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NOTICE OF ENTITLEMENT

I, John Gordon Hinde, of Spruson & Ferguson, St Martins Tower, 31 Market Street, Sydney, New South Wales 2000, Australia, being the patent attorney for the Applicant(s)/Nominated Person(s) in respect of Application No 32504/93 state the following:-

The Applicant(s)/Nominated Person(s) has/have entitlement from the actual inventor(s) as follows:-

The Applicant(s)/Nominated Person(s) is/are the assignee(s) of the actual inventor(s).

The Applicant(s)/Nominated Person(s) is/are the applicant(s) of the application(s) listed in the Declaration under Article 8 of the PCT. The basic application(s) listed on the Declaration under Article 8 of the PCT is/are the first application(s) made in a Convention country in respect of the invention.

DATED this Seventh day of July 1994.

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- (57) Claim

1. A method for the control of pitch in a pulp, characterised in that the method comprises sequentially adding to and mixing with the pulp while in a thick stock stage (1) 1-10kg/tonne of pulp dry solids of a low molecular weight, high cationic charge, polymeric, water-soluble species and (2) 0.5 to less than 10kg/tonne of pulp dry solids of montmorillonite clay particles, which clay particles do not have a water-soluble cationic polymer adsorbed thereon.

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(21) International Application Number: PCT/US92/10835 (22) International Filing Date: 15 December 1992 (15.12.92) (30) Priority data: 9127173.4 21 December 1991 (21.12.91) GB (71) Applicant: VININGS INDUSTRIES INC. [US/US]; 3950 Cumberland Parkway, Atlanta, GA 30339-4510 (US). (72) Inventor: DERRICK, Arthur, Percy ; 3/4 Ingalara Avenue, Cronulla, NSW 2230 (AU). (74) Agent: EISELE, Joseph, T.; 711 Third Avenue (20th Floor), New York, NY 10017 (US).		(81) Designated States: AU, BR, CA, FI, JP, NO, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published <i>With international search report.</i> 657389
(54) Title: METHOD FOR CONTROLLING PITCH (57) Abstract A method for the control of pitch in a pulp is characterised in that the method comprises sequentially adding to and mixing with the pulp (1) 1-10 Kg/tonne of pulp of a low molecular weight, high cationic charge, polymeric, water-soluble species, such as those derived from inorganic aluminium salts or quaternary polyamines, and (2) 0.5 to less than 10 Kg/tonne of pulp of montmorillonite clay particles, such as bentonite, which clay particles may be chemically modified to improve adsorption capacity or otherwise treated to increase the anionic charge density of the dispersion but which do not have a water-soluble cationic polymer associated therewith.		

Method for controlling pitch

This invention concerns a method of controlling pitch in pulps for use in making paper. More particularly, this invention concerns a method of controlling the buildup of pitch on equipment used for pulping and the improved dispersion of pitch in pulps.

Pitch, carried over from wood or bark pulped to make papermaking furnishes, is formed of a complex mixture of oleophilic, water insoluble, low molecular weight, essentially non-polar resins comprising fatty and resin acids with ionisable hydrophilic groups, fatty and resin acid esters, sterols, di and tri-glycerides, terpenes and waxes and various alcohols, hydrocarbons and neutral compounds associated with these resins. The resins tend to agglomerate to form sticky films or pitch balls, which in turn can give rise to spotting in the final paper product, wire spots, localised sticky spots on rolls, holes in the paper sheet, poor paper formation, felt plugging or sticking on dryer and calender rolls.

Effective pitch control, particularly in paper mills which produce paper from groundwood, thermo-mechanical and semi-chemical pulps, has traditionally been difficult. The important parameters for pitch control are elegantly summarised in US-A-4964955 as follows:

"To control pitch effectively it is not necessary to remove all of the pitch from the pulp. Different types of pulp mills have different tolerance levels for pitch particles which must be exceeded before pitch caused problems occur. What

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is necessary, however, is that the pitch be dispersed as tiny non-harmful aggregated or agglomerated particles and retained in this fine particulate form rather than as larger harmful globs in the paper sheet. Simply flocculating pitch will not give good pitch control; the pitch must be made to attach to either the cellulosic fibres or to any particulate matter used as filler in the paper sheet, or preferably to both. Pitch retained in large flocs causes breaks and undesirable dark specks in the finished paper sheet, while pitch not retained but in small flocs tends to accumulate in the papermaking facility's white water system".

