(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number: **0 020 672**
B1

(12) # EUROPEAN PATENT SPECIFICATION

(45) Date of publication of patent specification: **29.02.84**

(51) Int. Cl.³: **D 06 M 13/24,**
C 04 B 31/00

(21) Application number: **80900011.0**

(22) Date of filing: **30.11.79**

(86) International application number:
PCT/DK79/00055

(87) International publication number:
WO 80/01176 12.06.80 Gazette 80/13

(54) **FIBER MATERIAL AND PROCESS FOR ITS PREPARATION.**

(30) Priority: **01.12.78 DK 5478/78**

(43) Date of publication of application:
07.01.81 Bulletin 81/1

(45) Publication of the grant of the patent:
29.02.84 Bulletin 84/9

(84) Designated Contracting States:
AT CH DE FR GB NL SE

(56) References cited:
DE - A - 2 402 661
GB - A - 869 952
US - A - 3 087 949
US - A - 3 243 447
US - A - 3 296 242
US - A - 3 546 262
US - A - 3 673 229

(73) Proprietor: **KJELD HOLBEK APS**
74, Lejrevej
DK-4320 Lejre (DK)

(72) Inventor: **HOLBEK, Kjeld**
Lejrevej 74
DK-4320 Lejre (DK)

(74) Representative: **Marchant, James Ian et al,**
Elkington and Fife High Holborn House 52/54
High Holborn
London WC1V 6SH (GB)

Courier Press, Leamington Spa, England.

EP 0 020 672 B1

Fibre material and process for its preparation

The present invention relates to the production of substantially discrete metal oxide acylate-treated fibres and to the use thereof; more particularly, it relates to such a process wherein the fibres are dried and are treated with a metal oxide acylate prior to, during or subsequent to dry-defibration.

GB—A—1604910 describes a process for impregnating cellulose fibres and fields of application of these impregnated fibres, especially the use of the fibres as reinforcing fibre element in composite materials based on organic or inorganic binders or a combination of organic and inorganic binders.

One class of the treatment or impregnation agents described in the above-mentioned patent application has been found to have especially valuable properties, as treatment with these can provide not only cellulose fibres, but also other fibre materials, with unexpected and extremely desirable property improvements. The fibres in question, which when treated with metal oxide acylates in accordance with the present invention, may be modified in the desired way as regards their properties, are cellulose fibres of any kind, animal fibres and synthetic fibres of both inorganic and organic origin, also including metal fibres.

The fibres in question may be discrete fibres or fibre bundles or "coarser" materials, such as wood vlies or wood wool.

Main groups of fibre materials which may be advantageously treated with metal oxide acylates in accordance with the present invention are the following:

1) Cellulose-based materials:

Finely divided wood such as wood wool, wood chips, shavings, cellulose fibres from the paper industry, including, e.g.

mechanical wood pulp
semichemical wood pulp
fully-chemical wood pulp and
other cellulose pulps of the types mentioned in the above GB—A—1604910;
chemical cellulose fibres (rayon);
recycled material, e.g. old papers, cardboard and paste-board;
vegetable fibres, e.g. flax, bast, coconut fibres, sisal.

2) Animal fibres, e.g. cattle's hair, camel hair, wool and pigs' bristles.

3) Synthetic organic fibres, e.g. fibres of polyamide, polyester, polyvinyl chloride.

4) Inorganic fibres (glass wool, slag wool, rock wool, kaolin wool, glass mono-filaments and fibres of e.g. iron, steel, brass and aluminium).

The effect obtained by treating the fibres in question with metal oxide acylates in accordance with the present invention is supposed to result primarily from the chemical reaction between reactive groups on the fibre surfaces and the metal oxide acylates, secondarily from the metal acylate groups fixed on the fibre surface by the reaction.

The effect obtained in the case of cellulose fibre is particularly interesting. This effect can be described as a stabilization of the cellulose fibre towards influence from the surroundings on a number of fibre properties. This stabilization includes a number of properties, and, according to the particular choice of metal oxide acylate, manifests itself in a blocking of the water absorption, an increase in the alkali resistance, an increase in rigidity, thermal stability, rot resistance and a decrease in inflammability and obtaining of antistatic properties. When treated with metal oxide acylates in accordance with the present invention both cellulose fibres and synthetic fibres will obtain a considerable improvement of the ability to disperse in water or solvents and a considerably increased coupling effect towards both organic and inorganic binders, which is of decisive importance when using the fibres in composite materials.

