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(71) **Demandeur/Applicant**:
DEGUSSA AG, DE

(72) **Inventeurs/Inventors**:
LINDNER, GOTTLIEB-GEORG, DE;
KUHLMANN, ROBERT, DE;
DREXEL, CLAUS-PETER, DE

(74) **Agent**: FETHERSTONHAUGH & CO.

(54) **Titre** : SILICE OBTENUE PAR PRECIPITATION, A ALCALINITE CONSTANTE, ET UTILISATION DUDIT PRODUIT
(54) **Title**: SILICA BY PRECIPITATION AT CONSTANT ALKALI NUMBER, AND ITS USE

(57) **Abrégé/Abstract**:

Disclosed are precipitated silicas obtainable by acid precipitation of aqueous silicate solutions while maintaining a constant alkali number of at least 1, having good sorption characteristics for polar compounds. The precipitated silicas are useful as carrier material for choline chloride for example, and feed additives, chemical intermediates and in laundry detergent.

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Abstract:

Disclosed are precipitated silicas obtainable by acid precipitation of aqueous silicate solutions while maintaining a constant alkali number of at least 1, having good sorption characteristics for polar compounds. The precipitated silicas are useful as carrier material for choline chloride for example, and feed additives, chemical intermediates and in laundry detergent.

Silica by precipitation at constant alkali number, and its use

The invention relates to silicas obtainable by acid precipitation of alkali metal silicates at constant alkali number, and to their use as carrier silicas.

Precipitated silicas as carrier material, particularly for vitamin E acetate or choline chloride, have long been known. For example, it is described in EP 0 937 755 how a precipitated silica is prepared by a pH-controlled precipitation reaction and then spray-drying. A precipitated silica prepared in this way is particularly suitable for use for adsorbing liquid active substances such as a solution of choline chloride or vitamin E, for example.

German Patent Publication (DE) 198 60 441 discloses how an active substance adsorbate may be prepared from a precipitated silica and an active substance by spraying or injecting a silica suspension together with one or more active substances into a fluidized bed generated using hot air.

It is likewise possible to use hydrophobic precipitated silicas for these purposes, as described in DE 198 25 687.

In the context of their use as carriers, the following properties of silicas are important: adsorption capacity, good sorption kinetics, and low fine dust fraction. Owing to heightened safety requirements and the need to prepare adsorbates with ever higher concentrations, there is therefore a demand for carrier silicas which have a very low fines fraction with an adsorptiveness which is heightened at the same time. The known silicas generally do not possess pronounced sorption characteristics for polar compounds. Since silicas are frequently used as carrier material for polar compounds such as choline chloride, propionic acid or formic acid, for example, it is an object of the present invention to provide a silica which possesses particularly good sorbency for polar compounds.

It has surprisingly been found that by preparing the precipitated silicas at a constant alkali number, products can be obtained which have good sorption characteristics for polar compounds.

The present invention provides a process for preparing silicates by:

introducing an aqueous silicate solution;

simultaneously metering in an aqueous silicate solution and a Lewis and/or Brønsted acid;

reacidifying to a pH of 7-3.0;

filtering; and

drying,

wherein the metered addition of the aqueous silicate solution and the Lewis and/or Brønsted acid is carried out while maintaining an alkali number of a mixture thereof at a substantially constant value that is at least 1.

The high alkali number of the precipitated silicas of the invention results in a high silanol group density and so enhances a high absorbency for polar absorbates.

The present invention likewise provides for the use of the silicates of the invention as carrier material for feed additives, chemical intermediates, or for use in the laundry detergent industry, for example.

It is possible to add an electrolyte prior to, or during the simultaneous addition of the aqueous silicate solution and the acid.

Electrolytes for the purposes of the present invention are alkali or alkaline earth metal salts (or their aqueous solutions) which are not incorporated into the amorphous SiO_2 structure. Examples include Na, K, Rb, Ba, which in each case is in the sulfate, acetate, halide or carbonate form. The amount of the electrolyte is preferably 0.01-26%, more preferably 0.1-3%, by weight (calculated as the metal ion).

