CONVENTION APPLICATION FOR A PATENT

(a) Insert full
name(s) of
applicant(s).

O/We (a) BAYER AKTIENGESELLSCHAFT

(b) Insert
country(ies)
of applicant(s).

of (b) Leverkusen,

GERMANY

(c) Insert title
of invention.

d) "AGENT FOR USE IN PULP PRODUCTION"

which is described in the accompanying complete specification. This application is a Convention application and is based on the application or applications for a patent or patents or similar protection made in the following country or countries on the following date or dates:

in (d) Germany in on (e) June 21, 1978 in No. (f) P 28 27 111.6

My/Our address for service is care of ARTHUR S. CAVE & CO., Patent and Trade Mark Attorneys, 1 Alfred Street, Sydney, New South Wales, Australia 2000.

Dated this (g) 5th day of JUNE 1979.

The Commissioner of Patents,
COMMONWEALTH OF AUSTRALIA

ARTHUR S. CAVE & CO.
PATENT AND TRADE MARK ATTORNEYS
SYDNEY
AUSTRALIA

PATENT DECLARATION FORM (CONVENTION)
COMMONWEALTH OF AUSTRALIA
"Patents Act 1952-1973"

DECLARATION IN SUPPORT OF A CONVENTION APPLICATION UNDER
PART XVI FOR A PATENT OR PATENT OF ADDITION

To be signed by the applicant(s) or in the case of a Company, to be signed by a person authorised by the Company.

(a) Insert title of invention.

(b) Insert full name(s) of declarant(s).

(c) Insert address(es) of declarant(s).

In support of the Convention application made for a patent or patent of addition for an invention entitled "Agent for use in pulp production"

To (b) Joachim Gremm and Erwin Dill

Of (c) Leverkusen, Bayerwerk, Germany

Do solemnly and sincerely declare as follows:

1. I am/we are the applicant(s) for the patent or patent of addition.

2. The basic application(s) as defined by Section 141 of the Act were made in the following country or countries on the following date(s) namely:

   (d) Germany on (e) June 21, 1978 No. (f) P 28 27 111 6

   by (g) Bayer Aktiengesellschaft

3. I am/we are the actual inventor(s) of the invention referred to in the basic application.

   (h) 1) Dieter Bauer 2) Volker Haßmann 3) Paul Schiller

4. The basic application(s) referred to in paragraph 2 of this Declaration were the first application(s) made in a Convention country in respect of the invention the subject of the application.

Declared at Leverkusen this 9th day of May 1979

Joachim Gremm

Secretary

The Commissioner of Patents.
Agent for use in pulp production, characterised in that it contains organic, cyclic compounds containing keto groups and/or hydroxyl groups, in a finely divided form and surface-active agents.
TO BE COMPLETED BY APPLICANT

Name of Applicant: BAYER AKTIENGESELLSCHAFT

Address of Applicant: Leverkusen, GERMANY

Actual Inventor: DIETER BAUER, VOLKER HABMANN, PAUL SCHILLER, KONRAD NONN, KARLHEINZ WOLF


Complete Specification for the invention entitled

"AGENT FOR USE IN PULP PRODUCTION"

The following statement is a full description of this invention, including the best method of performing it known to me:-
It is described (see, for example, Bach and G. Fiehn, Zellstoff und Papier 21, 3 (1972); H.H. Holton, Pulp and Paper Canada 78, 19 (1977); U.S. Patent Specification 4,012,280; U.S. Patent Specification 4,036,680; U.S. Patent Specification 4,036,681, Canadian Patent Specification 986,662, Japanese Laid-Open Specification 112,903/75, Japanese Laid-Open Specification 43,803/76, Japanese Laid-Open Specification 109,303/76 and German Democratic Republic Patent Specification 98,549) that anthraquinone, certain anthraquinone derivatives and certain diketohydroanthracenes exert a favourable effect in certain processes for the production and bleaching of pulp from lignocellulose materials, such as wood, straw and bagasse, when from 0.001 to 10% by weight thereof, relative to the lignocellulose material, are employed. In addition to anthraquinone, anthrahydroquinone and Diels-Alder adducts of butadiene and its derivatives with p-benzoquinone or 1,4-naphthoquinone, the mono- and poly-alkyl, -alkoxy, -amino, -hydroxyl and/or -sulpho derivatives of these compounds are recommended for this. In the following text, these substances are collectively designated additives.

