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(71) Applicant: SEIKO KAGAKU KOGYO CO., LTD.
1-1, Tateishi 1-chome
Akashi-shi Hyogo 673(JP)

(71) Applicant: HOKUETSU PAPER MILLS, LTD.
5-1, Nishizaoh 3-chome
Nagaoka-shi Niigata 940(JP)

(71) Applicant: NIPPON OIL CO. LTD.
3-12, Nishi Shimbashi 1-chome
Minato-ku Tokyo(JP)

(72) Inventor: Yoshioka, Shigehiko
No. 93-1, Morita Okubo-Cho
Akashi-Shi Hyogo(JP)

(72) Inventor: Yamada, Hideto
No. 5-16-404, Oji 1-Chome
Akashi-Shi Hyogo(JP)

(72) Inventor: Yata, Akihiro
No. 1-8, Higashi Aramachi 2-Chome
Nagaoka-Shi Niigata(JP)

(72) Inventor: Sato, Hisatake
No. 22-12, Nakatehara 1-Chome Kohoku-Ku
Yokohama-Shi Kanagawa(JP)

(74) Representative: Heath, Derek James et al,
Bromhead & Co. 30 Cursitor Street Chancery Lane
London EC4A 1LT(GB)

(54) **Aqueous dispersed solution of substituted succinic anhydride.**

(57) An aqueous dispersion of a substituted succinic anhydride comprising an aqueous soluble copolymer A_1 or an aqueous soluble copolymer A_2 or a mixture of these two copolymers, the copolymer A_1 having 50 to 98.5 per cent by mol of (meth)acrylamide (a), 1 to 49.5 per cent by mol of N,N-dialkylamidealkyl(meth)acrylamide (b), and 0.5 to 20 per cent by mol of (meth)acrylic acid (c) as monomer ingredients, the aqueous soluble copolymer A_2 having 70 per cent by weight or more of a mixture monomer ingredient having 50 to 98.5 per cent by mol of (meth)acrylamide (a), 1 to 49.5 per cent by mol of N,N-dialkylaminoalkyl(meth)acrylamide (b) and 0.5 to 20 per cent by mol of (meth)acrylic acid (c) and other copolymer monomer (d) as monomer ingredients. The dispersion is used as a sizing agent and shows a dilute stability in paper-making steps as well as a sufficient sizing effect in a low content addition range, even in acidic paper making without using a calcium carbonate filler, and in neutral paper making using alum.

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The present invention relates to an aqueous dispersed solution of a substituted succinic anhydride. More particularly, the present invention relates to such an aqueous dispersion of a substituted succinic anhydride used as a sizing agent having excellent sizing effect and preferable operability in paper making steps and exhibiting excellent operations even in a dilute stability.

There is known a sizing paper employing an aqueous dispersed solution of a substituted succinic anhydride, and various types of aqueous dispersions have been heretofore proposed.

For example, an aqueous dispersion prepared by mixing an aqueous solution of cationic starch and a substituted succinic anhydride by a strong mechanical agitation in a homogeneous state (disclosed in Japanese Patent Publication No. 2305/1964 official gazette), an aqueous dispersion prepared by dispersing a substituted succinic anhydride in water or an aqueous solution of specific aqueous soluble polymer substance with the aid of a selected surfactant (disclosed in Japanese Patent Publication No. 36044/1978 official gazette, Japanese Patent Laid-open No. 45731/1983 official gazette, Japanese Patent Laid-open No. 47498/1984 official gazette), and further an aqueous dispersion prepared by dispersing a substituted alkyl-substituted or alkenyl-substituted succinic anhydride or a mixture (a) of the succinic anhydrides which contain 70 to 99.9 percent by weight and a mixture (b) of polyoxyethylene alkyl-ether phosphoric acid ester, polyoxyethylene alkylaryl ether phosphoric acid ester or the mixture of these phosphoric acid esters which

contain 0.1 to 30 percent by weight in water with the aid of an aqueous solution of aqueous soluble polymer compound (disclosed in Japanese Patent Laid-open No. 28598/1985).

However, in the abovementioned aqueous dispersion employing the former cationic starch of the aqueous dispersions of the substituted succinic anhydrides, a considerably large apparatus is not only required to disperse the substituted succinic anhydride in water, but it is difficult to sufficiently pulverize the aqueous dispersed solution thus obtained into fine dispersion particles, and the resultant dispersion has such a disadvantage that the dispersion cannot avoid the slight decrease in the sizing performance.

