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NOTICE OF ENTITLEMENT

We, **COURTAULDS FIBRES (HOLDINGS) LIMITED** of, 50 George Street, London W1A 2BB, United Kingdom and **BUSS AG** of, CH-4052 Basel, Lautengartenstrasse 7, Switzerland, being the applicants in respect of Application No. **48251/93** state the following:-

The Persons nominated for the grant of the patent have entitlement from the actual inventors as follows:

- **COURTAULDS FIBRES (HOLDINGS) LIMITED** is entitled by virtue of an assignment from Michael Colin Quigley; and
- **BUSS AG** is entitled by virtue of an assignment from Rainer Felix Naef.

The persons nominated for the grant of the patent have entitlement from the applicant of the application listed in the declaration under Article 8 of the PCT as follows:

- An assignment from Courtaulds Fibres Limited to Courtaulds Fibres (Holdings) Limited dated May 23, 1994;
- An assignment from Courtaulds Fibres (Holdings) Limited to Courtaulds Fibres (Holdings) Limited and Buss AG dated June 26, 1996.

The basic application listed on the request form is the first application made in a Convention country in respect of the invention

COURTAULDS FIBRES (HOLDINGS) LIMITED
and **BUSS AG**

By our Patent Attorneys,
WATERMARK PATENT & TRADEMARK ATTORNEYS


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Ian A. Scott
Registered Patent Attorney

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- (71) Applicant(s)
COURTAULDS FIBRES (HOLDINGS) LIMITED; BUSS AG
- (72) Inventor(s)
MICHAEL COLIN QUIGLEY; RAINER FELIX NAEF
- (74) Attorney or Agent
WATERMARK PATENT & TRADEMARK ATTORNEYS, Locked Bag 5, HAWTHORN VIC 3122
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- (57) Claim



1. A method of forming a solution of cellulose in a tertiary amine N-oxide which comprises the steps of:

- (i) forming a premix of tertiary amine N-oxide, water and cellulose,
- (ii) heating the premix under a vacuum in the range 25 millibars to 100 millibars in a vertically mounted thin-film evaporator having a heated inner surface and a rotor comprising a central shaft with paddle blades disposed along the shaft so that, on rotation of the shaft, material in the evaporator will be contacted by a plurality of blades annularly spaced around the shaft,
- (iii) evaporating the water from the premix to a level such that the cellulose forms a solution in the amine oxide, and
- (iv) removing the solution from the thin-film evaporator at a temperature in the range 90°C to 135°C,

characterised by providing said heated surface with an area in the range 1m² to 125m², rotating the rotor such that the tips of the blades have a velocity of up to 8 metres/sec., and providing a circumferential spacing between the tips of

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adjacent blades in the range of 65mm to 175mm, the drop off point for material dropping off one rotor blade being less than 90° before the pick up point for the next rotor blade.

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(21) International Application Number: PCT/GB93/01956 (22) International Filing Date: 15 September 1993 (15.09.93) (30) Priority data: 9219693.0 17 September 1992 (17.09.92) GB <i>COURTAULDS FIBRES (HOLDINGS) LIMITED</i> (71) Applicant: COURTAULDS FIBRES LIMITED [GB/ GB]; 50 George Street, London W1A 2BB (GB). <i>Buss AG</i> CH-4133 Basel, Hohenrainstrasse 10, Pratteln, <i>Switzerland</i> (72) Inventor: Q'IGLEY, Michael, Colin ; 41 Bonneville <i>Switzerland</i> Close, Coventry CV5 9QH (GB). (74) Agent: NEWBY, John, Ross; J.Y. & G.W. Johnson, Fur- nival House, 14-18 High Holborn, London WC1V 6DE (GB). 673540		(81) Designated States: AT, AU, BB, BG, BR, BY, CA, CH, CZ, DE, DK, ES, FI, GB, HU, JP, KP, KR, KZ, LK, LU, MG, MN, MW, NL, NO, NZ, PL, PT, RO, RU, SD, SE, SK, UA, VN, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published With international search report. - Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.  
(54) Title: FORMING SOLUTIONS (57) Abstract A solution of cellulose is formed by moving a premix of a tertiary amine N-oxide, water and cellulose through a thin-film evaporator using a bladed rotor under treatment conditions which specify, an area of heated surface (1m² to 125²), a circumferential spacing between blade tips (65 to 175 mm), a rotational speed of the blade tips (less than 8 m/sec.), a pressure (25 to 100 mb) and a temperature (90 °C to 135 °C).		

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Forming Solutions

Background of the Invention

1. Field of the Invention

This invention relates to a method of forming solutions
5 and has particular reference to forming solutions of
cellulose in tertiary amine N-oxide solvents.

2. Description of the Related Art

For many years shaped cellulose products such as fibres
have been produced from cellulose by the formation of a
10 cellulose compound which can be processed. Typically, the
cellulose compound is cellulose xanthate.

More recently, proposals have been made to form shaped
cellulose articles by forming true solutions of cellulose in
a solvent and spinning the solvent to form a fibre or film.

15 A preferred form of solvent is a tertiary amine N-
oxide, typically N-methylmorpholine N-oxide. Although
cellulose can be dissolved in such cyclic compounds, it has
proved difficult to dissolve it at rates which are
commercially acceptable.

20 In US Patent 4,246,221 there is described a process for
forming a true solution of cellulose in which there is
prepared a premix of cellulose, a solvent such as a tertiary
amine N-oxide and a non-solvent for cellulose such as water.
A premix of the three components is made, and on heating the
25 premix to evaporate the water, a true solution can be
formed.

The US patent referred to above describes forming the
solution particularly by heating the mixture in an extruder.
There is however description of alternative various types of
30 equipment, in particular thin-film evaporators. Thin-film
evaporators are well known as being suitable for the

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evaporation of moisture or liquid from a viscous solution. The premix of cellulose solvent and non-solvent is a viscous mixture.

5 An unusual aspect of the process of heating the premix and evaporating the water is that the result is to form a true solution of cellulose in the amine oxide. This solution is quite viscous - having a viscosity of typically 50,000 to 10,000,000 centipoise - and is more viscous than
10 the premix which is the starting material. Thus, the resultant product from the thin-film evaporator has a higher viscosity than the starting material but is a true liquid whereas the premix is not. In normal operations the evaporation of a product from a material tends to result in
15 the production of a solid as the material is processed.

For the production of a solution capable of being spun into fibres it is necessary to form a solution containing very little, if any, undissolved cellulose. It will be appreciated that the solution is formed into fibres by being
20 spun or extruded through a jet plate containing many fine-diameter holes. Typically, the holes will have a diameter of 250 microns or less. Such holes quickly become blocked if the product passing through them is not a true solution.

Although the use of a thin-film evaporator is described
25 in US-A-4,246,221 and is illustrated in EP-A-0,356,419, it has been discovered that there are economic problems associated with the scaling-up of the process for forming a true solution from a laboratory-scale to a large commercial-scale system.

