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(54) **Reaktív szolok és eljárás előállításukra**

The invention relates to reactive sols and methods for the manufacture thereof according to the preambles of claims 1 and 16.

The sol is characterized in that it contains at least one monomer and/or at least one oligomer having 2 to 15 repeating structural units which comprise at least two functional chemical groups as well as at least one intercalating agent likewise having at least two reactive chemical groups, where the intercalating agent is selected so that its functional groups do not react with the functional groups of the monomer and/or oligomer.

Dispersions in water, solvents or polyether alcohols known from the prior art are usually characterized by particle sizes of 100 nm to far beyond 1 µm. Thus, the manufacture of micro-emulsions having particles which have a plurality of functional groups is known. Such micro-emulsions having particles with diameters of about 5 to 100 µm are manufactured, for example, from bifunctional primary, optionally N- or S-containing alcohols and diisocyanates in higher-molecular, liquid polyaddition or polycondensation products containing exclusively secondary hydroxyl groups at temperatures between 60 and 80°C. Instead of alcohols, compounds with two primary or secondary amino groups can also be used and dispersed polyurea particles are obtained in this way.

US 2007/0238796 A1 describes the manufacture of stable microdispersion polyols, where polyols with polyisocyanate polyaddition products are obtained by reacting one or more isocyanate components with at least one compound containing an amino group, e.g. amines, alkanoamines or mixtures thereof as well as polyether polyols in the presence of urea and water. The urea acts as a stabilizer for the dispersion.

Since the properties of polymer materials are not only determined by the structural components of the macromolecules but to a high degree by additives including reactive or inert fillers, the form and type of these fillers has attracted great attention. Thus, the use of metal oxide nanoparticles in foams is described in DE 10 2005 003 299 A1. Polyurea dispersion polyols can be obtained by depolymerization of poly(urethane ureas) such as are present, for example, in the form of polyurethane soft foams (DE 10 2009 000604 A1). By reacting poly(urethane ureas) in one or more glycols, optionally with the addition of polyether alcohols, in the presence of secondary aliphatic amines or polyamines, polyurea particles dispersed in polyether alcohols are obtained.

However, in order to achieve a high effect of organic particles, discrete, completely solvatized particles are required which are present freely in a solvation agent acting as an intercalating agent and which are segregated from one another by this, so that no aggregation of the discrete particles can be made and these are intercalated by the solvating agent.

It is therefore the object of the invention to accordingly provide reactive sols with functionalized intercalated particles which can be manufactured by means of a simple process and which can be further processed to form end products.

The object is solved with the features of claims 1 to 16.

Advantageous further developments are specified in the subclaims.

Accordingly, a reactive sol according to the invention containing a liquid phase and nanoscale particles with functional groups is characterized in that the particles have a maximum size distribution of < 50 nm and a particle contains at least one monomer and/or at least one oligomer with 2 to 15 repeating structural units,

wherein the monomer and/or the oligomer is an adduct E from a compound A,

selected from one of the groups of diisocyanates, dicarboxylic acids, diepoxides and phenols with at least two A-end groups and

a compound D,

selected from one of the groups of diols, triols, diamines, triamines, polyamines, aldehydes or mixtures of these compounds with at least two D-end groups,

and the liquid phase contains a compound B acting as an intercalating agent selected from one of the groups of diols, triols, polyols, diamines, triamines, polyamines, amino-alcohols, dithiols and aldehydes having at least two B-end groups,

wherein the A-end groups are carboxyl, epoxy, isocyanate, phenol groups, the B-end groups are hydroxyl, amino, aldehyde groups and the D-end groups are hydroxyl, amino, aldehyde groups.

The method according to the invention for manufacturing a reactive sol by converting reactants by means of addition, condensation or substitution reactions, is characterized in that

a compound A,

selected from one of the groups of diisocyanates, dicarboxylic acids, diepoxides and phenols having at least two A end groups and

a compound B,

selected from one of the groups of diols, triols, polyols, diamines, triamines, polyamines, amino alcohols and aldehydes having at least two B end groups

in the equivalent ratio of 1:10 to 10:1 at between 0° and 250°C,

is converted into monomers and/or oligomers C of the type of a urethane, urethane urea, urea, resole, resite, a Mannich base or an ester and

into the compound A remaining as excess with the A end groups or the compound B with the B end groups,

wherein the A end groups are carboxyl, epoxy, isocyanate, phenol groups and

the B end groups are hydroxyl, amino and aldehyde groups

and

simultaneously with or after the start of the reaction,

a compound D selected from the group of diols, triols, diamines, triamines, polyamines, aldehydes or mixtures of these compounds is converted with the D-end groups hydroxyl, amino or aldehyde groups

at between 25 and 200 °C with the monomers and/or oligomers C into an adduct E of the compound A and the compound D,

releasing the compound B from the monomers and/or oligomers C with at least two B end groups.

In this case, the method in embodiments according to the invention is performed in one, two or multiple stages.

One further development of the invention provides that the monomer and/or the oligomer are prepolymers.

One embodiment of the invention is characterized in that the reactive sol contains 15-80 wt.% monomer and/or oligomer (adduct E) and 85-20 wt.% of the compound B acting as an intercalating agent.

In a further embodiment the monomer and/or oligomer (adduct E) is selected from the group comprising hydroxyl functional-group (-OH), amino functional-group (primary, secondary, tertiary) and carboxyl functional-group (-COOH) monomers and/or oligomers.

In a further embodiment the hydroxyl functional-group monomers and/or oligomers are selected from ester alcohols, resoles, resites, polyhydroxyl alcanoates or oligo (ω-hydroxyalkyl) terephthalates,

the amino functional-group monomers or oligomers are selected from oligo ureas, oligo ester-amides or oligomeric Mannich bases (β-amino ketones),

the carboxyl functional-group monomers or oligomers are selected from oligo-adipinates, oligo-sebacates, oligo-azelainates, oligo-succinates, oligo-phthalates, oligo-terephthalates, oligo-sophthalates, oligo-mesitylates, oligo-hydroxybutyrates, oligo-hydroxyvalerates, oligo-hydroxypropionates.

A further development is characterized in that the compound B acting as an intercalating agent exists in a concentration of 80 wt.% monomers and/or oligomers (adduct E) as a solvate therewith.

In a further embodiment the compound B acting as an intercalating agent is selected from water, (at least 2 C)-diols, triols with a molar mass of 62 to 6000, diamines, triamines, polyether alcohols, polyester alcohols and/or poly(ether ester alcohols), modified native oils, lactams, amides, thiols.

A further embodiment provides that at least one organic solvent with heteroatoms is contained, which is used to regulate the viscosity and/or behaviour in surface coatings.

In a further embodiment at least one OH-functional or NH_2-functional monomer and/or oligomer is contained in an intercalating agent/intercalating mixture in the form of one or a plurality of diols and/or triols with molar masses of between 62 and 6000.

In a further development of the invention the OH-functional monomer and/or oligomer is selected from terephthalates, phthalates, hydroxylalkanoates and/or ω-hydroxyalkyladipinates with a molar mass of 254 to 2000, resoles with molar masses of between 200 and 10,000 or resites with molar masses of between 1000 and 10,000.

A further embodiment of the invention is characterized in that the NH_2-functional monomer and/or oligomer are selected from oligo-ureas containing amino groups or Mannich bases having molar masses of between 200 and 10,000.

In a further embodiment an NH_2-functional monomer and/or oligomer in an intercalating agent or intercalating mixture is contained in the form of one or a plurality of amines with molar masses of between 56 and 10,000 and/or in ω-amino-polyethers with molar masses of between 200 and 5000.

In a further advantageous embodiment the reactive sol contains catalysts, stabilizers, flame protection means, organic fillers, inorganic filler materials, exfoliated layer silicates and nanoscale metal oxides, metal hydroxides and/or metal (oxide hydroxides) of the alkaline earth metals, earth metals, zinc, indium, other rare earth metals and mixtures thereof.

In the reactive sol at least one monomer and/or oligomer is present in the sol as a nanoparticle with a size of 2 - 20 nm.

In a further development up to 25 % of the oligomer contains one or a plurality of monomers.

In a further embodiment of the method according to the invention for forming further and additional intercalating agents, a further compound F with at least two D end groups is added to hydroxyl, amino, phosphate or carboxyl groups,

wherein when using oligo esters or polyesters as the compound F, a diol or a dicarboxylic acid is used,

when using urethane or urethane urea monomer and/or oligomer as the compound F, one or a plurality of diols, organic phosphates, amines, diamines, polyamines, triols and/or thiols are used,

when using resoles or resites as the compound F, one or a plurality of diols and/or phenols are used, when using Mannich bases as the compound F, one or a plurality of diols, amines, diamines, polyamines and/or triols are used.

Advantageously the conversion takes place in an extruder/reaction extruder/mixer-kneader as a single-step method in a temperature-controllable reactor.

The reactive sol according to the invention is used for the production of polymer coating agents, adhesives, elastomers, sealing compounds or moulding materials and for producing coatings, memory-foam polymers, composites, composite materials, multilayer material, elements of medical equipment, prosthetics and elements of rehabilitation equipment.

Monomers and/or oligomers form so-called functionalized intercalated organic particles in the intercalating agent. The particles comprise particles which are capable of forming intermolecular compounds and therefore capable of congregating of any kind whatsoever and which optionally can be detected at least partially in the form of nanoscale particles by laser light scattering. Nanoscale particles are designated as those particles which have a distribution maximum of 1 to 800 nm measured by laser light scattering.

Organic particles capable of aggregation are understood in the sense of the invention as species which can be solid but also elastic to gel-like, which can form a solvation shell and which are present in solvated form in intercalating agents. This species can be detected by means of laser light scattering, but it cannot be detected in general by laser light scattering but can also remain "invisible" in the measurement as a result of the measurement principle. This species also includes monomers which can be formed during one of the production processes.

Solid particles are those particles which are solid per se at room temperature and are at least partially crystalline, i.e. have a Mohs hardness of 4 to 10.

Elastic particles are understood as forms which are rubber-elastic per se and substantially amorphous at room temperature, i.e. have a glass transition below room temperature.

Gel-like particles are those particles which are rubber-elastic per se and substantially amorphous at room temperature, i.e. have a glass transition below room temperature but as a result of their low molecular weight of 200 to 5000, cannot be an elastomer.