In the above-described aqueous dispersion employing the latter mixture of the aid, the prepared mixture loses its stability in preservation and cannot secure a sufficient dispersibility of the substituted succinic anhydride in water, and the resultant aqueous dispersion not only loses its sizing effect with the lapse of time after preparation but deteriorates in the dispersibility of the substituted succinic anhydride having an internal olefin structure adapted for a sizing agent in water. Incidentally, a process for producing an aqueous dispersed solution by the prepared mixture has drawbacks such that, if the prepared mixture is dispersed in the aqueous solution dissolved in advance in a cationic starch when the aqueous dispersed solution is prepared from the prepared mixture, an aqueous dispersed solution having preferable dilute stability can be produced, but since the aqueous solution itself of the cationic starch is unstable in an ageing manner, the cationic starch must be dissolved in

water immediately before using the aqueous dispersed solution as a sizing agent, and since the underwater dispersibility fo the substituted succinic anhydrides of the cationic starch is low, a strong (large-scale) emulsifying apparatus for preparing the aqueous dispersed solution is required.

Further, the aqueous dispersion utilizing the substituted-alkyl succinic anhydride or substituted-alkenyl succinic anhydride or a mixture (a) of the succinic anhydrides which contain 70 to 99.9 percent by weight and a mixture (b) of polyoxyethylene alkyl-ether phosphoric acid ester, polyoxyethylene alkylaryl ether phosphoric acid ester or the mixture of these phosphoric acid esters which contain 0.1 to 30 percent by weight in water with the aid of an aqueous solution of aqueous soluble polymer compound, and the resultant aqueous dispersion further provides preferable dispersibility in water for the substituted succinic anhydride having an internal olefin structure as a sizing agent as its merits, but when the aqueous dispersion obtained from this mixture is used as a sizing agent in an industrial scale, some problems are still retained. In other words, the aqueous dispersion of the substituted succinic anhydride obtained from the mixture proposed in Japanese Patent Laid-open No. 28598/1985 exhibits dilute stability of the aqueous dispersion in case that the cationic starch dissolved in water of continuous state is contained in the aqueous dispersion, but, in other case, causes insufficient dilute stability, and when this aqueous dispersion is employed as a sizing agent in the practical paper making process, the dispersion has such a drawback that it contaminates the wires, rolls and carpets of the paper making machine and cannot operate continuously for a

long period of time.

Therefore, the inventors of the present invention have discovered the fact that the aqueous dispersed solution of the substituted succinic anhydrides which contained specific aqueous soluble copolymer obtained with (meth)acrylic amide and N,N-dialkylaminoalkyl(meth)acrylamide as indispensable monomer ingredients could eliminate the various types of drawbacks with the aqueous dispersed solution described above, and has proposed the aqueous dispersed solution of the substituted succinic anhydrides which contained the specific aqueous copolymer, but have discovered other drawbacks in the operation that, when the aqueous dispersed solution of the substituted succinic anhydrides was utilized as a paper making sizing agent, the sizing effect in the low content addition range of the sizing agent was not always satisfactory, and, when applied to the acidic paper making step which did not use calcium carbonate filler and neutral paper making step of middle quality paper frequently produced by adding small amount of sulfuric acid band by using calcium carbonate filler, the separation of wet paper and rolls was deteriorated to cause the paper to be cut.

Accordingly, an object of the present invention is to provide an aqueous dispersed solution of a substituted succinic anhydride which can perform the operation of the aqueous dispersed solution of a substituted succinic anhydride which contained a specific aqueous soluble copolymer produced with the (meth)acrylamide and N,N-dialkylaminoalkyl(meth)acrylamide as indispensable monomer ingredients, i.e., the operation of excellent sizing effect

and preferable dilute stability of the aqueous dispersed solution in paper making steps as well as perform a sizing effect sufficiently satisfied in a low content sizing agent addition range and achieve the entirely similar excellent operability to the other cases even in acidic paper making step without using calcium carbonate filler, and neutral paper making step including the case using sulfuric acid band.