Efforts to control pitch deposit problems by dispersing or precipitating the pitch have traditionally involved the use of alum salts; ultrafine crystalline talc; anionic, cationic and non-ionic dispersants, polymeric retention aids and mixtures of these materials. More recently, water soluble zirconium salt complexes and organotitanium chemicals have been suggested as pitch control agents.

Papermakers alum ($\text{Al}_2(\text{SO}_4)_3 \cdot 14\text{H}_2\text{O}$), liquid sodium aluminate (20-25.5% Al_2O_3) and polyaluminium chloride are the primary alumina sources used for pitch control. Above pH 3 hydrolysis of the salts occur and a variety of hydroxy-aluminium complexes are formed depending upon the hydroxide ion concentration. Hydroxyl bridging occurs forming dimers and higher order polymer species (ligands). The formation of these desirable short chain relatively low molecular weight polymer species is primarily influenced by the system pH, temperature (the higher the better), the

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concentration of aluminium ions and the cationic demand of the stock. In pitch control, the optimum pH is preferably around 4.5 although in practice an operating range of 4.0-5.5 is fairly typical. It is believed that pitch control with aluminium salts is a combination of a flocculation mechanism, where the pitch particles are entrapped by the polymeric aluminosilicate structure, and the formation of a cationic complex with the resin and fatty acid components of the pitch, which in turn is then adsorbed onto the pulp fibres. A sodium aluminate/alum combination is generally preferred because there is no need for caustic soda to be used to control pH, it minimises sulphate ion build up in closed white water systems and it limits iron contamination thus making it easier to meet brightness specifications. Typical consumption figures are: alum 5-25 Kg/tonne and 45% sodium aluminate 5-10 Kg/tonne.

Ultrafine talc with a surface area of from 10 to 15m²/gm has been used for pitch control in pulp paper mills and in particular in those mills that are processing Kraft or sulphite pulps, i.e. where the pitch is present in a partially saponified state. The dispersed pitch particles are adsorbed on to the hydrophobic talc platelets and then retained in the paper. Typically, a use rate of 10-40 Kg/tonne is required.

A wide range of surfactants and short chain polymeric dispersants have been used to maintain pitch in a dispersed state within the paper making circuit. These dispersants function by charge and/or steric hindrance mechanisms. Anionic dispersants, such as lignin sulphonates,

polynaphthalene sulphonates and polyacrylates, increase the electronegative charge on the pitch particles, i.e. they peptize the pitch particles and inhibit agglomeration. Nonionic surfactants result in steric repulsion of the pitch and depending on the type and concentration can soften and emulsify pitch deposits already formed. Since dispersants do not attach the pitch to the pulp fibres, they have limited use in controlling severe pitch problems such as is generated in TMP mills. An excess of dispersant can result in a substantial build up in the concentration of pitch in closed white water systems. Usage rates for pitch dispersants are typically in the range of 5-100 ppm on the stock.

The use of polyquaternary ammonium polymers for pitch control has been promoted in recent years. The control mechanism is described by T Hassler, Tappi Journal, June 1988, p 195 onwards. The low molecular weight cationic polymers which are commercially available are believed to be linear and/or partially crosslinked polydiallyldimethyl ammonium chloride (polyDADMAC) and copolymers of epichlorohydrin and short chain aliphatic secondary amines. A typical use rate would be from 1.25- 2.3 Kg/tonne.

The use of current retention and drainage aid technologies e.g. "Lapotaín" (Laporte Industries Limited), "Composil" (Eka-Nobel) and -Positek- (Nalco Chemical Co) etc, give significant improvements in pitch control on the machines, but reliance on this type of technology however is only possible when the mill can justify a retention and drainage aid programme.