It is likewise possible to add metal salts (or their aqueous solutions) which are incorporated into the SiO_2 structure to the precipitation mixture, thereby giving silicates. The amount of these metal ions may be between 1 and 50, preferably about 10% by weight; customary ions are Al, Zr, Ti, Fe, Ca and Mg.

There are known preparation processes for precipitated silicas in the course of which a constant pH is maintained. A precipitation reaction at a constant alkali number, on the other hand, means that the concentration of freely available sodium ions is kept constant.

As a result of the acid-base reactions during the precipitation, for example, of waterglass with sulfuric acid, sodium ions are on the one hand released in the form of sodium sulfate; on the other hand, sodium ions are incorporated into the silicate agglomerates which form. Since these two reactions proceed independently of one another kinetically, the course of precipitations at constant pH is different than that of precipitations conducted in accordance with the invention.

In the case of a precipitation reaction at a substantially constant alkali number, the pH changes analogously: for example, at a constant alkali number of 30, the pH falls from about 10.35 to levels between 8 and 10, depending on the duration of the precipitation reaction (simultaneous addition of an alkaline silicate solution and an acid). The longer such a precipitation reaction lasts, the lower the pH is at the end of the reaction. The intercalation of sodium ions into the silica structure is probably responsible for this.

The precipitated silicas of the invention are prepared at an alkali number of at least 1, in particular at least 15, preferably at an alkali number of from 15 to 60, with particular preference at an alkali number of from 25 to 50, and with very particular preference at an alkali number of from 30 to 40, the alkali number being kept substantially constant during the precipitation reaction. The alkali number during the simultaneous addition of the silicate and the acid may vary somewhat, for example, within 10% of the target alkali number.

The alkali number (AN) is determined by measuring the consumption of hydrochloric acid in a direct potentiometric titration of the precipitation suspension at a pH of 8.3, i.e., the color change point of phenolphthalein. The consumption of hydrochloric acid is a measure of the free alkali content of the solution or suspension. Owing to the temperature dependency of the pH, this measurement is made at 40°C and after a waiting time of 15 minutes. A precise description of the measurement protocol is given in the examples.

The invention provides a precipitated silica, characterized by the following physicochemical data:

BET surface area	from 50 to 700 m^2/g with the preferential ranges 100-300 m^2/g, 150-220 m^2/g, 180-210 m^2/g.
DBP absorption	from 100 to 450 g/100 g with the preferential ranges 250-450 g/100 g, 280-450 g/100 g,
Choline chloride absorption	from 50 to 400 g/100 g with the preferential ranges 240-400 g/100 g, 280-400 g/100 g (as measured in a 75% by weight aqueous solution),
CTAB surface area	from 50 to 350 m^2/g with the preferential ranges 100-250 m^2/g, 130-200 m^2/g,

the ratio of the DBP absorption to the choline chloride absorption, as a measure of the adsorption of a nonpolar substance and of a polar substance, being less than 1.07, preferably less than 1.05, with very particular preference less than 1.03. The ratio is usually 0.98 or more.

Since silicas and silicates possess different affinities for hydrophobic (i.e., nonpolar) compounds and hydrophilic (i.e., polar) compounds, two measurements are necessary for complete characterization of this property. The DBP number is used as a measure of an affinity of silicas for hydrophobic compounds; the choline chloride absorption is used as a measure of the affinity of silicas for hydrophilic compounds. The ratio of these DBP/choline chloride absorption measurements therefore reflects a new physical property.

The silicates or precipitated silicas of the invention may additionally be characterized by the modified Sears number. The modified Sears number is determined by the techniques described in the examples/methods and may be greater than 20, preferably greater than 25, with particular preference greater than 28.