"Solvation" is understood as the attachment of solvent molecules to dissolved particles. The "complexes" thereby formed from the intercalating agent functioning as solvent and (solvated) particles are called solvates. In the case of water as intercalating agent, we talk of hydrates. The stability of the solvates is dependent on the particle size of the dissolved substance [the smaller the particle size, the better is the solvation].

"Sol" is the designation for a colloidal solution in which a solid or liquid substance is dispersed in extremely fine solution in a solid, liquid or gaseous medium.

Designated as "solution" are mixtures of low-molecular functional monomers and/or oligomers in a liquid which form homogeneous mixtures and in which the functional groups and/or the entire molecule of the monomers and/or oligomers are surrounded with a shell of the liquid in the sense of the solution theory.

"Colloidal solutions" are designated as those mixtures in which a solution is apparently present but a sidewards-directed light scattering can be detected by means of the Tyndal effect.

"Colloidal dispersions" are designated as those mixtures in which the dispersed particles are less than 1 µm in size and have a solvation shell, an inhomogeneity is not identifiable for the human eye and a sidewards-directed light scattering can be detected by means of the Tyndal effect.

"Nanoscale dispersions" are designated as those mixtures in which the dispersed particles are 1 to 800 nm, preferably 1 to 100 nm, in size, can have a solvation shell, an inhomogeneity is not identifiable for the human eye and a sidewards-directed light scattering can be detected by means of the Tyndal effect.

According to the invention, the reactive sols with functionalized intercalated particles can be produced by single-, two- or multi-stage synthesis. In this case, a monomer and/or oligomer is produced with the formation of an intercalating agent or intercalating agent mixture.

A "reactive sol" in the sense of the invention is designated as those mixtures which consist of at least one species of functional, organic particle capable of aggregation which are formed from monomers and/or oligomers and at least one liquid compound having intercalating properties which can optionally have a viscosity of 5 to 20,000 mPas (25°C). Both species have at least two functional end groups, where these groups cannot however react with one another.

The viscosity of the intercalating agent should lie between 5 mPas (25°C) and 10,000 mPas (25°C), preferably between 15 mPas (25°C) and 4,000 mPas (25°C). Since the functional organic particles capable of aggregation can have different states, no general viscosity can be given for these under normal conditions. According to the invention, the viscosity of the intercalating agent is adapted to the compounds to be intercalated in such a manner that the sol according to the invention has a preferred maximum viscosity of 100,000 mPas (25°C).

The reactive sols according to the invention have a solid content of ≥ 15 wt.% up to 70 wt.%, preferably from 15 to 30 wt.%. Solid content is understood as the quantity of particles which can be obtained by removing the liquid compound(s) (intercalating agents and optionally additives).

Such reactive sols with functionalized intercalated organic particles had hitherto not been known or described.

In the mixtures designated as reactive sols, the functional groups can be hydroxyl groups, amino groups, thiol groups, carboxyl groups, isocyanate groups, nitrile groups, keto groups, aldehyde groups or cyanate groups, i.e. substantially those groups which contain at least one heteroatom. In this case, a requirement of the method according to the invention and the sols with functionalized intercalated particles according to the invention is that the particles present in the sol and the intercalating agent comprise those groups which do not undergo any chemical reaction with one another since otherwise the sol can become unstable.

Compounds having free or capped hydroxyl groups, amino groups, thiol groups, carboxyl groups, isocyanate groups, nitrile groups, keto groups, aldehyde groups or cyanate groups can now be used as functional particles based on monomers and/or oligomers in the sols according to the invention or they are formed in the intercalating agents.

The base bodies (monomer and oligomer) of the functional particles can in this case be of an aliphatic or aromatic nature. These base bodies can be in the form of monomers or in the form of oligomers with groups inserted between the monomers which can be both pure hydrocarbon groups e.g. methylene groups (-CH2-) and also groups based on heteroatoms. Examples of such groups are ether groups (-O-), ester groups (-COO-), urethane groups (-NHCO-), urea groups (-NHCONH-), mercapto groups (-S-), keto groups (-C=O-) etc.

Preferably reactive sols according to the invention are provided in which the functional, chemically reactive groups of the at least one monomer and/or the at least one oligomer are selected from the group -OH, -COOH, -SH, -NHR and/or $-NH_2$, and the functional chemically reactive groups of the at least one intercalating agent are selected from the group -OH, $-NH_2$, -SH, -NHR.

The composition of the reactive sol comprising the functional particles and the intercalating agent(s) can be varied widely. These reactive sols according to the invention accordingly contain 5-80 wt.% monomer and/or oligomer as functional particles and 95-20 wt.% intercalating agent.

In one embodiment of the reactive sols according to the invention, the functional particles are selected from hydroxyl functional-group monomers and oligomers, amino functional-group monomers and oligomers, thiol functional monomers and oligomers or carboxygroup functional monomers and oligomers.

Preferably used as monomers are those compounds which have at least two functional groups and in which intercalating agents are insoluble partially or in a concentration-dependent manner.

The monomers and/or oligomers are preferably selected from the group consisting of hydroxyl functional-group (OH), amino functional-group (NH_2) and carboxy functional-group (COOH) monomers or oligomers.

The hydroxyl functional-group monomers or oligomers can be selected, for example, from ester alcohols, resoles, resites, polyhydroxyalkanoates or oligoterephthalates or oligo(□-hydroxy-alkyl)terephthalates.

The amino functional-group monomers or oligomers can be selected, for example, from oligo ureas, oligo ester amides or oligomeric Mannich bases (β-aminoketone).

Carboxy functional-group monomers or oligomers are preferably selected from oligoadipinates, oligosebacates, oligoazelainates, oligosuccinates, oligophthalates, oligoterephthalates, oligoisophthalates, oligomesitylates, oligohydoxybutyrates, oligohydroxyvalerates and oligohydroxypropionates.

According to the invention, the intercalating agents contained in the reactive sol are those compounds which allow a solvation of the monomers and polymers to be represented as functional particles to a sol. The intercalating agents are usually compounds which preferably up to a concentration of 80 wt.% of monomers and/or oligomers form a solvate with these.

They are preferably selected from the group consisting of water, (at least 2 C)-diols, triols having a molar mass of 92 to 6000, diamines, aminoalcohols, alkoxylated di- or polyamines, triamines, polyether alcohols, polyester alcohols and/or poly(ether-ester alcohols), modified native oils, lactams, amides and thiols. The reactive sols can comprise, in addition to the intercalating agent, at least one organic solvent with heteroatoms which is used to regulate the viscosity and the behaviour with surface coatings.

(At least 2-C)-diols are selected from the group consisting of ethylene glycol, diethylene glycol, triethylene glycol, tetraethylene glycol, pentaethylene glycol, hexaethylene glycol, higher ethylene glycols or mixtures thereof, propylene glycol-1,2 or -1,3, dipropylene glycol, tripropylene glycol, higher propylene glycols up to a molar mass of 4000, copolyether alcohols of ethylene oxide and propylene oxide having molar masses of up to 5000, polytetramethylene glycols having a molar mass of 200 to 6000, butane diol-1,4 or -1,3, polycaprolactone diols having a molar mass of 400 to 8000, pentane-1,5-diol, hexane-1,6-diol, octane 1,8-diol.

Triols having a molar mass of 92 to 6000 are, for example, selected from glycerin, octanetriol-1,3,8, hexanetriol-1,3,6 or its homologues, trimethylolpropane, glycerin-propyleneoxide adducts having a molar mass of 260 to 4000, glycerin-propyleneoxide-ethyleneoxide adducts having a molar mass of 400 to 8000, trimethylolpropane-propyleneoxide adducts having a molar mass of 260 to 4000, trimethylolpropane-propyleneoxide-ethyleneoxide adducts having a molar mass of 400 to 8000.

Diamines are, for example, selected from ethylenediamine, propylenediamine, butane-1,4-diamine, hexane-1,4-diamine, piperazine, n-methylpiperazine, n-(2-n-methylaminoethyl)piperazine, n-ethyl-cyclohexane-amine, piperidine, imidazolidine.

Triamines are, for example, selected from diethylenetriamine, dipropylenetriamine, dibutylenetriamine, hexahydrotriazine, 2,4,6-triaminophenol.

Polyether alcohols are, for example, selected from those listed above under diol and triol.

Polyester alcohols are, for example, selected from adipic acid-ethylene glycol condensates having a molar mass of 400 to 2000, adipic acid-diethylene glycol condensates having a molar mass of 400 to 2000, adipic acid-diethylene glycol-hexanediol condensates having a molar mass of 400 to 2000, adipic acid-neopentyl glycol condensates having a molar mass of 400 to 2000, adipic acid-neopentyl glycol-glycerin condensates having a molar mass of 400 to 1000, adipic acid-neopentyl glycol-trimethylol propane condensates having a molar mass of 400 to 1000, adipic acid-neopentyl glycol-pentaerythrite condensates having a molar mass of 400 to 1000, phthalic acid-diethylene glycol condensates having a molar mass of 260 to 1000, phthalic acid-tetraethylene glycol condensates having a molar mass of 300 to 1000, isophthalic acid-diethylene glycol condensates having a molar mass of 260 to 1000, terephthalic acid-diethylene glycol condensates having a molar mass of 260 to 2000, terephthalic acid-diethylene glycol-glycerin-condensates having a molar mass of 260 to 2000, terephthalic acid adipinic acid-diethylene glycol-glycerin-condensates having a molar mass of 260 to 2000, terephthalic acid-adipinic acid-ethylene glycol-glycerin condensates having a molar mass of 260 to 2000, terephthalic acid-adipic acid-diethylene glycol-octanediol condensates having a molar mass of 260 to 2000, terephthalic acid-adipinic acid-2-ethyl-1,3-hexanediol-glycerin condensates having a molar mass of 260 to 2000.

Poly(ether-ester alcohols) are, for example, selected from terephthalic acid-diethylene glycol condensates having a molar mass of 260 to 2000, terephthalic acid-polyethylene glycol condensates having a molar mass of 260 to 3000, terephthalic acid-polypropylene glycol condensates having a molar mass of 260 to 3000.

Modified native oils are, for example, selected from epoxidized soya oil, epoxidized castor oil, air-oxidized rapeseed oil, air-oxidized fish oil, sulfonated rapeseed oil, sulfonated coconut oil.