According to an aspect of the present invention, there is provided an aqueous dispersed solution of a substituted succinic anhydride which comprises an aqueous soluble copolymer (A_1) having 50 to 98.5 percent by mol of (meth)acrylamide (a), 1 to 49.5 percent by mol of N,N-dialkylaminoalkyl(meth)acrylamide (b), and 0.5 to 20 percent by mol of (meth)acrylic acid (c) as monomer in ingredients, an aqueous soluble copolymer (A_2) having 70 percent by weight or more of a mixture monomer ingredient having 50 to 98.5 percent by mol of (meth)acrylamide (a), 1 to 49.5 percent by mol of N,N-dialkylaminoalkyl(meth)acrylamide (b) and 0.5 to 20 percent by mol of (meth)acrylic acid (c) and other copolymer monomer (d) as monomer ingredients, or both of the aqueous soluble copolymer (A_1) and similarly the aqueous soluble copolymer (A_2). More particularly,
there is provided an aqueous dispersed solution of a substituted succinic anhydride which comprises an aqueous soluble copolymer (A_1) produced by copolymerizing 50 to 98.5 percent by mol of (meth)acrylamide (a), 1 to 49.5 percent by mol of N,N-dialkylaminoalkyl(meth)acrylamide (b), and 0.5 to 20 percent by mol of (meth)acrylic acid (c) as monomer in ingredients, an aqueous soluble copolymer (A_2) produced by copolymerizing 70 percent by weight or more of a mixture

monomer ingredient having 50 to 98.5 percent by mol of (meth)acrylamide (a), 1 to 49.5 percent by mol of N,N-dialkylaminoalkyl(meth)acrylamide (b) and 0.5 to 20 percent by mol of (meth)acrylic acid (c) and 30 percent by weight or less of other copolymer monomer (d) as monomer ingredients, or both of the aqueous soluble copolymer (A_1) and similarly the aqueous soluble copolymer (A_2), and a substituted succinic anhydride (B) as indispensable ingredients.

The substituted succinic anhydrides (B) in the aqueous dispersed solution of the substituted succinic anhydride according to the present invention can employ all of the known substituted succinic anhydrides used heretofore as a sizing agent, and more particularly comprise those substituted succinic anhydrides which contain hydrophobic hydrocarbon groups each having at least 8 and more preferably 12 to 36 carbon atoms such as substituted succinic anhydrides (having alkyl or alkenyl groups). Generally, the substituted succinic anhydrides comprise those feasibly produced by addition reaction of alpha-olefins, inner olefins or olefins of the mixture of them and maleic anhydride. Typically, when the substituted succinic anhydrides are, for example, octadecen-9, tetradecen-7, hexadecen-7, eicosene-11 or the mixture thereof; inner olefin selected from straight-chain internal inner olefin mixture having double bonds produced by the dehydrogenation reaction of straight-chain paraffin are distributed substantially uniformly at the respective positions except alpha-positions; and straight-chain inner olefin mixture having 70 percent or more by weight of total amount of inner olefins disposed at 2-, 3- and 4-positions of

double bonds produced by anisotropic reaction of straight-chain alpha-olefin in the presence of catalyst; and the mixture produced by addition reaction of the anhydrides and maleic anhydride, i.e., when the substituted succinic anhydrides are those of alkenyl succinic anhydride having succinyl group in the substituted group, the sizing effect performed by the application of the substituted succinic anhydride of the present invention to the sizing agent for making paper in the paper making process is further improved.

The monomer ingredients of the aqueous soluble copolymer (A_1) or (A_2) used together with the substituted succinic anhydrides in the aqueous dispersed solution of the substituted succinic anhydrides are acrylamide, methacrylamide or both of them, i.e., (meth)acrylamide (a), N,N-dialkylamino. alkylacrylamide, N,N-dialkylamino.alkylmethacrylamide (b), acrylic acid, methacrylic acid or both of them, i.e., N,N-dialkylamino.alkyl(meth)acrylamide (b), i.e., (meth)acrylic acid (c). Incidentally, the N,N-dialkylamino.alkyl(meth)acrylamide (b) is basic third class amino group containing monomer represented by the following general formula

$$CH_2=\overset{R}{\overset{|}{C}}-CONH-(CH_2)n-N\begin{matrix}R_1\\R_2\end{matrix}$$

where, in the formula, R is a hydrogen or methyl group, R_1 and R_2 represent a methyl group, ethyl group or proply group, and n represents an integer of 2 to 4.

