

Dispersions

The invention provides dispersions, a process for preparing these and their use in the preparation of coating mixtures to prepare inkjet media.

It is known that dispersions, for example water-based dispersions, can be prepared from pyrogenically prepared oxides. From these aqueous dispersions can be prepared, for example, coating mixtures which are applied to paper or films. The coated films may then be printed using an inkjet printer. In this case, one objective is to obtain dispersions which are filled as highly as possible (high solids content) but with a low viscosity.

The invention provides:

dispersions which are characterised in that they consist of a liquid phase, preferably water, and a solid phase, wherein the solid phase consists of a pyrogenic oxide, doped using an aerosol, the BET surface area of which is between 5 and 600 m²/g, the basic component in the pyrogenic oxide is silica prepared by the method of flame hydrolysis or flame oxidation which has been doped with one or more doping component(s), preferably an aluminium oxide, doped by the method of aerosol application, wherein the amount of doped material is between 1 and 200 000 ppm and the doping component(s) are applied via a salt or a salt mixture of the doping component(s), and the solid phase in the dispersion is present in a proportion by weight between 0.001 and 80 wt.%.

The invention also provides a process for preparing dispersions according to the invention which is characterised in that a pyrogenic oxide, doped using an aerosol, is introduced into a liquid, preferably water, in a proportion by weight between 0.001 and 80 wt.% and this dispersion is then subjected to a milling step, wherein the milling procedure itself may be performed using a ball mill or pearl mill or a high-pressure mill or any other known

mill, preferably using a dispersing system in accordance with the rotor-stator principle (Ultra-Turrax).

The invention also provides the use of dispersions according to the invention to prepare coating mixtures in particular for inkjet papers or inkjet films or other inkjet materials, as a ceramic raw material, in the electrical industry, as a polishing agent (CMP applications), in the cosmetics industry, in the silicone and rubber industry, to adjust the rheology of liquid systems, as a starting material for preparing glass or glass coatings or melting crucibles, in dentistry, in the pharmaceutical industry, in PET film applications, as a starting material for preparing filter ceramics or filters, as a rust prevention agent, in inks and in battery separators.

The advantage of using highly filled and low viscosity dispersions according to the invention in paper production (that is in the preparation of coating mixtures for coating papers and other media) is based on the fact that proportionately less water has to be evaporated in the drying step after applying the dispersion (coating mixture). This provides a clear saving in energy.

Examples

EP 850 876 discloses pyrogenic oxides which are based on silicon dioxide which has been doped with one or more components, wherein doping takes place by introducing an aerosol to the flame. A pyrogenic silica doped with aluminium oxide is prepared in accordance with the process described in that patent. From this doped pyrogenic silica is prepared a highly filled aqueous dispersion which has a low viscosity. From this aqueous dispersion are prepared, by adding further components, coating mixtures which are applied to a film and are printed with an inkjet printer. The films prepared in this way have excellent print quality.

Example 1: Preparing a pyrogenic silicon oxide which is doped using an aerosol.

A doped pyrogenically prepared silicon dioxide is prepared in a burner arrangement like the one described in EP 0 850 876, in accordance with example 2 in that patent.

85 kg/h of SiCl_4 are evaporated and mixed with $51 \text{ Nm}^3/\text{h}$ of hydrogen and with $70 \text{ Nm}^3/\text{h}$ of a nitrogen/oxygen mixture (containing 35 vol.% O_2 , remainder N_2) and fed into the central tube in the burner. The gas mixture flows out of a nozzle and burns in a water-cooled combustion chamber.

$4 \text{ Nm}^3/\text{h}$ of (jacket) hydrogen flows out of the jacket nozzle which surrounds the central nozzle, in order to avoid incrustations. $70 \text{ Nm}^3/\text{h}$ of secondary air are also introduced into the combustion chamber.

An aerosol flows into the central tube out of an axial tube located in the central tube. The aerosol is an aluminium chloride aerosol which is produced by atomising a 15 percent aqueous AlCl_3 solution, using a two-fluid nozzle. An aerosol flow of 1 kg/h (aqueous salt solution) is produced, wherein a carrier gas stream of $16 \text{ Nm}^3/\text{h}$ of air conveys the aerosol through a heating section where it is thus heated. The air/aerosol gas mixture then enters the central tube from the axial tube at about 180°C .

