



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

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<b>(21) International Application Number:</b> PCT/AU97/00182 <b>(22) International Filing Date:</b> 2 April 1997 (02.04.97) <b>(30) Priority Data:</b> PN 9096 3 April 1996 (03.04.96) AU <b>(71) Applicant (for all designated States except US):</b> PARTICIPANT PROJECT IP LIMITED [AU/AU]; John Street, Hawthorn, VIC 3122 (AU). <b>(72) Inventor; and</b> <b>(75) Inventor/Applicant (for US only):</b> LASCARIS, Edmond [AU/AU]; John Street, Hawthorn, VIC 3122 (AU). <b>(74) Agent:</b> MISCHLEWSKI, Darryl; P.O. Box 1254, Camberwell, VIC 3124 (AU).		<b>(81) Designated States:</b> AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ARIPO patent (GH, KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, 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).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> PAPER PULP DRAINAGE AID  <b>(57) Abstract</b>  To improve the drainage and consequently the machine speed in making paper or paperboard from recycled paper, a starch hydrolysing enzyme [alpha-amylase] is added prior to the forming section. The enzyme must be active in the temperature range of 40 to 90 °C and in the pH range of 6 to 9. Amounts as low as 0.05 % by weight of the oven dried paper forming the pulp can allow increases in machine speeds of about 5 % or more.		

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## PAPER PULP DRAINAGE AID

### Field of the Invention

This invention relates to improvements in treating pulp and process water in paper mills and in particular to a drainage aid for use in recycled paper mills

### Background to the Invention

The use of recycled paper to produce paper pulp creates problems in the free drainage of the paper pulp and the treatment and recycling of the process water. Because of the wide range of source material, a large range of components are present in the backwater. These include the organic components fibres, fibrils, pitch, starch derivatives, lignin, synthetic polymers and inorganic components to include clay, titanium dioxide, gypsum, talc, chalk, zinc oxide, aluminium sulfate, ferrous sulphate, and calcium carbonate. Much of this material is present as fines[ i.e. below 120 micrometres]. It is preferable to retain some of the fines, which may comprise up to 5% of the paper sheet on the filter mat, without reducing drainage rates and paper properties and consequently affecting process times.

Enzyme treatment of paper pulps has been proposed to improve the freeness and physical properties of paper pulps with some claiming success with recycled fibre as well as virgin fibre. The enzymes that have been proposed are xylanases, cellulases, and other hemicellulases. These are commercially available as crude mixtures characterised by the dominant presence of the named enzyme. One defect of these treatments is that careful regulation of the addition and treatment conditions is required, because excessive treatment can lead to a decline in drainage. Another difficulty is that paper pulps are usually neutral and many cellulases are active in the acidic pH range.

U S A patent 4923565 proposed the use of a cellulase (having cellulase and xylanase activity), as a drainage aid for recycled paper pulp. One shortcoming of this proposal is that the pH must be adjusted to within the range 3 to 7.

An improvement to this patent is U S A patent 5116474 which utilises cellulases  
5 active in the pH range of 7 to 9.

U S A patents 5169497 and 5423946 suggest using the drainage aid of the '565 patent with cationic polymers such as acrylamides.

Patent specification WO 91/17243 discloses that a cellulase having an endo - glucanase component active between pH 6 and 10 can be used as a drainage aid  
10 for paper pulps including waste paper pulp. The purpose of the enzyme is to reduce harshness or clarify colour in cellulose containing fabrics

### **Brief description of the invention**

It is an object of this invention to improve the drainage of recycled paper pulp using a more effective enzymatic treatment. The term "recycled paper" is  
15 intended to cover paper pulp, which includes a major portion of paper or paperboard that has been recycled.

To this end the present invention provides, as a drainage aid for improving the free drainage of recycled paper pulp slurries, a preparation containing a major portion of a starch hydrolysing enzyme, preferably an amylase and more  
20 preferably an alpha-amylase.

