

The present invention relates to partially crystalline, substantially spherical particles of cellulose esters of aromatic or aromatic-aliphatic carboxylic acids, to a process for their preparation, and to the use thereof as stationary phase in chromatographic methods.

Microcrystalline cellulose triacetate, which is obtainable by heterogeneous esterification, has been widely used with success for the separation of racemates into their stereoisomers by chromatography. Cellulose, other polysaccharides and derivatives thereof are also suitable for use as stationary phase for such separations, q.v. T. Shibata et al., J. of Liquid Chrom., 9, 313-40 (1986).

A chromatographic separating material consisting of macroporous silica gel coated with cellulose triacetate or cellulose tribenzoate is described by Y. Okamoto et al. in Chemistry Letters, pp. 739-742 (1984). This carrier material is expensive. Moreover, only the layer of the cellulose derivative is available for the resolution of the enantiomers, so that the separating ability in respect of capacity is not entirely satisfactory. In J. of Chromatography, 351, pp. 346-350 (1986), K.-H. Rimböck proposes the use of powdered microcrystalline tribenzoyl cellulose, which is obtained by simple precipitation from a solution, as stationary phase. The separating ability of the powdered material is insufficient, and the utility is limited to the solving of analytical problems. In addition, the particle size and external particle form cannot be controlled in this method.

Finely particulate and porous cellulose ester beads of aromatic carboxylic acids, which can be used for resolving racemates, are described in EP-A-0 147 804. However, no preparation process is indicated, particularly for pressure-stable and partially crystalline porous cellulose ester beads which are particularly suitable for resolving racemates.

A process for the preparation of spherical, porous cellulose particles is described in EP-A-0 025 639. In this process, an organic solution of cellulose triacetate which contains a longer-chain alcohol is suspended, with stirring, in an aqueous phase that contains a higher molecular

weight surfactant, e.g. polyvinyl alcohol or gelatin. After removal of the organic solvent, the cellulose triacetate particles obtained are isolated and purified and then hydrolysed. Our own investigations have revealed that the cellulose triacetate particles obtained by these processes have only a low specific surface area and, as stationary phase in methods of liquid chromatography, have only a limited ability to resolve racemates.

The invention relates to finely particulate cellulose esters of aromatic or aromatic-aliphatic carboxylic acids in the form of substantially spherical, partially crystalline particles having an average diameter of 1 to 200 μm , a specific surface area of 10 to 300 m^2/g , measured in accordance with the BET method, and a calorimetrically determined heat of fusion of 1 to 50 J/g.

The average particle diameter is preferably 3 to 100 μm , in particular 5 to 80 μm . For analytical separations, an average particle diameter of about 5 to 50 μm , and for preparative separations of about 10 to 100 μm , has proved useful. The average particle diameter can also comprise only a fairly narrow range. Such materials can be prepared by fractionation by conventional methods, for example sedimentation, sieving or sifting.

The specific surface area is preferably 20 to 80 m^2/g , in particular 30 to 70 m^2/g .

Surprisingly, the finely particulate cellulose esters of this invention exhibit the partially crystalline character suitable for the chromatographic separation ability. The relative crystallinity can be indicated by the heat of fusion of the partially crystalline components of the cellulose esters, which is suitably determined by calorimetry, for example differential scanning calorimetry (DSC). The heat of fusion is preferably 2 to 50 J/g, in particular 5 to 30 J/g and, especially, 10 to 25 J/g.