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More recently, a method of reducing pitch in pulping and papermaking operations by a process which comprises (1) adding to a pitch-containing furnish a particulate composite substance comprising (a) a water soluble cationic polymer adsorbed onto (b) an essentially water insoluble particulate substrate carrying an anionic charge, and (2) adsorbing pitch onto said composite to form discrete, finely dispersed pitch-containing aggregates in the furnish, is disclosed in JS-A-4964955. On Column 8, line 67, to Column 9, line 15, of the US patent, it is disclosed that any essentially water-insoluble particulate organic or inorganic substance may be employed as the substrate, including phyllosilicate minerals, kaolin, talc, mica, montmorillonite, chlorite and pseudolayer silicates, though Kaolin is particularly preferred. Indeed, this preference is clearly demonstrated in the examples in that Kaolin is the only substrate exemplified. It is demonstrated in Examples IV to VI, IX and X that improved pitch control may be achieved by pre-treating a groundwood pulp with alum prior to treatment with a cationic kaolin. In Example XII, on Column 19, lines 12 to 16, it is disclosed that using 60-80 lbs/ton of cationic kaolin slurry with 35 lbs/ton of alum will essentially eliminate all pitch. From a practical point of view, however, the amount of cationic kaolin required in the process tends to make a papermaking process uneconomic. A similar method is disclosed in EP-A-0349311.

Japanese patent application No 62-245908 discloses a method for reinforcing paper strength by adding bentonite to a pulp after a cationic paper strength reinforcing agent has been mixed in the pulp. It is noted in the patent application that the process, apart from improving the paper strength, also solves pitch troubles. It is disclosed in Example 1 and Example 2 that a minimum of 10 Kg/tonne (1% relative to pulp) of bentonite was added to a pulp which had been previously treated with reinforcing agent and alum. Whilst this method appears suitable for improving paper strength, retention and pitch problems, as a method for controlling pitch alone, this process would tend to be uneconomic.

It is an object of the present invention to provide a process for controlling pitch in pulps, which process is technically at least as efficient at eliminating or reducing pitch in pulps as the process described in US-A-4964955 but which is economically more acceptable.

In accordance with the present invention, there is provided a method for the control of pitch in a pulp and is characterised in that the method comprises sequentially adding to and mixing with the pulp (1) 1-10 Kg/tonne of pulp dry solids of a low molecular weight, high cationic charge, polymeric, water-soluble species and (2) 0.5- < 1 0 Kg/tonne of pulp dry solids of montmorillonite clay particles, which clay particles do not have a water-soluble cationic polymer adsorbed thereon.

The low molecular weight, high cationic charge, polymeric, watersoluble species may be organic but is preferably inorganic and is

preferably present in an amount of 2 to 6 Kg/Tonne of pulp. The polymeric species has a low molecular weight, preferably below 1million and more preferably below 0.5 million.

If an organic species is used, it is preferably a polymer of the type disclosed on column 9, line 50, to column 10, line 34, of US-A-4964955. The polymer preferably has a cationic charge density of at least 5 m.eq/gm preferably a charge density of from 6 to 7 m.eq./g or more. Preferably, the polymer is not a Mannich polymer.

If an inorganic species is used, it is preferably papermakers alum, sodium aluminate, poly aluminium chloride, poly aluminium silicate or an other inorganic water soluble salt of aluminium. Preferably sodium aluminate is used in the present invention.

Montmorillonite clays are distinguished by their ability to be water swellable. Synthetic water swellable clays, such as Laponite (Laporte Industries Limited) , are understood to have similar properties to montmorillonite clays and are therefore included within the scope of the present invention. Bentonite is the preferred montmorillonite. Preferably, the amount of montmorillonite added to the pulp is from about 1 Kg/Tonne to less than 10 Kg/Tonne, more preferably from about 2 Kg/Tonne to about 5 Kg/Tonne.

Whilst for cost purposes it is preferred to use bentonite which has not been chemically modified in any way, in some instances it may be technically preferable to employ a bentonite which has been chemically modified, e.g. by alkali treatment to convert calcium bentonite

substantially to alkali (e.g. sodium, potassium or ammonium) bentonite, or a bentonite in intimate association with a water soluble, highly charged anionic polymer, e.g. Lapotain available from Laporte Industries Limited.

Preferably, the pulp contains less than 0.15 % polyacrylamide which has been submitted to a Mannich reaction and preferably the pulp contains no such polymer.