The invention also includes a method of improving drainage of recycled paper pulp which includes the step of adding to the pulp a drainage aid containing a major portion of a starch hydrolysing enzyme, preferably amylase, prior to the separation of the water from the pulp. The drainage aid is added in an amount  
25 of at least 0.05% by weight of oven dried paper forming the pulp, for a time and at a temperature sufficient to produce an improved drainage rate and at a pH at which the enzymes are active. At the lower concentration the addition of enzyme

will improve drainage to the extent that the paper machine speed and production can be increased by about 5% or more.

In part this invention is predicated on the discovery that drainage of recycled paper pulps is hampered by coatings on solid components in the pulp, particularly by the fine particles in the pulp. A significant proportion of recycled paper contains starch or starch based materials and in part this invention is based on the realisation that that enzymatic treatment may remove this coating. This invention is also founded on the discovery that amylase is able to act on the coatings of particles in the pulp regardless of its inner composition which is not the case with previously proposed enzymes such as cellulase. It is thought that the coatings are starch based and cellulases have little effect on the starch, but are more likely to act on the particles which are primarily cellulose. This further discovery also indicates that amylase is specific in its action on the starch components. This has the consequence that careful regulation of the treatment is not necessary and excessive treatment does not decrease the drainage improvement. The improved effectiveness of a drainage aid having a major amylase component may be due to the coatings on the fines being largely composed of starch and thus more susceptible to amylase or other starch hydrolysing enzyme treatment. Because the amylase has little action on the cellulose component of the fibres, excess treatment is unlikely to cause any deleterious effect. This contrasts with the prior art enzymes which are primarily cellulases which are usually recommended for papers made with a high content of virgin fibre.

Although amylase is a major component of the drainage aid, other enzymes, particularly cellulases active in the neutral- alkaline pH range, particularly pH 6 to 9 may be present. Some cellulases do improve drainage which is inhibited by cellulose based materials in the pulp and part of drainage inhibition in recycled pulps may be reduced with cellulases. For many recycled pulps or mixed virgin and recycled fibres a mixture of amylase and cellulase may provide the optimum improvement.

### Detailed description of the invention

Process conditions for dewatering paper pulp using the drainage aid of this invention are typically:

	temperature	40 to 90 °C
5	pH	6 to 9
	enzyme dosage	2.5x10 <sup>6</sup> to 15x10 <sup>6</sup> IU of activity per tonne of pulp which for an alpha amylase such as BAN 240L is equivalent to 0.5 to 3 Kg per oven dried tonne of recycled paper.
	contact times	15 to 120 minutes

- 10 The International Unit [I U] of enzyme activity is the amount of enzyme necessary to produce 1 micromole of reducing sugar product per minute.

The enzymes used in the drainage aid can comprise an amylase alone preferably a crude amylase or an amylase in combination with a cellulase such as endoglucanase, xylanase, or other hemi cellulases such as mannanase, all of which are preferably active in the neutral to alkaline pH range of 6 to 9. A preferred crude alpha- amylase is that derived from *Bacillus subtilis*. Starch hydrolysing enzymes from other microbial species may also be used including *B.licheniformis* and *Aspergillus oryzae*

- 20 In addition to the amylase drainage aid the conventional polymeric flocculants, such as cationic polyacrylamide, which also aid in drainage can be used. The amylase presence will allow a reduction in the amount of the flocculants required.

- 25 Laboratory scale drainage tests were carried out on backwater samples from a recycled paper plant. A crude and a purified alpha- amylase was used to treat the backwater samples. The purification was achieved by chromatographic focussing. Drainage performance was measured using a modified Schopper-Riegler drainage jar. A brass woven screen with 50 micron gaps captured the fines to form a filter cake. This filter cake sufficiently impeded the backwater to permit accurate drainage readings to be taken by continuously collecting and

weighing the filtrate. Drainage improvement was evaluated by comparing the difference between control and enzyme treated samples over 30 minutes.

### Example 1

Experiments were conducted using both crude and purified alpha amylase as identified in table 1. The backwater characteristics and fines size distribution are set out in tables 2 and 3.