30 Laboratory-scale thin-film evaporators having four blades and a heated surface area of about 0.5 m² have been found readily to produce a solution in accordance with the teachings of US-A-4,246,221 and as illustrated in EP-A-0,356,419.

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On attempting to carry out the same process utilising thin-film evaporators having a heated surface area of 1m^2 or above it has been found that a large increase in electrical (or rotor-induced) energy is required to obtain a complete solution of commercially viable quantities of the cellulose in the tertiary amine N-oxide. As the rate of production of solution is increased, there is a disproportionate increase in energy consumption.

A thin-film evaporator comprises an evacuable vertically oriented cylinder heated on the outside and containing a central rotatable shaft provided with paddle blades which distribute the material to be heated over the internal surface of the cylinder. As the central shaft is rotated the material passes down the evaporator, under the combined action of gravity and the paddles, to be subjected to heat and a vacuum within the thin-film evaporator. At the lower end of the cylinder the heated and processed material is removed by any suitable means. The normal arrangement for the paddles is to provide them in four vertical rows spaced at 90° around the central shaft.

It has now been discovered that with larger thin-film evaporators (by which is meant thin-film evaporators having a heated surface area in excess of 1m^2) it is not possible to operate such a thin-film evaporator as described in the prior art in a method which will produce a satisfactory true solution of cellulose in tertiary amine N-oxide at an energy consumption rate in kilowatt hours of consumption per kg per m^2 (kw/kg/m^2) which is economic.

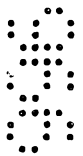
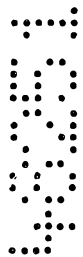
Summary of the Invention

By the present invention there is provided a method of forming a solution of cellulose in a tertiary amine N-oxide which comprises the steps of: forming a premix of a tertiary amine N-oxide, water and cellulose, heating the premix under a pressure lower than atmospheric in a vertically mounted thin-film evaporator having a heated inner surface and a

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Summary of the Invention

By the present invention there is provided a method of forming a solution of cellulose in a tertiary amine N-oxide which comprises the steps of: forming a premix of a tertiary amine N-oxide, water and cellulose; heating the premix
5 under a vacuum in the range 25 millibars to 100 millibars in a vertically mounted thin-film evaporator having a heated inner surface and a rotor comprising a central shaft with paddle blades disposed along the shaft so that, on rotation of the shaft, material in the evaporator will be contacted by a plurality of blades annularly spaced around the shaft; evaporating the water from the premix to a
10 level such that the cellulose forms a solution in the amine oxide; and removing the solution from the thin-film evaporator at a temperature in the range 90°C to 135°C, which method is characterised by providing said heated surface with an area in the range 1m² to 125m², by rotating the rotor such that the tips of the blades have a velocity of up to 8 metres/sec., and providing a circumferential
15 spacing between the tips of adjacent blades in the range of 65mm to 175mm, the drop off point for material dropping off one rotor blade being less than 90° before the pick up point for the next rotor blade.



~~central shaft with paddle blades disposed along the shaft so that, on rotation of the shaft, material in the evaporator will be contacted by two or more blades, evaporating the water from the premix to a level such that the cellulose~~
5 ~~forms a solution in the amine oxide and removing the solution from the thin-film evaporator, characterised in that the heated surface has an area in the range 1m^2 to 125m^2 , the rotor is rotated to give a tip velocity of up to 8 metres/sec., a separation is provided between tips of~~
10 ~~adjacent blades in the range of 65mm to 175mm, a vacuum in the range 25 millibars to 100 millibars is applied and the heated surface is heated to a temperature such that, under the conditions of applied vacuum, the solution is removed at a temperature in the range 90°C to 135°C .~~

15 Suitably the surface area is in the range 10m^2 to 125m^2 preferably 10m^2 to 75m^2 and the separation between blade tips is preferably in the range 75mm to 150mm.

 Preferably the rotor blades are located at intervals of 5° to 15° or 5° to 10° around the circumference of the
20 rotor.

 The paddle blades may be symmetrically or asymmetrically arranged about the central shaft. The paddle blades may be disposed in sets vertically spaced along the shaft. Each set may have the same number of blades, or the
25 upper half of the shaft may have more blades than the lower half, or alternatively the blade density may be varied along the shaft.

 The blades may be spirally arranged about the shaft with adjacent blades being angularly and axially spaced from
30 one another.

 The blades may be fixed blades and may be provided with teeth.



The blades may be aligned in the same direction as the rotational axis of the shaft, or may be inclined so as to force material downwards through the thin-film evaporator.

The central shaft may conveniently be revolved at a speed in the range 40 to 75 revolutions per minute (RPM). A speed of the tips of the blades in the range 2.5 to 5, preferably 2.5 to 4.5, metres/second would be typical.

The tertiary amine N-oxide is preferably a cyclic mono(N-methyl amine- N-oxide) such as N-methylmorpholine N-oxide.

Suitably the solution is manufactured at an exit temperature in the range 100°C to 115°C. Conveniently the premix is fed into the thin film evaporator at a temperature between ambient and 95°C, preferably between 75°C and 85°C.

15

The cellulose preferably has been derived from wood but may have been derived from cotton linters or other suitable cellulosic sources.

Preferably the solution contains 7% to 35% by weight cellulose, 15% to 4% water and the balance amine oxide.

The central shaft may be hollow and may be provided with integrally formed projecting blade root members to which the blades may be attached. The blades may comprise strips with integrally formed teeth. The teeth may comprise 10 to 40% of the vertical length of the strip.

Brief Description of the Drawings

By way of example embodiments of the present invention will now be described with reference to the accompanying drawings, of which:-

Figure 1 is a schematic sectional view of a system in accordance with the present invention,

Figure 2 is an end view of the top of the rotor of the system shown in Figure 1,

Figure 3 is a more detailed view of a blade to rotor connection,

5 Figure 4 is a perspective view of the arrangement of Figure 3,

Figure 5 is a schematic view of an alternative structure of rotor,

10 Figures 6a and 6b are schematic perspective views of different rotor designs,

Figure 7 is a graph of energy consumption and capacity against rotor speed,

Figure 8 is a graph of energy consumption against 15 capacity, and

Figure 9 is an extended graph of energy consumption against capacity, derived from Figure 8.

Description of Preferred Embodiments

Referring to Figure 1, this shows schematically a 20 system for performing the process of forming a cellulose solution in a tertiary amine N-oxide.

A tertiary amine N-oxide, such as N-methylmorpholine N-oxide, is fed into a container 1 along line 2. Feed lines are also provided for cellulose, 3, and water, 4. Typically, 25 the premix formed by mixing these components contains 10% by weight of cellulose, 50% by weight of water and 40% by weight of N-methylmorpholine N-oxide. The three components are mixed in the container 1 by means of a paddle screw 5 rotated by an electric motor 6. The paddle screw agitates 30 the mixture and passes the mixed components along a pipeline

7 to a thin-film evaporator indicated generally by 8. The pipeline preferably has a diameter such that it is always full, otherwise a restriction may be provided in its outlet into the thin film evaporator, so that material in the pipeline 7 is not exposed to the vacuum in the evaporator 8.