Lactams are, for example, selected from caprolactam.

Amides are, for example, selected from dimethylformamide, diethylformamide, hexamethylphosphoric acid triamide, diethylaminoethylphosphonic acid diethylester, aminophosphonic acid-bis(2-hydroxyethyl)ester.

Thiols are aliphatic or aromatic compounds containing SH groups including bis-, tris or tetrakis-thiolalkylene esters of dicarboxylic acids.

Organic solvents with heteroatoms are, for example, selected from dimethylsulfoxide, dimethylformamide, cyclohexanone, 3,5-dialkylphenoloxyethylene ether, alkozyphenolene, dibutyl-ether, dioxane.

With a higher number of repeating structural units in the intercalating agent, the solvatable quantity decreases further so that the reactive sol according to the invention is present, for example as a solvate in the form of a mixture of sol, solution, colloidal solution, colloidal dispersion and nanoscale dispersion. These different solvation/dispersion ratios are also part of a reactive sol according to the invention.

In particular, reactive sols according to the invention are those which contain at least one OH-functional or NH2-functional monomer or oligomer in an intercalating agent/mixture in the form of one or a plurality of diols and/or triols having molar masses between 64 and 6000. In this case, the OH-functional monomer or oligomer is selected from terephthalates, phthalates, hydroxyalkanoates and/or ω-hydroxyalkyladipinates having a molar mass of 254 to 2000, resoles having molar masses between 200 and 10,000 or resites having molar masses between 1000 and 10,000. The NH_2-functional monomer or oligomer is selected from amino-group-containing oligo ureas or Mannich bases having molar masses between 200 and 10,000.

A special variant of the reactive sols according to the invention are those which contain an NH_2-functional monomer or oligomer in an intercalating agent or mixture in the form of one or a plurality of amines having molar masses between 56 and 10,000 and/or in ω-amino-polyethers having molar masses between 200 and 5000.

With the aid of these additives, the properties of the reactive sols can advantageously be further adjusted to the specially required properties. In particular, nanoscale inorganic fillers contribute to the fact that already at very low concetrations, e.g. below 5 wt.%, quite different properties can be achieved. Thus, for example due to the metal oxides, the conductivity or due to the silicon carbide, the thermal conductivity can be substantially increased. Furthermore, exfoliated graphites can be used to increase the intumescence effect in the sense of flame retardant. In addition, the use of nanoscale metal or hydroxides allows a substantial increase in the flame retardancy even at low concentrations.

Catalysts are, for example, selected from tertiary amines, metallorganic compounds, amino alcohols, Lewis acids, Lewis bases, mineral acids, alkali or alkaline-earth hydroxides or carboxylates.

Stabilizers are, for example, selected from alkylsulfonates, alkali salts of alkylsulfonates, epoxydized oils, silicones, organic silicates, alkylsilicon polyether alcohols, polyether silanols, silazanes, hexamethyl disilazans, bis-aromatic carbodiimides such as 3,5-bis-(diisopropylphenyl)carbodiimide, stilbene derivatives, titanium dioxide, thiazolene, benzthiazolene, alkylimidazoles, alkylthiadiazoles, bis-(dialkylaminoalkyl)piperazines.

Flame retardants are, for example, selected from phosphororganic compounds such as triphenylphosphate, triphenylphosphite, tricresylphosphate, tris(2-chloroethyl)phosphate, tris-(2,3-dibromphenyl)phosphate, tris(2-chloropropyl)phosphate, red phosphorus, ammonium polyphosphates, ammonium polyphosphate II, aluminium hydroxide, aluminium oxide hydrate, magnesium hydroxide, magnesium oxide, antimony trioxide, graphite, expandable graphite, expanded clay, nanoscale montmorillonite, surface-modified layer silicates, Böhmite, decabromopiphenyl, octabromobiphenylether, pentabromobiphenylether, melamine, melamine cyanurate.

Organic fillers are, for example, selected from cellulose fibres, microcrystalline cellulose, reclaimed cellulose fibres, lignin, lignin sulfonate, wood flour, wood fibres, natural fibres such as hemp, sisal, flax or similar, natural oils, silsesquioxane.

Inorganic fillers are, for example, selected from talc, chalk, barium sulfate (heavy spar), rock flour, porcelain flour, titanium dioxide, aluminium oxide, zinc oxide, glass fibres, glass flour, expanded glass, graphite, iron oxide, cadmium sulfide, cadmium selenide, carbon fibres, carbon nanofibres (carbon nanotubes), silver powder.

Exfoliated layer silicates are, for example, selected from Montmorillonite, laponite, cloisite.

Nanoscale metal oxides, metal hydroxides and/or metal (oxide-hydroxides) of alkaline earth metals, earth metals, zinc, indium, other rare earth metals, for example, selected from aluminium hydroxide, aluminium oxide hydroxide, magnesium hydroxide, yttrium oxide, indium hydroxide, zinc indium hydroxide, zinc indium hydroxide oxide, in addition iron oxide, iron oxide hydroxide, iron-II-hydroxide.

The sols according to the invention are manufactured continuously or discontinuously by means of a single- or two-stage reaction. Preferred methods are described in detail in the examples.

The functional particles are in this case contained in the intercalating agent or mixture up to 80 wt.%, preferably up to 30 wt.%. Since these functional particles can have different solvation radii depending on their structure and composition, measurement using conventional laser light scattering devices is problematical and can lead to completely incorrect results. Thus, measurements were made, for example, on those sols according to the invention using laser light scattering by means of NANOFOXX in which the solvated monomer has a molecular size of 3.2 nm. A value of 1.2 nm was determined as a result of the measurements (own results). This contrasts with studies from the literature which for crystals, for example, of tris-(4,4'-diaminodiphenylmethane) and sodium chloride give molecular sizes over 18 nm (J. W. Swardstrom, L. A. Duvall, D. P. Miller, The Crystal and Molecular Structure of Tris-(4,4'-diaminodiphenylmethane)-Sodium Chloride, Acta Cryst. B28, 2510-2514 (1972)).

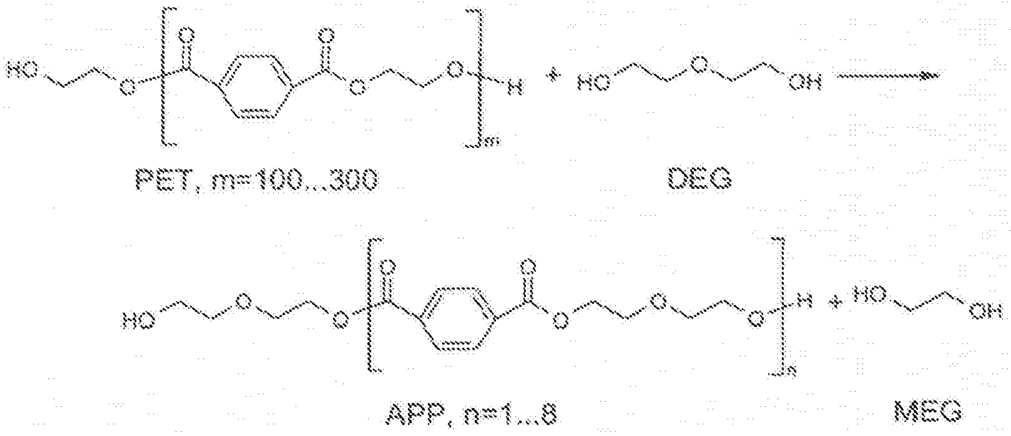
For a further sol according to the invention whose oligomer had a molar mass of 812 and whose molecular length on the basis of its structure should be 14.1 nm, a value of over 80 nm as maximum was determined by means of laser light scattering (NANOFOXX). The specification of particle sizes which are determined by laser light scattering is consequently only significant in part since the solvation conditions in the sols determine the measurement data.

In the sols according to the invention, the size of the fraction of the functional particles which can be measured by laser light scattering preferably lies in the range from 1 nm to 800 nm, preferably 1 to 400 nm, particularly preferably 1 to 50 nm. Quite particularly preferred sols have at least one monomer and/or oligomer as nanoparticle having a size of 2-20 nm in the sol.

However, these values are not a characterizing component of the invention, as explained hereinbefore.

As an example, mention is made of a reactive sol by solution/dispersion of bis-(2-hydroxyethyl)terephthalate in diethylene glycol where only a small fraction of the functional compound is dissolved and a larger fraction is solvated. As a result of the molecular size of the compounds, the dispersed particles usually have dimensions over 1 nm, in the said example the molecular size is given as 1.7 nm.

Compounds which have at least two and at most 15 repeating structural units are designated as oligomers. As an example, mention is again made of a terephthalic acid ester which is produced on the basis of diethylene glycol and terephthalic acid and is characterized by the structural formula



Whereas the monomer bis-(□-hydroxy-ethoxy-ethyl-) terephthalate in low-molecular diols can still be solvated up to concentrations of about 12%, a colloidal solution is formed above this which shows a clear Tyndall effect. The oligomer with n = 2 can be solvated up to about 14% without a demonstrable Tyndall effect being observed.

The manufacture of the functional sols with functionalized particles is accomplished according to the invention whereby the higher-molecular oligomers and/or polymers used as starting material are obtained by producing a prepolymer which is obtained by reacting at least two monomers with interreactive chemical groups.

A prepolymer is produced, for example, from at least one diol and/or polyether alcohol and at least one di- and/or polyisocyanate.

Diols are, for example, selected from ethylene glycol, diethylene glycol, triethylene glycol, tetraethylene glycol, pentaethylene glycol, hexaethylene glycol, higher ethylene glycols or mixtures thereof, propylene glycol-1,2 or -1,3, dipropylene glycol, tripropylene glycol, higher propylene glycols up to a molar mass of 4000, copolyether alcohols of ethylene oxide and propylene oxide having molar masses up to 5000, polytetramethylene glycols having a molar mass of 200 to 6000, butane diol-1,4 or -1,3, polycaprolactone diols having a molar mass of 400 to 8000, pentan-1,5-diol, hexan-1,6-diol, octan-1,8-diol.

Polyether alcohols are preferably selected from glycerin-propylene oxide-adducts having a molar mass of 260 to 4000, glycerin-propylene oxide-ethylene oxide adducts having a molar mass of 400 to 8000, trimethylolpropane-propylene oxide adducts having a molar mass of 260 to 4000, trimethylolpropane-propylene oxide ethylene oxide adducts having a molar mass of 400 to 8000.