The order of addition of the components to the pulp is not important in achieving the advantages of the present invention; component (1) may be added after component (2), though it is preferred to add component (1) before component (2). It is important, however, that in any event, the first component added to the pulp must be thoroughly mixed into the pulp before the second component is added. Mixing may be achieved by subjecting the pulp to high shear conditions, such as by passing the pulp through a centriscreeen or through mixing pumps or fan pumps, though simple turbulence mixing obtainable by passing the pulp along a length of pipeline may be just as effective.

The process of the present invention has a number of advantages over the process described in US-A-4964955, including the surprising fact that the process of the present invention has improved pitch control over the process of the prior art. Other advantages, including the obvious cost advantage, will be immediately apparent to a person skilled in the art.

The invention will now be further described, without limitation, by reference to the examples set forth below.

Examples

Samples of a virginTMP pulp, obtained from a major pulp/paper manufacturer located in the south east of U.S.A., were used in the following examples. The pulp was selected on the basis that southern pine pulps from this part of U.S.A. may contain significant concentrations of troublesome pitch. It should be appreciated that comparison of one series of results obtained from one pulp sample cannot be compared with the series of results obtained from a different pulp sample)

In each evaluation, the thick stock samples, taken from either the refiner or the latency stock chest, were diluted and aged at 180°F with tap water processed through a laboratory scale disintegrator and adjusted to 1 % consistency.

The samples were subjected to the colloidal dispersed solids test (CDS) the procedure of which involves

- 1 . Pulp stock samples (after treatment) are sieved through a 60 mesh screen, the resulting low solids solution is placed in 50 ml or 100 ml centrifuge tubes and spun for 30 mins at 4000rpm.

2. The supernatent is carefully removed by syringe and transferred into a small bottle (need approx 60mls of supernatent)

3. A suitable aliquot is then transferred to tared beaker. It is recommended that a minimum of 25gms of liquid should be filtered. Care is necessary to prevent any extraneous material contaminating the beaker.

4. Record the weight of the sample taken using a 2 decimal place balance.

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5. The liquid sample is then filtered through a previously dried, dessicated and weighed 0.45 micron millipore filter using maximum vacuum. The filter is previously "wet out" with distilled water.

6. After the fluid has completely drained, remove the filter pad, dry at 50°C for 15 mins, dessicate and reweigh using 4 decimal place balance.

7. Record this weight.

The weight of the filter and solids minus the weight of the filter is equal to the weight of colloidal dispersed solids (CDS)

$$\frac{\text{Weight of colloidal dispersed solids}}{\text{Weight of supernatant liquid taken}} \times 10^6 = \text{ppm CDS}$$

This test procedure has good correlation with the prediction of pitch problems in operating newsprint and linerboard mills. CDS measurements also correlate well with microscopic pitch counting procedures as described in Pitch Particle Concentration: an Important Parameter in Pitch Problems by L.H. Allen, Pulp & Paper Research Institute of Canada, Pointe Claire, Que, pl-9.

PITCH ADSORPTION RESULTS

<u>Optimisation Series I</u>	Selected Components
Stock consistency	0.62%
pH	4.8
Blank C.D.S	98 ppm

Chemical	Dose Rate kg/tonne	Reduction In CDS (Pitch)
Alum (17% Al_2O_3) System adjusted to pH 5.3	5	25
"	10	27
"	25	88
'Proset' 1820	1	16
"	2	35
"	4	57
Polymin SK	1	-
"	2	31
"	4	53

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Chemical	Dose Rate kg/tonne	% Reduction In CDS (Pitch)
PolyDADMAC	1	20
"	2	47
"	4	61
Southern Clay SCPX 572	3	43
"	5	63
"	10	92
"	20	96
Fulgel 300/2	3	61
"	5	78
"	10	80
"	20	84

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OPTIMISATION SERIES II

Stock-Consistency	0.65%
pH	4.9
Blank CDS	121 ppm

Activity of Simple Components

A. Cationic Components

Chemical	Dose Rate kg/tonne	% Reduction In CDS (Pitch)
VSA 45 - pH of System adjusted to 5.3	3	34
"	5	42
Alum (17% Al_2O_3) pH adjustment to 5.3	3	22
"	5	27
PAC (17% Al_2O_3) pH adjustment to 5.3	3	22
"	5	30
'Proset' 2830	0.5	21
"	1.0	51

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B. Bentonite substrate Components (Preferred)