The thin-film evaporator 8 comprises a rotor within a cylindrical member 9 which is heated on the outside by means of a heating element 10. The heating element may be an electrical heating element, or an oil-filled element, or a complete steam-filled or hot-water-filled heating jacket. At the lower end of the cylindrical portion 9 there is a tapered portion 11 leading to a discharge line 12.

At the upper end of the cylindrical portion 9 there is a feed chamber 13 provided with an exit duct 14 through which evaporated vapours may be removed. Premixed material passes into the chamber 13 through the pipeline 7 and is distributed around the thin-film evaporator by a distributor plate 15 on rotation of the rotor, indicated generally by 16.

The central shaft of the rotor 16 is rotated by means of an external electric motor 17.

The rotor 16 is provided with a series of blades 18 which are described in more detail below. In operation, a reduced pressure is applied through the duct 14, whereby, on

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heating of the premix by means of the heating element 10, the water is evaporated during operation of the thin-film evaporator to reduce the water content of the premix as it is heated.

5 A description of a thin film evaporator, trademark Filmtruder is to be found in Devolatalisation of Plastics, English translation by S. Welling, published by VDI-Verlag GmbH, Düsseldorf 1980, pp69-97, the contents of which are incorporated herein by way of reference.

10 This continuous heating and evaporation results in a lowering of the water, i.e. the non-solvent, component in the premix to such an extent that the cellulose forms a true solution in the tertiary amine N-oxide.

In the lower portions of the thin-film evaporator 8, 15 therefore, there is formed a viscous solution which is pushed downwards by inclined blades 19 on a conical member 20 into the neck at the bottom of the tapered portion 11 of the thin-film evaporator. By rotation of a screw member 21 the solution of cellulose in solvent is passed to a pump 22 20 driven by an electric motor 23. From there the solution is passed by means of suitable pipework 24 to a spin nozzle 25.

The screw member 21 is rotated by means of an electric motor 26', and control of the electric motor 26' in conjunction with control of the electric motors 6 and 17 25 controls the flow of solution through the system.

Figures 2 to 4 show in more detail the structure of the rotor illustrated generally at 16 in Figure 1. It can be seen from Figures 2 and 3 that the rotor comprises a cylindrical central portion 26 having at its lower end a 30 tapered conical portion. At its upper end the cylindrical portion has a terminating plate 28 to which the rotational shaft from motor 15 is connected.

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The rotor central portion 26 is essentially a hollow cylinder having integrally projecting from it a series of six parallel blade roots 29, 30 etc. These blade roots extend the length of the central portion 26 of the rotor. 5 The blade roots are welded to and form an integral portion of the central region of the rotor.

Bolted to the roots such as root 30 are a series of plates 31, 32, 33, 34, 35 and 36 which form the actual blades of the thin-film evaporator. As is shown more clearly in 10 Figure 3, a blade plate 38 is bolted to a blade root 39 by means of a conventional bolt 40. As can be seen in Figure 4, a blade plate 41 has a series of teeth members 42, 43, 44 and 45 which extend to the far edges of the blade plate 41 and the blade plate 41 is secured to a blade root 46 by 15 means of bolts 47, 48 and 49.

The blade teeth 42 to 45 may be angled to push the viscous premix and viscous solution downwards through the thin-film evaporator. Because the rotor 16 of the thin-film evaporator 8 is arranged in a vertical disposition, the 20 action of the inclined blades cooperates with the action of gravity to enhance movement of the premix and solution downwards through the evaporator.

As is shown schematically in Figure 5, the blades on the central rotor portion may be staggered around the rotor 25 rather than being in straight lines as is shown in Figures 1 to 4. In the case of the rotor shown in Figure 5 a blade 51 is located axially and angularly spaced from its nearest adjacent blade 52. In the case of staggered paddle blades, as is shown in Figure 5, the angle between the blades should 30 be no more than 60°.

In EP-A-0,356,419 referred to above, it is indicated that the thin-film evaporator should incorporate four paddle wheels located around a small-diameter central shaft, there being a large region in the centre of the evaporator in

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which the cellulose may mix with the solvent and non-solvent.

It has been found, however, that scaling-up the apparatus, whilst still using four blades in the thin-film evaporator, results in a large and uneconomic increase in the unit costs for the formation of a true solution of cellulose in the solvent. It might have been thought that the rotor in the evaporator was not operating quickly enough to form a true solution. On manufacture, therefore, of a thin-film evaporator having a surface area in excess of 1 m², attempts were made to increase the rotational speed of the central rotor, so as to increase the number of wipes per minute experienced by the premix to increase the rate of formation of solution. However, this increased the unit cost significantly.

A series of tests was, therefore, carried out using a different number of rotor blades on the rotor rather than increasing the speed of rotation of the central rotor. For ease of understanding how the number of rotor blades on a rotor can be varied, it is desirable to refer to Figures 6a and 6b which are schematic cross-sections of portions of a rotor. In Figure 6a a central rotor shaft 60 carries a pair of flat rotor blades 61, 62 which are disposed at an angle of 90° to one another. The blade 61 would have an opposing blade at the other end of its diameter and the blade 62 would also have an opposing blade at the other end of its diameter. Thus as the rotor 60 was rotated the material in the thin-film evaporator would first be agitated by blade 61. The blades 61 and 62 are shown in a vertically staggered position and it will be appreciated that material being agitated by the blade 61 will drop off the blade at its lower edge 63, which is its drop-off point. The material will then stay in a fixed position until rotation of the rotor 60 results in the same material being contacted by the blade 62. This constitutes its pick-up point. Thus, the angular disposition of the flat blades 61 and 62 is 90°

and each particle of material will be contacted four times for every revolution of the rotor 60.

The concept of 90° located blades and four wipes per revolution implies that the blades 61,62 are arranged 5 perpendicularly along the axis of the rotor 60. In many cases the blades 61 and 62 would extend over the complete length of the rotor 60 without any interruption. In such cases agitation takes place between the tip of the rotor and the interior wall of the thin-film evaporator.

10 It is possible, however, to use rotor blades which are not completely vertically oriented. Referring to Figure 6b, therefore, rotor blades 71,72 are again disposed on a central rotor shaft 70. In this case, however, the rotor blades have inclined lower edges. The tops of the rotor
15 blades are therefore arranged at 90° angles as shown by the angle 73 generated between the upper edges of the blades 71 and 72. However, material being wiped by the blade 71 will be moved downwardly to leave the blade at its lower edge 74. In this case, the angle 75 between the drop-off point of the
20 blade 71, i.e. edge 74, and the pick-up point of the blade 72 is less than 90°. By dividing the angle 75 into 360° a notional number of rotor blades per rotor can be generated. Thus, if the angle 75 is 75° then there will be a notional 4.8 blades per rotor. Thus, the notional number of blades
25 per rotor can be increased either by including more sets of rotor blades disposed around the rotor or by angling the rotor blades so that the drop-off point for one rotor blade is less than 90° before the pick-up point for the next rotor blade.