Di and/or polyisocyanates are, for example, selected from 4,4'-diphenylmethane diisocyanate, toluylene-2,4-diisocyanate, toluylene-2,6-diisocyanate, hexamethylene diisocyanate, isophorone diisocyanate, 4,4'-Ddicyclohexylmethane diisocyanate, 2,4-xylylene diisocyanate, 2,6-xylylene diisocyanate, hexamethylene diisocyanate-Biuret, trimeric toluylene diisocyanate, polymeric 4,4'-diphenylmethane diisocyanate.

These prepolymers are reacted with amines in the sense of an amidation of the carbamic acid ester group (substitution reaction). This conversion is a hitherto little studied reaction, the rate of which is determined by the type of amine used for the amidation and its basicity.

For amidation of the carbamic acid ester group, use is made of amines selected, for example from di-n-butylamine, di-n-hexylamine, N-methyl-N-hexylamine, N-methylbenzylamine, diethylene triamine, ethylene diamine, triethylene tetramine, dipropylene triamine, tripropylene tetramine, piperazine, N-ethyl-N'-ethyl-piperazine, morpholin, piperidine, imidozolidine, diethylamine.

In a particular embodiment of the invention, the reactive sols are obtained by the specific structure of functional amines in the intercalating agent. This is accomplished by

- a synthesis of oligomers which are readily accessible to a conversion by suitable reagents and structural units which support the solvation of functional particles and specifically co-determines their properties for the subsequent use for manufacturing end products and

- the further reaction with reagents directed at the conversion of suitable groups which in turn have a multiplicity of functional groups of which a part are used for substitution and a further part are used for their functionalization.

Thus, the subsequent steps and the properties of the functional particles to be achieved and the end products to be produced therefrom can already be predefined in the synthesis and taken account by the structure of the components. The selection of the monomers, oligomers and intercalating agents is made in such a manner that

- the functional particles produced in the intercalating agents are permanently stably solvated,

- the functional particles produced have a very narrow particle size distribution,

- the functional particles yield predetermined properties of the end products as a result of the type of compounds used and the number thereof,

- the functional particles have a predetermined number of functional groups as a result of the type of compounds used and

- the quantity and the size of the functional particles in the sol is adjusted by the quantity of cleavable monomers and/or oligomers used,

- the stability of the sol due to the released monomers and/or oligomers is produced by the quantity of cleavable monomers and/or oligomers used.

The reaction conditions for carrying out the method according to the invention can be adjusted widely as a function of the type and quantity of monomers and oligomers which form the functional particles and the intercalating agent. Typically the sol is produced at temperatures between 40°C and 250°C within 10 minutes to 24 hours.

Preferably a temperature range of 40 to 250°C is selected for the first stage. When using, for example, Mannich bases, it lies, for example, between 30 and 80°C, for products containing urethane groups this preferably lies in the lower range of 40 to 200°C, when using resoles it preferably lies in the middle range of 100 to 220°C, and for oligo esters it preferably lies in the upper range of 180 to 250°C. For the second stage, possibly to be executed simultaneously, a temperature range of preferably 60 to 240°C is selected. In this case, this is adjusted to the nature and the properties of the bonds to be reacted.

As an example for suitable monomer or oligomer components which are solvated, the following are mentioned:

- ester alcohols, preferably bis-(2-hydroxyethyl)terephthalic acid ester, bis-(2-hydroxyethyl-ethoxy)terephthalic acid ester, bis-(2-hydroxyethyl) phthalic acid ester, bis-(2-hydroxyethyl-ethoxy)phthalic acid ester, bis-(2-hydroxyethylethoxy)isophthalic acid ester and bis-(2-

hydroxyethyl-ethoxy)1,5-naphthylenene dicarboxylic acid ester,

- ester amides, preferably bis-(2-aminoethyl)terephthalic acid diamide, bis-(2-aminoethyl)phthalic acid diamide, bis-(2-aminopropyl-N-methyl-N-propyl)terephthalic acid diamide, bis-(2-aminopropyl-N-methyl-N- propyl)phthalic acid diamide, bis-(2-aminopropyl-N-methyl-N-propyl)isophthalic acid diamide, bis-(2-aminoethyl)adipinic acid diamide, bis-(2-aminopropyl-N-methyl-N-propyl)apidic acid diamide, oligomers thereof with up to twelve repeating structural units or mixtures thereof,

- ureas, preferably N,N'-bis-(4-amino-diphenylmethyl) urea, bis-([4-amino-propyl-N-methyl-N-propylene-ureyl]diphenylmethyl) urea, bis-(4-amino2-methyl-phenyl) urea, bis-(2-amino-4-methylphenyl) urea, bis-(4-aminodicyclohexyl-methyl) urea, bis-([4-amino-propyl-N-methyl-N-propylene-ureyl] dicyclohexylmethyl) urea, bis-([4-amino-ethyl-N-ethylene ureyl]diphenylmethyl) urea, bis-([4-amino-pro-yl-N-propylene ureyl]diphenylmethyl) urea, oligomers thereof with up to twelve repeating structural units or mixtures thereof,

- resoles, preferably bis-(4-hydroxymethyl-phenyl)methylene, oligomers thereof with up to twelve repeating structural units or mixtures thereof,

- resites, preferably bis-(4-hydroxymethyl-phenyl)methylene, oligomers thereof with up to twelve repeating structural units or mixtures thereof,

- polyoxymethylene oligomers thereof with up to twelve repeating structural units or mixtures thereof,

- bis-(2-hydroxyethyl)terephthalate and oligomers thereof with up to twelve repeating structural units or mixtures thereof,

- bis-(□-hydroxy-ethoxy-ethyl-)terephthalate and oligomers thereof with up to twelve repeating structural units or mixtures thereof,

- bis-(2-hydroxyethyl)adipinate and oligomers thereof with up to twelve repeating structural units or mixtures thereof,

- bis-(□)-hydrozy-ethoxy-ethyl)adipinate and oligomers thereof with up to twelve repeating structural units or mixtures thereof,

- bis-(4-hydroxybutyl)adipinate and oligomers thereof with up to twelve repeating structural units or mixtures thereof,

- bis-(6-hydroxyhexyl)adipinate and oligomers thereof with up to twelve repeating structural units or mixtures thereof,

- bis-(2-ethyl-1 -hydroxy-hexyl)terephthalate and oligomers thereof with up to four repeating structural units or mixtures thereof,

- 1-oxy-2-methylene-(N,N-bis-2-hydroxyethylamino)4-tert.-butyl-benzene.

Intercalating agents can, for example, be:

- simple chemical compounds with at least one oxygen and/or nitrogen atom in the molecule, e.g. cyclohexanone, tetrahydrofuran, di-n-butylether, dimethyl formamide, dimethyl acetamide, dimethyl sulfoxide,

- longer-chain polyether and/or polyester and/or poly(etherester), e.g. diethylene glycol, triethylene glycol, polyethylene glycols having a molar mass of 200 to 6000, dipropylene glycol, tripropylene glycol, polypropylene glycols having a molar mass of 200 to 3000, poly(propylene-ethylene)copolyether alcohols having a molar mass of 400 to 6000, polypropylene-ether triols based on glycerin or trimethylol propane having a molar mass of 200 to 5000, poly(propylene-ethylene)copolyether triols having a molar mass of 400 to 8000, polyether esters based on adipic acid, phthalic acid, isophthalic acid, terephthalic acid, sebacic acid, azelaic acid etc. and propylene oxide or propylene oxide-ethylene oxide-copolymer blocks, bis-(1-hydroxy-2-ethylhexyloxy)adipic acid ester, bis-(1-hydroxy-2-ethyl-hexyloxy) succinic acid ester,

- modified native oils, e.g. epoxidized soya oil, hydrated and alkoxylated castor oil, cleaved and alkoxylated oils such as rapeseed oil or soya oil, oxidized and alkoxylated native oils such as rapeseed oil or soya oil,

- lactams such as caprolactam.

Thus, for example, a polyester or polyester mixture having a molar mass of 40,000 to 200,000 in a diol, a diol mixture, a mixture of diols and triols, a monomomer and/or oligomer, a mixture of diols and native oils, a mixture of diols and modified native oils and/or a mixture of diols, oligoesterols and native oils optionally with added metallorganic compounds and/or metal salts as catalysts for the esterification reaction can be specifically cleaved to give uniform olioesters having a molar mass of 250 to 2000 by heating to temperatures of 200 to 270°C whilst maintaining defined conditions. For example, a polybutylene terephthalate having a molar mass of 68,000 in an excess of diethylene glycol can be reacted within eight hours at 230 to 250°C by distilling off butane-1,4-diol to a sol consisting substantially of bis-(2-hydroxyethoxy-terephthalate) and the excess diethylene glycol as intercalating agent.

Alternatively a partially crystalline oligoester can be produced whereby by reacting a stoichiometric excess of a diol and/or triol with one or more dicarboxylic acids in this or in an oligoester or a hydroxyether ester optionally in the presence of metallorganic compounds, the molar mass of the oligoester can be adjusted to a maximum of 2000. Thus, for example, a bis-(□-hydroxylalkyl-adipinate) can be produced as a sol by reaction of adipic acid in an excess of alkylene diol, e.g. butane-1,4- diol, in the presence of dibutyl tin diacetate within 3 hours at a temperature of 170 to 180°C.

Diols are, for example, diethylene glycol, triethylene glycol, tetraethylene glycol, pentaethylene glycol, hexaethylene glycol, higher ethylene glycols or mixtures thereof.

Triols are, for example, glycerin, octane triol-1,3,8, hexane triol-1,3,6 or homologues thereof, trimethylol propane, glycerin-propylene oxide adducts having a molar mass of 260 to 800.

Dicarboxylic acids are, for example, adipic acid, sebacic acid, azelaic acid, phthalic acid, isophthalic acid, terephthalic acid, naphthylene-1,5-dicarboxylic acid.

Oligoesters are, for example, simple condensates of one of the aforesaid dicarboxylic acids and one of the aforesaid diols with 2 to 8 repeating structural units having molar masses up to 1000.

Hydroxyetheresters are those oligoesters which were produced using lower homologues of ethylene glycol or propylene glycol having molar masses up to 600.