A preferred aqueous silicate solution is sodium silicate solution. A particularly preferred aqueous silicate solution is waterglass. As for the Brønsted acid, sulfuric acid, hydrochloric acid, carbonic acid or acetic acid may be used. As the Lewis acid it is possible to use Al^{3+} ions, in the form, for example, of the sulfate.

After the simultaneous metering of the aqueous silicate solution and the acid, the acid is further added to the mixture until a pH of 7-3.0, preferably 4.0-3.3, is reached (reacidification), to obtain a suspension.

The BET surface area is determined in accordance with ISO 5794/1, Annex D, the CTAB surface area in accordance with ASTM D 3765-92, the DBP absorption in accordance with the protocol described in the annex.

The precipitated silica suspensions prepared by the process of the invention are filtered conventionally and the filter cake is washed with water. The filter cake obtained in this way is liquefied, where appropriate, and dried by the customary drying process, such as rotary tube furnace, Büttner dryer, spin-flash dryer, pulse combustion dryer, spray dryer, or in a nozzle tower. Further, purely physical treatment by granulation and/or grinding is likewise possible. Also possible is a water repellency treatment or coating with waxes.

The silicates or precipitated silicas of the invention may be used in particular as carriers for feed additives such as formic acid, propionic acid, lactic acid, phosphoric acid, choline chloride solution or plant extracts, tagatese extract for example.

Furthermore, the precipitated silicas of the invention may be used as carrier material for chemical intermediates such as melamine resins or coatings additives or in the laundry detergent industry as carriers for fragrances or detergents.

Moreover, the silicates or precipitated silicas of the invention may be used as a filler in elastomers/plastics, battery separators, toothpastes, catalyst supports, or as a flocculation assistant.

The following examples and measurement protocols are intended to illustrate the invention without restricting its scope.

Examples

General experimental protocol: (Example 1-8)

Water is charged to a precipitation vessel with a capacity of 2 m^3 (applies to all pilot-scale trials; laboratory trials: 40 l; plant trials: 80 m^3) and a certain amount of waterglass (i.e., sodium silicate solution) is metered in. The values for the density of the sodium silicate solution, sulfuric acid, the SiO_2 content, Na_2O content, temperature, and the alkali number (AN number) are evident from the tables. After the target temperature has been reached, sodium silicate solution and sulfuric acid are metered in. Thereafter, sulfuric acid continues to be metered in at the same rate until a pH of 3.5 is reached. The suspension with the described solids content is filtered on filter presses (membrane filter presses) and then prepared for drying. The filter cake is liquefied by adding sulfuric acid, using a shearing unit, until the desired viscosity and pH are reached. The feed is then dried.

List of abbreviations:

AN = alkali number

WGL charge = waterglass charge = initial charge of sodium silicate solution

WGL = waterglass

VA = time at which the viscosity increases sharply, also referred to as the gel point

Fc = precipitation rate in [mol/(l•min)], defined by

$$\frac{\text{ml/min (acid inflow)}\bullet\text{mol/l (acid molarity)}}{\text{l (initial charge)}}$$

% TS feed = % solids content of feed to dryer

GV-Din = loss on ignition to DIN

LF = conductivity

CC absorption = choline chloride absorption

Example	1	2	3	4
Trial No.	7508	7504	7487	7491
AN	30	40	40	40
Water charge (l)	1359	1220	1314	1220
WGL charge (l)	145.3	186.1	197.6	184.1
Temperature (°C)	85	85	85	85
WGL metered (l)	344.2	211.4	247.9	249.2
Acid metered (l)	26.32	16.42	19.32	20.15
VA (min)	22.50	37.00	38.50	41.00
Precipitation period (min)	60	55	55	65
Reacidification amount (l)	20.00	22.30	25.11	22.86
Reacidification time (min)	21		39	156
Fc	5.52	4.01	4.39	4.17
g/l (solids content of the earth suspension)	94.8	92.5	97.7	99.2
Waterglass analysis				
Density (g/ml)	1.346	1.346	1.349	1.349
% SiO_2	27.2	27.2	27.3	27.3
% Na_2O	7.99	7.99	8.08	8.08
Sulfuric acid (mol/l H_2SO_4)	18.14	18.14	18.14	18.14
Dryer	**Nozzle tower dryer**	**Nozzle tower dryer**	**Nozzle tower dryer**	**Nozzle tower dryer**
pH of feed	3.9	3.8	3.8	3.8
Viscosity (mPa*s)	40	75	110	120
% TS feed	17.3	18.3	19.7	20.3

