

(19)



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(11)

EP 0 672 213 B1

(12)

EUROPEAN PATENT SPECIFICATION

(45) Date of publication and mention
of the grant of the patent:

05.02.1997 Bulletin 1997/06

(21) Application number: **94900166.3**

(22) Date of filing: **15.11.1993**

(51) Int Cl.⁶: **D21H 21/16, D21H 17/17**

(86) International application number:
PCT/FI93/00481

(87) International publication number:
WO 94/13883 (23.06.1994 Gazette 1994/14)

(54) **PROCESS FOR THE PREPARATION OF A HYDROPHOBIC SIZING AGENT DISPERSION**

VERFAHREN ZUR HERSTELLUNG VON EINEM HYDROPHOBEN LEIMUNGSMITTEL

PROCEDE DE PREPARATION D'UNE DISPERSION D'AGENT D'ENCOLLAGE HYDROFUGE

(84) Designated Contracting States:
AT BE CH DE FR GB IE IT LI NL PT SE

(30) Priority: **04.12.1992 FI 925535**

(43) Date of publication of application:
20.09.1995 Bulletin 1995/38

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EP 0 672 213 B1

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Description

Hydrophobic sizing refers to the making of paper or board more or less water repellent. This property may be measured e.g. by the so-called Cobb test, in which the penetration of water into paper or board is measured as a function of time. The result is indicated as an absorption g/m^2 . In a properly hydrophobically sized product, the value is generally $< 25 \text{ g/m}^2$, whereas in a product, which has not been treated with sizing agents increasing the hydrophobicity, the value may be $> 100 \text{ g/m}^2$.

Conventional compounds used as a hydrophobic sizing agent include wood-based resin acids and their derivatives.

Nowadays, the paper and board industry has increasingly transferred to using for hydrophobic sizing so-called neutral sizing agents, which generally are compounds based on alkyl-ketene-dimer (AKD) or alkenyl-succinic-acid-anhydride (ASA).

Hydrophobic sizing agents are generally delivered to the paper and board mills as a water dispersion, in which the share of the active agent (AKD, ASA, resin) of the product is within the range of 5-50%. When a sizing agent of the ASA type is concerned, the sizing agent dispersion is primarily prepared at a paper or board mill.

Hydrophobic sizing agents are added to a pulp at the paper or board machine at the starting step of the manufacturing process. In certain cases, these sizing agents may be added also at a later step of the manufacturing process, e.g. at the size press. The quantities of the hydrophobic sizing agents to be used generally amount to 0.05-2.0% of the dry weight of the paper or board.

In addition to the active agent (AKD, ASA or wood resin), a hydrophobic sizing agent dispersion contains stabilizers, surfactants and other additional chemicals. As a stabilizer e.g. cationic starch, cationic polyethylene imines, cationic polyacryl amides, gelatin or casein are used.

As surfactants e.g. sodium lignosulphonate or alkyl sulphonates are used.

Typical additional chemicals include biocides, alum and pH adjusting chemicals.

A conventional way of preparing a hydrophobic sizing agent dispersion from these components is to prepare, by mixing and heating in a reactor, a homogenous premixture containing in a suitable ratio water, an active agent, a stabilizer, surfactants and other potential additional chemicals.

After obtaining the homogenous premixture, it is circulated via a high-pressure homogenizer, a colloid mill or a high-revolution centrifugal pump generally several times, until a sufficiently small particle size has been obtained (generally $0.1 - 10 \mu\text{m}$). The pressure varies within the range of 50 - 500 bar and the temperature within 50 - 100°C . The dispersion obtained is thereafter run via a heat exchanger into a product container. In the heat exchanger, the temperature of the dispersion is decreased to $< 30^\circ\text{C}$.

Surprisingly, it has now been found that a high-pressure digester, a steam jet digester, primarily designed for cooking starch acts as an extremely good dispersing device in the preparation of a hydrophobic sizing agent dispersion.

The invention will be explained more detailed with reference to the enclosed drawing. A steam jet digester process acts as follows:

A premixture is prepared in a reactor 1 according to the same principle as in the afore-mentioned conventional process. After this, the mixture is pumped into a digester portion 2 itself, wherein the mixture contacts a high-pressure steam flow 3 and the temperature and the pressure increase (the pressure generally to 2 - 15 bar and the temperature to 100 - 200°C). The dispersion formed is thereafter run into a tubular reactor 4, after which the pressure and the temperature are decreased in an expander device 5 and the excess of steam may be separated by means of a steam condenser 6. The homogenous product formed is then run via a heat exchanger into a product container.

This process deviates from a conventional process as follows:

Conventional process:

Pressure 50 - 500 bar
Temperature 50 - 100°C

Steam jet digester process

Pressure 2 - 15 bar
Temperature 100 - 200°C

The preparation of a hydrophobic sizing agent dispersion in a process of a steam jet digester type makes it possible to use high solids contents during the processing, and since the delay in the digester and reactor tubes is extremely short, usually < 2 minutes, this makes it possible to use very small units having a large production capacity.

