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Preparing and stabilizing functional metal nanoparticle comprises reduction of soluble metal salts with hydrogen in ionic fluids, where the particle size of obtained nanoparticle is adjustable by variation of anions of the ionic fluids

Patent Number(s): DE102007038879-A1 ; WO2009024312-A2 ; WO2009024312-A3

Inventor(s): BEYERSDORFF T, JANIAC C, KLINGELE M, REDEL E, SCHUBERT T

Patent Assignee Name(s) and Code(s): UNIV FREIBURG ALBERT-LUDWIGS (UYFR-Non-standard)

IOLITEC IONIC LIQUID TECHNOLOGIES GMBH (IOLI-Non-standard)

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Derwent Primary Accession Number: 2009-F01872 [10]

Patents Cited by Examiner: 1

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Abstract: NOVELTY - Preparing and stabilizing functional metal nanoparticle with a particle size of 0.1-1000 nm comprises reduction of soluble metal salts with hydrogen in ionic fluids, where the particle size of the resulting metal nanoparticle is adjustable by variation of anions of the used ionic fluids.

USE - The method is useful for preparing and stabilizing functional metal nanoparticle, where the prepared nanoparticle is silver-, copper-, cobalt-, iron-, iridium-, rhodium-, palladium-, platinum-, gold-, ruthenium-, nickel-, zinc-, cadmium-, manganese-, rhenium-, chromium-, molybdenum-, tungsten-, vanadium-, niobium-, tantalum-, titanium-, zirconium-, hafnium-, scandium- or yttrium-nanoparticle. The initially obtained metal nanoparticle-dispersion is used directly as active material in the catalyst process; or as active sensor component. The initially obtained metal nanoparticle-dispersion directly or after a downstream formulation step is used as antimicrobial- or decontamination-coating (all claimed).

ADVANTAGE - The method allows for the adjustment of the particle size of the metal nanoparticles and the used ionic fluid is reusable. The obtained metal nanoparticle is stable. The method is suitable for large-scale industrial preparation of the nanoparticles without any contamination. The ionic fluid used is inflammable, non-toxic or non-volatile.

Technology Focus/Extension Abstract: TECHNOLOGY FOCUS - INORGANIC CHEMISTRY - Preferred Method: In the method, the particle size distribution is attained by the addition of a proton catcher to the reaction mixture, preferably a nitrogen base is added as the proton catcher in the reaction mixture, which in its protonated form has a molar volume similar to that of the cations of the ionic fluids. The nitrogen base lies at the bottom to the cations of the ionic fluids. The reduction with hydrogen is carried out in an autoclave at a pressure of less than or equal to 5 bar and at less than or equal to 120 degrees C. The prepared metal nanoparticle is isolated from an initially obtained metal nanoparticle-dispersion. After the isolation of the metal nanoparticle, the ionic fluid is recovered, cleaned and reused for further reactions. The initially obtained metal nanoparticle-dispersion is treated with air, oxygen or other oxidant to obtain a metal oxide nanoparticle. Preferred Components: The ionic fluid is a salt or mixture of salts of formula ((Q-n+)_m(Z-m-n) (I) formed from cations of formula ((Q-n+)) (II) and anions of formula

Additional information

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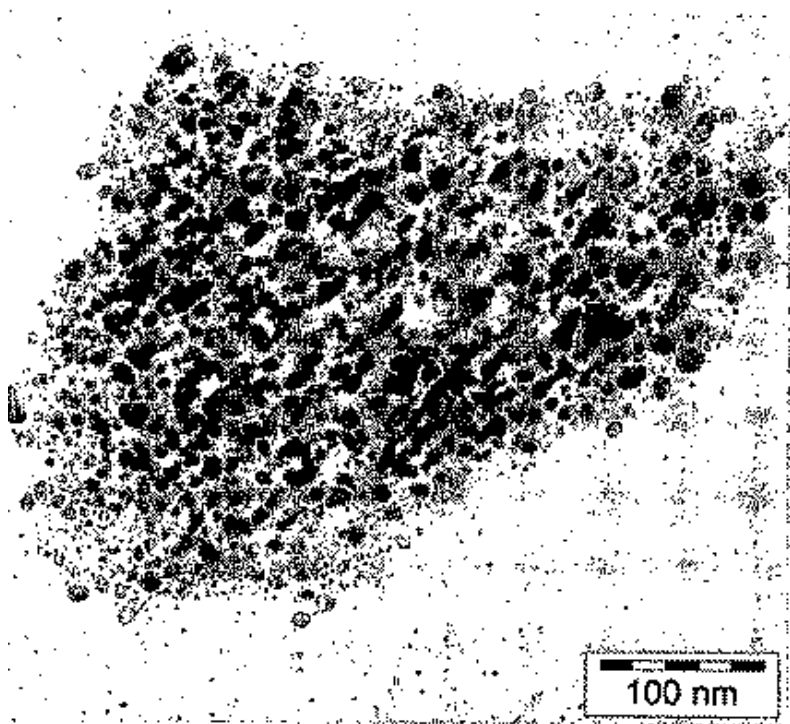
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((Z-m-)) (III). The ionic fluid has a melting point of less than 180 degrees C (preferably less than 100 degrees C). The ionic fluid is hydrophobic. The ionic fluid has the same anion as that of the metal salt. Q = imidazolium-, ammonium-, pyrrolidinium-, piperidinium-, pyridinium-, morpholinium-, guanidinium-, benzotriazolium-, quinolinium-, isoquinolinium-, pyrazolium-, 1,4-diaza-bicyclo(2.2.2)-octan-1-ium-, 1,2,4-triazolium-, pyridazinium-, pyrimidinium-, pyrazinium-, 1,3,5-triazinium-, 1,2,3-triazolium-, piperazinium-, oxazolium-, oxazolidinium-, thiazolium-, quinoxalinium-, benzimidazolium-, imidazolidinium-, indolinium- or thiomorpholinium-cation; Z = halide, polyhalide, pseudohalide, phosphate, phosphite, phosphonate, phosphinate, sulfate, sulfonate, borate, boronate, carboxylate, carbonate, alkoxide, bis(trifluoromethylsulfonyl)imide, bis(pentafluoroethylsulfonyl)imide, tricyanomethide, tris(trifluoromethylsulfonyl)methide, hexafluoroantimonate, hexafluoroarsenate, nitrate, nitrite, tetrachloroferrate(III), tetrabromoferrate(III), tetrachloroaluminate, heptachlorodialuminate, decachlorotrialuminate, tetrabromoaluminate, hexafluorosilicate or hexacyanoferrate(III) anion; and n, m = 1-4 (preferably 1).