The aerosol is burned together with the air/ SiCl_4 mixture. The reaction gases and the pyrogenically prepared silica doped with aluminium oxide produced are removed under suction through a cooling system, and cooled thereby, in a known manner by applying a reduced pressure. The solid material (doped pyrogenic oxide) is separated from the gas stream in a filter or a cyclone.

The doped pyrogenically prepared silica is produced as a white finely divided powder. Adhering residues of hydrochloric acid are removed by treating at elevated temperature with air which contains water vapour.

The pyrogenic silicon dioxide doped using an aerosol has the following physico-chemical characteristics:

BET:	60 m ² /g
pH (4% aqu. disp.):	3.9
Compacted bulk density:	142 g/l
Chloride content:	180 ppm
Al ₂ O ₃ content	0.19 wt. %
DBP absorption:	73 g/100 g

(DBP: dibutyl phthalate)

An aqueous dispersion is prepared with the doped pyrogenic oxide. The commercially available Aerosils (pyrogenically prepared silica) provided by Degussa-Hüls AG /Frankfurt, OX 50 and Aerosil 90, are used as comparison examples.

Table 1 gives the characteristics of the oxides:

Table 1:

Physico-chemical characteristics of the doped pyrogenic oxide in accordance with example 1 and the comparison examples

	Doped pyrogenic oxide according to ex. 1	OX 50	Aerosil 90
BET m²/g	60	50	90
pH (4% aqu. disp.)	3.9	3.8 - 4.8	3.7 - 4.7
Compacted bulk density g/l	142	130	80
Chloride content ppm	180	< 250	< 250
Al ₂ O ₃ content wt.%	0.19	<0.08	0.05
SiO ₂ content wt.%	99.8	>99.8	>99.8

An aqueous dispersion is prepared using these three different pyrogenic oxides.

This is achieved using a rotor-stator system (Ultra-Turrax) with a dispersion time of 30 minutes in a double-walled vessel (with water cooling). It is attempted to prepare a 40% (with respect to solids) dispersion ($w = 0.40$). Basically, this dispersion may also be prepared, however, by using other dispersion equipment such as, for example, ball mills or pearl mills or various types of jet or high-pressure mills (jets of liquid directed towards each other).

It is shown that it is not possible to produce a 40% dispersion with Aerosil 90 using this system because the system is too highly viscous.

The viscosity of the dispersions prepared in this way (doped oxide and Aerosil OX 50) is measured after 2 h with a Brookfield viscometer.

Table: 2

Viscosity of the 40% aqueous dispersion

	Doped pyrogenic oxide according to example 1	OX 50	Aerosil 90: not possible to prepare a 40% dispersion using Ultra-Turrax
5 rpm	2420	2320	> 10,000
10 rpm	1520	1320	
20 rpm	970	745	
50 rpm	554	372	
100 rpm	370	256	

rpm = revolutions per minute of the Brookfield viscometer

Inkjet coating mixtures are prepared from these 40% aqueous dispersions.

Formulation for preparing an inkjet coating mixture:

2 dispersions, A and B, are prepared.

Dispersion A is a 40% ($w = 0.40$) aqueous dispersion which contains the pyrogenic oxide (or the doped pyrogenic oxide). This is prepared by dispersing the pyrogenic oxide or doped oxide for 30 minutes with an Ultra-Turrax system in a water-cooled double-jacket system.

Dispersion B is a 10% (with respect to PVA) aqueous dispersion of polyvinyl alcohol (solid, abbreviated to PVA), Mowiol 26-88 from the Clariant Co.

The two dispersions A and B are combined over the course of 10 minutes by stirring at 500 rpm with a dissolver disc to give a dispersion C.

Dispersions A and B are mixed in such a way that a ratio by weight of Aerosil (or doped pyrogenic oxide) to PVA of 100:20 is produced in subsequent dispersion C. In the case of a 40% dispersion A, this is mixed with dispersion B in the ratio by weight of 1.25:1 in order to achieve the required ratio by weight (100:20 for the solids).

Furthermore (if required) enough water is added in order to produce a 24% dispersion C, with respect to the sum of the solids (pyrogenic oxide + PVA).

The viscosity of this dispersion C, the inkjet coating mixture, is measured after 24 h using a Brookfield viscometer.