Example	1	2	3	4
Analysis				
GV-DIN (%)	5.2	4.9	4.9	4.9
Water (%)	6.2	5.3	6.3	6.0
pH	6.5	6.5	6.9	6.1
LF (µS/cm)	610	600	550	650
N_2 surface area (m^2/g)	199	189	167	165
CTAB surface area (m^2/g)	148	127	120	113
DBP number (g/100 g) (corrected, i.e., for anhydrous material)	279	281	270	270
Sears number (V2)	31.3	30.9	29.4	29.5
CC absorption	276	282	270	275
Ratio of DBP/CC absorption	1.0109	0.9965	1.0000	0.9818

Example	**5**	**6**	**7**	**8**
Trial No.	7397	7362	7370	7420
AN	20	30	30	40
Water charge (l)	1651	1499	1459	1224
WGL charge (l)	108.4	148.8	148.8	185.3
Temperature (°C)	85	85	85	85
WGL metered (l)	345.0	441.8	542.5	224.4
Acid metered (l)	28.40	36.00	32.78	18.44
VA (min)	24.75	27.75	27.25	40.25
Precipitation period (min)	40	65	60	60
Reacidification amount (l)	20.42	28.41	24.70	26.37
Reacidification time (min)	41	63	56	94
Fc	7.21	5.96	6.03	3.86
g/l (solids content of the earth suspension)	79.3	104.0	100.0	92.5
Waterglass analysis				
Density (g/ml)	1.346	1.348	1.348	1.351
% SiO_2	27.4	27.2	27.2	27.6
% Na_2O	8.03	8.02	8.02	8.01
Sulfuric acid (mol/l H_2SO_4)	18.87	17.74	17.64	17.70
Dryer	**Nozzle tower dryer**	**Nozzle tower dryer**	**Nozzle tower dryer**	**Nozzle tower dryer**
pH of feed	3.6	3.6	3.4	3.6
Viscosity (mPa*s)	75	60	33	90
% TS feed	16.3	16.9	15.3	19.8

Example	5	6	7	8
Analysis				
GV-DIN (%)	5.1	5.3	5.8	5.6
Water (%)	5.8	6.2	5.0	5.9
pH	6.8	6.7	6.3	6.6
LF (µS/cm)	490	510	530	550
N_2 surface area (m^2/g)	240	175	185	137
CTAB surface area (m^2/g)	200	135	148	115
DBP number (g/100 g) (corrected, i.e., for anhydrous material)	247	293	292	276
Sears number (V2)	31.0	31.5	30.8	28.5
CC absorption	233	285	294	279
Ratio of DBP/CC absorption	1.0601	1.0281	0.9932	0.9892

Example	**Hubersil 5170** **9**	**HiSil SC72** **10**	**Sipernat 22** **11**	**Sipernat 2200** **12**
Trial No.				
AN				
Water charge (l)				
WGL charge (l)				
Temperature (°C)				
WGL metered (l)				
Acid metered (l)				
VA (min)				
Precipitation period (min)				
Reacidification amount (l)				
Reacidification time (min)				
Fc				
g/l (solids content of the earth suspension)				
Waterglass analysis				
Density (g/ml)				
% SiO_2				
% Na_2O				
Sulfuric acid (mol/l H_2SO_4)				
Dryer	**Granulation**	**Nozzle tower dryer**	**Spray dryer**	**Nozzle tower dryer**
pH of feed				
Viscosity (mPa*s)				
% TS feed				