The carboxylic acids from which the cellulose esters are derived preferably have the formula R-X-COOH , in which X is a direct bond, $\text{C}_1\text{-C}_4$ alkylene, $\text{C}_2\text{-C}_4$ alkenylene, $\text{C}_2\text{-C}_4$ alkylidene or $\text{C}_2\text{-C}_4$ alkynylene, and R

is monocyclic, bicyclic or tricyclic, preferably monocyclic, C₆-C₁₄aryl or heteroaryl, each having 5 or 6 ring atoms and one to three, preferably one or two, hetero atoms selected from the group consisting of N, O or S, R being unsubstituted or monosubstituted or polysubstituted by

C₁-C₁₂alkyl, preferably C₁-C₄alkyl, C₁-C₁₂alkoxy, preferably C₁-C₄alkoxy, C₁-C₁₂alkylthio, preferably C₁-C₄alkylthio, phenyl, phenoxy, phenylthio, benzyl, benzoxy, benzylthio, cyano, halogen, C₁-C₈acyl, C₁-C₈acyloxy, hydroxyl, C₁-C₁₂alkoxycarbonyl, phenoxy carbonyl, benzoxy carbonyl, nitro or di(C₁-C₆alkyl)amino. Preferred substituents are chlorine, methyl, ethyl and methoxy.

X as alkylene preferably contains 1 to 3 and, in particular, 1 or 2, carbon atoms. X as alkenylene, alkylidene or alkynylene preferably contains 2 or 3 carbon atoms. Alkylene, alkenylene and alkynylene can be linear or branched. Examples of such groups are methylene, ethylene, ethylidene, ethenylene, ethynylene, 1,2- or 1,3-prop-ylene, 1,1- or 2,2-propylidene, 1,2- or 1,3-prop-1-enylene or 1,2- or 1,3-prop-2-enylene, 1,3-propynylene, 1,2-, 1,3-, 1,4- or 2,3-butylene, 1,1- or 2,2-butylidene, 1,2-, 1,3- or 1,4-but-1-enylene, 1,2-, 1,3- or 1,4-but-2-enylene or 1,2-, 1,3- or 1,4-but-3-enylene, 1,2-, 1,3- or 1,4-but-1-ynylene, 1,2-, 1,3- or 1,4-but-2-ynylene or 1,2-, 1,3- or 1,4-but-3-ynylene. Particularly preferably X is C₁-C₃alkylene, C₂-C₃alkylidene or C₂-C₃alkenylene or a direct bond. In particular, X is a direct bond, methylene, ethylene or ethenylene. In a preferred embodiment of the invention, X is a direct bond or 1,2-ethenylene.

R as aryl preferably contains 6 to 10 carbon atoms. The aryl can be condensed with alkylene or alkenylene of 2 to 4 carbon atoms. Examples of aryl are phenyl, naphthyl, phenanthryl, anthracyl, indenyl, indanyl, fluorenyl, fluorenonyl, anthraquinonyl, xanthonyl, thioxanthonyl. Preferably R is naphthyl and, in particular, phenyl.

R as heteroaryl preferably contains 1 hetero atom selected from the group consisting of O, S and N. The heteroaryl can be condensed with phenylene. Examples of heteroaryl are furyl, thionyl, pyrrolyl, benzofuryl, benzthionyl, indyl, pyridyl, pyrimidyl, quinoliny and

isoquinolinyl. Pyridyl is particularly preferred.

The radical R can be monosubstituted or polysubstituted, preferably mono- to trisubstituted, in particular monosubstituted or disubstituted by C₁-C₁₂alkyl, in particular C₁-C₄alkyl, C₁-C₁₂alkoxy, in particular C₁-C₄alkoxy, or C₁-C₁₂alkylthio, in particular C₁-C₄alkylthio, for example methyl, ethyl, n- or isopropyl, n-butyl, isobutyl or tert-butyl, pentyl, hexyl, heptyl, octyl, nonyl, decyl, undecyl, dodecyl and corresponding alkoxy or alkylthio radicals, with methyl, ethyl, methoxy and ethoxy being particularly preferred; phenyl, phenoxy, phenylthio, benzyl, benzoxy and benzylthio, preferably phenyl or benzyl; hydroxyl, cyano, halogen, in particular, F, Cl and Br; C₁-C₈acyl or C₁-C₈acyloxy, preferably C₁-C₄acyl or C₁-C₄acyloxy, for example acetyl, chloroacetyl, dichloroacetyl, trichloroacetyl, fluoroacetyl, trifluoroacetyl, propionyl, butanoyl, benzoyl and phenylacetyl, and corresponding acyloxy radicals; C₁-C₁₂alkoxycarbonyl, preferably C₁-C₄alkoxycarbonyl, phenoxycarbonyl and benzoxy carbonyl.