In general, the additives are accessible in the form of powders. However, the incorporation of pulverulent additives of this type into processes for the production of pulps from lignocellulose materials and for the bleaching thereof presents problems. If the pulverulent additives are added to the lignocellulose material to be employed, it must be expected, in this case, that the finer constituents of the additives pass into the environment in the form of a dust and are thus partly withdrawn from the intended use, are troublesome to people working near the place of addition and can give rise to the danger of dust explosions. In addition, uniform distribution is difficult with the relatively small amount of additives required. However, uniform distribution of the additives is desirable in order to achieve uniform quality of the pulp.
Uniform distribution of the additives is also made difficult by the fact that the additives are generally only very slightly soluble in water and in the aqueous electrolyte solutions used in pulp production (for example, only $6.10^{-4}$ g of 9,10-anthraquinone dissolves in 1 litre of water at 50°C).

Furthermore, the additives are so poorly wetted by water and aqueous electrolyte solutions, such as are used in pulp production, that the finer constituents of the pulverulent additives cannot be stirred in, or can only be stirred in to a poor extent, but float non-wetted on the surface, air sometimes being occluded. Moreover, the additives have a relatively high specific gravity (for example anthraquinone has a specific gravity of 1.438 g/cm³ at 20°C), so that the coarser constituents of the pulverulent additives, which can be stirred into water or an electrolyte solution, rapidly settle again and, after standing for a short time, form on the floor of the vessel a compact layer which can be stirred up again only with difficulty. The addition of the additives directly to the cooking liquor, to the mixture of lignocellulose material and cooking liquor or in the form of a paste in water or dilute electrolyte solutions is thus also no way of achieving with certainty a uniform distribution of the additives.

An agent has now been found for use in pulp production, which is characterised in that it contains organic, cyclic compounds containing keto groups and/or hydroxyl groups, in a finely divided form, and surface-active agents.

Here and in the following text, the expression pulp production is understood as all processes and process stages in which the lignin in materials containing lignin and cellulose is acted upon in a chemical manner. Examples of these processes are alkaline, neutral and acid pulping processes with lignocellulose materials, such as wood, straw, bagasse and grasses, and bleaching processes with partially or substantially pulped lignocellulose materials.
Possible organic, cyclic compounds containing keto groups and/or hydroxyl groups are preferably carbocyclic compounds, for example monocyclic, bicyclic and/or polycyclic compounds, in particular monocyclic, bicyclic and/or tricyclic compounds, and particularly preferably tricyclic compounds, especially fused ring tricyclic compounds which contain two keto groups and/or two hydroxyl groups in each case and are preferably hydrocarbons except for the keto or hydroxyl groups and/or other substituents. Compounds which can be used here are, preferably, p-benzoquinone, 1,4-naphthoquinone, 9,10-anthraquinone and/or Diels-Alder adducts of 1,3-dienes, for example of unsubstituted or substituted butadienes, with p-benzoquinone and/or 1,4-naphthoquinone, and/or mono- and poly-alkyl, -hydroxyl, -amino, -alkoxy, -alkylamino and/or sulpho derivatives thereof. The alkyl, alkoxy and alkylamino moieties can each contain, for example, 1 to 12 C atoms, preferably 1 to 4, carbon atoms. For example, the dispersions according to the invention can contain 9,10-anthraquinone, 2-methylantraquinone, 2-ethylanthraquinone, 2,3-dimethyl-9,10-anthraquinone, 2,6-dimethylantraquinone, 2,7-dimethylantraquinone, 2-aminoanthraquinone, 1-methoxyanthraquinone, 1,4,4a,9a-tetrahydro-9,10-diketoanthracene, 2-ethyl-1,4,4a,9a-tetrahydro-9,10-diketoanthracene, 2,3-dimethyl-1,4,4a,9a-tetrahydro-9,10-diketoanthracene, 1,4,4a,5,8,8a,9a,10a-octahydro-9,10-diketoanthracene, 1,3-dimethyl-1,4,4a,9a-tetrahydro-9,10-diketoanthracene and 2,3,6,7-tetramethyl-1,4,4a,5,8,8a-9a,10a-octahydro-9,10-diketoanthracene. Compounds which can also be used are the above named compounds in reduced form containing hydroxy groups instead of keto groups, for example hydroquinone and anthrahydroquinone. The dispersion according to the invention can contain two or more of these substances, in particular two or more of these substances which have specific gravities which are close to one another. It is also possible to employ compounds which carry two or more of the substituents mentioned, for example hydroxyl groups and amino groups. However, the dispersion according to the invention preferably contains...
only one of these substances, very particularly preferably 9,10-anthraquinone. In the following text, the organic, cyclic compounds containing keto groups and/or hydroxyl groups are designated as pulping auxiliaries.

It is a particular feature of the agent according to the invention that the pulping auxiliaries, in particular 9,10-anthraquinone, are present in a finely divided form. For example, the pulping auxiliaries, in particular 9,10-anthraquinone, can consist to the extent of at least 80% by weight of particles with particle sizes of less than 10 μm. The pulping auxiliaries, in particular 9,10-anthraquinone, preferably consist to the extent of at least 80% by weight of particles with a particle size of less than 5 μm.