Metallorganic compounds are, for example, compounds of tin such as tin dioctoate, dibutyl tin dilaurate, dibutyl tin diacetate, bis-(tributyl tin)oxide, titanium tetrabutylate, antimony tributylate, germanium tetrabutylate.

The reaction can preferably take place in a heatable stainless steel reactor, extruder, reaction extruder and mixer kneader. Preferably the reaction can also be carried out as a single-step process in a temperature-controllable reactor.

In summary it can be ascertained that reactive sols according to the invention comprising organic solvated, functionalized, i.e. reactive with respect to other compounds, particles in an intercalating agent are prepared. These have the major advantage that on the one hand the formation of aggregates of particles is avoided but on the other hand the smallest possible particles with a narrow distribution are formed as sol.

Polymer solutions are provided as reactive sols which have the advantage that matrix polymers can thus be prepared which can have a broad spectrum of properties and therefore can be used flexibly. It is known that the effect of solid additives is inversely related to their particle size, i.e. the smaller the particles, the greater their effect on the property spectrum of the matrix polymer. Consequently there is a need for such particles which are as small as possible in a form suitable for the production of material.

Consequently the reactive sols can be used as required to produce polymer coating agents, adhesives, elastomers, potting compounds or moulding materials.

Other possible applications are, for example, in the production of foams, cellular elastomers, coatings, memory-foam polymers, composites, composite materials, multilayer material, elements of medical equipment, prosthetics and elements of rehabilitation equipment.

Exemplary embodiments

Example 1

Synthesis of the preliminary product (1st stage)

2.5 kg of 4,4'-diphenylmethane diisocyanate (scaly) is added to a 10 l double-walled stainless steel reactor with agitator, thermal oil heating, metering for liquids and solids, nitrogen line and heat exchanger, heated to 45°C and fused. 5.5 kg of polypropylene glycol MG 1100 (e.g. Lupranol® 1100, Elastogran AG) is slowly added to the liquid isocyanate whilst stirring so that the temperature does not exceed 55°C. The mixture is then agitated at 46°C for another 1 h. A homogeneous, viscous yellowish product having an isocyanate content of 5.32% is obtained.

Conversion of the preliminary product (2nd stage)

3.2 kg of dipropylene glycol, 0.8 kg of diethylene triamine and 0.5 kg of di-n-butylamine are weighed out into a 20 l stainless steel reactor with agitator, thermal oil heating, metering for liquids and solids, nitrogen line and heat exchanger and this mixture was heated to 160°C whilst agitating. Within 40 minutes 5.5 kg of the preliminary product produced above is added. After addition has ended, the mixture is agitated at 180°C for a further 45 min. The reaction mixture is drained off by means of a bottom valve. This is homogeneous, easily flowing, brown-orange and clear. The reactive sol contains 31.4% oligo ureas with amine end groups. The determination of the particle size of amine-functional oligo ureas in the reactive sol by means of Nanophox® (Sympatec GmbH, PCCS) yielded the maximum of the distribution curve at 12 nm and a distribution from 10 to 14 nm. The reactive sol has a hydroxyl number of 410 mg KOH/g, an amine number of 92 mg KOH/g and a viscosity (rotation) of 720 mPas (25°C).

The reactive sol is processed with polymeric 4,4'-diphenylmethane diisocyanate (p-MDI, Lupranat® M20S from BASF SE) to give a 2 mm thick coating. Before mixing with p-MDI, 1% synthetic sodium aluminium silicate (NAS) and 0.5% acetyl acetone are added to the reactive sol. The mixing ratio of reactive sol to p-MDI is therefore as 100 : 119. A very smooth layer is obtained having a Shore-D-hardness of 80 and a glass transition temperature (DSC) of 126°C.

Example 2

The method described in Example 1 is carried out as a continuous method by means of a reaction extruder.

In this case, the synthesis of the preliminary product is initially accomplished whilst uniformly metering and mixing the fused diisocyanate and the diol (molar ratio 2 : 1) with 5.0 kg 4,4'-diphenylmethane diisocyanate and 11.0 kg polypropylene glycol MG 1100 (e.g. Lupranol® 1100, Elastogran AG,) per hour at 45°C to 70°C (temperature gradient) in the front part of the extruder.

The conversion of the preliminary product formed is accomplished directly following in the second part of the extruder (heating zones 4 to 8) at 180°C, whilst metering the premixed solvolysis mixture (32 parts of dipropylene glycol, 8 parts of diethylene triamine and 5 parts of di-n-butyl-amine).

The product is homogeneous, flowable, brown-orange and clear. The reactive sol contains 31.4% oligo ureas. The determination of the particle size of amine-functional oligo ureas in the reactive sol by means of Nanophox® (Sympatec GmbH, PCCS) yielded the maximum of the distribution curve at 12 nm and a distribution from 10 to 14 nm. The reactive sol has a hydroxyl number of 425 mg KOH/g, an amine number of 95 mg KOH/g and a viscosity (rotation) of 720 mPas (25°C).

The reactive sol is processed with polymeric 4,4'-Dphenylmethane diisocyanate (p-MDI, Lupranat® M20S from BASF SE) to give a 2 mm thick coating. Before mixing with p-MDI, 1% synthetic sodium aluminium silicate (NAS) and 0.5% acetyl acetone are added to the reactive sol. The mixing ratio of reactive sol to p-MDI is therefore as 100 : 117.5. A very smooth layer is obtained having a Shore-D-hardness of 85 and a glass transition temperature (DSC) of 102°C.

Example 3

Synthesis of the preliminary product

2.7 kg of 4,4'-dicyclohexylmethane diisocyanate is heated to 45°C in a 10 l stainless steel reactor with agitator, thermal oil heating, metering for liquids and solids, nitrogen line and heat exchanger, and 5 kg of PTMO 1000 is added slowly (about 1.5 hours) to this whilst stirring so that the temperature does not exceed 75°C. The mixture is then agitated at 60°C for about 1 h. A homogeneous, viscous yellowish product having an isocyanate content of 5.5% is obtained.

Conversion of the preliminary product

5.5 kg of this preliminary product is added directly via a bottom drain line into a 20 l stainless steel reactor with agitator, thermal oil heating, nitrogen line and heat exchanger with a mixture heated to 120°C comprising 3.2 kg of dipropylene glycol, 0.9 kg of dipropylene triamine and 0.5 kg of di-n-butylamine and after addition is complete, the mixture is heated to 180°C whilst agitating. Agitation is carried out for a further 30 minutes at 180°C, and then the reaction mixture is drained off by means of a bottom valve. This is homogeneous, flowing, and clear. The reactive sol contains 19.2% amine-functional oligo ureas. The determination of the particle size of amine-functional oligo ureas in the reactive sol by means of Zetasizer (Malvern) yielded the maximum of the distribution curve at 11 nm. The reactive sol has a hydroxyl number of 340 mg KOH/g, an amine number of 92 mg KOH/g and a viscosity (rotation) of 400 mPas (25°C).

The reactive sol is processed with polymeric 4,4'-Dphenylmethane diisocyanate (p-MDI, Lupranat® M20S from BASF SE) to give a 2 mm thick coating. Before mixing with p-MDI, 1% synthetic sodium aluminium silicate (NAS) and 0.5% acetyl acetone are added to the reactive sol. The mixing ratio of reactive sol to p-MDI is therefore as 100 : 101. A very smooth layer is obtained having a Shore-D-hardness of 70 and a glass transition temperature (DSC) of 96°C.

Example 4

Synthesis of the preliminary product

5 kg of 4,4'-diphenylmethane diisocyanate (scaly) is added to a 10 l double-walled stainless steel reactor with agitator, thermal oil heating, metering for liquids and

solids, nitrogen line and heat exchanger, heated to 45°C and fused. 4.5 kg of polypropylene glycol MG 450 (e.g. Lupranol® 1200, BASF SE) is slowly added to the liquid isocyanate whilst stirring so that the temperature does not exceed 55°C. The mixture is then agitated at 46°C for another 1 h. A homogeneous, viscous yellowish product having an isocyanate content of 6.8% is obtained.

Conversion of the preliminary product

2.1 kg of dipropylene glycol, 1.5 kg of diethylene triamine, and 0.4 kg of di-n-butylamine and 1 kg of butane diol are weighed out into a 20 l stainless steel reactor with agitator, thermal oil heating, metering for liquids and solids, nitrogen line and heat exchanger and this mixture is heated to 160°C whilst agitating. Within 40 minutes, 5 kg of the preliminary product produced above is added. After addition has ended, the mixture is agitated for a further 45 minutes at 180°C. The reaction mixture is drained off by means of a bottom valve. This is homogeneous, easily flowing, yellow and clear. The reactive sol contains about 50% oligo ureas with amine end groups. The determination of the particle size of amine-functional oligo ureas in the reactive sol by means of Zetasizer (Malvern) yielded the maximum of the distribution curve at 3.5 nm and a distribution from 2 to 6 nm. The reactive sol has a hydroxyl number of 568 mg KOH/g, an amine number of 187 mg KOH/g and a viscosity (rotation) of 909 mPas (25°C).

The reactive sol is processed with polymeric 4,4'-Dphenylmethane diisocyanate (p-MDI, Lupranat® M20S from BASF SE) to give a 2 mm thick coating. Before mixing with p-MDI, 1% synthetic sodium aluminium silicate (NAS) and 0.5% acetyl acetone are added to the reactive sol. The mixing ratio of reactive sol to p-MDI is therefore as 100 : 133. A very smooth layer is obtained having a Shore-D-hardness of 82 and a glass transition temperature (DSC) of 132°C.

Example 5

Synthesis of the preliminary product

3.0 kg of 4,4'-diphenylmethane diisocyanate (scaly) is heated to 45°C in a 10 l double-walled stainless steel reactor with agitator, thermal oil heating, metering for liquids and solids, nitrogen line and heat exchanger, and thereby fused and this is mixed slowly with 3.9 kg of PolyTHF 650 S whilst agitating so that the reaction temperature does not exceed 60°C. The mixture is then agitated at 55°C for another 1 h. A homogeneous, viscous yellowish slightly turbid product having an isocyanate content of 7.0% is obtained.