Example	**Hubersil 5170** **9**	**HiSil SC72** **10**	**Sipernat 22** **11**	**Sipernat 2200** **12**
Analysis				
GV-DIN (%)				
Water (%)	6.0	5.7	5.0	5.0
pH				
LF (µS/cm)				
N_2 surface area (m^2/g)				
CTAB surface area (m^2/g)				
DBP number (g/100 g) (corrected, i.e., for anhydrous material)	204	311	270	255
Sears number (V2)	18.4	23.5		
CC absorption	165	270	235	231
Ratio of DBP/CC absorption	1.2364	1.1519	1.149	1.1039

Determining the modified Sears number of silicas, silicates and hydrophobic silicas

1. Scope

Free OH groups are detectable by titration with 0.1 N KOH in the range from pH 6 to pH 9.

2. Apparatus

2.1 Precision balance to 0.01 g precisely
2.2 Memotitrator DL 70 (made by Mettler) equipped with 10 ml and 20 ml buret, 1 pH electrode and 1 pump (e.g., NOUVAG™ pump, type SP 40/6)
2.3 Printer
2.4 Titration vessel 250 ml (made by Mettler)
2.5 Ultra-Turrax™ 8000-24,000 rpm
2.6 Thermostated waterbath
2.7 2 dispenser 10-100 ml for metering methanol and deionized water
2.8 1 dispenser 10-50 ml for metering deionized water
2.9 1 measuring cylinder 100 ml
2.10 IKA™ universal mill M20

3. Reagents

3.1 Methanol p.a.
3.2 Sodium chloride solution (250 g NaCl p.a. in 1000 ml deionized water)
3.3 0.1 N hydrochloric acid
3.4 0.1 N potassium hydroxide solution
3.5 Deionized water
3.6 Buffer solutions pH 7 and pH 9

4. Procedure

4.1 Sample preparation

Grind about 10 g of sample for 60 seconds in the IKA™ universal mill M20.

Important: Since only very finely ground samples lead to reproducible results, these conditions must be observed strictly.

4.2 Analytical procedure

4.2.1 Weigh out 2.50 g of the sample prepared in accordance with section 4.1 into a 250 ml titration vessel.

4.2.2 Add 60 ml of methanol p.a.

4.2.3 After complete wetting of the sample, add 40 ml of deionized water.

4.2.4 Disperse for 30 seconds using the Ultra-Turrax™ at a speed of about 18,000 rpm.

4.2.5 Rinse sample particles adhering to the vessel edge and stirrer into the suspension using 100 ml of deionized water.

4.2.6 Condition sample to 25°C in a thermostated water bath (for at least 20 minutes).

4.2.7 Calibrate pH electrode with the buffer solutions pH 7 and pH 9.

4.2.8 The sample is titrated in the Memotitrator™ DL 70 in accordance with method S 911. If the course of titration is unclear, a duplicate determination is carried out subsequently.

The results printed out are as follows:

pH
V_1 in ml/5 g
V_2 in ml/5g

Principle:

First of all the initial pH of the suspension is measured, then according to the result the pH is adjusted to 6 using KOH or HCl. Then 20 ml of NaCl solution are metered in. The titration is then continued to a pH of 9 using 0.1 N KOH.

Sears numbers:

$$\mathrm{Si-OH + NaCl} \Rightarrow \mathrm{Si-ONa + HCl}$$

$$\mathrm{HCl + KOH} \Rightarrow \mathrm{KCl + H_2O}$$

Calculation

$$V_1 = \frac{V*5}{E}$$

$$V_2 = \frac{V*5}{E}$$

V_1 = ml KOH or ml HCl to pH 6/5 g of substance

V_2 = ml KOH consumption to pH 9/5 g of substance

E = initial mass

6. **Appendix**

Titration conditions for Analysis S 911 on the Memotitrator™ DL 70.