Chemical	Dose Rate kg/tonne	% Reduction In CDS (Pitch)
"GADORGEL" X	2	13
"	3	16
"	5	23
"FULGEL" 300	2	16
"	3	29
"	5	43
SCPX590	2	20
"	3	26
"	5	36
SCPX592	2	14
"	3	22
"	5	27

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Chemical	Dose Rate kg/tonne	% Reduction In CDS (Pitch)
SCPX590/2	3	29
"	5	37
SCPX592/2	3	30
"	5	42

Note: SCPX-/2 Substrates are the basic bentonite modified with 2% ^w/W Accumer 9000 (available from Rohm and Haas Co) in solids

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Optimisation Series III

Stock-Consistency 0.66%

pH 4.8

Blank CDS 133-134 ppm

Chemical Programme	Dose Rate of Components kg/tonne	% Reduction In CDS (Pitch)
VSA 45/'Fulgel' 300 pH to 5.3 After VSA addition	3/2	20
"		
VSA 45/'Fulgel' 300 pH to 5.3 After VSA addition	5/2	90
"	3/3	23
"	5/3	87
VSA 45 Alone pH to 5.3	5	74
VSA 45/Fulgel 300/2 pH to 5.3 After VSA addition	3/2	12
"	5/2	45
"	3/3	17
"	5/3	73
Fulgel 300/2/VSA 45 ie Reverse Addition	2/3	27

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Chemical Programme	Dose Rate of Components kg/tonne	% Reduction In CDS (Pitch)
'Proset' 2830/'Fulgel' 300	0.5/2	24
"	1/2	43
"	0.5/3	31
"	1/3	46
"	1/5	49
'Proset' 2830 Alone	0.5	20
"	1.0	33
'Proset' 2930/'Fulgel' 300/2	0.5/2	22
"	1/2	33
"	0.5/3	20
"	1/3	32

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Chemical Programme	Dose Rate of Components kg/tonne	% Reduction In CDS (Pitch)
Alum Alone (17%Al ₂ O ₃) pH adjusted to 5.3	5	22
Alum/'Fulgel' 300	5/3	33
"	5/5	42
Alum/'Fulgel' 300/2	5/3	28
Alum/'Fulgel' 300/2	5/5	16
Alum @ Equivalent Al ₂ O ₃ to VSA 45 + SCPX 572	7.5/5	55

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Chemical	Dose Rate Components kg/tonne	% Reduction In CDS (Pitch)
VSA 45 Alone pH to 5.3	5	72
VSA 45/SCPX 572	5/3	83
"	5/5	87
VSA 45/SCPX 572/2	5/3	67
VSA 45/scpx 572/2	5/5	73

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Chemical	Dose Rate Components kg/tonne	% Reduction In CDS (Pitch)
'Proset' 2830/SCPX 572	1/3	53
"	1/5	57
'Proset' 2830/SCPX 572/2	1/3	35
"	1/5	38

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Stock Sample Consistency - 0.9%
pH - 4.8
Blank CDS
Concentration - 158 ppm

Chemical	Dose Rate of Components kg/tonne	% Reduction In CDS (Pitch)
VSA 45 (alone)	5	43
VSA 45/SCPX 590	5/5	71
'Proset' 2830 (alone)	1	29
'Proset' 2830 (alone)	1.5	75
'Proset' 2830/SCPX 590	1/5	77
'Proset' 2830/SCPX 590	1.5/5	94

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PITCH ADSORPTION ACTIVITY:

PAC / PAC & ACCOFLOC HCX
@ 5Kg/t

	% Reduction in CDS ('PITCH')
PAC @ 5Kg/l	44
PAC @ 10Kg/l	44
PAC @ 25Kg/l	97
PAC @ 5Kg/l & HCX	60
PAC @ 10Kg/l & HCX	72
PAC @ 25Kg/l & HCX	98

ACCOFLOC HCX / HCX & PROSET 2830
@ 2Kg/l

	Reduction in CDS ('PITCH')
HCX @ 3Kg/t	20
HCX @ 5Kg/t	45
HCX @ 10Kg/T	54
HCX @ 20Kg/t	73
HCX @ 3Kg/t & P2830	88
HCX @ 5Kg/t & P2830	91
HCX @ 10Kg/t & P2830	96
HCX @ 20Kg/t & P2830	95