Conversion of the preliminary product

2.0 kg of dipropylene glycol, 1.2 kg of diethylene glycol, 0.9 kg of bis-N,N-(2-aminopropyl)methylamine, and 0.5 kg of di-n-butylamine are weighed out into a 20 l stainless steel reactor with agitator, thermal oil heating, metering for liquids and solids, nitrogen line and heat exchanger and this mixture is heated to 160°C whilst agitating. Within 60 minutes, 5.5 kg of the preliminary product produced above is added. After addition has ended, the mixture is agitated for a further 30 minutes at 180°C. The reaction mixture is drained off by means of a bottom valve. This is homogeneous, flowing, and clear at 35°C. The reactive sol contains 24.2% amine-functional oligo ureas. The determination of the particle size of amine-functional oligo ureas in the reactive sol by means of Zetasizer (Malvern) yielded the maximum of the distribution curve at 5.8 nm. The reactive sol has a hydroxyl number of 422 mg

KOH/g, an amine number of 127 mg KOH/g and a viscosity (rotation) of 590 mPas (25°C).

The reactive sol is processed with polymeric 4,4'-Dphenylmethane diisocyanate (p-MDI, Lupranat® M20S from BASF SE) to give a 2 mm thick coating. Before mixing with p-MDI, 1% synthetic sodium aluminium silicate (NAS) and 0.5% acetyl acetone are added to the reactive sol. The mixing ratio of reactive sol to p-MDI is therefore as 100 : 128. A very smooth layer is obtained having a Shore-D-hardness of 85 and a glass transition temperature (DSC) of 122°C.

Example 6

Synthesis of the preliminary product

5 kg of 4,4'-diphenylmethane diisocyanate (scaly) is added into a 20 l double-walled stainless steel reactor with agitator, thermal oil heating, metering for liquids and solids, nitrogen line and heat exchanger, heated to 45°C and thereby fused. To the liquid isocyanate is slowly added 6.5 kg of Poly THF 650S (MG 650) whilst agitating so that the temperature does not exceed 55°C. The mixture is then agitated at 46°C for about another 1 h. A homogeneous, viscous yellowish product having an isocyanate content of 7% is obtained.

Conversion of the preliminary product

3.8 kg of dipropylene glycol, 0.7 kg of diethylene triamine, and 1.2 kg of polypropylene triol MG 3100 (Lupranol® 2032, BASF SE) are weighed out into a 20 l stainless steel reactor with agitator, thermal oil heating, metering for liquids and solids, nitrogen line and heat exchanger and this mixture is heated to 160°C whilst agitating. Within 40 minutes, 4.3 kg of the preliminary product produced above is added. After addition has ended, the mixture is agitated for a further 45 minutes at 180°C. The reaction mixture is drained off by means of a bottom valve. This is homogeneous, easily flowing, yellow and clear. The reactive sol contains 31.4% oligo ureas with amine end groups. The determination of the particle size of amine-functional oligo ureas in the reactive sol by means of Zetasizer (Malvern) yielded the maximum of the distribution curve at 5.4 nm and a distribution from 4 to 8 nm. The reactive sol has a hydroxyl number of 416 mg KOH/g, an amine number of 74 mg KOH/g and a viscosity (rotation) of 1690 mPas (25°C).

The reactive sol is processed with polymeric 4,4'-Dphenylmethane diisocyanate (p-MDI, Lupranat® M20S from BASF SE) to give a 2 mm thick coating. Before mixing with p-MDI, 1% synthetic sodium aluminium silicate (NAS) and 0.5% acetyl acetone are added to the reactive sol. The mixing ratio of reactive sol to p-MDI is therefore as 100 : 114. A very smooth layer is obtained having a Shore-D-hardness of 90 and a glass transition temperature (DSC) of 132°C.

Example 7

Synthesis of the preliminary product

5 kg of 4,4'-diphenylmethane diisocyanate (scaly) is added into a 20 l double-walled stainless steel reactor with agitator, thermal oil heating, metering for liquids and solids, nitrogen line and heat exchanger, heated to 45°C and thereby fused. To the liquid isocyanate is slowly added 6.5 kg of Poly THF 650S (MG 650) whilst agitating so that the temperature does not exceed 55°C. The mixture is then agitated at 46°C for about another 1 h. A homogeneous, viscous yellowish product having an isocyanate content of 7% is obtained.

Conversion of the preliminary product

3.7 kg of dipropylene glycol, 1 kg of diethylene triamine, 0.4 kg dibutyl amine and 0.9 kg of polypropylene MG 3100 (Lupranol® 2032, BASF SE) are weighed out into a 20 l stainless steel reactor with agitator, thermal oil heating, metering for liquids and solids, nitrogen line and heat exchanger and this mixture is heated to 160°C whilst agitating. Within 40 minutes, 4 kg of the preliminary product produced above is added. After addition has ended, the mixture is agitated for a further 45 minutes at 180°C. The reaction mixture is drained off by means of a bottom valve. This is homogeneous, easily flowing, yellow and clear. The reactive sol contains about 27% oligo ureas with amine end groups. The determination of the particle size of amine-functional oligo ureas in the reactive sol by means of Zetasizer (Malvern) yielded the maximum of the distribution curve at 25 nm and the distribution from 14 to 35 nm. The reactive sol has a hydroxyl number of 445 mg KOH/g, an amine number of 115 mg KOH/g and a viscosity (rotation) of 1090 mPas (25°C).

The reactive sol is processed with polymeric 4,4'-Dphenylmethane diisocyanate (p-MDI, Lupranat® M20S from BASF SE) to give a 2 mm thick coating. Before mixing with p-MDI, 1% synthetic sodium aluminium silicate (NAS) and 0.5% acetyl acetone are added to the reactive sol. The mixing ratio of reactive sol to p-MDI is therefore as 100 : 130.7. A very smooth layer is obtained having a Shore-D-hardness of 88 and a glass transition temperature (DSC) of 129°C.

Example 8

Synthesis of a reactive sol, which contains aminofunctional and biofunctional mono- and oligoureas from 4,4'-MDI and N-aminoethylpiperazine (NAEP) and diethyltriamine (DETA) in a stainless steel reactor (two-stage process).

Synthesis of the preliminary product

2.5 kg of 4,4'-diphenylmethane diisocyanate (scaly) is added into a 10 l double-walled stainless steel reactor with agitator, thermal oil heating, metering for liquids and solids, nitrogen line and heat exchanger, heated to 45°C and thereby fused. To the liquid isocyanate is slowly added 5.5 kg of polypropylene glycol MG 1100 (e.g. Lupranol® 1100, Elastogran AG) whilst agitating so that the temperature does not exceed 55°C. The mixture is then agitated at 46°C for about another 1 h. A homogeneous, viscous yellowish product having an isocyanate content of 5.32% is obtained.

Conversion of the preliminary product

4.14 kg of dipropylene glycol, 0.37 kg of diethylene triamine, and 0.67 kg of N-aminoethyl piperazine (NAEP) are weighed out into a 20 l stainless steel reactor with agitator, thermal oil heating, metering for liquids and solids, nitrogen line and heat exchanger and this mixture is heated to 160°C whilst agitating. Within 40 minutes, 4.82 kg of the preliminary product produced above is added. After addition has ended, the mixture is agitated for a further 45 minutes at 180°C. The reaction mixture is drained off by means of a bottom valve. This is homogeneous, easily flowing, brown-orange and clear. The reactive sol contains about 24% oligo ureas with amine end groups. The determination of the particle size of amine-functional oligo ureas in the reactive sol by means of Zetasizer (Malvern) yielded the maximum of the distribution curve at 5.2 nm and a distribution from 3 to 8 nm. The reactive sol has a hydroxyl number of 434 mg KOH/g, an amine number of 102 mg KOH/g and a viscosity (rotation) of 1860 mPas (25°C).

The reactive sol is processed with polymeric 4,4'-Dphenylmethane diisocyanate (p-MDI, Lupranat® M20S from BASF SE) to give a 2 mm thick coating. Before mixing with p-MDI, 1% synthetic sodium aluminium silicate (NAS) and 0.5% acetyl acetone are added to the reactive sol. The mixing ratio of reactive sol to p-MDI is therefore as 100 : 125. A very smooth layer is obtained having a Shore-D-hardness of 83 and a glass transition temperature (DSC) of 121°C.

The reactive sol has a biocide effect against algae, daphnia and bacteria. It can be used to produce coatings having a biocide effect. In water basins at 16-22°C and 1% blue algae culture, no growth was determined after 6 months; the coating was smooth and unchanged.

Example 9

Single-stage process

1.5 kg of 4,4'-diphenylmethane diisocyanate (scaly) is added into a 20 l double-walled stainless steel reactor with agitator, thermal oil heating, metering for liquids and solids, nitrogen line and heat exchanger, heated to 45°C and thereby fused. To the liquid isocyanate is added a mixture of 3.44 kg of polypropylene glycol MG 1100 (e.g. Lupranol® 1100, BASF SE), 4.3 kg DPG and 0.7 kg DETA whilst agitating.

At the same time the mixture is heated to 180°C whilst agitating and agitated for 30 minutes at this temperature.

The reaction mixture is drained off by means of a bottom valve. This is homogeneous, easily flowing, yellowish and visually clear. The reactive sol contains about 22.6% oligo ureas with amine end groups. The determination of the particle size of amine-functional oligo ureas in the reactive sol by means of Nanophox® (Sympatec GmbH, PCCS) yielded the maximum of the distribution curve at 12 nm and a distribution from 8 to 15 nm. The reactive sol has a hydroxyl number of 457 mg KOH/g, an amine number of 78 mg KOH/g and a viscosity (rotation) of 851 mPas (25°C).

The reactive sol can be used to produce coatings. To this end the reactive sol is processed with polymeric 4,4'-Dphenylmethane diisocyanate (p-MDI, Lupranat® M20S from BASF SE) to give a 2 mm thick coating. Before mixing with p-MDI, 1% synthetic sodium aluminium silicate (NAS) and 0.5% acetyl acetone are added to the reactive sol. The mixing ratio of reactive sol to p-MDI is therefore as 100 : 128. A very smooth layer is obtained having a Shore-D-hardness of 87 and a glass transition temperature (DSC) of 132°C.

The reactive sol has a biocide effect against algae, daphnia and bacteria. It can be used to produce coatings having a biocide effect. In water basins at 16-22°C and 1% blue algae culture, no growth was determined after 8 months; the coating was smooth and unchanged.