One of the most important applications of a number of the above-mentioned fibres in modified form is the composite field where the modified fibres may replace usually applied mineral fibres, such as asbestos. Cellulose fibres treated with metal oxide acylates in accordance with the present invention may be used in composite materials as a substitute for not only asbestos, but also glass wool, mineral fibres of the rock wool type, slag wool, kaolin wool, etc., but they are also advantageously applicable in composite materials already containing cellulose-based materials, e.g. paper and cardboard, and thus add new and improved properties to these materials.

On the other hand, mineral fibres treated with metal oxide acylates in accordance with the present invention also show improved properties when incorporated in composite materials, as their ability to become defibrated and uniformly dispersed in the final composite material has been found to be dramatically increased when they are treated with the metal oxide acylates in the present manner.

One of the most important improvements ascertained in the cellulose fibres treated with suitable metal oxide acylates in accordance with the present invention is a blocking or hindrance in the reaction of the fibre towards water. Cellulose fibres are hygroscopic *per se*, and on water absorption primarily an increase of volume caused by swelling occurs, which will result in disadvantageous internal material tensions in a composite material. When the tendency to swell is reduced or completely eliminated by the present treatment with a metal oxide acylate, composite materials containing such fibres obtain

correspondingly improved material properties.

As cellulose fibres modified with metal oxide acylates in accordance with the present invention appear as discrete fibres not having a tendency to conglomerate, the treated fibres *per se* may also be used directly, e.g. as insulating material in cavities or as stuffing in pillows, bedding, etc.

As described in GB—A—1604910 metal oxide acylates are a class of compounds invented by Dr. Jacobus Rinse and e.g. described in Belgian patent No. 555,969, Dutch patent No. 104,261, US Patents Nos. 3,987,949, 3,243,447, 3,177,238, 3,518,287, 3,625,934, 3,546,262, 3,346,674 and 3,673,229, Belgian patent No. 735,548 and British patents Nos. 1,230,412 and 1,274,718.

Some of such references will now be discussed in a little more detail.

US Patent No. 3,087,949 relates to metal oxide acylates of tetravalent metals, such as zirconium and tin, and states that they have surface active properties and that some of them are also useful for water-proofing and as driers for paints. For example, Example 1 relates to the preparation of a zirconium oxide stearate and states that the product has excellent water-proofing for textiles.

US Patent No. 3,296,242 relates to metal oxide acylates containing at least one trivalent element selected from aluminium and boron and at least one divalent metal, in other words, combined metal oxide acylates. This reference states that the products have outstanding pigment and surface-wetting characteristics and are useful in surface-coatings, metal lubricants, elastomers and plastic compositions. For example, Example 20 states that a 2 per cent solution of aluminium-zirconium compound is used to water-proof cotton and is found to give good results in water-proofing and in resistance to dry cleaning.

US Patent No. 3,546,262 relates to the preparation of divalent metal oxide acylates from virtually any divalent metal or combination of two such metals ans states that these metal oxide acylates are soluble in numerous organic solvents, with which liquid solutions containing from 60 to 90 percent, by weight, solids have frequently been obtained. When such solutions are applied to a solid substrate, they dry to tack-free, glossy protective coatings having excellent adherence. For example, Example 1 relates to a copper compound of this type and states that the product is a preservative for wood and burlap bags. Also, Example 11 states that a zinc compound is useful as a wood preservative. Furthermore Example 14 states that various compounds are useful as coating materials for glass fibres.

US Patent No. 3,673,229 relates to mixed metal oxide acylates of trivalent and divalent metals and states that the products are useful as resin additives, fungicides, anti-corrosives and colourants. For example, Examples 16 and 21 state that the respective products are useful as preservative stain for wood. Also Example 22 states that a similar compound (zinc-chromium compound) is useful as a wood preservative and anti-corrosive.