In a preferred embodiment of the invention, the substituents are C₁-C₄alkyl or halogen, in particular methyl, ethyl, F or Cl.

In a preferred embodiment of the invention, X is a direct bond, C₁-C₃alkylene or C₂-C₃alkenylene, and R is unsubstituted or substituted phenyl or pyridyl. Particularly preferably, X is a direct bond, methylene, ethylene and ethenylene.

The invention further relates to a process for the preparation of the cellulose esters of the invention which comprises slowly adding, with efficient stirring, a solution of a cellulose ester of aromatic or aromatic-aliphatic carboxylic acids in an organic solvent that contains 1 to 50 % by volume, based on the amount of the solvent, of a C₃-C₂₂alkanol, to a solution of an anionic surfactant in water, removing the organic solvent with continued stirring, isolating the solid particles and washing and drying them.

The cellulose esters can be prepared by known methods of esterification. The degree of polymerisation can be from 5 to 1000, preferably from 5 to

500 and, in particular, from 10 to 50.

The degree of esterification of the cellulose ester can be 1 to 3, preferably 2 to 3 and, in particular, 2.5 to 3. The attainable degree of esterification can depend on the reactivity of the carboxylic acid used.

The particle size can be influenced by the stirring rate, which can be, for example, 100 to 1000 rpm, and by the amount of cellulose ester in the organic solvent and the rate of addition of the organic solution to the aqueous phase.

The specific surface area can be influenced by the reaction conditions, for example the choice of solvent, alkanol and surfactant, by the reaction parameters, for example the rate of addition and stirring, the evaporation rate, and by the ratios of solvent, water, alkanol, surfactant and cellulose ester.

The amount of cellulose ester in the organic solvent can be, for example, 1 to 20 % by weight, preferably 1 to 12 % by weight, based on the solvent.

It can be advantageous to use a substantially water-insoluble solvent.

The organic solvent preferably has a lower boiling point than that of water. Examples of suitable solvents are aromatic hydrocarbons, halogenated hydrocarbons, in particular fluorinated and/or chlorinated hydrocarbons, cyclic ethers, carboxylic acid esters and ketones. Examples of such solvents are benzene, toluene, xylene, methylene chloride, chloroform, trichlorofluoromethane, chloroethane, trifluorotrichloroethane, dichlorotetrafluoroethane, acetone, methyl ethyl ketone, diethyl ketone, cyclohexanone, tetrahydrofuran and dioxane or mixtures of such solvents. The particularly preferred solvents are methylene chloride and tetrahydrofuran.

It is also possible to use solvents that have a boiling point higher than that of water, for example, toluene, xylene, chlorobenzene,

ethylene glycol or diethylene glycol ether. The solvent is then removed together with water. In doing so it is expedient to replace the water that has been removed, for example during the distillation, or to use a correspondingly larger amount of water.

The organic solution preferably contains an alkanol in an amount of 5 to 40 % by volume. The alkanol can be branched and, preferably, linear. It preferably contains 7 to 16 and, in particular, 7 to 12, carbon atoms. Examples of such alkanols are pentanol, hexanol, heptanol, octanol, nonanol, decanol, undecanol, dodecanol, tridecanol, tetradecanol, hexadecanol, octadecanol and eicosanol. The alkanols are preferably substantially water-insoluble. Especially preferred alkanols are linear C₇-C₁₀alkanols.

The volume ratio of the organic solution to the aqueous phase is preferably 10:1 to 1:10, in particular 5:1 to 1:5 and, especially, 3:1 to 1:3.