Example 10

44 kg of diethylene glycol is added into a 100 l double-walled stainless steel reactor with propeller agitator, distillation column with heat exchanger, nitrogen line from the bottom of the reactor, four thermal sensors, supply for liquids via a metering pump and filling port for filling with solids, which is heated with thermal oil by means of an external heating unit, and heated to 150°C. During the heating-up phase 25 g of dibutyl tin diacetate is added to the diol. As soon as the temperature of 150°C is reached,

38.8 kg of terephthalic acid dimethylester (DMT) is added via the filling port within 30 minutes. The temperature is slowly increased to 230°C, the nitrogen flow is adjusted to 5 l/min and thus methanol is distilled off from the reaction mixture. As soon as the amount of methanol has reached about 600 g, the nitrogen flow is increased to 10 l/min and at 230°C further agitation is carried out until about 30 ml of methanol has been collected. Then during the cooling phase nitrogen with 10 l/min is passed through until the temperature has reached 120°C. Then the mixture is drained via the bottom drain. In this way a reactive sol having a hydroxyl number of 285 mg KOH/g and a viscosity of 28700 mPas (25°C) is obtained. In a particle size investigation by means of Nanophox® (Sympatec GmbH, PCCS) functional particles having a maximum at 15 nm and a distribution between 12 and 18 nm were found.

The reactive sol is processed with polymeric 4,4'-Dphenylmethane diisocyanate (p-MDI, Lupranat® M20S from BASF SE) to form a sealant in the form of a 4 mm thick plate. Before mixing with p-MDI, 2% synthetic sodium aluminium silicate (NAS) and 0.05% dibutyl tin diacetate are added to the reactive sol. The mixing ratio of reactive sol to p-MDI is therefore as 100 : 66. A very hard plate is obtained having a Shore-D-hardness of 78 and a glass transition temperature (DSC) of 92°C.

Example 11

25 kg of a low-molecular resole is produced from 20 kg of o-cresol and 23 l of 30% formaldehyde solution in the presence of 200 g of concentrated hydrochloric acid. About 12 kg is distilled off from the water.

59 kg of polypropylene glycol MG 400 is added into a 100 l double-walled stainless steel reactor with propeller agitator, distillation column with heat exchanger, nitrogen

line from the bottom of the reactor, four thermal sensors, supply for liquids via a metering pump and filling port for filling with solids, which is heated with thermal oil by means of an external heating unit, and heated to 85°C whilst agitating. At this temperature the previously produced condensate is added slowly in such a manner that the temperature does not fall below 80°C. As soon as the resole solution has been completely added, the temperature is slowly increased to 110°C whilst agitating. As soon as the distillation of the water commences, a nitrogen stream of 7.5 l/min is passed through and the water is further distilled off. When the amount of distillate becomes smaller, the temperature is slowly increased to 125°C. As soon as 4 l of water is collected, the nitrogen stream is increased to 12 l/min and further increased to 20 l/min. When no more distillate is collected, the heating is switched over to cooling. At 65°C the product is drained into containers.

A reactive sol having a hydroxyl number of 195 mg KOH/g and a viscosity of 8450 mPas (25°C) is obtained. In a particle size investigation by means of Nanophox® (Sympatec GmbH, PCCS) functional particles having a maximum at 10 nm were found.

The reactive sol is processed with polymeric 4,4'-Dphenylmethane diisocyanate (p-MDI, Lupranat® M20S from BASF SE) to form a sealant in the form of a 4 mm thick plate. Before mixing with p-MDI, 2% synthetic sodium aluminium silicate (NAS) and 0.5% dibutyl tin diacetate are added to the reactive sol. The mixing ratio of reactive sol to p-MDI is therefore as 100 : 45. A very hard plate is obtained having a Shore-D-hardness of 81 and a glass transition temperature (DSC) of 102°C.

Example 12

Synthesis of the preliminary product

2.5 kg of 4,4'-diphenylmethane diisocyanate (scaly) is added into a 10 l double-walled stainless steel reactor with agitator, thermal oil heating, metering for liquids and solids, nitrogen line and heat exchanger, heated to 45°C and thereby fused. To the liquid isocyanate is slowly added 5.5 kg of polypropylene glycol MG 1100 (e.g. Lupranol® 1100, Elastogran AG) whilst agitating so that the temperature does not exceed 55°C. The mixture is then agitated at 46°C for about another 1 h. A homogeneous, viscous yellowish product having an isocyanate content of 5.32% is obtained.

Conversion of the preliminary product

3.95 kg of dipropylene glycol, 0.83 kg of diethylene triamine, and 0.64 kg of tricyclo-diamine-decane-diamine (TCD) are weighed out into a 20 l stainless steel reactor with agitator, thermal oil heating, metering for liquids and solids, nitrogen line and heat exchanger and this mixture is heated to 160°C whilst agitating. Within 20 minutes, 4.58 kg of the preliminary product produced above is added. After addition has ended, the mixture is agitated for a further 30 minutes at 180°C. The reaction mixture is drained off by means of a bottom valve. This is homogeneous, slightly thin-liquid. yellow and clear. The reactive sol contains about 30% oligo ureas with amine end groups. The determination of the particle size of amine-functional oligo ureas in the reactive sol by means of Zetasizer (Malvern) yielded the maximum of the distribution curve at 4.7 nm and a distribution from 3 to 8 nm. The reactive sol has a hydroxyl number of 496 mg KOH/g, an amine number of 143 mg KOH/g and a viscosity (rotation) of 707 mPas (25°C).

Example 13

Synthesis of a reactive sol which contains aminofunctional mono- and oligoureas from 4,4'-MDI and diethyltriamine (DETA) and N-aminoethylpiperazine (NAEP) in polypropylene glycol MG1100 (e.g. Lupranol® 1100, Elastogran AG) in a sulphonating flask (single-stage method).

15.6g of 4,4'-diphenylmethane diisocyanate is fused under exposure to nitrogen gas (47°C) in a 250 ml sulphonating flask (intensive cooler, nitrogen supply and temperature sensor, magnetic agitator) and then a mixture of 4 g DETA, 6 g NAEP, 34.4g Lupranol 1100 and 40 g DPG is added and agitated whilst increasing the temperature to 180°C. The mixture initially solidifies and after a reaction time of 30 min gives a visually clear product with an oligourea content of about 23%, a hydroxyl number of 437, an amine number of 86 and a viscosity (rotation) of 1850 mPas (25°C).

The reactive sol can be used to produce coatings. To this end the reactive sol is processed with polymeric 4,4'-Dphenylmethane diisocyanate (p-MDI, Lupranat® M20S from BASF SE) to give a 2 mm thick coating. Before mixing with p-MDI, 1% synthetic sodium aluminium silicate (NAS) and 0.5% acetyl acetone are added to the reactive sol. The mixing ratio of reactive sol to p-MDI is therefore as 100 : 121.5. A very smooth layer is obtained having a Shore-D-hardness of 83 and a glass transition temperature (DSC) of 121°C.

The reactive sol has a biocide effect against algae, daphnia and bacteria. It can be used to produce coatings having a biocide effect. In water basins at 16-22°C and 1% blue algae culture, no growth was determined after 9 months; the coating was smooth and unchanged.

Example 14

Synthesis of a reactive sol which contains aminofunctional mono- and oligoureas Vorastar HB 6549 (caprolactone prepolymer (Dow), NCO 16%), diethyltriamine (DETA) in a sulphonating flask (single-stage process).

41.1 g of DETA is heated to 160°C whilst agitating a 250 ml sulphonating flask (intensive cooler, nitrogen supply and temperature sensor, magnetic agitator) and 58.9 g of Vorastar HB 6549 is added. After a reaction time of 60 min at 180°C, a yellow, homogeneous slightly turbid product is obtained which contains about 50% oligo ureas having a viscosity of 7420 mPas (25°C), a hydroxyl number of 548 and an amine number of 428.

The sol can be used to produce coatings.

Example 15

A Mannich base is produced by placing 3.05 kg of p-tert.-butylphenol and 3.1 kg of diethanol amine in a 10 l stirred reactor with propeller agitator, two metering pumps, nitrogen inlet and thermometer, and heated to 40°C. 2.85 kg of 32% formaldehyde solution is added to the mixture whilst agitating and gassing with nitrogen and agitated for an hour at this temperature. Agitation is then carried out for a further three hours at 80°C. The mixture is then cooled to about 55 - 60°C and the pressure is reduced gradually to remove the water. Finally at 1 mbar and 55°C the water is completely removed.

This method is modified to produce the sol. The reaction is carried out as described above. However the reaction is carried out without the dewatering step. From the product thus produced, which is still extremely hydrous, 5.3 kg is removed before the dewatering and mixed with 2.85 kg of a polyether triol based on glycerin and propylene oxide (hydroxyl number 420 mg KOH/g) and agitated for three hours at 75 - 80°C. The mixture is then cooled to about 55 - 60°C and the pressure is reduced gradually to remove the water. Finally at 1 mbar and 55°C the water is completely removed.

A reactive sol is obtained having a hydroxyl number 492 mg KOH/g and OH-functionality 3.4 as well as a viscosity (25°C) of 1450 mPas.

Films having a thickness of 2 mm are produced from the reactive sol and 4,4'-dicyclohexylmethane diisocyanate with an equivalent ratio of 1.00 : 1.06 by reaction at 80°C in a Matis®-LabCoater. These have a Shore-D hardness of 72 and an ultimate elongation of 12%. These films are models for a very hard but elastic coating.

Example 16

The approach of Example 15 is repeated but 2.25 kg of bisphenol A and 4.5 kg of N-methyl-N,N-bis(i-propylamine) is reacted with formaldehyde. The reactive sol is again produced as in Example 15 by adding 3.5 kg of dipropylene glycol to 5.0 kg of the reaction product and proceeding further as in Example 15.

A reactive sol is obtained having the hydroxyl number 685 mg KOH/g and the OH-functionality 3.0 as well as a viscosity (25°C) of 1290 mPas.

Films having a thickness of 2 mm are produced from the reactive sol and 4,4'-dicyclohexylmethane diisocyanate with an equivalent ratio of 1.00 : 1.06 by reaction at 80°C in a Matis®-LabCoater. These have a Shore-D hardness of 76 and an ultimate elongation of 19%. These films are models for a very hard but elastic coating.