30 It has now unexpectedly been discovered that by reducing the angle between the drop-off point for one rotor blade and the pick-up point for the next rotor blade to engage that material, significant increases in productivity can be obtained in the thin-film evaporator and, more
35 importantly, the increases in productivity can be obtained

whilst reducing the total absorbed power required to produce each kg of processed material.

Tables 1, 2 and 3 below show the effect of varying the average number of blade sets on product output versus rotor speed for a thin-film evaporator having an effective surface area of 5.5 m². Table 1 shows the rotor speed, product (solution) output, absorbed power, total absorbed power and total absorbed power in kilowatts (kw) divided by kilograms (kg) per hour output for a rotor having an average of 4.8 blade sets. Tables 2 and 3 show the same factors but for rotors having an average of 8.8 blade sets in the case of Table 2 and 12 blade sets in the case of Table 3.

Table 1

4.8 Blade Sets, 75' spacing

Rotor Speed (rpm)	Solution Output (kg/hr)	Absorbed Power (kw)	Total Absorbed Power (kw)	Total Absorbed Power (kw)/kg hour
90	460	8	13	0.0283
110	530	9.7	19	0.0358
140	670	18.3	26.5	0.0395
180	765	24.9	35.5	0.0464
220	910	30.5	45.5	0.0500

Table 2

8.8 Blade Sets, 40.9' spacing

Rotor Speed (rpm)	Solution Output (kg/hr)	Absorbed Power (kw)	Total Absorbed Power (kw)	Total Absorbed Power (kw)/kg/hr
93	735	20.7	26	0.0353
100	790	23.3	29	0.0295
105	840	25.0	31	0.0369
110	875	26.7	33	0.0377
120	950	30.1	37	0.0389
130	975	31	38.5	0.0395
140	1050	32.3	40.5	0.0386

Table 3

12 Blade Sets, 30' spacing

Rotor Speed (rpm)	Solution Output (kg/hr)	Absorbed Power (kw)	Total Absorbed Power (kw)	Total Absorbed Power (kw)/kg/hour
100	1074	23.8	30	0.0279
130	1380	28.0	43	0.0312

The total absorbed power includes the power absorbed by the bearings and gears and inefficiencies in the electrical and mechanical system used to drive the thin-film evaporator. The absorbed power is deduced by operating the 5 thin-film evaporator at the various speeds, but whilst empty, and determining the power output required to drive the thin-film evaporator itself. Thus, the absorbed power is the power required merely to form a solution of the cellulose in the solvent.

10 The particularly important figure is, however, the total absorbed power, as this is the factor which effects the true cost of operating the thin-film evaporator.

It will readily be appreciated that it is normally

preferable from a mechanical viewpoint to operate equipment at a lower rather than a higher speed, because wear rates increase dramatically as speeds increase.

An immediate survey of the three tables indicates clearly therefore that by increasing the number of blade sets the total dope output in terms of kg per hour available by operating the thin-film evaporator at a constant speed increases dramatically. Thus, operating the thin-film evaporator with 4.8 blade sets at 140 rpm gives a total output of only 670 kg per hour. Operating the same thin-film evaporator with an average of 8.8 blade sets gives a dope output of 1,050 kg per hour, whereas using 12 blade sets even at a lower rotor speed of 130 rpm increases the dope output to 1,380 kg per hour.

Although, therefore, it is important that the output of dope can be increased by increasing the number of blade sets it has unexpectedly been discovered that this distinct advantage (meaning that fewer thin-film evaporators are required to produce a given dope output) is also accompanied by a decrease in the power required to produce material at higher production rates.

This may most easily be seen by referring to the accompanying graph, Figure 7, which is a graphical representation of the information set out in Tables 1 to 3 above.

In the graph the rotor speed is shown on the x axis as varying between 80 and 220 revolutions per minute. On the left-hand y axis the total absorbed power in kw per kg per hour of dope output is shown. On the right-hand y axis is shown the output in kg per hour. Comparing first the dotted lines 81, 82 and 83, these show the total output for a thin-film evaporator having 4.8 blade sets, 8.8 blade sets and 12 blade sets, respectively. It can be seen that very considerable improvements in productivity are to be obtained

by increasing the number of blade sets. In Figure 7 the crosses within circles represent the total absorbed power per kg per hour for the 4.8 blade-set thin-film evaporator, the squares within circles show the same factor for the 8.8 blade-set thin-film evaporator and the triangles within circles show the same factor for the 12 blade-set thin-film evaporator.

The solid lines 84, 85 and 86 in Figure 7 show the power consumption in kw per kg per hour of dope output, i.e. the unit power consumption. It can first be seen that the 12 blade-set thin-film evaporator not only has the highest productivity (line 83) but also produces dope at the lowest power consumption in terms of kw per kg per hour (line 86). The line 84 shows that the unit power consumption for the 4.8 blade-set rotor is lower at lower rotational speeds than for the 8.8 blade-set rotor (line 85). The lines cross, however, showing that as the rotational speed and output increases the 8.8 blade-set rotor becomes more efficient.

To enable the information shown graphically in Figure 7 to be used to derive the actual power consumption per kg/hour of product manufactured it is necessary to prepare a further graphical representation as shown in Figure 8. Although the information on power compared to productivity is available in Figure 7 it is more easily understood in Figure 8. Figure 8, therefore, is a graph of solution output in kg per hour on the y axis against power consumption in terms of total absorbed power in kw per kg per hour of output on the x axis. Line 90 is the line graphically representing the output of the rotor having an average of 4.8 blade sets, line 91 is the average 8.8 blade-set rotor line and line 92 is the average 12 blade-set rotor line.

It can first be seen that for any given power input into the system the production of dope is dependent on the number of rotor blades on the rotor. Thus, reading along

the x axis to any given power input in terms of total absorbed power per kg per hour of dope produced, the greater the number of rotors the higher the position of the line joining the various points and hence the higher the total output of dope. Thus, inputting between points 0.027 and 0.030 kw hours per kg would give a productivity of about 400 kg per hour in the case of a 4.8 blade-set rotor, about 600 to 800 kg per hour in the case of an 8.8 blade-set rotor and about 1000 to 1200 kg per hour in the case of a 12 blade-set rotor thin-film evaporator.

The information in Figure 8 has to be extrapolated to give the full implication of the improvement to be obtained by moving from a 4.8 blade-set rotor to a 12 blade-set rotor. Figure 9 is such an extrapolation. Figure 9 is again a graph of output in kg/hr on the y axis versus unit power requirement in kw/kg/hr on the x axis. Line 92 is the same as in Figure 8, but line 93A is line 93 from Figure 8 extended to higher outputs. The actual measured points are shown on the graph and are derived from Tables 1 and 3. From Figure 9 it can be seen that a 12 blade-set rotor can produce 1075 kg/hr output at a unit energy requirement of 0.028 kw/kg/hr, whereas the 4.8 blade-set rotor would require a unit energy input of 0.0575 kw/kg/hr, over twice as much energy. Similarly the 12 blade-set rotor can produce 1380 kg/hr at a unit energy requirement of 0.031 kw/kg/hr, whereas the 4.8 blade-set rotor would require an energy input of about 0.0725 kw/kg/hr, considerably more than twice the unit energy requirement.