Example 1

In a laboratory scale a stearine/palmitine alkyl ketene dimer (AKD) premixture was prepared having the following composition:

water	800 g
AKD	200 g
Orotan SN*	3.0 g
Raisamyl 150E**	22 g

* Naphthalene formaldehyde sulphonate condensate, sodium salt

** Cationic potato starch (DS = 0.05).

The starch was cooked before adding.

This premixture was divided into two portions, one of which was run 3 times through the laboratory high-pressure homogenizer (Rannie). The pressure was 200 bar, the temperature 70°C. The other portion was run once through a laboratory-scale steam-jet digester, the temperature being 120°C and the pressure 3 bar. Both emulsions were diluted into a solids content of 12,5% and cooled into room temperature.

The following characteristics were measured from each of the dispersions: viscosity (Brookfield) and average particle size.

High-pressure homogenizer/Steam-jet digester

Viscosity	10 cP	8 cP
Average particle size	0.56 µm	0.60 µm

The results show that the dispersions are by their characteristics nearly identical.

The dispersions were kept at room temperature for one month, and the same measurements were performed again, whereby the result was as follows:

High-pressure homogenizer/Steam-jet digester

Viscosity	15 cP	16 cP
Average particle size	0.86 µm	0.95 µm

The results show that the storage stabilities of the two dispersions are of the same order.

Example 2

In a laboratory scale an alkenyl succinic-acid anhydride* (ASA) premixture was prepared having the following composition:

water	900 g
ASA	50 g
Orotan SN	0.5 g
Raisamyl 150E	100 g

This premixture was divided into two portions as well, one of which was run through a high-pressure homogenizer (pressure 200 bar, temperature 50°C) and the other portion was run through a steam-jet digester, the temperature being 110°C and the pressure 1.5 bar.

The dispersions produced were diluted into a solids content of 7.5%, and their viscosity and average particle size were measured.

* As an alkenyl succinic-acid anhydride anhydrides derived from straight-chain C₁₃ - C₂₀ α-olefines were used.

High-pressure homogenizer/Steam-jet digester

Viscosity	600 cP	520 cP
Average particle size	0.50 μm	0.65 μm

The results show that the dispersions are nearly identical.

A value < 1 μm is regarded as a generally accepted particle size in products of this type.

Claims

1. A method for preparing a hydrophobic sizing agent especially for a paper making process, wherein from basic sizing agent components based on an alkenyl succinic acid anhydride (ASA), an alkyl ketene dimer (AKD) or a resin and from additives preferably added thereto a sizing agent premixture is prepared by heating and mixing and this premixture is subjected to a pressurized thermal and mechanical treatment for obtaining a sizing agent dispersion, **characterized** in that the pressurized treatment is performed at a pressure of 2 - 15 bar and at a temperature of 100 - 200°C during a period of less than 2 minutes by subjecting the sizing agent premixture to a high-pressure water steam directed at a high speed.

Patentansprüche

1. Verfahren zur Herstellung von einem hydrophoben Leimungsmittel, insbesondere für einen Papierherstellungsprozess, wobei aus einer Leimungsmittelgrundkomponente auf Alkenylbernsteinsäure- (ASA), Alkylketendimer- (AKD) oder Harzbasis und aus vorzugsweise zu dieser Komponente zugesetzten Zusatzstoffen durch Erwärmen und Mischen eine Vormischung des Leimungsmittels hergestellt wird, und diese Vormischung zur Erreichung einer Leimungsmitteldispersion einer thermischen und mechanischen Behandlung unter Druck ausgesetzt wird, dadurch **gekennzeichnet**, daß die Druckbehandlung unter einem Druck von 2 bis 15 bar und bei einer Temperatur von 100 bis 200°C während weniger als 2 Minuten durchgeführt wird, indem die Leimungsmittelvormischung dem Einfluß eines mit einer hohen Geschwindigkeit geleiteten Hochdruckwasserdampfes ausgesetzt wird.

Revendications

1. Procédé de préparation d'un agent d'encollage hydrofuge spécialement pour un procédé de fabrication de papier, dans lequel à partir de composants d'agents d'encollage de base à base d'un anhydride d'acide alcénylsuccinique (ASA), d'un dimère d'alkylcétène (AKD) ou d'une résine et à partir d'additifs qui y sont de préférence ajoutés, un prémélange d'agents d'encollage est préparé par chauffage et mélange, et ce prémélange est soumis à un traitement thermique et mécanique sous pression pour obtenir une dispersion d'agents d'encollage, caractérisé en ce que le traitement sous pression est effectué à une pression de 2 à 15 bars et à une température de 100 à 200°C pendant une période inférieure à 2 minutes en soumettant le prémélange d'agents d'encollage à une vapeur d'eau à haute pression dirigée à une grande vitesse.