**Table 1**  
alpha-amylase characterisation

alpha-amylase	CMC IU/mg	Xylan oat spelts IU/mg	Xylan birchwood IU/mg	Starch IU/mg	Amylo-pectin IU/mg
crude	0.006	0.649	0.395	243.750	291.667
purified	0	19.875	0	175.624	182.500

**Table 2**

Backwater characterisation

Batch	pH	Conductance micro S/cm	Dry mass g/L	Ash %
1	7.23	850	6.31	18.19
2	7.12	980	6.53	20.55
3	7.34	860	7.08	19.04

**Table 3**

Particle size analysis

Number density parameters (microns)	Batch 1	Batch 2	Batch 3
Median	1.20	0.89	0.96
Mean	1.80	1.35	1.55
SD	2.13	1.56	2.00
Mode	1.25	0.75	0.75

The results of the experiments are set out in table 4.

Because the fines in the backwater samples are in a higher concentration than in the pulp the dosage rates are higher because the surface area to volume ratios for the fines is 20 to 30 times the ratio for pulp. Thus the dosage requirement for treating pulps will be significantly less than the table suggests.

**Table 4**  
Drainage analysis

Batch	amylase purity	amylase dosage mg/L	amylase dosage units/Kg	Drainage vol. After 30 mins mL	Drainage improvement %
1	control	0	0	768.57	na
1	pure	0.15	4,171	791.07	2.93
1	crude	0.15	5789	804.56	4.68
1	crude	1.5	57,890	874.9	13.83
1	crude	15.0	578,900	932.71	21.36
2	control	0	0	764.19	na
2	pure	0.15	4032	774.59	1.36
2	crude	0.15	5597	793.86	3.88
2	crude	1.5	55,970	822.92	7.69
2	crude	15.0	559,700	902.34	18.08
3	control	0	0	707.93	na
3	pure	0.15	3720	725.6	2.50
3	crude	0.15	5163	733.87	3.66
3	crude	1.5	51,630	794.06	12.17
3	crude	15.0	516,300	861.49	21.69

10 These results show that amylase acts on the surface of all the components in the backwater regardless of inner composition, by removing starch coatings on both

fines and fibres. Excessive treatment with cellulases tends to increase the fines leading to a decline in drainage performance and paper strength.

The large surface area and particulate nature of the fines is regarded as the major contributor to poor drainage performance of recycled pulps. By selectively  
5 hydrolysing the surface material on fines and fibres, the interaction between particle surface, retention and drainage aids and water is thought to be enhanced. This consequently encourages better drainage in the forming section of the paper plant.

### Example 2

10 A mill trial was conducted on a twin wire fourdrinier machine producing liners and medium for corrugated boxes. The machine is capable of producing between 12 and 15 tons of paper per hour from furnish consisting of mixed waste paper, newsprint and OCC.

The enzyme selected for the mill trial was BAN 240L which is an alpha amylase  
15 preparation produced by Novo Nordisk. It is a clear brown watery liquid obtained from a purified culture of *Bacillus subtilis* variety *amyloliquefaciens*.

The enzyme addition commenced from 9am on day 1 and ceased 12pm on day 3 [approximately 48hours]. The enzyme was dosed at 30 l/hr [as received basis] in at two points in the stock preparation area.

20 Table 5 shows the mill performance over 7 days with the enzyme addition occurring on days 1 to 3. During the period of enzyme addition the machine speed increased by 4.49% compared to the operation without enzyme addition. In the table production rates have been extrapolated to remove lost time. Product output increased 6.5% but when adjusted for trim the increase was  
25 4.48%.

TABLE 5

Trial Days	1	2	3	4	5	6	7
Av. Dry end speed m/min	516.21	523.4	507.34	493.05	490.12	493.28	497.31
Production T/day	314.71	318.87	319.91	300.38	298.65	296.27	298.43
Trim mm	2788.50	2843.00	2800.46	2717.26	2806.65	2775.48	2729.96

An improvement in the freeness of the pulp as measured by the Schopper-Reigler [SR] drainage values in the headbox and machine chests, and for both  
 5 top and bottom lines was observed.

TABLE 6

## Schopper-Reigler Freeness values of pulp

	Headbox top	Headbox bottom	Machine chest -top	Machine chest - bottom
During trial	35	40	31	40
After trial	57	59	51	63

10 This shows that the enzyme decreases the SR freeness values by 32% to 39% depending on the sample origin.

This compares favourably with the performance of the cellulases with recycled paper pulp disclosed in USA patent 5116474 which obtained decreases in SR values of between 5% and 23%

15 This mill trial showed that a cost effective enzyme treatment produced an additional 19.4 tonnes of paper per day, increased machine speed by 22.14 m/min and improved the S R values.