Stock Consistency 0.95

pH 4.8

Blank CDS 273 ppm

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PITCH ADSORPTION ACTIVITY:

PROSET 2830/ P2830 & IICX/ P2830
& GADORGEL X (HCX & CA ORGEL @ 5Kg/l)

	% REDUCTION IN CDS ('PITCH')
PROSET 2830 @ 1Kg/l	58
PROSET 2830 @ 2Kg/l	82
PROSET 2830 @ 4Kg/l	96
P2830 @ 1Kg/l & HCX	77
P2830 @ 2Kg/l & HCX	87
P2830 @ 4Kg/l & HCX	97
P2830 @ 1Kg/l\GADORGEL	60
P2830 @ 2Kg/l\GADORGEL	88
P2830 @ 4Kg/l\GADORGEL	95

CLAY SUBSTRATES @ 10Kg/t

	% REDUCTION IN CDS ('PITCH')
FULBOND X	53
GADORGEL X	60
AX SAPONITE	59
ACCOFLOC HCX	59
FULGEL 300	59

PITCH ADSORPTION ACTIVITY:

PROSET 2830 @ 2Kg/t
& CLAY SUBSTRATES @ 5Kg/t

	% Reduction of CDS ('PITCH')
P2830 only	82
P2830 & HCX	87
P2830 & GADORGEL X	88
P2830 & GADORGEL X/2	91
P2830 & FULGEL 300	100
P2830 & FULGEL 300/2	97

PROSET 2830 @ 1Kg/t
& CLAY SUBSTRATES @ 5Kg/t

	% Reduction of CDS ('PITCH')
P2830 only	58
P2830 & HCX	77
P2830 & GADORGEL X	60
P2830 & GADORGEL X/2	94
P2830 & FULGEL 300	94
P2830 & FULGEL 300/2	81

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The performance of the process of the present invention was compared with the process described in US-A4964955. A 2gm per litre dispersion of SCPX 572 bentonite was modified by reacting with a dilute solution (2%) of a polyDADMAC product. The Zeta potential of the composite material was measured at 40 mv on a Matec Applied Sciences Electrokinetic Sonic Analysis System. The ratio of cationic polymer to bentonite was used to produce a 7g/l total actives dispersion for testing. The results were as follows.

Stock Sample

Consistency	-	0.9% (100% TMP)
pH	-	4.8
Blank CDS Concentration	-	158 ppm

Chemical Programme	Dose Rate of Components kg/tonne	% Reduction in CDS (Pitch)
SCPX 572 Bentonite	10	67
Cation Modified SCPX 572 Bentonite	10	68
Cation Modified SCPX572 Bentonite	14	82
Poly DADMAC @ 4kg/tonne followed by SCOX572 Bentonite @ 10kg. tonne	4/10	81

Note All concentrations are expressed as 100% Active Materials

Alum (17% Al ₂ O ₃) System adjusted to pH 5.3	5	37
Alum followed by SCPX572 Bentonite	5/10	85
Alum followed by Cation modified SCPX572 Bentonite	5/10	90

As can be seen from the results, the cation modified bentonite demonstrates substantially no advantage over the normal anionic charge bentonite at equivalent use rates. This result is technically most surprising and clearly demonstrates the commercial advantage of the present invention.

The following trade marks, which may be registered trade marks, have been used in the Examples:

PROSET 1820 is an epichlorohydrindimethylamine polymer available from Vinings Industries Inc.

POLYMIN SK is a polyamidoamine available from BASF

SCPX 572 is a high swelling bentonite clay available from Southern Clay Products

Fulgel 300/2 is an anionically modified bentonite available from Laporte Industries Limited

VSA 45 Is a 45% sodium aluminate solution available from Vinings Industries Inc

PAC is polyaluminium chloride

PROSET 2830 is an epichlorohydrindimethylamine crosslinked with a short chain aliphatic amine available from Vinings Industries Inc

GADORGEL is a high swelling bentonite clay available from Laporte Industries Ltd

FULGEL 300 is a high swelling bentonite clay available from Laporte Industries Ltd

SCPX 590 is a high swelling bentonite clay available from Southern Clay Products

SCPX 592 Is a high swelling bentonite clay modified with a low molecular weight anionic polymer available from Southern Clay Products

SCPX 590/2 is a bentonite clay modified with anionic polymer available from Southern Clay Products

SCPX 592/2 is a bentonite clay modified with an anionic polymer available from Southern Clay Products

ACCOFLOC HCX is an hectorite clay available from American Colloids Inc.