The metal oxide acylates comprise a large group of substances, as both metal and acylate groups may be varied within a wide range. The general composition of the metal oxide acylates is supposed to be

divalent metal $(R—Me—O—Me—R)_x$ linear compounds

trivalent metal $(O_3—Me_3—R_3)_y$ planar compounds

tetravalent metal $(O_6—Me_4—R_4)_z$ tetrahedron shaped-compounds

wherein Me is a metal atom, R is an acylate group which is an organic acid residue with at least 12 carbon atoms and x, y and $z = 3 - 7$ is a supposed molecular size in solution. It should be noted that metal oxide acylates wherein each molecule contains two or more different metals possibly of different valence, may also be produced, and such types of compounds are also described in the above-mentioned patent specifications.

The metal oxides tend to react with OH groups and other reactive groups present as free groups on the fibre surfaces. The reaction rate is supposed to decrease with increasing complexity of the molecular structure so that the metal oxide acylates of tetravalent metals require higher temperatures or more time in order to obtain reaction.

When choosing the metal oxide acylates to be used according to the present invention, the desired effects of the end product and the effects on the processing properties of the fibres when treated with the metal oxide acylate are to be considered. This may be illustrated by the treatment of cellulose fibre, as a suitable choice of the combination of metal and acylate residue in the metal oxide acylate may confer a number of further properties besides the obtainment of cellulose fibres which are easily dispersible and have an effective coupling to binders. Thus e.g. fire resistance may be obtained by using aluminium or iron oxide phthalates or metal oxide acylates wherein the metal is antimony, Zn- or Cu-oxide acylates provide rot resistance, Mg- and Fe-containing oxide acylates will usually increase to antistatic properties of the fibres, and titanium oxide phthalate will increase the rigidity of the fibres, while the use of styrene modified aliphatic acids in the acylate residue results in an improvement of the alkali-resistance of the fibres.

As the different metal oxide acylates are mutually fully compatible combined effects may be obtained by correct combination of the metal oxide acylate treatment agent.

In connection with the treatment of e.g. cellulose fibres and other fibres, where an increase of the water repellent properties is important, it is important that metal oxide acylates containing titanium show a considerably higher effect with respect to hydrophobization that other metal oxide acylates which make them applicable in considerably smaller concentrations and thus permit admixture of other

metal oxide acylates in order to obtain combination effects.

Examples of desired properties are e.g.: when the end product to be produced with the treated fibre is a composite material for electrical insulation, a minimization of the water sensitivity of the fibres is desired, whereas it would be advantageous with a moderate water absorption, when the fibres are to be used in connection with inorganic binders such as e.g. cement or plaster.

A possible explanation of the rot resistance-improving effect, which the metal oxide acylates impart to organic fibres including cellulose fibres, may be that the free alcohol groups on the surface of the native cellulose fibres play an important part in the initiation of both the bacterial and fungal deterioration mechanism, and that such free alcohol groups are blocked when the fibres are treated with a metal oxide acylate in accordance with the present invention.

According to the invention it is of special importance that a pre-treatment of the fibres with other types of wood impregnation or a treatment of the fibres with other types of wood impregnation together with the present treatment with metal oxide acylates does not seem to influence the effect of the metal oxide acylate treatment. Thus, it has been found possible to combine a treatment with e.g. paraffin with a treatment with e.g. aluminium oxide stearate, as these two treatment agents were applied from the same solution. Thus, it will also be possible to combine a treatment with metal oxide acylate in accordance with the present invention with simultaneous application of a polymer, e.g. a polyolefin, applied from the same solution.

Chlorinated paraffins and other materials of the wax type may also be combined with the metal oxide acylates and be applied on fibres from the same solution in accordance with the present invention.

Besides, it has been found that e.g. cellulose fibres may be pre-treated with inorganic compounds, e.g. inorganic wood preservation agents, especially the so-called "CZC", "CCZC" and "CCA" types, and thereafter successfully be treated with metal oxide acylate in accordance with the present invention, these cases, however, requiring an intermediate removal of water provided this has been used when impregnating with an inorganic preservation agent. A suitable way of removing the water is azeotropic distillation with e.g. xylene.