The aqueous soluble copolymer (A_1) comprises a copolymer of 50 to 98.5 percent or more by mol of the (meth)acrylamide (a), 1 to 49.5 percent or more by mol of

N,N-dialkylamino.alkyl(meth)acrylamide (b), 0.5 to 20 percent by mol of (meth)acrylic acid (c) and the copolymer (A_1) can be readily produced by the ordinary polymerization of the monomer ingredients prepared at the abovementioned ratio of the compositions, but it is confirmed that the copolymer (A_1) of 75 to 94 percent by mol of the (meth)acrylamide (a), 5 to 20 percent by mol of N,N-dialkylaminoalkyl(meth)acrylamide (b), and 1 to 10 percent by mol of (meth)acrylic acid (c) obtains more preferable results by the sizing effect in the low sizing agent addition range of the quality of the case using the aqueous dispersed solution of the substituted succinic anhydride of the present invention as a sizing agent or preferable operability in the acidic paper making step or neutral paper making step. The copolymerization is preferably aqueous soluble polymerization, and the molecular weight of the aqueous soluble copolymer (A_1) is approx. 5,000 or more and more preferably 20,000 or more to be effectively used.

The aqueous soluble copolymer (A_2) is a copolymer of monomer ingredients of 70 percent or more by weight of mixture monomer ingredients having 50 to 98.5 percent by mol of the (meth)acrylamide (a), 1 to 49.5 percent by mol of N,N-dialkylaminoalkyl(meth)acrylamide (b), and 0.5 to 20 percent by mol of (meth)acrylic acid (c), 30 percent or less by weight of other copolymer monomer as monomer ingredients, and can be readily produced similarly to the case o the aqueous soluble copolymer (A_1). This aqueous soluble copolymer (A_2) becomes preferable in the quality of the sizing agent when utilizing the aqueous dispersed solution of the substituted succinic anhydride of the present invention as sizing agent of the case that, similarly to the aqueous

soluble copolymer (A_1), regarding the (meth)acrylamide (a), N,N-dialkylaminoalkyl(meth)alkylamide (b) and (meth)acrylic acid (c), 75 to 94 percent by mol of (meth)acrylamde (a), 5 to 20 percent by mol of N,N-dialkylaminoalkyl(meth)acrylamide (b) and 1 to 10 percent by mol of (meth)acrylic acid (c) are contained. Further, the case of the aqueous soluble copolymer (A_2), containing 5,000 or more and more preferably having 20,000 or more of molecular weight is effectively used. Incidentally, as other copolymer monomer ingredients of 30 percent or less by weight of the copolymer (A) in the aqueous soluble copolymer (A_2), there are, for example, aromatic vinyl monomer such as styrene, alkylstyrene; alpha-, beta-unsaturated carboxylic acid esters such as alkylester, hydroxyalkylester, aminoalkylester of alpha-, beta-unsaturated carboxylic acid, vinyl acetate or other fatty acid vinylesters; other vinyl compounds such as vinylether or vinylamide; aryl compounds such as arylalcohol or arylether and arylamine or diarylamine and their quaternay ammonium salt; polymerizable quaternary ammonium compounds quaternarized from the N,N-dialkylaminoalkyl(meth)acrylamide, aminoalkyl(meth)acrylate and vinylpyridine, or other polymerizable unsaturated compounds.

The contents of the substituted succinic anhydride in the aqueous dispersion of the substituted succinic anhydride according to the present invention can be arbitrary selected, but generally ranging from 0.01 percent by weight to 25 percent by weight to obtain stable aqueous dispersion. The aqueous dispersion of the present invention has particularly excellent utility in the stability at diluting time. If less than the concentration of less than 1 percent by weight, the

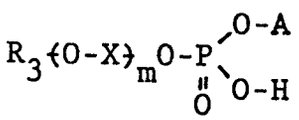
effect performed by the diluting stability further becomes apparent.

The total amount of the contents of aqueous soluble copolymers (A_1) and (A_2) contained in the aqueous dispersed solution according to the present invention are generally preferably ranged from 0.05 percent by weight to 20 percent by weight, and the total of the aqueous soluble copolymers (A_1) and (A_2) is preferably selected in a range from about 0.2 to 2 parts by weight.