**Example 3 – Comparative**

The enzymes as described in tables 7a 7b and 7c were tested for comparison purposes.

“na” means the data is not available

5

**TABLE 7a**

Name	AMG 300L	BAN 240L	Celluclast 1.5 L	Denimax L
Enzyme	Amylo-glucosidase	Alpha amylase	Cellulase	Endo-glucanase
Organism of origin	<i>Aspergillus niger</i>	<i>Bacillus subtilis</i>	<i>Trichoderma reesei</i>	Non pathogenic mould
Density g/mL	1.2	1.2	1.2	na
pH optima	4.5	5-7	4.5-6.0	4.5-5.5
Temp. °C	60	50-70	50-60	45-55
Reaction time	na	na	na	na
Dosage	na	na	na	na

**TABLE 7b**

Name	Fungamyl 800L	Irgazyme 40	Novozyme 342	Pergalase A40
Enzyme	Alpha-amylase	Xylanase [no cellulase activity]	Cellulase, glu canase and hemicellulase	Cellulase Predominant
Organism of origin	<i>Aspergillus oryzae</i>	<i>Trichoderma longibrachiat um</i>	na	<i>Trichoderma longibrachiat um</i>
Density g/mL	1.25	1.01-1.15	na	1.01-1.15
pH optima	4.5-6.0	7.0-8.5	7.5-8.5	5-7
Temp. °C	55-60	50-70	40	35-55
Reaction time	na	0.5-3 hrs	1hr	0.5-2 hrs
Dosage	na	0.2-0.7 l/t	4l/t	0.5-1.8l/t

TABLE 7c

Name	Promozyme 200L	Pulpzyme HB	San Super 240L
Enzyme	Pullanase Pullan6-glucano- hydrolase	Xylanase Endo-1,4-beta-D- xylanase	Amyloglucosidase [and alpha amylase]
Organism of origin	<i>Bacillus</i> <i>acidopullulytics</i>	Bacterial	na
Density g/mL	1.25	na	1.25
pH optima	4.0-6.0	7-8	5.5
Temp. °C	50-60	50	55
Reaction time	na	2-3	na
Dosage	na	1kg/t	na

These enzymes were tested in the laboratory to assess the drainage  
 5 improvement with pulp backwater in similar fashion to example 1. The results  
 are shown in table 8. The reducing sugar increase is an indication that the  
 enzyme was active. These results show that the alpha amylases BAN 240L and  
 FUNGAMYL out performed the other enzyme types with the exception of the  
 cellulase CELLUCLAST.

TABLE 8

**Treatment of backwater containing fines  
with enzymes listed in tables 7a, 7b and 7c**

Enzyme	Drainage improvement (%)	Reducing sugar increase (mM)
AMG 300L	0.7	9.2
BAN 240L	4.0	0.77
CELLUCLAST 1.5L	2.8	0.28
DENIMAX L	1.7	0.94
FUNGAMYL 800L	2.5	2.75
IRGAZYME 40	0.9	0.78
NOVAZYME 342	1.3	0.78
PERGALASE A40	0.4	0.0
PROMOZYME 200L	0.0	1.35
PULPZYME HB	1.0	0.31
SANSUPER 240L	0.9	4.81

#### 5 Example 4 – Flocculants

Backwater samples having a consistency at 0.631%, were treated with alpha amylase obtained from *Bacillus licheniformis*, incubations were performed at 0 and 300 IU/ml at 50°C for 3 hours. High molecular weight cationic polyacrylamide [CATPAM] was added at a set dosage of 0.3 micrograms per 10 1000mL of backwater. The backwater containing the polymer was inverted 30 times in a glass measuring cylinder. Particle size analysis was performed in the range 0.2 to 60 micrometres.