Possible surface-active agents contained in the agent according to the invention can be virtually any cationic, non-ionic and anionic surface-active agents.

Examples of possible cationic surface-active agents are quaternary long-chain and/or oxethylated amines, quaternary pyridinium compounds or long-chain phosphonium compounds. Long-chain means here a carbon chain of at least 4 C atoms, preferably of at least 6 C atoms.

Examples of cationic surface-active agents are in particular: trimethyl-hexadexyl-ammonium bromide, cetlylpyridinium bromide, lauryl-dimethylbenzyl-ammonium chloride, monoesters of triethanolamine with stearic acid as formic acid salts or acetic acid salts, N-lauryl-methylbenzimidazole hydrochloride and dodecyl-trimethyl-phosphonium bromide.

A large part of these compounds and further cationic surface-active agents which can be used are described, for example, in K. Lindner, Tenside - Textilhilfsmittel - Waschrohstoffe [Surface-active Agents - Textile Auxiliaries - Detergent Raw Materials], volume 1, pages 963-1041 (1964).

Examples of non-ionic surface-active agents which can be used are adducts of alkylene oxides, in particular ethylene oxide, with higher fatty acids, alcohols, phenols, acid amides, mercaptans, amines or alkylphenols. The adducts can be obtained, for example, from 5 to 50 mols of
alkylene oxide and 1 mol of fatty acid, alcohol, phenol, acid amide, mercaptan, amine or alkylphenol, these compounds containing at least 4, preferably at least 6, C atoms. Non-ionic surface-active agents which can be used are also 5 adducts of alkylen oxides, in particular ethylene oxide, with polypropylene oxide or with sugars, as well as oxethyl- and non-oxethylated sugar derivatives, such as fatty acid esters of pentaerythritol or of sucrose.

Particular examples of non-ionic surface-active 10 agents are: adducts of 5 to 20 mols of ethylene oxide with stearic acid, oleyl alcohol, polypropylene glycol, nonylphenol, oleic acid amide and dodecylamine.

A large part of these compounds and further non-ionic surface-active agents which can be used are described, for example, in N. Schöpfeldt, Grenzflächenaktive Äthylenoxid-Addukte [Surface-active Ethylene Oxide Adducts], pages 42 to 95 (1976), and in K. Lindner, Tenside-Textilhilfs-mittel - Waschrohstoffe [Surface-active Agents - Textile Auxiliaries - Detergent Raw Materials], volume 1, pages 837 to 917 (1964).

Preferably, the agent according to the invention contains anionic surface-active agents. Examples of anionic surface-active agents which can be used are alkylsulphonates, sulphated unsaturated higher fatty acids, sulphonates of polycarboxylic acid esters, alkylbenzenesulphonates, sulphated aliphatic alcohols, adducts of ethylene oxide with higher amines, acids, phenols or alcohols, which adducts have been converted into acid esters with an inorganic polybasic acid, such as phosphoric acid or particularly sulphuric acid, and also ligninsulphonates or derivatives of ligninsulphonates, condensation products of aromatic sulphonic acids with formaldehyde, and polyphosphates.

Particular examples of anionic surface-active agents are: sodium dodecylsulphonate, sodium laurylsulphate, sodium dodecylbenzenesulphonate, dibutylnapthalenesulphonate, acid sulphuric acid esters of the adduct of 2 mols of ethy- lene oxide with 1 mol of nonylphenol, sodium dioctyl-sul-
phosphatocinate, condensation products of cresol, 2-naphthol-
6-sulphonic acid and formaldehyde, and also sulphonic acids
of naphthalene, terphenyl or ditolyl ether in each case
condensed with formaldehyde. All the anionic surface-
active agents are preferably employed in the form of their
alkali metal salts and/or ammonium salts.

A large part of these compounds and further anionic
surface-active agents which can be used are described in
K. Lindner, Tenside-Textilhilfsmittel - Waschrohstoffe
[Surface-active Agents - Textile Auxiliaries - Detergent
Raw Materials], volume 1, pages 571 to 835 (1964).

Particularly preferably, the agent according to the
invention contains ligninsulphonates and/or condensation
products of aromatic sulphonie acids and formaldehyde as the
surface-active agents.

The agent according to the invention can also con-
tain any desired mixtures of anionic and non-ionic surface-
active agents.