Further, the aqueous dispersed solution of the substituted succinic anhydride according to the present invention can, of course, additionally comprise other surfactants or aqueous soluble polymer substances. More specifically, when small amount of an oil-in-water type surfactant made, for example, of polyoxyalkylenealkyl(aryl)ether and its sulfuric acid half ester or phosphoric acid ester, the dispersed particle diameter of the substituted succinic anhydride in the aqueous dispersion can be very finely pulverized, and the aqueous dispersed solution of the substituted succinic anhydride which additionally contain small amount of such oil-in-water type surfactant is most preferable for the aqueous dispersed solution of the present invention. In the aqueous dispersion containing the oil-in-water type surfactant, 0.1 to 10 percent by weight of oil-in-water type surfactant contained in the substituted succinic anhydride is ordinarily preferable.

The aqueous dispersed solution of the substituted succinic anhydride according to the present invention can be readily produced by adding a substituted succinic anhydride (B) to an aqueous solution which contains the aqueous soluble

copolymer (A_1) or (A_2), or both, and mechanically emulsifying and dispersing the mixture by various types of a homogenizer or a mixer. When a small amount of oil-in-water type surfactant is additionally employed, it is preferable to use the prepared mixture of the oil-in-water type surfactant and the substituted succinic anhydride (B). Incidentally, the oil-in-water type surfactant used for the prepared mixture may use any having mutual solubility with the substituted succinic anhydride (B), and it is preferable to selectively use the surfactant which provides excellent underwater dispersing ability in the substituted succinic anhydride (B) and less affects the adverse influence to the sizing effect such as, for example, polyoxyalkylenealkyl(aryl)ether and its sulfuric half-ester or phosphoric acid ester. The content of the oil-in-water type surfactant in the prepared mixture is preferably 0.1 to 10 percent by weight. More preferable oil-in-water type surfactant is a polyoxyalkylenealkyl(aryl)ether phosphoric acid ester represented by the following general formula



where R_3 is an alkyl group or alkylaryl group having 8 or more carbon atoms, X represents an alkylene group, A represents a hydrogen or formula $R'-(O-X)_l-$(in the formula R' is an alkyl group or alkylacryl group, X represents an alkylene group, l represents an integer equal to or greater than 1), and m represents an integer equal to or greater than 1. This is particularly excellent in the solubility with the substituted succinic anhydride or underwater dispersibility of the

substituted succinic anhydride. When the oil-in-water surfactant in which R_3 in the general formula is an alkyl group or alkylaryl group having 10 to 20 carbon atoms, X is an ethylene group, m is an integer equal to or greater than 5 is employed, the underwater dispersed particles of the substituted succinic anhydride produced by dispersing the mixture of the substituted succinic anhydride and the oil-in-water surfactant in the aqueous soluble copolymer (A_1), the aqueous soluble copolymer (A_2) or in the aqueous solution of the both aqueous soluble copolymers become very ultrafine. When the sizing effect of the the aqueous dispersed solution used as paper making sizing agent is further excellent, and becomes more preferable aqueous dispersed solution of the substituted succinic anhydride. To produce the aqueous dispersed solution of the substituted succinic anhydride according to one aspect of the present invention by employing the mixture of the substituted succinic anhydride (B) and the oil-in-water type surfactant, 1 part by weight of the mixture, and the aqueous solution containing 0.1 to 2 percent by weight of the aqueous soluble copolymer (A_1) are mixed and agitated at arbitrary ratio. The agitator used in this case includes an ordinary propeller type agitater or other various homogenizer or a mixer.

The aqueous dispersed solution of the substituted succinic anhydride of the present invention as described above is mainly utilized as a paper making sizing agent, but may be utilized for a sizing agent in various fibrous industrial relations. Incidentally, in the utility as the paper making sizing agent, the aqueous dispersed solution of the substituted succinic anhydride is prepared in arbitrary or

desired concentration, and can be applied to all paper making steps such as acidic paper making step using sulfuric acid band and neutral paper making step using no sulfuric acid band as sizing agent added to a pulp slurry as a paper making raw material or surface sizing agent to be coated by arbitrary means on wet or dry sheet after formed.

In the aqueous dispersed solution of the substituted succinic anhydride according to the present invention, the abovementioned aqueous soluble copolymer (A_1) and the aqueous soluble copolymer (A_2) added and contained in the dispersed solution exhibit high adsorbility to pulp. Thus, the ultrafine substituted succinic anhydride particles in the dispersed solution can be efficiently adhered to the surface of the fiber of the pulp to perform the advantages of the present invention.