It has been found that the amount of metal oxide acylate usually to be used in accordance with the present invention in order to obtain the desired effect is normally within the range of 0.5—8% by weight of metal oxide acylate, calculated on the weight of the fibre, as in many cases a sufficient effect is obtained when adding between 1 and 4% by weight, calculated on the fibre.

In principle the treatment of fibre materials with metal oxide acylates in accordance with the present invention comprises a reaction with the metal oxide acylate on active groups on the fibre surface, and it is therefore usual to treat discrete fibres or fibre bundles instead of treating coherent fibre materials such as sheets, rolls, etc. Synthetic organic and inorganic fibres and animal fibres will normally be in the desired discrete form already from the producer, but this is not always the case with cellulose fibres. The cellulose fibres may e.g. be in the form of compact pulps such as raw cardboard from the wood cellulose manufacture or recycled cardboard and paper or semi-compact pulps such as the so-called "flash-dry" fibre from the wood cellulose production or as rags.

In such cases where the starting material is present in such a coherent or semi-coherent form the starting material must be submitted to a dry-defibration process. Such a dry-defibration process may be performed according to known methods wherein the starting material is transformed into loose fibres by defibration in an impact mill or hammer mill.

The dry defibration may be immediately combined with a treatment with a metal oxide acylate in accordance with the present invention, as the heat developed during the mechanical treatment may serve partly to dry out the cellulose fibres to the desired degree and partly to keep the temperature at a level where the reaction between the fibre surface and the metal oxide acylate proceeds rapidly, at the same time while the elevated temperature contributes to evaporation of the solvent in which the metal oxide acylate is applied. In accordance with the present invention, when, for example, a cellulose material is dry defibrated, the metal oxide acylate may also be applied immediately before the dry defibration, as the immediately subsequent mechanical treatment and temperature increase result in the fact that the fibres obtain the necessary dryness and the temperature necessary for the reaction with the metal oxide acylate.

The temperatures which are preferred for obtaining the reaction between the fibres and the metal oxide acylate will depend on the identify of the metal oxide acylate. Aluminium oxide stearate, for example, reacts reasonably rapidly already at temperatures between 20 and 40°C, while oxide acylates of titanium require a higher reaction temperature in the range of 40—80°C. In principle, any elevated temperature of up to about 200°C may be used, as the metal oxide acylates are perfectly capable of withstanding these temperatures, and thus it is often the temperature resistance of the fibres which is decisive for the temperature at which the present treatment is to take place.

Besides an application wherein the fibres are immersed in or wetted with a solution of metal oxide acylates, the metal oxide acylates may be applied e.g. by spraying while the fibres are in airborne condition immediately subsequent to the dry defibration in accordance with the present invention.

During the present treatment the amount of solution and its concentration is adapted to the desired degree of reaction, i.e. the amount of metal oxide acylate which it is desired to apply on the

fibre. The solvent is suitably a hydrocarbon or a chlorinated hydrocarbon which, for application in liquid phase, has suitably a boiling point which is higher than the reaction temperature at which the coupling process proceeds. Thus, an irregular depositing of the metal oxide acylate caused by a too rapid evaporation of the solvent is avoided. On the other hand it may be desirable for certain purposes to apply the metal oxide acylate in a solvent which evaporates very quickly, e.g. Freon such as described in GB—A—1604910. When the reaction between the fibres and the metal oxide acylate has proceeded, a filtration or centrifugation of the solvent and an evaporation of the solvent remaining on the fibres is performed, when a solvent with a considerably higher boiling point than the reaction temperature has been used, so that the fibres become practically free of the solvent.

It is of considerable importance for the successful reaction between the various types of fibres and metal oxide acylates in accordance with the present invention that the fibres are dry when treated, as the metal oxide acylates show a very high reactivity with water with formation of soaps and under such circumstances would not enter into the desired chemical reaction with reactive groups on the fibre surfaces. It has been found that a suitable dryness has been obtained, when the fibre materials have been dried to a constant weight at moderate temperatures, e.g. 40—60°C.