Determining the alkali number:

The alkali number determination, referred to below for short as AN determination, is the consumption of hydrochloric acid in a direct potentiometric titration of alkaline charges or suspensions to a pH of 8.3 (viewed historically: pH 8.3 corresponds to the color change point of phenolphthalein); this gives the free alkali content of the solution or suspension.

The pH meter is calibrated at room temperature, the combined electrode is equilibrated to 40°C, and the sample mixture is then conditioned at 40°C, and on reaching that temperature the titration is conducted.

Because of the fairly long time for equilibrium to be established between the silica/silicate at the specified pH - in this case 8.3 - a waiting time is necessary until the consumption of acid is finally read off. Extensive investigations have found that for the AN determination a waiting time of 15 minutes must be observed, after which the equilibrium has established itself stably and good reproducibility is ensured.

Description of method:

pH meter calibration:

- Calibrating temperature of buffer solutions 20°C
- Temperature equilibration 20°C

Measurement of suspension:

- Temperature equilibration of pH meter at 40°C
- 50 ml of suspension
- 50 ml of distilled water
- Hydrochloric acid c = 0.5 mol/l
- Condition suspension to 40°C
- Determine acid consumption after 15 min titration time
- Titration end at pH 8.3

Accuracy of method: +/- 0.1 ml acid consumption

Determining the maximum choline chloride absorption:

Test means:

A. Test apparatus:

250 ml glass beaker, high form

Spatula
Precision balance

B. Test substances:

75% strength choline chloride solution [choline chloride, ultrapure (Merck)]
Silica under test

Calibration notes

When a new delivery test solution is received, it must be examined comparatively with the quality used up until that time.
Before use, the balances are to be tested for functionality and serviced annually.

Procedure:

10 g of the carrier silica under test are weighed out into a 250 ml glass beaker, high form, and 75% strength choline chloride solution is added dropwise, while stirring with the spatula. The mixture is observed continuously to check when the maximum absorption has been reached. When viewed closely, it is possible to make out white silica particles which stand out distinctly from waxlike (saturated) particles. The maximum choline chloride absorption has been achieved when there are no longer any unladen particles in the mixture and this mixture is not yet waxlike/greasy.

Evaluation:

$$\text{Max. choline chloride absorption in g/100 g} = \frac{(a-10)\text{x}100}{10}$$

a = total weight

CLAIMS:

1. A precipitated silica having:

a BET surface area of from 50 to 700 m^2/g;

a DBP absorption of from 100 to 450 g/100 g;

a choline chloride absorption of from 50 to 400 g/100 g (as measured in a 75% by weight aqueous solution);

a CTAB surface area of from 50 to 350 m^2/g; and

a DBP/choline chloride absorption of less than 1.07.

2. The precipitated silica of claim 1, having a BET surface area of from 100 to 300 m^2/g.

3. The precipitated silica of claim 1, having a BET surface area of from 150 to 220 m^2/g.

4. The precipitated silica of claim 1, having a BET surface area of from 180 to 210 m^2/g.

5. The precipitated silica of any one of claims 1-4, having a DBP absorption of from 250 to 450 g/100g.

6. The precipitated silica of any one of claims 1-4, having a DBP absorption of from 280 to 450 g/100g.

7. The precipitated silica of any one of claims 1-6, having a choline chloride absorption of from 240 to 400 g/100g (as measured in a 75% by weight aqueous solution).

8. The precipitated silica of any one of claims 1-6, having a choline chloride absorption of from 280 to 400 g/100g (as measured in a 75% by weight aqueous solution).