Reaktív szolok és eljárás előállításukra

Szabadalmi igénypontok

1. Reaktív szol, amely folyadék fázist és funkciós csoportokkal rendelkező nanorészecskéket tartalmaz, **azzal jellemezve**, hogy
a részecskék maximális méreteloszlása 50 nm-nél kisebb, és
a részecske legalább egy monomert és/vagy legalább egy, 2-15 ismétlődő szerkezeti egységből álló oligomert tartalmaz,
ahol a monomer és/vagy az oligomer egy A vegyületből és egy D vegyületből álló E adduktum,
amely A vegyület a legalább két A-végcsoportot tartalmazó diizocianátok, dikarbonsavak, diepoxidok és fenolok közül van kiválasztva, és amely D vegyület a legalább két D-végcsoportot tartalmazó diolok, triolok, diaminok, triaminok, poliaminok, aldehidek és ezek keverékei közül van kiválasztva,
és a folyadék fázis tartalmaz egy interkaláló ágensként funkcionáló B vegyületet, amely legalább két B-végcsoportot tartalmazó diolok, triolok, poliolok, diaminok, triaminok, poliaminok, amino-alkoholok és aldehidek közül van kiválasztva,
ahol az A-végcsoportok karboxil-, epoxi-, izocianát-, fenolcsoportok, a B-végcsoportok hidroxilcsoportok, aminocsoportok, aldehidcsoportok,
és a D-végcsoportok hidroxil-, amino-, aldehidcsoportok.

2. Az 1. igénypont szerinti reaktív szol, **azzal jellemezve**, hogy a monomer és/vagy oligomer pre-polimer.

3. Az 1. vagy 2. igénypont szerinti reaktív szol, **azzal jellemezve**, hogy tartalmaz 5-80 tömeg% monomert és/vagy oligomert (E adduktumot) és 95-20 tömeg% interkaláló ágensként funkcionáló B vegyületet.

4. Az 1-3. igénypontok bármelyike szerinti reaktív szol, **azzal jellemezve**, hogy a monomer és/vagy oligomer a következőkből álló csoportból van kiválasztva: hidroxilcsoportot (OH), aminocsoportot (primer, szekunder, tercier) és karboxilcsoportot (COOH) hordozó monomerek és/vagy oligomerek.

5. Az 1-4. igénypontok bármelyike szerinti reaktív szol, **azzal jellemezve**, hogy a hidroxilcsoportot hordozó monomerek és/vagy oligomerek a következők közül vannak kiválasztva: észteralkoholok, rezolok, rezitek, polihidroxil-alkanoátok és oligo(ω-hidroxi-alkil)-tereftalátok;
az aminocsoportot hordozó monomerek vagy oligomerek a következők közül vannak kiválasztva: oligoureák, oligo-észter-amidok és oligomer Mannich-bázisok (β-amino-ketonok);
a karboxilcsoportot hordozó monomerek vagy oligomerek a következők közül vannak kiválasztva: oligoadipinátok, oligoszebacátok, oligoazelainátok, oligoszukcinátok, oligoftalátok, oligotereftalátok, oligoszoftalátok, oligomezitilátok, oligohidroxibutirátok, oligohidroxivalerátok és oligohidroxipropionátok.

6. Az 1-5. igénypontok bármelyike szerinti reaktív szol, **azzal jellemezve**, hogy az interkaláló ágensként funkcionáló B vegyület a monomerek és/vagy oligomerek (E adduktum) 80 tömeg% koncentrációjáig azokkal alkotott szolvátként van jelen.

7. Az 1-6. igénypontok bármelyike szerinti reaktív szol, **azzal jellemezve**, hogy az interkaláló ágensként funkcionáló B vegyület a következők közül van kiválasztva: víz, (legalább 2C)-diolok, 92-6000 molekulatömegű triolok, diaminok, triaminok, poliéter-alkoholok, poliészter-alkoholok és/vagy poli(éter-észter-alkoholok), módosított természetes olajok, laktámok, amidok, tiolok.

8. Az 1-7. igénypontok bármelyike szerinti reaktív szol, **azzal jellemezve**, hogy tartalmaz legalább egy, heteroatomokat tartalmazó szerves oldószert, amelynek funkciója a viszkozitás és áramlóképesség szabályozása.

9. Az 1-8. igénypontok bármelyike szerinti reaktív szol, **azzal jellemezve**, hogy legalább egy interkaláló ágens/interkaláló elegy legalább egy, OH vagy NH_2 funkciós csoportot hordozó monomert és/vagy oligomert tartalmaz egy vagy több diol és/vagy 62 és 6000 közötti molekulatömegű triolok alakjában.

10. A 9. igénypont szerinti reaktív szol, **azzal jellemezve**, hogy az OH funkciós csoportot hordozó monomer és/vagy oligomer a következők közül van kiválasztva: tereftalátok, ftalátok, hidroxialkanoátok és/vagy ω-hidroxi-alkiladipinátok, amelyek molekulatömege 254-2000, rezolok, amelyek molekulatömege 200 és 10 000 közötti, és rezitek, amelyek molekulatömege 1000 és 10 000 közötti.

11. A 9. igénypont szerinti reaktív szol, **azzal jellemezve**, hogy az NH_2 funkciós csoportot hordozó monomer és/vagy oligomer a következők közül van kiválasztva: oligoureák és Mannich-bázisok, amelyek molekulatömege 200 és 10 000 közötti

12. Az 1-8. igénypontok bármelyike szerinti reaktív szol, **azzal jellemezve**, hogy az NH_2 funkciós csoportot hordozó monomer és/vagy oligomer egy interkaláló ágensben vagy interkaláló keverékben egy vagy több 56 és 10 000 közötti molekulatömegű amin és/vagy 200 5000 közötti molekulatömegű ω-amino-poliéterek formájában van jelen

13. Az 1-12. igénypontok bármelyike szerinti reaktív szol, **azzal jellemezve**, hogy tartalmaz még katalizátorokat, stabilizálószereket, lánggátló anyagokat, szerves töltőanyagokat, szervetlen töltőanyagokat, hasított rétegszilikátokat, és alkáliföldfémek, földfémek, cink, indium, egyéb ritkaföldfémek és keverékeik nanoméretű fém-oxidjait, fém-hidroxidjait és/vagy fém-(oxid-hidroxid)-jait.

14. Az 1-13. igénypontok bármelyike szerinti reaktív szol, **azzal jellemezve**, hogy a szolban legalább egy monomer és/vagy oligomer 2-20 nm méretű nanorészecskeként van jelen.

15. Az 1-14. igénypontok bármelyike szerinti reaktív szol, **azzal jellemezve**, hogy az oligomer legfeljebb 25 % tartalmaz továbbá egy vagy több monomert.

16. Eljárás reaktív szol előállítására reaktánsok addíciós, kondenzációs vagy szubsztitúciós reakciókkal történő átalakításával, **azzal jellemezve**, hogy

egy A vegyületet,

amely a legalább két A-végcsoportot tartalmazó diizocianátok, dikarbonsavak, diepoxidok és fenolok közül van kiválasztva,

és egy B vegyületet,

amely a legalább két B-végcsoportot tartalmazó diolok, triolok, poliolok, diaminok, triaminok, poliaminok, aminoalkoholok és aldehidek közül van kiválasztva,

0 °C és 250 °C közötti hőmérsékleten, 1:10-től 10:1-ig terjedő egyenérték arányban

C monomerekké és/vagy oligomerekké alakítunk át, uretán, uretán-urea, urea, rezol, rezit, Mannich-bázis vagy észter formájában,

és A-végcsoportokat hordozó A vegyület vagy B-végcsoportokat hordozó B-vegyület feleslege marad vissza,

ahol az A-végcsoportok karboxil-, epoxi-, izocianát-, fenolcsoportok, és
a B-végcsoportok hidroxil-, amino- és aldehidcsoportok,

és a reakció megkezdésével egyidejűleg vagy azt követően egy diolok, triolok, diaminok, triaminok, poliaminok, aldehidek vagy e vegyületek keverékei közül kiválasztott D vegyületet, amely hidroxil-, amino- vagy aldehid D-végcsoportokat hordoz, 25 °C és 200 °C közötti hőmérsékleten a C monomerekkel és/vagy oligomerekkel az A vegyület és a D vegyület E adduktumává alakítjuk, miközben a C monomerekből és/vagy oligomerekből legalább két B-végcsoportot tartalmazó B vegyület szabadul fel.

17. A 16. igénypont szerinti eljárás, **azzal jellemezve**, hogy az eljárást egy, két vagy több szakaszban végezzük.

18. A 16. vagy 17. igénypont szerinti eljárás, **azzal jellemezve**, hogy egy további F vegyületet adagolunk, amely legalább két hidroxil, amino, foszfát vagy karboxil D-végcsoportot tartalmaz, ahol
oligoészterek vagy poliészterek alkalmazása esetén F vegyületként diolt vagy dikarbonsavat alkalmazunk,
uretán vagy uretán-urea monomer és/vagy oligomer alkalmazása esetén F vegyületként egy vagy több diolt, szerves foszfátot, amint, diamint, poliamint, triolt és/vagy tiolt alkalmazunk,
rezolok vagy rezitek alkalmazása esetén F vegyületként egy vagy több diolt és/vagy fenolt alkalmazunk,
Mannich-bázis alkalmazása esetén F-vegyületként egy vagy több diolt, amint, diamint, poliamint és/vagy triolt alkalmazunk.

19. A 16-18. igénypontok bármelyike szerinti eljárás, **azzal jellemezve**, hogy az átalakítást extruderben, extruder reaktorban, dagasztó-keverőgépben végezzük.

20. A 16-19. igénypontok bármelyike szerinti eljárás, **azzal jellemezve**, hogy az átalakítást szabályozható hőmérsékletű reaktorban végezzük.

21. Az 1-15. igénypontok bármelyike szerinti reaktív szol alkalmazása polimer bevonóanyagok, ragasztók, elasztomorerek, tömítőanyagok vagy fröccsanyagok előállítására.

22. Az 1-15. igénypontok bármelyike szerinti reaktív szol alkalmazása bevonóanyagok, emlékező hab-polimerek, kompozitok, társított anyagok, többrétegű anyagok, gyógyászati műszer alkatrészek, protézisek és rehabilitációs eszköz alkatrészek előállítására.