The fibres treated with metal oxide acylate in accordance with the present invention and optionally other agents may then in the usual way be incorporated in composite materials with inorganic or organic binder, and in this process it has been found that they are defibrated much more easily than corresponding fibres which have not been treated with metal oxide acylate. The composite material in question may be of the same kind as the materials described in GB—A—1604910. In such composite materials the present metal oxide acylate-treated fibres have been found to be distributed in the ideal uniform way, also in large concentrations, in contrast to the non-uniform distribution, often with lumps of non-defibrated material, which is often characteristic to the same materials when they have not been treated with metal oxide acylate in accordance with the present material.

The preparation of composite materials using fibres produced in accordance with the present invention is carried out in any known way for preparing such composite materials. When e.g. the composite material is prepared by wet defibration of the present metal oxide acylate-treated fibres and combination with an inorganic binder, it has been found to be suitable to flocculate the fibres together with the inorganic binder using a flocculating agent, e.g. a flocculating agent of the cationic type such as Prodefloc C1, which is a polyacrylamide product having attached quaternary ammonium groups. It has been found that when using the treated fibres a perfect "precipitation" of e.g. cement on the fibre surface is obtained together with a complete retention in the subsequent dehydration. The fibres treated according to the invention may also be used as a substitute for mineral fibres in the composite materials described in GB—A—1597369 and applications in other countries which claim priority from Danish patent application No. 5436/76.

It is evident that when treating the fibre material the metal oxide acylate in accordance with the present invention, any suitable impregnation method mentioned in GB—A—1604910 may be used, also including pressure impregnation and pressure/vacuum impregnation, and a dry defibration process may e.g. be combined with a pressure/vacuum impregnation by means of a suitable embodiment of a closed apparatus.

The invention is further illustrated in the following Examples.

Example 1

Treatment of a dry defibrated sulphate cellulose fibres with titanium oxide acylate.

50 g of sulphate cellulose fibres which had been dry defibrated in a hammer mill and (after intermediate storing) dried to a constant weight of 60°C, are treated with 440 ml of a solution in white spirit (50%) and extraction petrol (50%) containing the calculated amount, corresponding to the desired treatment, of partly titanium oxide acylate (Moaco D-42-00, wherein the acylate is a styrene-reacted conjugated fatty acid), and partly paraffin with a melting point of 55—60°C; in the calculations the fact that the fibres absorb 8 g of solvent per g of fibre has been taken into account.

After storing of the moistened fibres at room temperature for 2 hours, the reaction between the titanium oxide acylate and the fibres and drying out of the fibres is carried out by keeping the fibres at 100°C for 12 hours.

The resulting fibres are loose and cotton-like. Paper sheets are prepared from the fibres in a laboratory sheet former. The amounts of applied treatment agent and the test results appear from below table:

TABLE I

Test	1	2	3	4	5	6
Treatment addition, titanium oxide acylate paraffin	1%	2%	1%	1%	0%	0%
Effects on paper sheets:	0%	0%	2%	1%	2.5%	1%

The materials prepared according to tests 1—4 are felt-like without strength, strongly water-repellent and only moderately swelling when immersed in a 2N sodium hydroxide solution. The materials prepared according to tests 5—6 show almost normal strength, moderate water repellence and moderate swelling.

Example 2

Treatment of cellulose fibres combined with defibration in a hammer mill.

A hammer mill equipped with a feeder with adjustable feeding rate designed for supplying cellulose pulp in the form of webs is used for the treatment, an application device for the treatment medium having been placed immediately in front of the feeder. The applicatioin apparatus is an "Air-Less" flat spraying nozzle mounted in such a distance from the pulp web that the fan-shaped jet just moistens the web.

The supply of treatment medium is controlled by adjustment of the dosage from the nozzle and the rate of pulp supply.

Using the above arrangement two types of modified cellulose fibres are prepared:

1. 5% aluminium oxide stearate is added to 2500 g sulphate cellulose by spraying a solution (50%) thereof in a solution consisting of equal parts of white spirit and petroleum. Before the treatment the cellulose pulp has been dried to constant weight at 50°C for 18 hours. After the treatment the resulting fibres are dried out for 12 hours at 90°C in a ventilated oven in order to remove the remaining solvent.

2. A mixture of 4% aluminium oxide stearate and 4% liquid paraffin is added to 2500 g of sulphate cellulose. The admixture is carried out by spraying a mixture of 1.67 parts by weight of aluminium oxide stearate in solution (60%) in white spirit and 1 part by weight of liquid paraffin, 10% by weight of this mixture being supplied to the pulp. Also in this case, the fibres are dried as described above before and after defibration.