The anionic surfactant is preferably present in the aqueous solution in an amount of 0.05 to 10 % by weight, preferably 0.1 to 5 % by weight, based on the amount of water.

The anionic surfactant can be, for example, an acid, a semiester of dibasic to tetrabasic acids, in particular dibasic or tribasic acids or salts thereof. Examples of suitable salts are ammonium salts, alkali metal salts (in particular sodium and potassium salts), or alkaline earth metal salts (in particular magnesium and calcium salts).

The acids and semiesters contain, for example, linear or branched C₃-C₂₀alkyl groups or phenyl groups which are substituted by 1 to 3 C₁-C₂₀alkyl groups. The acids can also contain C₁-C₂₀perfluoroalkyl groups. Examples of suitable acids are mono- or dicarboxylic acids, sulfonic acids, phosphonic acids and phosphinic acids. Suitable polybasic acids for semiesters are, for example, sulfuric acid, phosphoric acid, phosphorous acid, malonic acid and maleic acid. A great number of such surfactants are described in the National Standard Reference Data System (NSRDS), Nat. Bur. Stand. (U.S.) 36, pp. 24-32,

U.S. Government Printing Office (1971). Preferred surfactants are C_8 - C_{16} sulfuric acid semiesters, in particular lauryl sulfate.

The process can be carried out such that the solution of the cellulose ester is added dropwise, with stirring, to the aqueous phase and subsequently, with continued stirring, the organic solvent is removed, expediently by distillation with heating up to the boiling point of the solvent and/or under vacuum. The particles are then isolated, for example by decantation or filtration. The particles are then purified, for example by washing with water and a hydrophilic solvent, advantageously with a C_1 - C_4 alkanol. The material is then dried.

Surprisingly, the process affords cellulose esters of aromatic or aromatic-aliphatic carboxylic acids in the form of substantially spherical particles which have a high porosity (specific surface area) and a partially crystalline character, which is important for the separation of enantiomers by chromatography. The use of macroporous carrier material (e.g. silica gel Si 1000/4000) can be avoided. The separating efficiency is surprisingly good and better than that of comparable products. It is advantageous that higher loadings can be used. It is particularly surprising that the cellulose esters of this invention have an exceedingly high pressure stability, which so far has only been possible with cellulose esters coated on silica gel carriers [q.v. Chemistry Letters, pp. 739-742 (1984)]. This high pressure stability makes the material particularly suitable for high-performance liquid chromatography.

The invention further relates to the use of the cellulose esters of the invention as stationary phase in chromatographic methods, especially for resolving racemates.

The following examples illustrate the invention in more detail. Percentages are by weight, unless otherwise stated.

A) Preparation Examples

Example 1: 5 g of cellulose tribenzoate in 150 ml of methylene chloride

are treated with 17 ml of heptanol. The resultant solution is added dropwise, at room temperature, to a 0.75 % solution of sodium lauryl sulfate (300 ml) which is stirred at 400 rpm. At the same rate of stirring, the methylene chloride is subsequently removed by evaporation at 40-42°C (bath temperature). The residue is isolated by filtration and washed with water and ethanol. The powdered product is dried in a vacuum drier at 80°C (20 hours). Yield: 4.8 g (96 % of theory). The spherical particles having a diameter of 10-30 μm can be fractionated, as required, by sieving or sedimentation. The physical properties of the product are determined as follows:

Specific surface area: 59.6 m^2/g .

The specific surface area (m^2/g) is determined by the BET method.

Crystallinity: 19.3 J/g.

The relative crystallinity is determined by DSC (differential scanning calorimetry; Mettler TA3000) from the heat of fusion ΔH (J/g).

Example 2: The process of Example 1 is repeated with different compositions and yields the products listed in Table 1.