TABLE 9

**alpha-Amylase and polymer treated backwater**

Particle Measurement	Volume	Volume
	No polymer addition	polymer addition
<b>CONTROL</b> - Median	19.3	23.46
- Mean	20.91	23.91
- SD	14.30	14.75
<b>alpha-Amylase-</b>		
Median	17.21	25.63
- Mean	19.68	25.31
- SD	14.32	15.67

Control particle count decreased from 212056 to 144617 while the a-Amylase  
 5 particle count decreased from 178404 to 128787.

In the absence of polymer the particle count of the enzyme treated backwater  
 decreased by 15% relative to the control. The particle count decreased further  
 with the addition of polymer to the backwater. The control decreased by 32%,  
 whereas the enzyme treated backwater decreased by 40% relative to the control  
 10 without polymer. The mean particle mass for the enzyme treated backwater,  
 with polymer, increased by 6% relative to the control with polymer. These  
 results imply that the enzyme treated fines are more receptive to polymer  
 interactions as demonstrated by the greater floc size and lower particle count.

These results compare favourably with the data in USA patent 5169497 which  
 15 show an improvement in freeness when a cationic polyacrylamide is added to a  
 cellulase treated recycled paper pulp.

**Summary of benefits**

From the above results it can be seen that the present invention by improving drainage allows the speed of the paper section to improve, increasing the output of paper from the recycled paper stock.

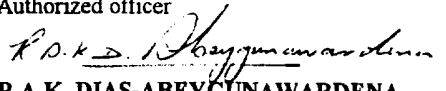
- 5 In addition the following ancillary benefits can also be recognised
- Avoids degradation of cellulose
  - Overdosing does not degrade paper
  - Iodine can be used to monitor the reaction
  - The alpha-Amylase is inactivated in the drying section
- 10 • alpha-Amylase is a food grade enzyme and is safe for use in making food packaging
- alpha-Amylase is commonly available

The claims defining the invention are as follows:

- 1) In a method of forming paper from recycled paper stock the improvement comprising the addition of a drainage improving amount of a starch hydrolysing enzyme, preferably amylase to the pulp suspension prior to the forming section of the paper making plant.
- 2) The improvement as claimed in claim 1 wherein the enzyme is an amylase active within the temperature range of 40 to 90°C and the pH range 6 to 9.
- 3) The improvement of claim 1 wherein the enzyme is added to the pulp suspension at least 15 minutes prior to entering the paper forming section.
- 4) The improvement of claim 1 wherein the enzyme is added in an amount of at least 0.05% by weight of the oven dried paper forming the pulp.
- 5) The improvement of claim 3 in which at least one cellulase is also added to the pulp with the starch hydrolysing enzyme.
- 6) A paper pulp drainage aid consisting of a major portion of a starch hydrolysing enzyme, preferably an amylase, active in the pH range 6 to 9 and the temperature range of 40 to 90 °C.
- 7) A drainage aid as claimed in claim 3 which also contains a cellulase active in the pH range 6 to 9.

# INTERNATIONAL SEARCH REPORT

International Application No.  
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<b>A. CLASSIFICATION OF SUBJECT MATTER</b>				
Int Cl <sup>6</sup> D21H 17/22, 21/10				
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Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.		
P,X	WO 97/10385 <u>A</u> (ROERMOND PAPIER) 20 March 1997 page 10, lines 15-18, page 11 lines 18-21	1		
P,X	WO 97/10386 <u>A</u> (ROERMOND PAPIER) 20 March 1997 see entire document	1		
P,X	WO 97/04168 <u>A</u> (SVERIGES STARKELS EPRODUCENTER) 6 February 1997 see entire document	1, 2, 6		
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Date of the actual completion of the international search 25 June 1997		Date of mailing of the international search report 03 JUL 1997		
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**PCT/AU 97/00182**

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Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 334739 <u>A</u> (JEAN-LUC FUENTES et al)	
A	EP 319846 <u>A</u> (SALKINOJA-SALONEN) 14 June 1989	

**INTERNATIONAL SEARCH REPORT**  
Information on patent family members

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Patent Document Cited in Search Report				Patent Family Member			
US	4923565	DE	3782602	EP	262040		
WO	9710385	AU	70995/96				
WO	9710386	AU	70994/96				
WO	9704168	AU	64734/96				
EP	334739	CA	1333317	FR	2629108	US	5110412
EP	319846	AU	618299	CA	1301688	JP	2006698
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