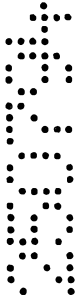
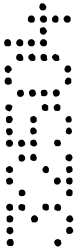
The claims defining the invention are as follows:-

1. A method for the control of pitch in a pulp, characterised in that the method comprises sequentially adding to and mixing with the pulp while in a thick stock stage (1) 1-10kg/tonne of pulp dry solids of a low molecular weight, high cationic charge,
5 polymeric, water-soluble species and (2) 0.5 to less than 10kg/tonne of pulp dry solids of montmorillonite clay particles, which clay particles do not have a water-soluble cationic polymer adsorbed thereon.
2. A method as claimed in claim 1, wherein the montmorillonite clay is bentonite.
- 10 3. A method as claimed in claim 1 or 2, wherein the amount of montmorillonite added to the pulp is from about 1kg/tonne to about 5kg/tonne.
4. A method as claimed in any one of the preceding claims, wherein the montmorillonite clay is bentonite which has been chemically modified, to convert calcium bentonite substantially to alkali bentonite, or a bentonite in intimate association with a
15 water soluble, highly charged anionic polymer.
5. A method as claimed in any one of the preceding claims, wherein the amount of component (1) added to the pulp is from about 2kg/tonne to about 6kg/tonne.
6. A method as claimed in claim 5, wherein the amount of component (1) added to the pulp is from about 2kg/tonne to 5kg/tonne.
- 20 7. A method as claimed in any one of the preceding claims wherein, component (1) is an organic polymer having a charge density of at least 5m.eq/g.
8. A method as claimed in any one of claims 1 to 6, wherein component (1) is an inorganic polymer selected from one or more of papermakers alum, sodium aluminate, poly aluminium chloride, poly aluminium silicate or another inorganic water soluble salt
25 of aluminium.
9. A method as claimed in any one of the preceding claims, wherein component (1) is added and mixed with the pulp before component (2) is added and mixed to the pulp.
10. A method of claim 4 wherein the montmorillonite clay is bentonite which has
30 been chemically modified by alkali treatment to convert calcium bentonite substantially to alkali bentonite said alkali bentonite being selected from the group consisting of sodium bentonite, potassium bentonite and ammonium bentonite.
11. A method as claimed in claim 1 and substantially as herein described with reference to any one of the Examples.



Dated 22 July, 1994
Vinings Industries Inc.

Patent Attorneys for the Applicant/Nominated Person
SPRUSON & FERGUSON



INTERNATIONAL SEARCH REPORT

PCT/US92/10835

A. CLASSIFICATION OF SUBJECT MATTER

IPC(5) : D21H 21/02

US CL : 162/164.6, 168.2, 181.3, 181.8, 162/199, DIG. 4

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)

U.S. : NONE

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 practicable, search terms used)

Pitch, Pulp, Paper, Montmorillonite, Bentonite, Alkali Bentonite, Polymer, Sodium Aluminate, Organic Polymer

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US, A, 4,913,755 (langley et al), 03 April 1990 see entire document.	1-3
Y	US, A, 4,964,955 (Lamar et al.) 23 October 1990 see entire document.	1



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:	*T	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
A document defining the general state of the art which is not considered to be part of particular relevance	*X*	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
E earlier document published on or after the international filing date	*Y*	document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
L document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	*Z*	document member of the same patent family
O document referring to an oral disclosure, use, exhibition or other means		
P document published prior to the international filing date but later than the priority date claimed		

Date of the actual completion of the international search

01 FEBRUARY 1993

Date of mailing of the international search report

23 FEB 1993

Name and mailing address of the ISA/US
Commissioner of Patents and Trademarks
Box PCT
Washington, D.C. 20231

Facsimile No. NOT APPLICABLE

Authorized officer

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/US92/10835

Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This international report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
3. ☒ Claims Nos.: 4-8
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.
2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest.
☐ No protest accompanied the payment of additional search fees.