Table 3:

Viscosity of the coating mixture measured after 24 h:

	Doped oxide according to example 1	OX 50	Aerosil 90
Solids content of the coating mixture (pyrogenic oxide + PVA) wt.%	24	24	22.5
Viscosity [mPas] at 100 rpm	3244	685	3352

(Note: In the case of preparing the coating mixture from Aerosil 90, a 30% aqueous dispersion is used initially.)

These coating mixtures are applied to an untreated polyester film (thickness 100 micrometres) with the aid of a shaped spreading rod. The wet film thickness of the coating mixture is 120 micrometres. The coating is dried at 105°C for 8 minutes.

The film with the applied coating is printed on an Epson Stylus Colour 800 using the highest resolution.

Table 4:

Assessing the printing results:

Property assessed	Doped pyrogenic oxide according to example 1		OX 50		Aerosil 90	
	Assessment	Score	Assessment	Score	Assessment	Score
Colour intensity	good	2	adequate	4	satisfactory	3
Resolution	very good	1	satisfactory	3	good	2
Colour running (bleeding)	no bleeding	1	fairly pronounced bleeding	4	small amount of bleeding	2.5
Drying time	very short	1	short	2.5	very short	1
Adhesion to film	good	2	good	2	poor	5
Average	very good to good	1.4	satisfactory	3.1	satisfactory	2.7

Best score 1, poorest score 6:

Taking the sum of all the properties of the coatings, in particular with regard to print quality, the aqueous dispersion prepared from the doped oxide and the coating mixture produced therefrom and the coating again prepared

therefrom exhibits by far the best results for printing with an inkjet printer with a very short drying time.

The viscosity of the aqueous dispersion of the doped oxide is much lower than that of a dispersion of Aerosil 90, with which, for example, a 40% aqueous dispersion cannot be prepared using this method of dispersion.

In the case of Aerosil OX 50, which has a comparable BET surface area to that of the doped oxide, although an aqueous dispersion which has a similar viscosity to that of the doped oxide can be prepared, the print quality of the layer prepared therefrom (using the coating mixture previously prepared therefrom) is not of acceptable quality.

Using the dispersion according to the invention, it is also possible to obtain a high solids content in the coating mixture which means that much less energy has to be expended when drying the coating.

If the results in accordance with table 4 for the coating mixtures are compared it can be seen quite clearly that the doped oxide, via the dispersion prepared therefrom, provides by far the best printing results. The adhesion of the coating mixture which was prepared from the aqueous dispersion according to the invention to the film was also very good.

Although dispersions made with Aerosil OX 50 also have a relatively low viscosity, the print quality of the coating mixtures, or coatings, prepared therefrom is not acceptable.

Claims

1. Dispersions, characterised in that they consist of a liquid phase, preferably water, and a solid phase, wherein the solid phase consists of a pyrogenic oxide, doped using an aerosol, the BET surface area of which is between 5 and 600 m²/g, the basic component in the pyrogenic oxide is silica prepared by the method of flame hydrolysis or flame oxidation which has been doped with one or more doping component(s), preferably an aluminium oxide doped by the method of aerosol application, wherein the amount of doped material is between 1 and 200 000 ppm and the doping component(s) are applied via a salt or a salt mixture of the doping component(s), and the solid phase in the dispersion is present in a proportion by weight between 0.001 and 80 wt.%.
2. A process for preparing dispersions according to Claim 1, characterised in that a pyrogenic oxide, doped using an aerosol, is introduced into a liquid, preferably water, in a proportion by weight between 0.001 and 80 wt.% , and this dispersion is then subjected to a milling procedure, wherein milling itself is performed using a ball mill or a pearl mill or a high-pressure mill or any other known mill, preferably using a dispersing system in accordance with the rotor-stator principle (Ultra-Turrax).
3. The use of dispersions according to Claim 1 to prepare coating mixtures, in particular for inkjet papers or inkjet films or other inkjet materials, as a ceramics raw material, in the electrical industry, as a polishing agent (CMP applications), in the cosmetics industry, in the silicone and rubber industry, to adjust the rheology of fluid systems, to prepare glass or glass coatings or melting crucibles, in dentistry, in the pharmaceutical industry, in PET film applications, as



a starting material for preparing filter ceramics or filters, as a rust prevention agent, in inks and in battery separators.

