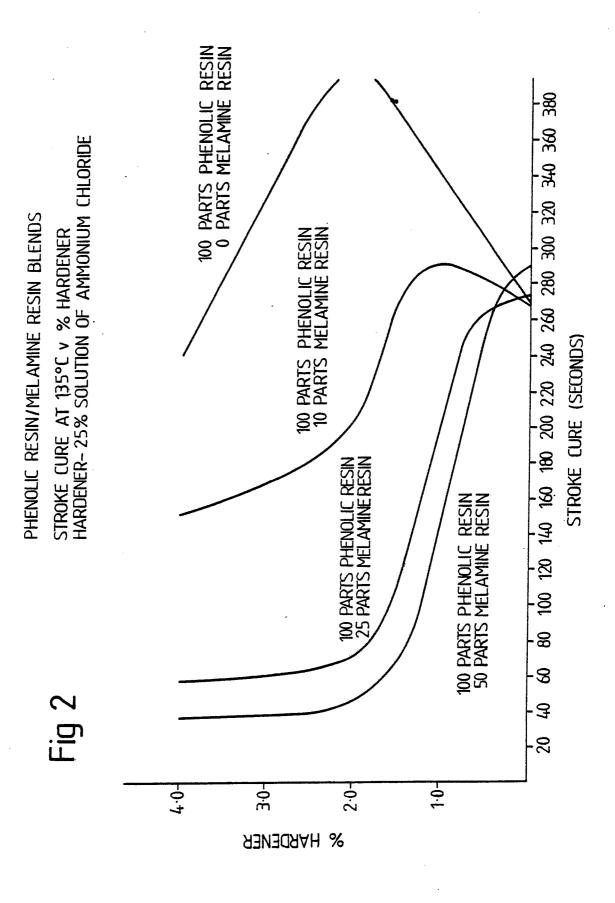
- 23. A method as claimed in claim 22, wherein curing is effected at a temperature of between 150 and 200°C.
- 24. A method as claimed in claim 22 or claim 23 wherein curing is effected at a pressure of between 10 and 30 kgm/sq cm.
- 25. A method as claimed in claim 24 wherein curing is effected at a pressure of between 15 and 25 kg/sq cm.
- 26. A method as claimed in any one of claims 22 to 25 wherein curing is effected for a period of less than 120 seconds.
- 27. A method as claimed in claim 4 or any claim appended thereto wherein said curing temperature is from 135 to 200°C.
- 28. A method as claimed in claim 27, wherein the curing pressure is between 10 and 60 kg/sq cm.
- 29. A method as claimed in claim 28, wherein the curing pressure is reduced progressively as the board is compressed during curing.
- 30. A method as claimed in claim 27, wherein said curing is effected for a period of less than 10 minutes.
  - 31. A method as claimed in claim 27, wherein said curing is effected in a continuous process.
  - 32. A method of making a board material substantially as hereinbefore described with reference to any one of the examples.
  - 33. A board produced by the method claimed in any preceding claim.
  - 34. A laminating paper construction produced by the method claimed in claim 1 and any preceding claim appended thereto.

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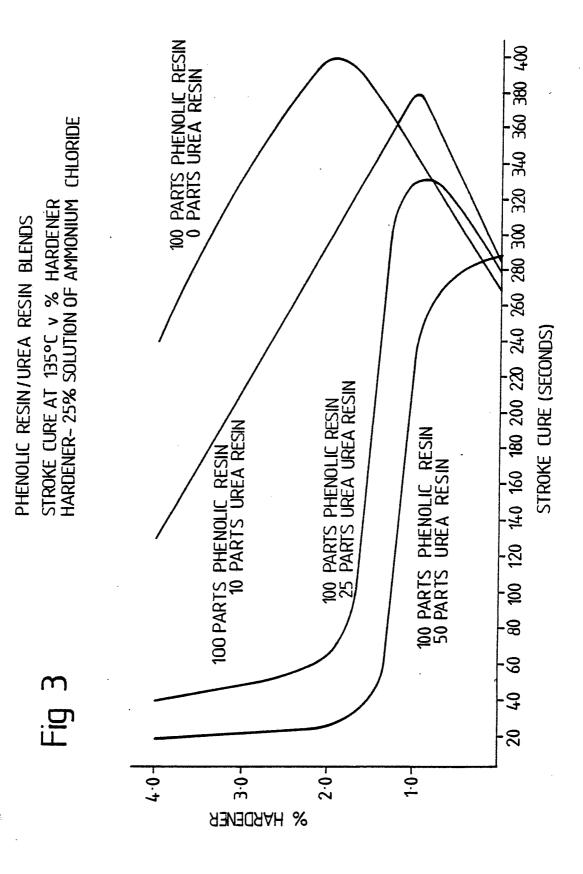








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#### INTERNATIONAL SEARCH REPORT

International Application No PCT/AU84/00050

## I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) 3 According to International Patent Classification (IPC) or to both National Classification and IPC

Int. Cl. 3 B32B 27/04, 27/16, 23/14, 29/06, B29J 5/02, CO8L 97/02

#### II. FIELDS SEARCHED

Minimum Documentation Searched 4

Classification System Classification Symbols

IPC

B32B 27/04, 23/12, 29/06, 27/26, 23/06

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched &

AU : IPC B32B 27/04, CO8L 97/02

#### III. DOCUMENTS CONSIDERED TO BE RELEVANT 14

Category •	Citation of Document, 16 with indication, where appropriate, of the relevant passages 17	Relevant to Claim No. 18
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A	GB, A, 1317427 (FORMICA INTERNATIONAL LIMITED) 16 May 1973 (16.05.73)	
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A	US, A, 3928706 (GIBBONS) 23 December 1975 (23.12.75)	
A	US, A, 4006048 (CANNADY, JR. ET. AL.) 1 February 1977 (01.02.77)	
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#### IV. CERTIFICATION

Date of the Actual Completion of the International Search 1

22 June 1984 (22.06.84)

International Searching Authority 1

AUSTRALIAN PATENT OFFICE

Date of Mailing of this International Search Report 2 28-06-84)

28 JUNE Signature of Authorized Officer 20

A. S. MOORE

# ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL APPLICATION NO. PCT/AU PCT/AU84/00050

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

	ent Document ed in Search Report	·		Paten	t Family Memb	ers	
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