The underwater dispersibility of the aqueous soluble copolymers (A_1) and (A_2) contained in the aqueous dispersed solution of the substituted succinic anhydride of the present invention for the substituted succinic anhydride is much superior to that of the copolymer of the (meth)acrylamide (a) and the N,N-dialkylaminoalkyl(meth)acrylamide (b), and such operations of the aqueous soluble copolymer (A_1) and (A_2) provides extremely high dilute stability of the aqueous dispersed solution of the substituted succinic anhydride of the present invention.

Further, the aqueous soluble copolymers (A_1) and (A_2) contained in the aqueous dispersed solution of the present invention are conducted in such a manner that the carboxyl group based on the (meth)acrylic acid monomer ingredient of the copolymer (A_1) or (A_2) reacts with aluminum multi-valency

cation in the sulfuric acid band. Consequently, when the aqueous dispersed solution of the substituted succinic anhydride according to the present invention is applied as paper making sizing agent, various troubles such as wires, rolls and carpets of the paper making machine to be contaminated in the paper making process can be avoided in addition to the excellent sizing effect.

EXAMPLES AND COMPARISON EXAMPLES

Parts by weight of the surfactants listed in the columns of Table 1 were added to 100 parts by weight of alkenyl succinic anhydride, agitated and mixed at ambient temperature, and the composition of the alkenyl succinic anhydride was produced. The alkenyl succinic anhydride was produced by reacting inner olefin having 90 percent by weight of olefin having 15 to 18 carbon atoms and double bonds at 2 to 4 positions with maleic anhydride in accordance with the ordinary process.

Then, the composition of the alkenyl succinic anhydride was mixed similarly by parts in the solution of 10 percent by weight of aqueous soluble polymer compound listed in the columns of Table 1, agitated by a homogenization mixer (HV: M type produced by Tokushu Kika Kogyo K.K.) at 50V for one minute, and the aqueous dispersed solutions of the substituted succinic anhydrides were prepared.

Table 1

	Disperesed solution	Surfactant	Wt.	Aqueous soluble polymer	Wt.
Example	(I)	Polyoxyethylenenonyl-phenol ether phosphoric acid ester (Polyoxyethylene: m=9)	4	Copolymer of 85 mol-% of acryl-amide, 10 mol-% of dimethyl-aminopropylacrylamide and 5 mol-% of acrylic acid (Molecular Wt: about 200,000)	500
	(II)	Polyoxyethylenedistryrene phenol ether phosphoric acid ester (Polyoxyethylene: m=13)	4	Copolymer of 78 mol-% of acryl-amide, 18 mol-% of dimethyl-aminopropylacrylamide and 4 mol-% of acrylic acid (Molecular Wt: about 500,000)	500
	(III)	Polyoxyethylene nonyl-phenol ether phosphoric acid ester (Polyoxyethylene: m=21.5)	3	Copolymer of 70 mol-% of acryl-amide, 20 mol-% of dimethyl-aminopropylacrylamide and 10 mol-% of acrylic acid (Molecular Wt: about 50,000)	500
Comparison Example	(I)	the same as solution in column (I)	4	Copolymer of 90 mol-% of acryl-amide and 10 mol-% of dimethyl-aminopropylacrylamide (Molecular Wt: about 200,000)	500
	(II)	the same as solution in column (I)	4	Copolymer of 90 mol-% of acryl-amide and 10 mol-% of dimethyl-aminoethylmethacrylate methyl-chloride (Molecular Wt: about 2000,000)	500
	(III)	Polyoxyethylenenonyl-phenol ether (Polyoxyethylene m=15)	5	Cationic starch (sold in the market)	2,000

EXPERIMENT 1

The aqueous dispersed solutions produced in the above examples and comparison examples were added to 2 percent by weight of pulp slurry (L-BKP, c.s.f.: 400cc.) containing 20 percent by weight of heavy calcium carbonate filler (Escaron # 800 produced by Sankyo Seifun K.K.) with respect to the weight of the pulp and similarly 1 percent by weight of pasted cationic starch with respect to the weight of the pulp so that the compositions of the substituted succinic anhydride and the surfactant in the aqueous dispersed solution became 0.15 percent by weight of the pulp, then, manual paper of the weight of 80 g/m^2 was prepared in accordance with the ordinary process with TAPPI Standard sheet machine, dried by a rotary drier at 110°C for 120 seconds, and manual paper was produced.