For larger units, of, for example, 1.40 m diameter, up to 20, 24, 26, 28, 30, 32, 34 or even more blade sets can be used, with operating pressures in the range 50 to 250 millibars or 35 to 100 mb and exit temperatures of 100-110°C.

It has unexpectedly been discovered, therefore, that increasing the number of wipes of the rotor blade on the

premix by increasing the rotational speed of the rotor does not increase the capacity of the apparatus to form a true solution in the same way as increasing the number of blades.

Given that increasing the number of blades results in
5 an increasing number of wipes per minute experienced by the premix as it passes through the solution, it might have been thought that this would be equivalent to increasing the rotational speed of the rotor. However, this has been found not to be the case and increasing the number of blades
10 around the rotor still gives a good-quality product in terms of a clear solution with insignificant amounts of undissolved cellulose in the solution.

It has further been discovered that by using larger numbers of blades, the thin film evaporator produced good
15 quality solution even when operating slowly, thus enabling the unit to be slowed down and produce smaller quantities of solution for times when less solution is required.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. A method of forming a solution of cellulose in a tertiary amine N-oxide which comprises the steps of:

- (i) forming a premix of tertiary amine N-oxide, water and cellulose,
- (ii) heating the premix under a vacuum in the range 25 millibars to 100 millibars in a vertically mounted thin-film evaporator having a heated inner surface and a rotor comprising a central shaft with paddle blades disposed along the shaft so that, on rotation of the shaft, material in the evaporator will be contacted by a plurality of blades annularly spaced around the shaft,
- (iii) evaporating the water from the premix to a level such that the cellulose forms a solution in the amine oxide, and
- (iv) removing the solution from the thin-film evaporator at a temperature in the range 90°C to 135°C,

characterised by providing said heated surface with an area in the range 1m² to 125m², rotating the rotor such that the tips of the blades have a velocity of up to 8 metres/sec., and providing a circumferential spacing between the tips of adjacent blades in the range of 65mm to 175mm, the drop off point for material dropping off one rotor blade being less than 90° before the pick up point for the next rotor blade.

2. A method as claimed in claim 1, characterised in that the surface area is in the range 10m² to 125m² preferably 10m² to 75m², the circumferential spacing between blade tips is in the range 75mm to 150mm, and the tip velocity is in the range 2.5 to 5 metres/sec.

3. A method as claimed in claim 1 or claim 2, characterised in that the tip velocity is in the range 2.5 to 4.5 metres/sec.

4. A method as claimed in any preceding claim, characterised in that the solution is removed at an exit temperature in the range 100°C to 115°C.



5. A method as claimed in any preceding claim, characterised in that the premix is fed into the thin-film evaporator at a temperature between ambient and 95°C, preferably between 75°C and 85°C.

6. A method as claimed in any preceding claim, characterised in that the rotor has blades located at ^{intervals} ~~internals~~ of 5° to 15° or 5° to 10° around the circumference of the rotor.

7. A method as claimed in any preceding claim, characterised in that the solution contains 7% to 35% by weight cellulose, 15% to 4% water and the balance amine N-oxide.

8. A method as claimed in any preceding claim, characterised in that the blades are spirally arranged about the shaft with adjacent blades being angularly and axially spaced from one another.

9. A method as claimed in any preceding claim, characterised in that some at least of the blades are inclined so as to force material downwards through the thin-film evaporator.

10. A method of forming a solution of cellulose in a tertiary amine N-oxide, substantially as herein described with reference to the accompanying drawings.

DATED this 1st day of August, 1996.