The agent according to the invention can be made
available in two forms, either in the form of a pulverulent
mixture which contains organic cyclic compounds, containing
keto groups and/or hydroxyl groups, and surface-active
agents in a finely divided form, or in the form of a dis-
persion which contains the above constituents dispersed in
water and/or an organic, water-miscible solvent having a
boiling point above 80°C. Preferably, the organic sol-
vent has a boiling point above 100°C. Examples of
organic solvents which can be used are alcohols, in parti-
cular polyhydric alcohols, and acid amides and substituted
acid amides. Examples of these are: dimethylformamide,
propylene glycol, glycol monoethyl ether, diethylene glycol
monoethyl ether, formamide, glycol, glycerol, diols, triols
and polyols. In conjunction with water, these solvents
having boiling points over 100°C also act as agents which
prevent drying out. If the dispersion according to
the invention contains water and organic solvents, these
two components can be present in any desired mixing ratio.
Preferably, a dispersion of this type contains 5 to 20 %
by weight of solvent (relative to the total dispersion).

The pulverulent mixture according to the invention can, for example, contain 30 to 98 % by weight of one or more pulping auxiliaries, in particular 9,10-anthraquinone, in a finely divided form and 2 to 70 % by weight of one or more surface-active agents. Preferably, the pulverulent mixture according to the invention contains 50 to 95 % by weight of one or more pulping auxiliaries, in particular 9,10-anthraquinone, in a finely divided form and 5 to 50 % by weight of one or more surface-active agents.

Pulverulent mixtures according to the invention can be prepared, for example, by dry grinding of the components individually and subsequent mixing or by first preparing the mixture of the components and grinding the mixture. Grinding is preferably carried out in such a way that, after the grinding procedure, at least 80 % by weight of the pulping auxiliaries have a particle size of less than 10 µm, preferably less than 5 µm. To carry out a grinding procedure of this type, conventional dry comminution apparatuses can be used, for example ball mills, rotor/stator mills, pinned disc mills, hammer mills and jet mills. Jet mills can be operated, for example, with air or steam.

The surface-active agents do not have to be present in a finely divided form, that is to say they do not necessarily have to be ground, since they generally dissolve readily when the pulverulent mixture is used in processes for the production of pulp. It is preferred, however, to add the surface-active agents before the grinding procedure. In this way, an agglomeration, which may occur, of the ground particles can be prevented.

If the pulverulent mixtures according to the invention are to be employed in processes for the production of pulp, carried out in an alkaline medium, it can be advantageous to add small quantities of a material, having an alkaline reaction, to the mixture. Substances of this type, for example NaOH, KOH, Na₂CO₃, K₂CO₃ or LiOH, can be added, for example, in quantities of 0.2 to 5 % by weight (relative to the total mixture).

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The pulverulent mixture according to the invention can be employed direct in processes for the production of pulp. It is also possible, however, first to convert the pulverulent mixture according to the invention into a dispersion by stirring it into water, for example into 30 to 300 % by weight of water (relative to the pulverulent mixture) and to employ this dispersion in the process for the production of pulp.

If the pulverulent mixture according to the invention is converted into an aqueous dispersion, before it is employed in a process for the production of pulp, it can be advantageous to add substances, which effect a stabilisation of dispersions, to the pulverulent mixture. Examples of substances of this type can be: highly disperse silica, magnesium silicates and aluminium silicates, montmorillonites which can also contain organic bases, chalk and/or thickeners, such as methylcellulose, hydroxymethylcellulose, hydroxyethylcellulose, carboxymethylcellulose as well as polyacrylates and/or polymethacrylates and/or copolymers thereof, and also combinations of the substances mentioned. Substances of this type can be added to the pulverulent mixture according to the invention, for example, in quantities of 0 to 5 % by weight. Preferably, substances of this type are added to the pulverulent mixture according to the invention in quantities of 0.1 to 1 % by weight.

The agent according to the invention in the form of a dispersion can, for example, contain 30 to 70 % by weight of one or more pulping auxiliaries in a finely divided form, in particular 9,10-anthraquinone, 0.5 to 30 % by weight of one or more surface-active agents and at least 30 % by weight of water and/or organic, water-miscible solvents having a boiling point above 80°C. Preferably, the agent according to the invention in the form of a dispersion contains 45 to 65 % by weight of pulping auxiliary in a finely divided form, in particular 9,10-anthraquinone, 1 to 10 % by weight of surface-active agents and at least 35 % by weight of water and/or organic solvent.

The agent according to the invention in the form of dispersion contains 45 to 65 % by weight of pulping auxiliary in a finely divided form, in particular 9,10-anthraquinone, 1 to 10 % by weight of surface-active agents and at least 35 % by weight of water and/or organic solvent.
a dispersion can additionally contain further substances, for example preserving agents, agents which prevent drying out, if one of the organic solvents mentioned has not already been added, and/or agents for stabilising dispersions.