Table 1

Benzoyl cellulose (g)	CH_2Cl_2 (ml)	n-Hept- anol (ml)	SLS ¹⁾ (%, ml)	Specific surface area (m^2/g)	DSC ΔH (J/g)	Dia- meter (μm)
36.6	1000	124	0.75, 2200	43.7	17.2	10-20
5	150	25	0.75, 300	64.2	18.9	10-30
5	75	25	0.75, 120	50.3	19.6	20-30
5	150	37.5	0.75, 300	40.9	17.6	10-20
50	750	150	0.75, 1200	46.5	15.4	20-30
4	120	20	1.50, 88	55.0	15.3	3-8
4	120	20	0.10, 88	63.1	15.6	5-15

1) SLS: solution of sodium lauryl sulfate in water.

Example 3: In accordance with the procedure described in Example 1, 6 g of cellulose tribenzoate in 180 ml of CHCl_3 are treated with 30 ml of heptanol and 145 ml of a 0.75 % solution of sodium lauryl sulfate. The chloroform is removed by evaporation at 65°C . The powdery material is isolated by filtration and washed with water and then with ethanol, and subsequently dried. Yield: 95 % of theory. (Specific surface area: $59.9 \text{ m}^2/\text{g}$; ΔH : 14.45 J/g ; diameter: $5\text{-}10 \text{ }\mu\text{m}$).

Example 4: In accordance with the procedure described in Example 1, 5 g of cellulose tribenzoate in 150 ml of methylene chloride are treated with 17 ml of decanol and 300 ml of a 0.75 % solution of sodium lauryl sulfate and the product is isolated. (Specific surface area: $31.6 \text{ m}^2/\text{g}$; ΔH : 16.0 J/g ; diameter: $10\text{-}20 \text{ }\mu\text{m}$).

Example 5: In accordance with the procedure described in Example 1, 2 g of cellulose tribenzoate in 60 ml of methylene chloride are treated with 10 ml of heptanol and 48 ml of a solution of sodium dodecylbenzyl-sulfonic acid (1 % in water) and the product is isolated. (Specific surface area: $53.2 \text{ m}^2/\text{g}$; ΔH : 15.8 J/g ; diameter: $5\text{-}10 \text{ }\mu\text{m}$).

Example 6: In accordance with the procedure described in Example 1, 10 g of cellulose tri(paramethylbenzoate) in 300 ml of methylene chloride are treated with 50 ml of heptanol and 240 ml of a 0.7 % solution of sodium lauryl sulfate and the product is isolated. (Specific surface area: $68.7 \text{ m}^2/\text{g}$; ΔH : 10.6 J/g ; diameter: $5\text{-}10 \text{ }\mu\text{m}$).

Example 7: In accordance with the procedure described in Example 1, 10 g of cellulose tri(metamethylbenzoate) in 300 ml of methylene chloride are treated with 50 ml of heptanol and 240 ml of a 0.7 % solution of sodium lauryl sulfate and the product is isolated. (Specific surface area: $57.8 \text{ m}^2/\text{g}$; ΔH : 12.7 J/g ; diameter: $10\text{-}20 \text{ }\mu\text{m}$).

Example 8: In accordance with the procedure described in Example 1, 5 g of cellulose tricinnamate in 150 ml of methylene chloride are treated with 45 ml of heptanol and 120 ml of a 0.1 % solution of sodium lauryl sulfate and the product is isolated. (Specific surface area: $28.7 \text{ m}^2/\text{g}$; ΔH : 8.2 J/g ; diameter: $5\text{-}20 \text{ }\mu\text{m}$).

Example 9: 5 g of o-methylbenzoyl cellulose in 300 ml of tetrahydrofuran are treated with 12 ml of heptanol. The polymer solution is added dropwise at room temperature to a 0.7 % solution of sodium lauryl sulfate in water (300 ml) which is stirred at 400 rpm. At the same rate of stirring, the tetrahydrofuran is subsequently distilled off at 70-75°C (bath temperature). The residue is isolated by filtration and washed with water and ethanol. The product is dried in a vacuum drier at 80°C. Yield: 4.9 g (98 % of theory). (Specific surface area: 36.1 m²/g; ΔH : 2.6 J/g; diameter: 5-20 μm).