COURTAULDS FIBRES (HOLDINGS) LIMITED and BUSS AG

WATERMARK PATENT & TRADEMARK ATTORNEYS
290 BURWOOD ROAD
HAWTHORN VICTORIA 3122
AUSTRALIA



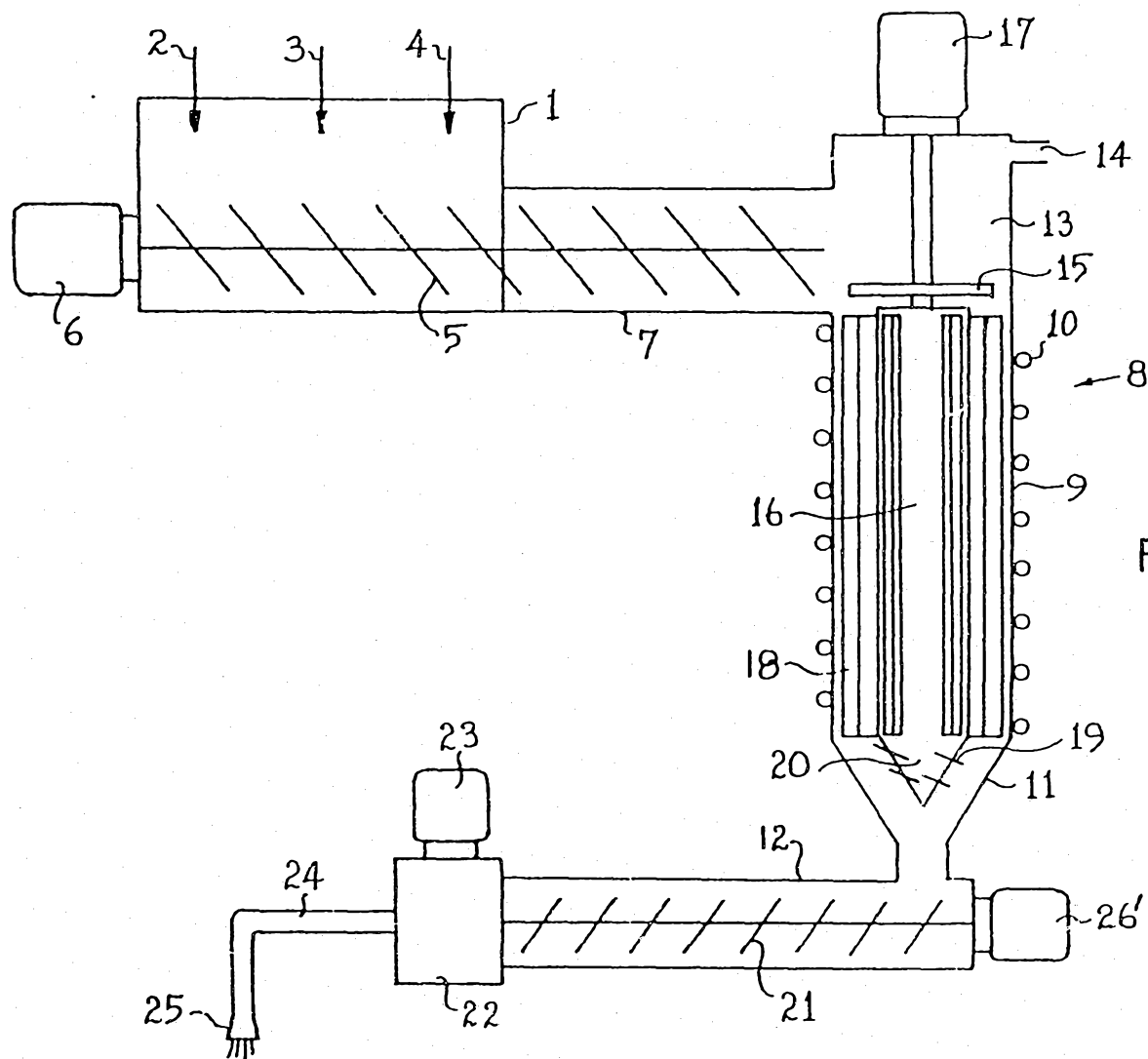


FIG 1

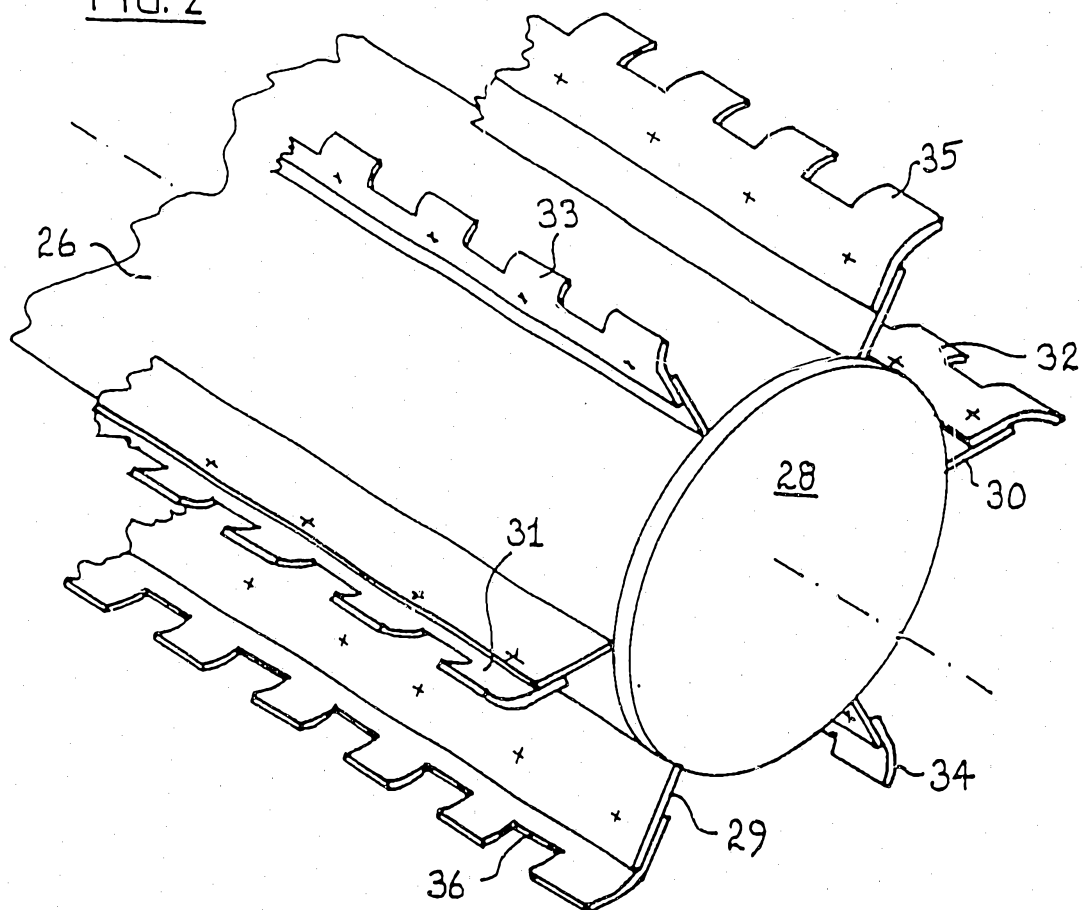
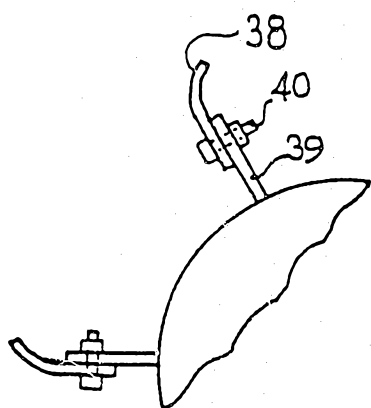
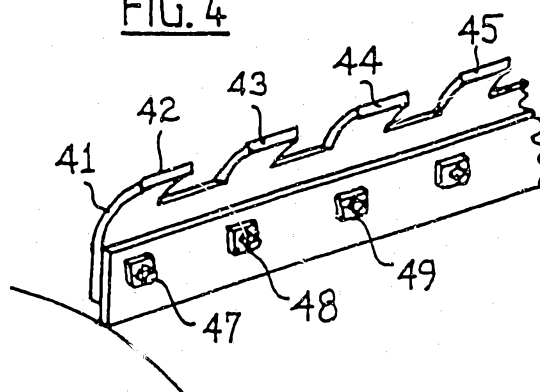
FIG. 2FIG. 3FIG. 4