Preserving agents which can be used are those substances which prevent, for example, the formation of mould and/or an attack by bacteria. Customary preserving agents, such as sodium pentachlorophenolate, adducts of paraformaldehyde with aromatic alcohols, in particular benzyl alcohol, and/or formaldehyde solutions are suitable for this purpose. Preserving agents can be added to the agent according to the invention in the form of a dispersion, for example, in quantities of 0 to 3 % by weight, preferably in quantities of 0.05 to 0.5 % by weight.

Examples of agents which prevent drying out, which can be used, are customary soluble agents which prevent drying out and which can be identical to the water-miscible solvents having a boiling point above 80°C, described above in more detail. For example, formamide, glycol, glycol derivatives, glycerol, diols, triols and polyols are particularly suitable. Agents which prevent drying out can be added to the agent according to the invention, if it is present in the form of a dispersion, for example in quantities of 2 to 20 % by weight, preferably in quantities of 5 to 10 % by weight (in each case relative to the total dispersion).

As the agents which effect a stabilisation of dispersions, the same agents which are indicated above for the pulverulent agents according to the invention can be employed. Stabilisers can be added to the agent according to the invention in the form of a dispersion, for example, in quantities of 0 to 2 % by weight, preferably in quantities of 0.1 to 1 % by weight.

Agents according to the invention in the form of a dispersion can, for example, be prepared by introducing water or mixtures of water and the solvents described above, or only one of the solvents described above, surface-active agents, one or more pulping auxiliaries, in particular 9,10-
anthraquinone, and, if appropriate, stabilisers, mixed in the desired ratio to a customary wet comminution apparatus. Suitable wet comminution apparatuses are, for example, kneaders, kneading screws, ball mills, rotor/stator mills, dissolvers, corundum disc mills and vibration mills. Preferably, high-speed stirred ball mills are used, the grinding bodies preferably having a diameter in the range from 0.1 to 10 mm. Grinding is preferably continued until the solid particles have, at least to the extent of 80 % by weight, a particle size of less than 10 μm, preferably less than 5 μm. If appropriate, the preserving agents and/or agents which prevent drying out can be added to the dispersion before or after grinding.

In addition to the possibilities, described above, for preparing the agent according to the invention in the form of a pulverulent mixture, pulverulent mixtures according to the invention can also be prepared by drying from agents according to the invention in the form of a dispersion. Such drying can be carried out for example, in a circulating air cabinet, in continuous or discontinuous paddle driers, in drum driers, in thin-layer contact driers and in freeze-driers. Advantageously, a spray-drier with atomiser discs, two-material nozzles or single-material nozzles is used for drying. In this way, free-flowing granules can be obtained.

If the agent according to the invention in the form of a dispersion is to be introduced into pulp production processes which are carried out in an alkaline medium, it can be advantageous to add small amounts of a substance having an alkaline reaction to the dispersion. Substances of this type, for example aqueous sodium hydroxide solution or aqueous potassium hydroxide solution, can be added, for example, in quantities of 0 to 2 % by weight.

The agent according to the invention, in particular an agent containing 9,10-anthraquinone, is used in processes for pulp production. In pulp production, the agent according to the invention can be fed in before the cooking, but advantageously already before the impregnation, in which
the lignocellulose material is impregnated with an aqueous solution of the pulping chemicals at a temperature of 80 to 100°C. The aqueous solution of the pulping chemicals also serves as a conveying medium for charging the impregnator and/or cooker with lignocellulose material. The agent according to the invention, in particular an agent containing 9,10-anthraquinone, can be metered into the refluxing solution or into the solution charged with chips, or optionally also directly into the impregnator or cooker. The amount of agent according to the invention used in pulp production can be chosen so that, for example, 0.001 to 10 % by weight, relative to the lignocellulose material, of the agent according to the invention are added. When using the agent according to the invention, the pulping auxiliary is finely distributed in the pulping liquid.

The agent according to the invention, in the form of a dispersion, in particular in the form of a dispersion containing 9,10-anthraquinone, has additional advantages. Thus, the preparation of this dispersion is simple. The agent according to the invention in the form of a dispersion can be pumped, that is to say it can be metered, and conveyed through pipelines, with the aid of a pump suitable for pumping dispersions, for example a peristaltic pump, an eccentric screw pump or a piston pump. The agent according to the invention in the form of a dispersion is stable for a relatively long time as a dispersion. A dispersion of this type can be stored for at least several days, in general for one or more weeks, during which the dispersed substances do not settle or float to the surface, or settle or float to the surface only to such a slight extent that they can be brought into the dispersed state again by simple means, for example a slow-speed stirrer. This has the advantage that a relatively large amount of the dispersion can be prepared all at once, the metering of which can then be effected, for example, by a simple measurement of volume or amount. Depending on which possibility appears to be more advantageous, the agent according to the invention can also
be made available as a pulverulent mixture from which a
dispersion can be prepared, if desired, only shortly before
use in pulp production.