Example 10: In accordance with the procedure described in Example 1, 3 g of p-ethylbenzoyl cellulose in 180 ml of methylene chloride are treated with 30 ml of heptanol and 180 ml of a 0.7 % solution of sodium lauryl sulfate and the product is isolated. (Specific surface area: 28.6 m²/g; ΔH : 2.8 J/g; diameter: 3-5 μm).

Example 11: In accordance with the procedure described in Example 9, 5 g of p-chlorobenzoyl cellulose in 150 ml of tetrahydrofuran are treated with 37.5 ml of heptanol and 300 ml of a 0.7 % solution of sodium lauryl sulfate and the product is isolated. (Specific surface area: 68.3 m²/g; ΔH : 12.7 J/g; diameter: 20-40 μm).

Example 12: In accordance with the procedure described in Example 9, 5 g of m-chlorobenzoyl cellulose in 150 ml of tetrahydrofuran are treated with 35 ml of heptanol and 300 ml of a 0.7 % solution of sodium lauryl sulfate and the product is isolated. (Specific surface area: 38.9 m²/g; ΔH : 10.8 J/g; diameter: 5-15 μm).

B) Use Examples

A number of the prepared products are packed into a steel column (25 cm x 0.46 cm) by the slurry method and tested with various test racemates.

Definition of the capacity factor k' and of the separation factor α :

$$\text{capacity factor (k')} = \frac{\left(\begin{array}{c} \text{retention volume of} \\ \text{the enantiomer} \end{array} \right) - \left(\begin{array}{c} \text{retention volume of a} \\ \text{non-retained compound} \end{array} \right)}{\left(\begin{array}{c} \text{retention volume of a} \\ \text{non-retained compound} \end{array} \right)}$$

$$\text{separation factor } (\alpha) = \frac{\text{capacity factor of the 2nd eluted enantiomer}}{\text{capacity factor of the 1st eluted enantiomer}}$$

The HPLC operations are carried out with a 110 B (Beckman) pump. Detection is made by UV spectroscopy (Shimadzu UV-120-02, fitted with HPLC cell No. 1200) and polarimetry (Perkin Elmer 241 LC). The conditions of the chromatography are: hexane/2-propanol (9:1) as eluant; rate of flow 1 ml/min; temperature 25°C. The results are reported in Tables 2 to 5.

Table 2: Resolution of enantiomers by chromatographing racemic compounds on the product of Example 1

Racemate	Capacity factor		Separation factor α
	k'_1	k'_2	
phenylethanol	1.29	2.42	1.88
phenylethanediol	2.0	2.86	1.43
phenyl vinyl sulfone	7.91	12.71	1.60
butane-1,3-diol diacetate	3.64	7.19	1.98
tetrahydro-1-naphthol	0.94	2.06	2.19

Table 3: Resolution of enantiomers by chromatographing racemic compounds on the product of Example 7

Racemate	Capacity factor		Separation factor α
	k'_1	k'_2	
trans-stilbene oxide	3.67	11.60	3.10
1-naphthylethanol	8.67	11.5	1.33

Table 4: Resolution of enantiomers by chromatographing racemic compounds on the product of Example 8

Racemate	Capacity factor		Separation factor
	k'_1	k'_2	α
1-naphthyl ethanol	10.3	11.96	1.16
methyl mandelate	13.07	15.11	1.16

Table 5: Resolution of enantiomers by chromatographing racemic compounds on the product of Example 6

Racemate	Capacity factor		Separation factor
	k'_1	k'_2	α
1-naphthylethanol	8.64	11.78	1.36
benzoin	11.52	15.18	1.32
Troegeer base	2.62	23.76	9.07