FIG. 5

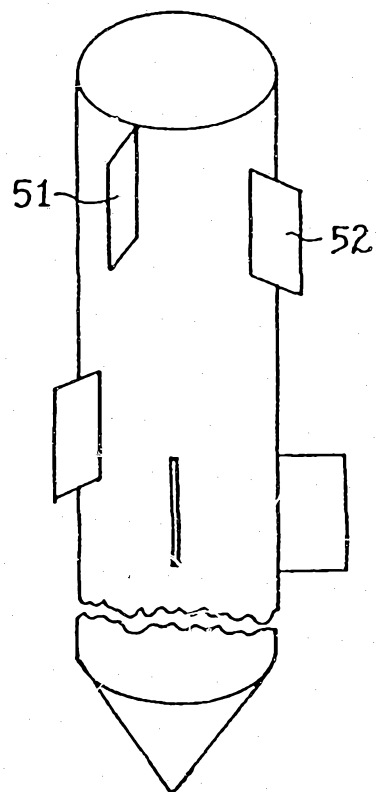


FIG. 6a

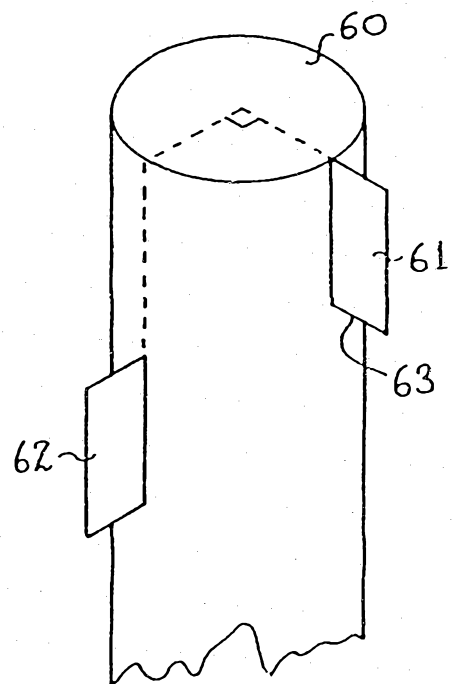


FIG. 6b

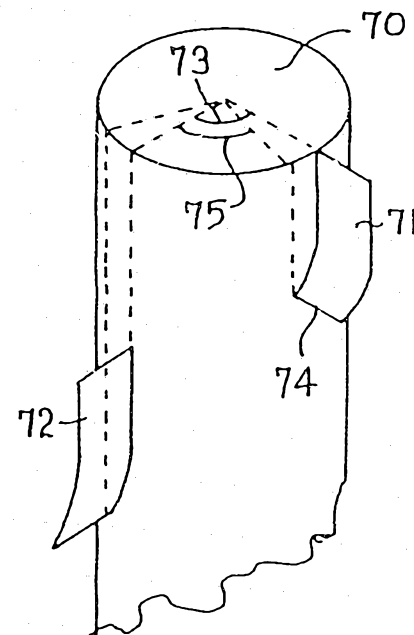


FIG 7

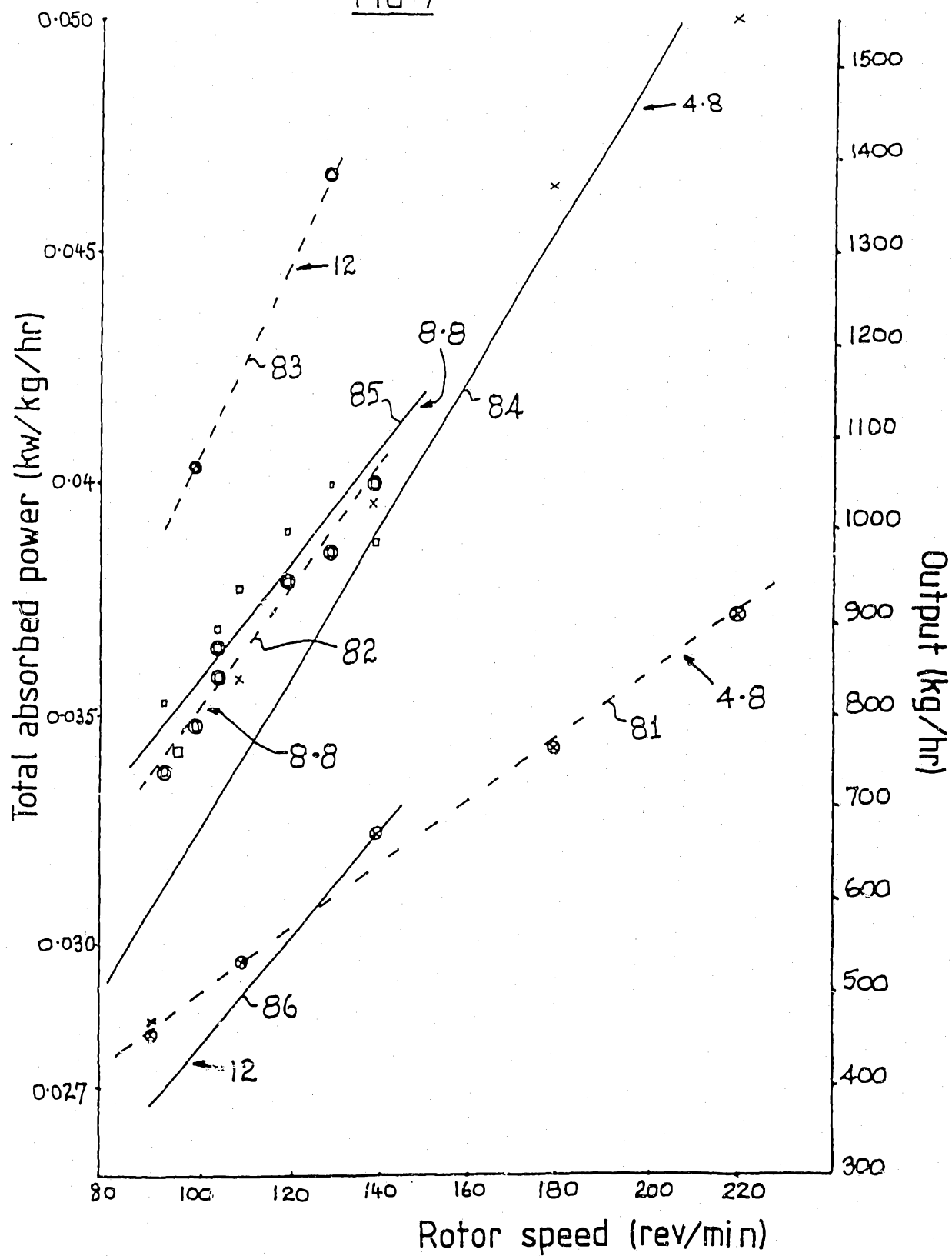
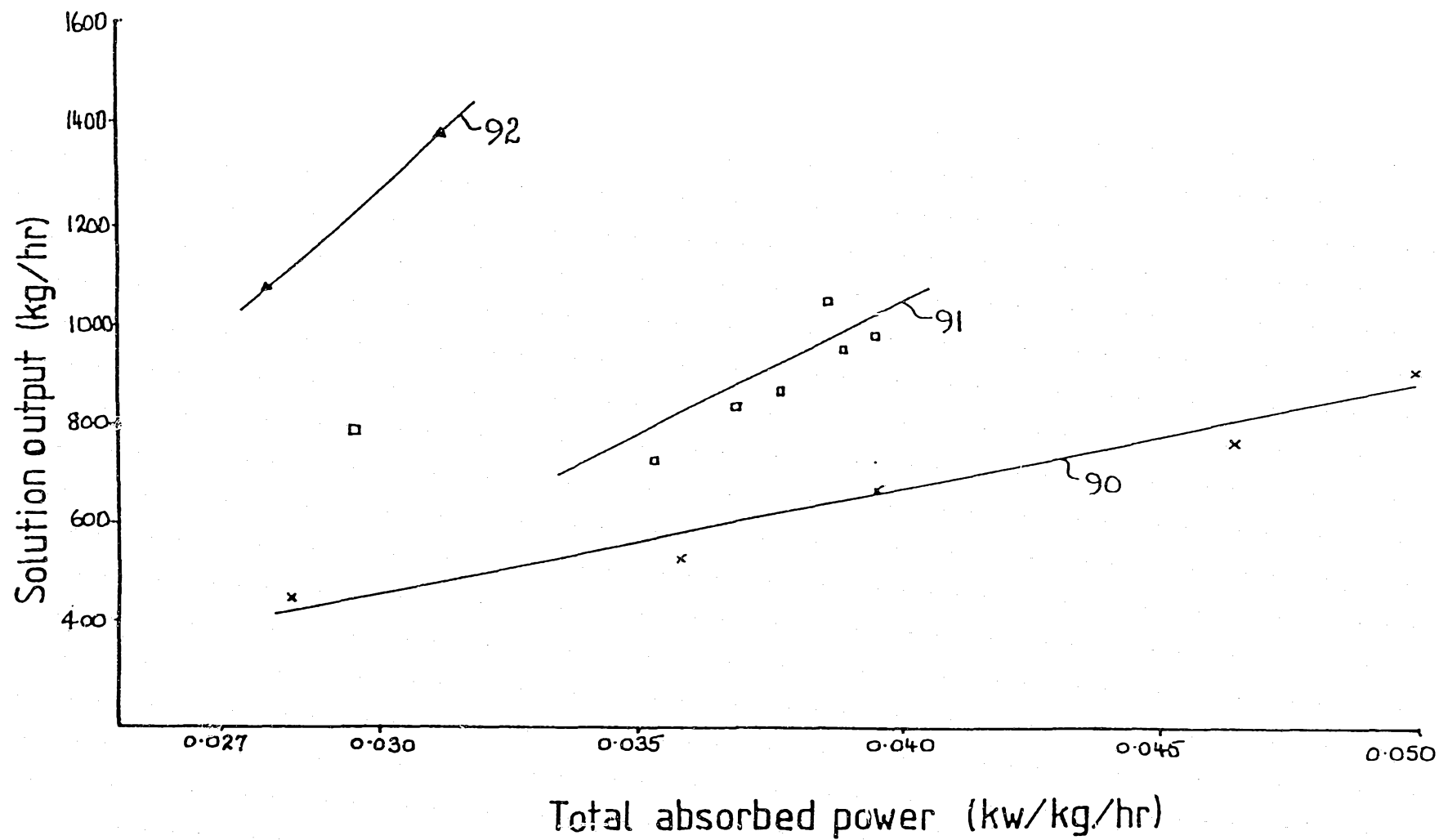
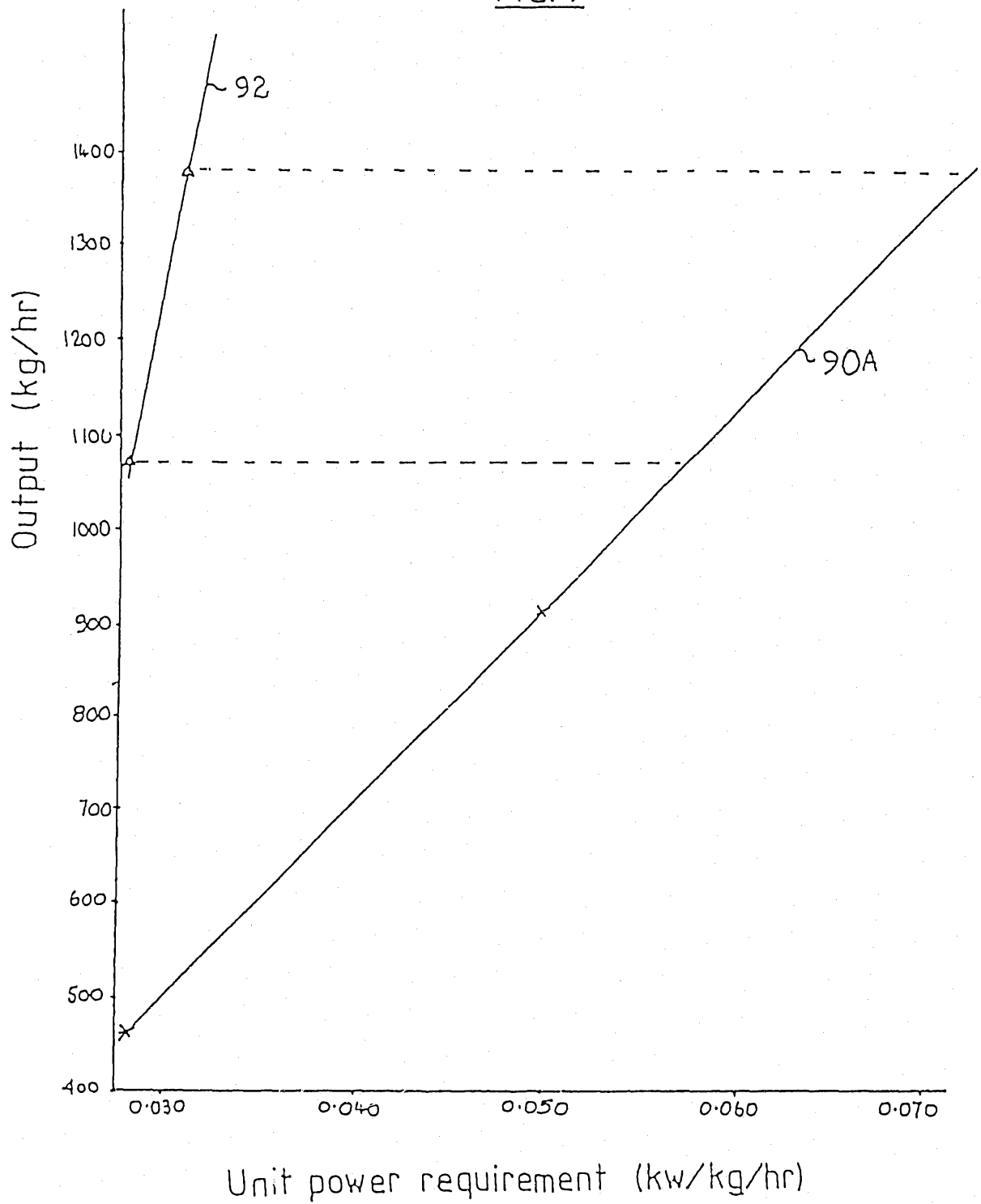


FIG 8

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FIG. 9



INTERNATIONAL SEARCH REPORT

International Application No.
PCT/GB 93/01956A. CLASSIFICATION OF SUBJECT MATTER
IPC 5 B01D1/22 B01F1/00 C08J3/09

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 5 B01D B01F C08J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP,A,0 356 419 (LENZING AG) 28 February 1990 cited in the application see the whole document -----	1

☐ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

17 December 1993

Date of mailing of the international search report

18.01.94

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax (+31-70) 340-3016

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Van Belleghem, W

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No.

PCT/GB 93/01956

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP-A-0356419	28-02-90	AT-B- 392972	25-07-91
		JP-A- 2127434	16-05-90
		SU-A- 1797630	23-02-93
		US-A- 5094690	10-03-92