By employing the agent according to the invention
in processes for pulp production, including the bleaching
of pulp, the favourable effects of the presence of organic,
cyclic substances containing keto groups and/or hydroxyl
groups can be utilised in an optimum manner, since a uniform
distribution thereof is obtained.

It is to be described as distinctly surprising that
the agents according to the invention completely fulfil the
requirements for use in pulp production, including the bleaching of pulp, since a recrystallisation with an enlargement
of the particles was to be expected due to the slight,
though significant solubility in water of the organic, cyclic
compounds containing keto groups and/or hydroxyl groups, in particular 9,10-anthraquinone, and it was thus not possible
to anticipate that the finely divided state and hence the stable dispersions could be maintained for prolonged periods.

Moreover, a process has been found for pulp production from lignocellulose materials in the presence of organic, cyclic compounds containing keto groups and/or hydroxyl groups, which is characterised in that the organic, cyclic compounds containing keto groups and/or hydroxyl groups are employed in the form of the agent according to the invention. With the exception of the use of the agent according to the invention, this process can be carried out in a manner which is in itself known. For example, this process can be carried out by digesting lignocellulose materials in a sulphite solution, which can be acid, neutral or alkaline, and feeding the agent according to the invention to the digestion solution, before or after adding the lignocellulose material. It is also possible to employ the agent according to the invention in the known processes for pulp production, which are called the Kraft process and polysulphide process. Furthermore, the agent according to the invention can be employed in the known oxygen/alkali process for pulp production and/or in the Le A 18 875
bleaching processes known for pulp production.

The agent according to the invention can be employed in the process according to the invention for pulp production and the bleaching of pulp in an amount of, for example, 0.001 to 10 % by weight (relative to the lignocellulose material).

Preferably, 9,10-anthraquinone is employed in the process according to the invention in the form of the agent according to the invention. In this case, it is particularly preferable to use the agents designated as particularly preferred within the scope of the agent according to the invention.

The process according to the invention has a number of advantages. Thus, for example, it is possible to meter and uniformly distribute the organic, cyclic compounds containing keto groups and/or hydroxyl groups without difficulties, and as a result thereof, pulps of uniform quality are obtained. Furthermore, it is possible to realise the advantageous effects, determined on a laboratory scale under ideal conditions, of the addition of organic, cyclic compounds containing keto groups and/or hydroxyl groups, in industrial installations for pulp production.

In the laboratory experiments, for example, the lignocellulose material were agitated in the pulping liquid or bleaching liquid, which facilitates distribution of the additives. In industrial installations for pulp production, this is the case only to a minor extent, and the distribution of the additives is thus made more difficult if they are not employed in the form of the agent according to the invention.

Examples

Example 1

A paste of 500 g of 9,10-anthraquinone in a mixture of 248 ml of water, 2 g of sodium pentachlorophenolate, 100 g of ethylene glycol and 150 g of 50 % strength sodium ligninsulphonate as obtained in pulp production, is ground for 15 minutes with glass beads of 0.3 to 0.4 mm diameter in a high-speed stirred ball mill as described in the Le A 18 875.
After the end of grinding, the particle size of the anthraquinone in the pumpable dispersion obtained is less than 10 μm. About 95% of the particles are smaller than 5 μm. Even after standing for several weeks at room temperature, the anthraquinone does not sediment.

**Example 2**

A paste of 600 g of 9,10-anthraquinone in 316 ml of water and 60 g of ethylene glycol is ground, with the addition of 20 g of sodium dinaphthylmethanesulphonate, in a high-speed stirred ball mill corresponding to Example 1. After the end of grinding, the particle size of the anthraquinone is as indicated in Example 1. Subsequently, 4 g of a swellable aluminium silicate or magnesium silicate are stirred as a thickener into the dispersion. The free-flowing dispersion of the anthraquinone does not sediment and, when stirred into the wood-pulping liquor of the Kraft process and soda process, gives a stable dispersion in the wood-pulping liquor.

**Example 3**

A paste of 600 g of 9,10-anthraquinone in a mixture of 312 g of water, 60 g of ethylene glycol, 20 g of a condensation product of naphthalenesulphonate and formaldehyde, 4 g of swellable aluminium silicate, 3 g of 45% strength sodium hydroxide solution and 2 g of a formaldehyde depot compound is premixed in a dissolver and ground in a mill corresponding to Example 1. After the end of grinding, the particle size of the anthraquinone is as indicated in Example 1. A pumpable dispersion is obtained which does not separate even after prolonged standing and can be readily dispersed in the so-called white liquor (= aqueous solution of the pulping chemicals) in the Kraft process and soda process.