EXAMPLE - Silver tetrafluoroborate (0.035 g) and 1-butyl-imidazole (0.040 g) were dissolved in 1-butyl-3-methyl-imidazolium-tetrafluoroborate (2 g) under argon and at room temperature. The solution was transferred under argon into a high grade steel autoclave and dried at hot vacuum for 20 minutes and 0.1 mbar. Subsequently the autoclave was filled directly with hydrogen to a pressure of 4 bar and the reaction mixture was heated for 2 hours at 85 degrees C. Subsequently, the autoclave was evacuated in high vacuum for 1 hour at 100 degrees C and 0.1 mbar to remove excess 1-butyl-imidazole from the reaction mixture and to obtain silver nanoparticle having a particle size of 2.80 plus minus 0.78 nm, where the minimal- and maximal-size were 1.25 nm and 4.68 nm, respectively.

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Drawing:



International Patent Classification: B01J-013/00; B22F-001/00; B22F-009/16; B22F-009/24; A01N-059/16; C02F-001/50; C09C-001/00; C09C-003/00

Derwent Class Code(s): D22 (Sterilising, bandages, dressing and skin-protection agents); E19 (Other organic compounds general - unknown structure, mixtures)

Derwent Manual Code(s): D09-A01; E05-S; E05-T; E06-D; E07-D; E07-E01; E07-E03; E07-F01; E07-F02; E10-A08C; E10-A10D; E10-A15A; E10-A17B; E10-A22; E10-C04J2U; E10-C04L; E10-E04L; E31-B03C; E31-B03D; E31-F05; E31-H05; E31-K05; E31-K07; E31-L; E31-M; E31-N05C; E31-P06B; E31-Q02; E31-Q07; E32-B; E34-C03; E35-U05

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DE102007038879-A1	19 Feb 2009	B01J-013/00	200916	Pages: 21	German
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WO2009024312-A3	23 Dec 2009		201002		German

Application Details:

DE102007038879-A1	DE10038879	17 Aug 2007
WO2009024312-A2	WOEP006768	18 Aug 2008
WO2009024312-A3	WOEP006768	18 Aug 2008

Priority Application Information and Date:

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Designated States:

WO2009024312-A2:

(National): AE; AG; AL; AM; AO; AT; AU; AZ; BA; BB; BG; BH; BR; BW; BY; BZ; CA; CH; CN; CO; CR; CU; CZ; DK; DM; DO; DZ; EC; EE; EG; ES; FI; GB; GD; GE; GH; GM; GT; HN; HR; HU; ID; IL; IN; IS; JP; KE; KG; KM; KN; KP; KR; KZ; LA; LC; LK; LR; LS; LT; LU; LY; MA; MD; ME; MG; MK; MN; MW; MX; MY; MZ; NA; NG; NI; NO; NZ; OM; PG; PH; PL; PT; RO; RS; RU; SC; SD; SE; SG; SK; SL; SM; ST; SV; SY; TJ; TM; TN; TR; TT; TZ; UA; UG; US; UZ; VC; VN; ZA; ZM; ZW

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Compound(s):

DCR Number	Role	DCR Number	Role	DCR Number	Role
180533-0-0-0	(K U)	298873-0-0-0	(K U)	180533-0-0-0	(EX USE)
298873-0-0-0	(EX USE)				

Markush Number:

Markush Number	Role	Markush Number	Role	Markush Number	Role
103986001	(K U)	103986002	(K U)	103986003	(K U)
103986004	(K U)	103986005	(K U)	103986006	(K U)
103986007	(K U)	103986008	(K U)	103986009	(K U)
103986010	(K U)	103986011	(K U)	103986012	(K U)
103986013	(K U)	103986014	(K U)	103986015	(K U)
103986016	(K U)	103986017	(K U)	103986018	(K U)
103986019	(K U)	103986020	(K U)	103986021	(K U)
103986022	(K U)	103986023	(K U)	103986024	(K U)
103986025	(K U)	103986026	(K U)	103986027	(K U)
103986028	(K U)	103986029	(K U)	103986030	(K U)
103986031	(K U)	103986032	(K U)	103986033	(K U)
103986034	(K U)	103986035	(K U)	103986036	(K U)
103986037	(K U)	103986038	(K U)	103986039	(K U)
103986040	(K U)	103986041	(K U)	103986042	(K U)
103986043	(K U)	103986044	(K U)	103986045	(K U)

Ring Index Number(s):

00245	00212	01595	00096	00094
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Derwent Compound Number(s):

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Compound Number	Role	Compound Number	Role	Compound Number	Role
RA0XLT	(K U)	RA1SLR	(K U)	RA21P5	(K U)

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◀ | Record 100 of 343 | ▶




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