The produced paper was prepared in moisture and measrued in accordance with JIS (the Japanese Industrial Standards) with respect to Stöckigt sizing degree (sec.) and listed in Table 2.

In Table 2, the emulsion mean particle diameters (micron) of the aqueous dispersed solution, the emulsion mean particle diameters (micron) of the dispersed solutions after allowed to stand for 10 hours, and the stabilize of the diluted dispersion diluted so that the composition of the alkenyl succinic anhydride and the surfactant became 0.05 percent by weight with civil water having 100 of total hardness in the dispersed solutions are listed.

Table 2

Dispersed solution	Sizing degree (sec.)	Mean particle size of emulsion (μm) Direct after preparation	After 10 hrs.	Diluted Stability (0.05wt.%)
(I)	23	0.5 or less	0.5 or less	Good
(II)	32	0.5 or less	0.5 or less	Good
(III)	28	0.5 or less	0.5 or less	Good
(I)	18	0.5 or less	0.5 or less	Good
(II)	9	1	7-10	Discrete
(III)	16	1	Precipitated	Good

EXPERIMENT 2

The aqueous dispersed solutions produced in the above examples and comparison examples were added to 2 percent by weight of pulp slurry (L-BKP, c.s.f.:400cc.) containing 20 percent by weight of talc with respect to the pulp weight and similarly 0.5 percent by eight of sulfuric acid band ($Al_2(SO_4)_3.16\text{-}18H_2O$) with respect to the pulp weight so that the compositions of the substituted succinic anhydride and the surfactant in the aqueous dispersed solution became 0.2 percent by weight of the pulp, then similarly to the EXPERIMENT 1 to obtain paper. The produced paper was prepared in moisture and measured according to JIS (the Japanese Industrial Standards) with respect to stöckist sizing degree (sec.) and listed in Table 3.

Table 3

Dispersed solution	(I)	(II)	(III)	(i)	(ii)	(iii)
Size (sec.)	29	23	25	14	5	12

EXPERIMENT 3

The aqueous dispersed solutions produced by the examples and the comparison examples were added to pulp slurry produced with the same compositions as those used in the EXPERIMENT 2 so that the compositions of the substituted succinic anhydride and the surfactant in the aqueous dispersed solution became 1 percent by weight of the pulp weight, paper was made similarly to the EXPERIMENT 1, and wet sheet was prepared. The produced moistened sheet was interposed between a chromium plated plate and filter paper, pressed to remove water for 5 min. under the pressure of 3.5 kg/cm^2, the moistened sheet was then removed from the plate, and the contaminants on the surface of the plate were observed with bare eyes (presence of adhesive such as fine fiber). The results were listed in Table 4.

Table 4

Dispersed solution	(I)	(II)	(III)	(i)	(ii)	(iii)
Contaminants of plate face	Non	Slight	Non	Many	More	Many

According to the present invention, the aqueous dispersed solution of the substituted succinic anhydride is as constituted and operated as described above, and the aqueous solution of the substituted succinic anhydride has the same diluted stability as the aqueous dispersion of the substituted succinic anhydride utilzing the conventional cationic starch

by the stabilization of the aqueous soluble copolymer (A_1) and/or the aqueous soluble copolymer (A_2) in the aqueous dispersed solution or in both the copolymers (A) and (B), and provides the feasibility of handling in case of utilizing the emulsion particle size further finer than the emulsion particle size in the aqueous dispersed solution of the substituted succinic anhydride and industrial scale equivalent to those of the aqueous dispersion of the substituted succinic anhydride using the conventional synthetic polymer. Accordingly, when the aqueous dispersed solution of the substituted succinic anhydride is utilized as the sizing agent in paper making steps, the addition of the substituted succinic anhydride is very low such as 0.15 percent by weight with respect to the pulp weight, sufficiently satisfactory sizing agent can be produced.