**Example 4**

Analogously to Example 2, 600 g of 9,10-anthraquinone are ground in 100 g of ethylene glycol, 250 g of water and 30 g of sodium ligninsulphonate and are thickened with 20 g of chalk or 5 g of methylcellulose and preserved by

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means of formaldehyde addition products. As in Example 1, a pumpable, stable dispersion is obtained.

Example 5

Analogously to Example 2, 600 g of 9,10-anthraquinone in 90 g of ethylene glycol monoethyl ether, 250 g of water and 20 g of a condensation product of naphthalene-sulphonate and formaldehyde are ground, thickened with 20 g of chalk and rendered alkaline with 3 g of 45 % strength sodium hydroxide solution. As in Example 4, a pumpable, stable dispersion is obtained which can readily be stirred into wood-pulping liquors of the Kraft process and soda process.

Example 6

500 g of 9,10-anthraquinone are ground, as in Example 1, in 490 g of an aqueous pulping solution (= black liquor) obtained in a pulp production process and containing ligninsulphonates, and are thickened with 1 % by weight of swellable aluminium silicate, a stable, pumpable anthraquinone dispersion being obtained which can readily be stirred into wood-pulping liquor.

Example 7

A paste of 500 g of 9,10-anthraquinone in a mixture of 323 ml of water, 2 g of sodium pentachlorophenolate, 100 g of ethylene glycol and 75 g of a condensation product of cresol, 2-naphthol-6-sulphonic acid and formaldehyde is comminuted corresponding to Example 1. The anthraquinone does not sediment out of the dispersion obtained.

Example 8

516 g of 50 % strength aqueous sodium ligninsulphonate are added to a dispersion, obtained according to Example 1, without sodium pentachlorophenolate and with the corresponding additional amount of water in place of ethylene glycol. Subsequently, this dispersion is dried in a spray drier (inlet temperature 180°C, outlet temperature 90°C). The granules obtained, which consist of 60 % by weight of anthraquinone and 40 % by weight of sodium ligninsulphonate, can readily be divided by stirring into water or wood-pulping liquor of the Kraft process or soda.
Example 9

Corresponding to Example 8, granules which can readily be divided in water or wood-pulping liquor of the Kraft process or soda process and which contain 50% by weight of anthraquinone and 50% by weight of sodium ligninsulphonate, are obtained by adding 850 g of 50% strength aqueous sodium ligninsulphonate.

Example 10

Corresponding to Example 8, granules which can very readily be divided in water or wood-pulping liquor of the Kraft process or soda process and which consist of 30% by weight of anthraquinone and 70% by weight of sodium ligninsulphonate, are obtained by adding 2,084 g of 50% strength aqueous sodium ligninsulphonate.

Example 11

Anthraquinone formulations which were prepared analogously to Examples 8 to 10 but in which a condensation product of naphthalenesulphonate and formaldehyde was employed in place of sodium ligninsulphonate, can readily be divided in water or wood-pulping liquor of the Kraft process or soda process by simply stirring in.

Anthraquinone formulations which were prepared in a corresponding manner, but with a condensation product of sulphonated ditolyl ether and formaldehyde, or a condensation product of sulphonated diphenyl ether and formaldehyde, or a condensation product of sulphonated terphenyl and formaldehyde, can likewise be readily divided in water or wood-pulping liquor of the Kraft process or soda process by simply stirring in.

Example 12

500 g of 9,10-anthraquinone which was comminuted by jet grinding with air to particle sizes of less than 10 μm, are intensively mixed with 333 g of sodium ligninsulphonate. A dispersion of good fine division is obtained by stirring this preparation into water or wood-pulping liquor of the Kraft process or soda process.
Example 13
A preparation which, when stirred into water or wood-pulping liquor of the Kraft process or soda process, gives a good fine division, is obtained by mixing 500 g of 9,10-anthraquinone which was comminuted according to Example 12, and 500 g of sodium ligninsulphonate.

Example 14
A preparation which contains 30 % by weight of anthraquinone and 70 % by weight of sodium ligninsulphonate is obtained by mixing 500 g of 9,10-anthraquinone which was comminuted corresponding to Example 12, with 1,117 g of sodium ligninsulphonate. When stirred into water or wood-pulping liquor of the Kraft process or soda process, this preparation leads to a dispersion of good fine division.

Example 15
500 g of 9,10-anthraquinone and 50 g of a condensation product of naphthalenesulphonate, diphenyl ether-sulphonate, or ditolyl ether-sulphonate and formaldehyde, were ground down to a particle size of less than 10 μm in a jet mill which was operated with air. When stirred into 290 g of water or corresponding amounts of Kraft pulping liquor or soda pulping liquor, this preparation leads to a pumpable dispersion which is stable for a prolonged time and which is readily distributed in further Kraft pulping liquor or soda pulping liquor.