9. The precipitated silica of any one of claims 1-8, having a CTAB surface area of from 100 to 250 m^2/g.

10. The precipitated silica of any one of claims 1-8, having a CTAB surface area of from 130 to 200 m^2/g.

11. The precipitated silica of any one of claims 1-10, having a DBP/choline chloride absorption ratio of less than 1.05.

12. The precipitated silica of any one of claims 1-10 having a DBP/choline chloride absorption ratio of less than 1.03.

13. The precipitated silica of any one of claims 1-12, containing between 0.01 and 26% by weight of an electrolyte.

14. The precipitated silica of claim 13, wherein the electrolyte is an acetate, halide or carbonate of a metal ion selected from the group consisting of Na, K, Rb and Ba.

15. The precipitated silica of any one of claims 1-12, containing between 1 and 50% by weight of metal ions from the group consisting of Al, Mg, Ca, Ti, Zr, and Fe.

16. The precipitated silica of any one of claims 1-12, containing about 10% by weight of metal ions selected from the group consisting of Al, Mg, Ca, Ti, Zr and Fe.

17. The precipitated silica of one of claims 1 to 16, which has a modified Sears number of at least 20.

18. The precipitated silica of any one of claims 1-16, which has a modified Sears number of at least 25.

19. The precipitated silica of any one of claims 1-16, which has a modified Sears number of at least 28.

20. A process for preparing precipitated silica, which comprises:

introducing an aqueous silicate solution;

simultaneously metering in an aqueous silicate solution and a Lewis or Brønsted acid;

reacidifying to a pH of 7-3.0;

filtering; and

drying,

wherein the metered addition of the aqueous silicate solution and the Lewis or Brønsted acid is carried out while maintaining a constant alkali number of at least 1.

21. The process of claim 20, wherein the constant alkali number is at least 15.

22. The process of claim 20, wherein the constant alkali number is between 15 and 60.

23. The process of claim 20, wherein the constant alkali number is between 25 and 50.

24. The process of claim 20, wherein the constant alkali number is between 30 and 40.

25. The process of one of claims 20-24, wherein an electrolyte is added prior to or during the simultaneous addition of the aqueous silicate solution and the Lewis or Brønsted acid.

26. The process of one of claims 20-24, wherein ions from the group consisting of Al, Ti, Zr, Fe, Mg and Ca are added prior to or during the simultaneous addition of the aqueous silicate solution and the Lewis or Brønsted acid.

27. A use of the precipitated silica of one of claims 1 to 20 as carrier material for feed additives, chemical intermediates, or in the laundry detergent industry.

28. A use of the precipitated silica of one of claims 1 to 20 as a carrier for formic acid, propionic acid, lactic acid, phosphoric acid, choline chloride solution, plant extracts, melamine resins, coatings additives, fragrances or detergents.

29. A use of the precipitated silica of one of claims 1 to 20 in elastomers/plastics, battery separators, toothpastes, catalyst supports or as a flocculation assistant.

30. A process for preparing the precipitated silica as defined in any one of claims 1 to 12, which comprises:

(A) initially introducing an aqueous solution of sodium silicate into a precipitation vessel;

(B) simultaneously metering in an aqueous solution of sodium silicate and sulfuric acid into the precipitation vessel, while maintaining an alkali number of a resulting mixture at a constant value that is within the range of from 15 to 60;

(C) further metering in sulfuric acid into the precipitation vessel until a pH of 7-3.0 is reached, thereby obtaining a suspension of precipitated silica;

(D) filtering the precipitated silica, thereby obtaining a filter cake of the precipitated silica;

(E) washing the filter cake of the precipitated silica with water; and

(F) drying the precipitated silica.

31. The process of claim 30, which further comprises:

(G) liquefying the filter cake washed with water in step (E), by adding sulfuric acid, before drying the precipitated silica in step (F).

32. The process of claim 30 or 31, wherein the sodium silicate is waterglass.

33. The process of any one of claims 30 to 32, wherein sulfuric acid is metered in until a pH of 4.0-3.3 is reached in step (C).

34. The process of any one of claims 30 to 33, wherein the constant alkali number maintained in step (B) is from 30 to 40.

FETHERSTONHAUGH & CO.
OTTAWA, CANADA

PATENT AGENTS