Patent claims

1. A finely particulate cellulose ester of an aromatic or aromatic-aliphatic carboxylic acid in the form of substantially spherical, partially crystalline particles having an average diameter of 1 to 200 μm , a specific surface area of 10 to 300 m^2/g , measured in accordance with the BET method, and a calorimetrically determined heat of fusion of 1 to 50 J/g.
2. A cellulose ester according to claim 1, having an average diameter of 3 to 100 μm .
3. A cellulose ester according to claim 1, having an average diameter of 5 to 80 μm .
4. A cellulose ester according to claim 1, having a specific surface area of 20 to 80 m^2/g .
5. A cellulose ester according to claim 1, having a specific surface area of 30 to 70 m^2/g .
6. A cellulose ester according to claim 1, having a heat of fusion of 5 to 30 J/g.
7. A cellulose ester according to claim 1, having a heat of fusion of 10 to 25 J/g.
8. A cellulose ester according to claim 1, in which the carboxylic acid has the formula



in which X is a direct bond, $\text{C}_1\text{-C}_4$ alkylene, $\text{C}_2\text{-C}_4$ alkenylene, $\text{C}_2\text{-C}_4$ alkylidene or $\text{C}_2\text{-C}_4$ alkynylene, and R is monocyclic, bicyclic or tricyclic $\text{C}_6\text{-C}_{14}$ aryl or heteroaryl, each having 5 or 6 ring atoms and one to three hetero atoms selected from the group consisting of N, O or S, R being unsubstituted or monosubstituted or polysubstituted by $\text{C}_1\text{-C}_{12}$ alkyl, $\text{C}_1\text{-C}_{12}$ alkoxy, $\text{C}_1\text{-C}_{12}$ alkylthio, phenyl, phenoxy, phenylthio, benzyl, benzoxy, benzylthio, cyano, halogen, $\text{C}_1\text{-C}_8$ acyl, $\text{C}_1\text{-C}_8$ acyloxy, hydroxyl,



C_1 - C_{12} alkoxycarbonyl, phenoxycarbonyl, benzyloxycarbonyl, nitro or di-
(C_1 - C_6 alkyl)amino.

9. A cellulose ester according to claim 8, in which X is a direct bond, C_1 - C_3 alkylene or C_2 - C_3 alkenylene, and R is unsubstituted or substituted phenyl, naphthyl or pyridyl.

10. A cellulose ester according to claim 9, in which R is unsubstituted or substituted phenyl, naphthyl or pyridyl, and X is a direct bond, methylene, ethylene or ethenylene.

11. A process for the preparation of a cellulose ester according to claim 1, which comprises slowly adding, with efficient stirring, a solution of a cellulose ester of an aromatic or aromatic-aliphatic carboxylic acid in an organic solvent that contains 1 to 50 % by volume, based on the amount of the solvent, of a C_5 - C_{22} alkanol, to a solution of an anionic surfactant in water, removing the organic solvent with continued stirring, isolating the solid particles and washing and drying them.

12. A process according to claim 11, wherein the organic solvent has a lower boiling point than that of water.

13. A process according to claim 12, wherein the solvent is an aromatic hydrocarbon, a halogenated hydrocarbon, a cyclic ether, a carboxylic acid ester or a ketone.

14. A process according to claim 11, wherein the alkanol contains 7 to 16 carbon atoms.

15. A process according to claim 14, wherein the alkanol contains 7 to 12 carbon atoms.

16. A process according to claim 11, wherein the alkanol is present in an amount of 5 to 40 % by volume, based on the amount of the organic solvent.



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17. A process according to claim 11, wherein the surfactant is an acid, a semiester of dibasic to tetrabasic acids or salts thereof.

18. A process according to claim 11, wherein the surfactant is lauryl sulfate.

19. A process according to claim 11, wherein the volume ratio of the organic solution to the aqueous phase is from 10:1 to 1:10.

20. Use of a cellulose ester according to claim 1 as stationary phase in chromatography, especially for resolving racemates.

21. A finely particulate cellulose ester of an aromatic or aromatic-aliphatic carboxylic acid, obtainable by the process according to claim 11.