Example 16
500 g of 9,10-anthraquinone and 10 g of a condensation product of naphthalenesulphonate and formaldehyde were ground together. The finely divided pulverulent mixture obtained gives, when stirred into 330 g of a water/glycol mixture in the ratio 6 : 2 : 1 to 10 : 1 or corresponding amounts of Kraft pulping liquor or soda pulping liquor, stable pumpable dispersions which are very readily distributed in Kraft pulping liquors or soda pulping liquors. The same results were obtained when 10 g of a condensation product of diphenylsulphonate and formaldehyde or 10 g of a condensation product of ditolylsulphonate and formaldehyde are employed.

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Example 17

600 g of 9,10-anthaquinone, 100 g of a condensation product of naphtalenesulphonate and formaldehyde, 2 g of aluminium silicate and 2 g of sodium hydroxide are jet-milled together to give a particle size of 5 to 20 μm. A preparation is obtained which gives a stable dispersion, when stirred with 600 g of water or wood-pulping liquor of the Kraft process or soda process.

Example 18

When Examples 1 to 17 are employed, using anthraquinone derivatives, such as monoalkyl-, dialkyl-, hydroxy-, dihydroxy-, amino-, alkoxy- and alkylamino-anthraquinone, or correspondingly substituted Diels-Alder adducts of 1,3-dienes with 1,4-naphthoquinones or 1,4-benzoquinone in place of anthraquinone, this gives stable pumpable dispersions or pulverulent mixtures which can be converted into stable pumpable dispersions, using the indicated amounts of liquid. The dispersions obtained are distinguished in that they can be dispersed without any problems in Kraft pulping liquors or soda pulping liquors.

Example 19

400 g of 9,10-anthraquinone were ground in 580 g of ethylene glycol and 20 g of sodium dinaphthylmethanesulphonate in a bead mill analogously to Example 1. As in Examples 3 and 4, a pumpable dispersion is obtained which is stable and can readily be stirred into wood-pulping liquors of the Kraft process and soda process.
The claims defining the invention are as follows:

1. Agent for use in pulp production, characterised in that it contains organic, cyclic compounds containing keto groups and/or hydroxyl groups, in a finely divided form and surface-active agents.

2. Agent according to Claim 1, characterised in that it contains monocyclic, dicyclic and/or polycyclic compounds containing two keto groups and/or two hydroxyl groups.

3. Agent according to Claims 1 and 2, characterised in that it contains 9,10-anthraquinone.

4. Agent according to Claims 1 to 3, characterised in that the organic, cyclic compounds containing keto groups and/or hydroxyl groups consist to the extent of at least 80% by weight of particles which have particle sizes of less than 10 μm.

5. Agent according to Claims 1 to 4, characterised in that the organic, cyclic compounds containing keto groups and/or hydroxyl groups consist to the extent of at least 80% by weight of particles which have a particle size of less than 5 μm.

6. Agent according to Claims 1 to 5, characterised in that it contains anionic surface-active agents.

7. Agent according to Claims 1 to 6, characterised in that it contains, as the surface-active agents, ligninsulphonates and/or condensation products of aromatic sulphonic acids and formaldehyde.

8. Agent according to Claims 1 to 7, characterised in that it consists of a pulverulent mixture which contains 30 to 98% by weight of one or more organic, cyclic compounds, containing keto groups and/or hydroxyl groups, and 2 to 70% by weight of one or more surface-active agents.

9. Agent according to Claims 1 to 7, characterised in that it consists of a dispersion which contains 30 to 70% by weight of one or more organic, cyclic compounds, containing keto groups and/or hydroxyl groups, 0.5 to 30% by weight of one or more surface-active agents and at least 30% by weight of water and/or organic water-miscible solvents having a boiling point above 80°C.

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10. Agent according to Claim 9, characterised in that it additionally contains preserving agents, agents which prevent drying out and/or agents for stabilising dispersions.

11. Process for pulp production from lignocellulose materials in the presence of organic, cyclic compounds containing keto groups and/or hydroxyl groups, characterised in that the organic, cyclic compounds containing keto groups and/or hydroxyl groups are employed in the form of an agent according to Claims 1 to 10.

12. Process according to Claim 11, characterised in that an agent according to Claims 1 to 10 is employed in an amount of 0.001 to 10 % by weight (relative to the lignocellulose material).

13. Process according to Claims 11 and 12, characterised in that 9,10-anthraquinone is employed in the form of an agent according to Claims 1 to 10.

14. The invention substantially as herein described.

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BAYER AKTIENGESELLSCHAFT,
By Its Patent Attorneys,
ARTHUR S. CAVE & CO.