Since the aqueous dispersed solution of the substituted succinic anhydride of the present invention has excellent fixing action to pulp fiber in the presence of sulfuric acid band, when the aqueous dispersed solution of the substituted succinic anhydride is used as paper making sizing agent, excellent sizing effect can be provided in all paper making steps including paper making stages using the sulfuric acid band. In addition, the paper making steps can be conducted without contaminants of wires, rolls and blanket of the paper making machine to provide excellent operability.

Claims

1. An aqueous dispersed solution of a substituted succinic anhydride comprising:

(i) an aqueous soluble copolymer (A_1) having 50 to 98.5 percent by mol of (meth)acrylamide (a), 1 to 49.5 percent by mol of N,N-dialkylaminoalkyl(meth)acrylamide (b), and 0.5 to 20 percent by mol of (meth)acrylic acid (c) as monomer in ingredients, and

(ii) an aqueous soluble copolymer (A_2) having 70 percent by weight or more of a mixture monomer ingredient having 50 to 98.5 percent by mol of (meth)acrylamide (a), 1 to 49.5 percent by mol of N,N-dialkylaminoalkyl(meth)acrylamide (b) and 0.5 to 20 percent by mol of (meth)acrylic acid (c) and other copolymer monomer (d) as monomer ingredients, or both of the aqueous soluble copolymer (A_1) and similarly the aqueous soluble copolymer (A_2).

2. The aqueous dispersed solution of a substituted succinic anhydride according to claim 1, wherein said aqueous soluble copolymer (A_1) or (A_2) is an aqueous soluble copolymer containing 75 to 94 percent by mol of (meth)acrylamide (a), 5 to 20 percent by mol of N,N-dialkylaminoalkyl(meth)acrylamide (b) and 1 to 10 percent by mol of (meth)acrylic acid (c) as monomer ingredients.

3. The aqueous dispersed solution of a substituted succinic anhydride according to any of claims 1 and 2, wherein said substituted succinic anhydride is an alkenyl succinic anhydride.

4. The aqueous dispersed solution of a substituted succinic anhydride according to any of claims 1, 2 and 3,

wherein an oil-in-water type surfactant is contained as a dispersing aid.

5. The aqueous dispersed solution of a substituted succinic anhydride according to claim 4, wherein said oil-in-water type surfactant is polyoxyalkylenealkyl(aryl)ether phosphoric acid ester.

European Patent Office

EUROPEAN SEARCH REPORT

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Application number

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DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int Cl 4)
X	US-A-4 533 434 (S. YOSHIOKA et al.) * Column 1, line 36 - column 3, line 10 *	1-4	D 21 H 3/02
A	ABSTRACT BULLETIN OF THE INSTITUTE OF PAPER CHEMISTRY, vol. 55, no. 2, August 1984, pages 245,246, abstract no. 2297, Appleton, Wisconsin, US; & JP-A-91 897/83 (KANZAKI PAPER MFG. CO. LTD; ARAKAWA CHEMICAL INDUSTRY LTD) 31-05-1983 * Whole abstract *	1	
A	ABSTRACT BULLETIN OF THE INSTITUTE OF PAPER CHEMISTRY, vol. 54, no. 11, May 1984, page 1340, abstract no. 12666, Appleton, Wisconsin, US; & JP-A-60 095/83 (ARAKAWA CHEMICAL INDUSTRY LTD) 09-04-1983 * Whole abstract *	1	TECHNICAL FIELDS SEARCHED (Int Cl 4) D 21 H
A	ABSTRACT BULLETIN OF THE INSTITUTE OF PAPER CHEMISTRY, vol. 54, no. 3, September 1983, page 350, abstract no. 3226, Appleton, Wisconsin, US; & JP-A-143 600/82 (ARAKAWA CHEMICAL INDUSTRY LTD) 04-09-1982 * Whole abstract *	1,2	

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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	10-02-1987	NESTBY K.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
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EUROPEAN SEARCH REPORT

Application number
0228801

EP 86 30 8989

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int Cl 4)
A	EP-A-0 151 646 (SEIKO KAGAKU KOGYO CO.; HOKUETSU PAPER MILLS; NIPPON OIL CO.) * Claim 1 * & JP-A-28 598/85 (Cat. D)	3-5	
P,X	EP-A-0 177 260 (SEIKO KAGAKU KOGYO CO.; NIPPON OIL CO.; HOKUETSU PAPER MILLS) * Whole document *	1-5	
			TECHNICAL FIELDS SEARCHED (Int. Cl.4)

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