The admixture of the treatment chemicals is a considerable advantage when carrying out the dry defibration process. This advantage especially appeared in that the treated fibres had a considerably smaller content of cellulose dust compared to the untreated fibres — a fact that is observed immediately when handling the fibres.

Example 3

Examination of the effect of the treatment of cellulose fibres described in Example 2.

The immediate effect of the treatment of the cellulose fibres is a reduction of the reaction of the cellulose fibres to water.

This was demonstrated as follows:

Paper samples with a sheet weight of 250 g/m^2 were prepared by means of the generally known art for the preparation of paper sheets in laboratory sheet former, partly from untreated fibres, partly from a mixture of 75% of treated fibres and 25% of untreated fibres, an admixture of untreated fibres being necessary in order to obtain a paper sheet with the necessary coherency for the implementation of measurements.

The measurements used are determination of the shrinkage of the paper sheets, i.e. its longitudinal change at the transition from completely moistened to dry condition.

The results of this examination are as follows:

untreated fibres	4.8% shrinkage
Fibres with 5% aluminium oxide stearate added	1.7% shrinkage
Fibres with 4% aluminium oxide stearate and 4% liquid paraffin added	1.1% shrinkage

It appears that the shrinkage of the paper has been considerably reduced as a result of the treatments; the measured shrinkage is mainly to be ascribed to the presence of the untreated fibres.

Example 4

Examination of treated cellulose fibres as fibre reinforcement in a cement matrix.

The modified cellulose fibres described in Example 2 are used in these examinations.

The following composition and preparation method are used in the fibre-cement experiments:

Composition:

Cellulose fibre	16 g	6.78% by weight
Cement powder	220 g	93.22% by weight
Water	400 ml	i.e. a water/dry matter ratio in the mixture of 1.69

The amount used corresponds to one sheet with a dimension of 200 x 80 x 8 mm^3.

Process:

A "Braun" blender with 3 speeds is used as a mixing machine. Cement and water are mixed, whereafter fibre is added within 1 minute, and the mixing is continued for 2 minutes.

A flocculation agent, 10 ml of a solution (0.2%) of Prodefloc C1, is then added, and the mixing is continued for a further 0.5 minute.

The mixture is transferred to a filter box with the length/width-dimensions of the sheet to be obtained. After filtering off the water the filter cake is transferred to a form and compressed to the final thickness without further draining of water. It was noted that the filtered water is clear without contents of cement particles.

The compressed sheets are cured at room temperature and 100% relative humidity for 2 weeks, whereafter the sheets were air dried.

The bending strength of the produced sheets was examined in both air dried condition and wet condition, the wet condition being obtained by immersing the air dried sheets in water for 48 hours.

The results of the test are as follows:

Type of fibre	Bending strength kp/cm^2	
	Dry condition	Wet condition
Untreated	220	105
5% aluminium oxide stearate added	225	130
4% aluminium oxide stearate + 4% liquid paraffin added	230	150

It appears that the treatment of cellulose fibres has an effect which, as expected, especially appear by the obtainment of higher bending strengths of sheets in wet condition.

The words "Freon", "Moaco" and "Prodefloc" used in the above are Registered Trade Marks.

Claims

1. A process for the production of substantially discrete metal oxide acylate-treated fibres characterised in that the fibres are dried and are treated with a metal oxide acylate prior to, during or subsequent to dry-defibration.

2. A process as claimed in claim 1 characterised in that the dry-defibration is performed in a hammer mill.

3. A process as claimed in claim 1 or claim 2 characterised in that the dried fibres are treated with the metal oxide acylate prior to dry-defibration.

4. A process as claimed in claim 1 or claim 2 characterised in that the dried fibres are treated with the metal oxide acylate while airborne subsequent to dry-defibration.

5. A process as claimed in any of claims 1 to 4 characterised in that the drying is performed by heating to constant weight.

6. A process as claimed in any of claims 1 to 4 characterised in that the drying is performed by azeotropic distillation with a hydrocarbon.

7. A process as claimed in any of claims 1 to 6 characterised in that the metal oxide acylate is an aluminium oxide acylate or a titanium oxide acylate.

8. A process as claimed in any of claims 1 to 7 characterised in that the fibres are inorganic fibres or cellulose fibres.

9. Substantially discrete metal oxide acylate-treated fibres when produced by a process as

claimed in any of claims 1 to 8.

10. The use of substantially discrete metal oxide acylate-treated fibres as claimed in claim 9 in composite materials.

Patentansprüche

1. Verfahren zur Herstellung von im wesentlichen diskreten, mit Metalloxidacylat behandelten Fasern, dadurch gekennzeichnet, daß die Fasern getrocknet und mit einem Metalloxidacylat behandelt werden vor, während oder nach der Trocken-Defibrierung.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die Trocken-Defibrierung in einer Hammermühle durchgeführt wird.

3. Verfahren nach Anspruch 1 oder 2, dadurch gekennzeichnet, daß die getrockneten Fasern vor der Trocken-Defibrierung mit dem Metalloxidacylat behandelt werden.

4. Verfahren nach Anspruch 1 oder 2, dadurch gekennzeichnet, daß die getrockneten Fasern nach der Trocken-Defibrierung, während sie in der Luft schweben, mit dem Metalloxidacylat behandelt werden.

5. Verfahren nach einem der Ansprüche 1 bis 4, dadurch gekennzeichnet, daß die Trocknung durch Erhitzen bis zur Gewichtskonstanz durchgeführt wird.

6. Verfahren nach einem der Ansprüche 1 bis 4, dadurch gekennzeichnet, daß die Trocknung durch azeotrope Destillation mit einem Kohlenwasserstoff durchgeführt wird.

7. Verfahren nach einem der Ansprüche 1 bis 6, dadurch gekennzeichnet, daß das Metalloxidacylat ein Aluminiumoxidacylat oder ein Titanoxidacylat ist.

8. Verfahren nach einem der Ansprüche 1 bis 7, dadurch gekennzeichnet, daß die Fasern anorganische Fasern oder Cellulosefasern sind.

9. Im wesentlichen diskrete, mit Metalloxidacylat behandelte Fasern, hergestellt nach einem Verfahren nach einem der Ansprüche 1 bis 8.

10. Verwendung der im wesentlichen diskreten, mit Metalloxidacylat behandelten Fasern gemäß Anspruch 9 in Verbundmaterialien.

Revendications

1. Un procédé pour la production de fibres sensiblement discrètes traitées par un acylate d'oxyde de métal, caractérisé en ce que les fibres sont séchées et sont traitées par un acylate d'oxyde de métal avant, pendant ou après défibration à sec.

2. Un procédé selon la revendication 1, caractérisé en ce que la défibration à sec est effectuée dans un broyeur à marteaux.

3. Un procédé selon la revendication 1 ou 2, caractérisé en ce que les fibres séchées sont traitées par l'acylate d'oxyde de métal avant la défibration à sec.

4. Un procédé selon la revendication 1 ou 2, caractérisé en ce que les fibres séchées sont traitées par l'acylate d'oxyde de métal tandis qu'elles sont supportées par l'air après la défibration à sec.

5. Un procédé selon l'une quelconque des revendications 1 à 4, caractérisé en ce que le séchage est effectué par chauffage à poids constant.

6. Un procédé selon l'une quelconque des revendications 1 à 4, caractérisé en ce que le séchage est effectué par distillation azéotropique avec un hydrocarbure.

7. Un procédé selon l'une quelconque des revendications 1 à 6, caractérisé en ce que l'acylate d'oxyde de métal est un acylate d'oxyde d'aluminium ou un acylate d'oxyde de titane.

8. Un procédé selon l'une quelconque des revendications 1 à 7, caractérisé en ce que les fibres sont des fibres inorganiques ou des fibres de cellulose.

9. Des fibres sensiblement discrètes traitées par un acylate d'oxyde de métal quand elles sont produites par un procédé selon l'une quelconque des revendications 1 à 8.

10. L'utilisation de fibres sensiblement discrètes traitées par un acylate d'oxyde de métal selon la revendication 9 dans des matières composites.