METHOD OF MAKING COMPOSITE PARTICLES WITH TAILED SURFACE CHARACTERISTICS

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Appl. No.: 11/035,168

Filed: Jan. 12, 2005

ABSTRACT

The invention describes a procedure to make metal containing composite particles and composite particle suspensions. The procedure is versatile and can produce particles with a variety of particle sizes and compositions. For some applications the metal composite particles can provide the functionality of wholly metallic particles including configurations where the metal is located on the particle surface. Such metals have application in a wide variety of fields, including accomplishing electrochemical reduction and catalysis.
METHOD OF MAKING COMPOSITE PARTICLES WITH TAILORED SURFACE CHARACTERISTICS

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BACKGROUND

Materials have properties that can be ascribed to either surface or bulk characteristics. These parameters include catalytic activity, electrical and thermal conductivity, optical, electronic, chemical and mechanical properties. Particles with metallic exteriors of tailored compositions and thickness can exploit these properties while providing control over other properties such as density, material cost and particle behavior.

Such metals have application in a wide variety of fields, including but not limited to water treatment, remediation, diagnostic medicine, drug delivery, and cosmetics. For example, zero valent metals may be useful as fillers, such as those for thermal and electrical conductivity, capacitance, charge dispersion and electromagnetic interference and absorbance. Such metals also may be used as magnetic and/or luminescent materials, tags, pigments, conductive inks and coatings, sensors, dopants, alloys, sintering aids, and catalysis supports such as in fuel cells and batteries, for example. Additionally, zero valent metals may be useful for infrared missile decoys, for example as chaff or pyrophoric tracers.

Zero valent metals are known to supply electrons that enable and sustain electrochemical reactions. Electrochemical reactions include those that convert toxic chemical compounds into innocuous products. For example, halogenated chemical compounds, such as trichloroethylene and carbon tetrachloride, can be converted to hydrocarbons such as ethane and methane, for example. Similarly, toxic compounds such as chromium (VI) (CrO$_4^{2-}$), lead (II) (Pb$^{2+}$) and arsenic (V) (As$^{5+}$), for example, can be converted to compounds having less toxic oxidation states or aqueous solubility. Therefore, zero valent metals have a wide variety of uses in the field of remediation, such as in ex-situ and in-situ electrochemical reduction, and in ex-situ and in-situ immobilization. The foregoing examples provide only an indication of the wide variety of uses for zero valent metals, uses being clearly understood and appreciated by anyone having ordinary skill within arts utilizing metal particles.

The field of remediation, the targeted substances are often located underground. A promising in-situ remediation protocol uses zero valent metals that are injected into the subsurface where they react with and destroy the targeted contaminants. It is difficult or impossible to transport coarse microscale particles through the subsurface as the particles settle rapidly and are strained or filtered before they reach the contaminant phase. Smaller sub-micrometer and nanoscopic particles can more easily fit within the interstices of soil particles and travel greater distances through the sub-surface to reach the contaminant phase.

In the field of remediation and catalysis, the targeted reactions take place on the particle surface. Therefore, it is desirable for the particles to have a high surface area. The relationship between particle size and surface area is shown in Equation (1) where $d$ is the diameter of a spherical particle, SSA is the specific surface area (area/mass) and $\rho$ is particle density (mass/volume).

$$d = \frac{6 \times 10^{-6}}{\rho \times \text{SSA}^{1/3}}$$ (1)

This expression reveals an inverse relationship between specific surface area and particle size. Smaller particles offer more surface area per unit mass and are often desirable for remediation applications.

Commercially available zero valent metal particles are typically microscale with dimensions greater than one micrometer and these large particles offer a low specific surface area. Using iron as an example, spray atomized and electrolytic iron particles typically offer dimensions greater than 10 micrometers and have an active surface area of only about 0.1 to 0.2 m$^2$/g. Carbonyl iron particles that are produced by condensation from the vapor phase typically offer dimensions of 1 to 10 micrometers and have an active surface area of only about 0.5 to 1 m$^2$/g. The large particle size makes this product unsuitable for many applications including in-situ remediation, for example.

Making nanocrystalline and sub-micrometer metal particles can alleviate problems associated with microscale product. Nanoscale zero valent metal particles having a surface area in excess of 20 m$^2$/g has been produced by various methods. One method involves the electrochemical reduction of a metal salt solution in water. Equation (2) shows the electrochemical reduction of a trivalent iron ion with a borohydride ion.

$$4\text{Fe}^{3+} + 3\text{BH}_4^- + 6\text{H}_2\text{O} \rightarrow 4\text{Fe}^{0} + 3\text{H}_2\text{BO}_3^- + 12\text{H}^+ + 6\text{H}_2$$ (2)

This is a slow and expensive process that results in material having very limited commercial use. The particles also have residual boron that is undesirable for some applications. These particles also tend to form aggregates of smaller particles. Aggregated particles still provide a high surface area but behave like coarse particles when injected underground and offer less than ideal sub-surface mobility.

Another method that is employed to make high surface area zero valent metal involves the thermal reduction of a metal oxide. The metal oxide particles are heated in a reducing atmosphere (typically hydrogen or carbon monoxide) to produce zero valent metal. An example is provided in equation (3) and involves the reaction of hematite (Fe$_2$O$_3$) with hydrogen gas.

$$\text{Fe}_2\text{O}_3 + 3\text{H}_2 \rightarrow 2\text{Fe}^{0} + 3\text{H}_2\text{O}$$ (3)

Although less expensive than chemically precipitated product, this product is also prohibitively expensive for most applications. This product is also aggregated with less than ideal underground mobility.

Another method for producing zero valent metal particles involves the chemical precipitation of zero valent iron onto the surface of an inert carrier particle, as described in U.S. Pat. No. 6,689,485. Using iron salt and borohydride precursors in water and a reaction similar to that shown in equation (2) produces nanocrystalline iron deposits on the carrier surface. This procedure also offers the disadvantages of expensive chemical precursors and difficulties in scaling the process.
Moreover, chemically precipitated particles are synthesized in water and all of the materials, including thermally reduced particles, are stored and shipped in water. Metal react with and electrochemically reduces water as depicted in equation (4). This consumes the iron prematurely and can also lead to the formation of solid surface deposits of water insoluble metal hydroxides (show in equation (5)), oxides, and oxyhydroxides. These non-metallic substances can prematurely passivate the particle surface and inhibit reactivity.

\[ \text{Fe}^{2+} + \text{H}_2 \rightarrow \text{Fe}^{3+} + 2\text{H}^+ \quad (4) \]

\[ \text{Fe}^{3+} + 2\text{OH}^- \rightarrow \text{Fe(OH)}_2 \quad (5) \]

Other efforts have focused on the mechanical attrition or comminution of coarse iron particles into material having smaller particles. This methodology is ineffective for producing discrete, sub-micrometer or nanoscale particles, since iron is ductile at room temperature and does not become brittle unless cooled to cryogenic temperatures. When electrolytic iron is processed in a stirred media mill with spherical steel media, instead of breaking down into smaller particles the particles are mechanically deformed and flattened into particles with a flake morphology and a surface area of 1-2 m²/g. FIG. 2 is a SEM micrograph showing the particles resulting from this process. The particles are very large, typically 10 micrometer to 100 micrometer. These are poorly suited for remediation applications.

To date, there is no cost-effective and easy way to manufacture large quantities of the high surface area zero valent metal particles of the type described herein, such particles having a wide variety of uses in a large number of fields.

SUMMARY OF THE INVENTION

The present invention is a cost-effective and scalable technique for producing sub-micrometer and nanocrystalline zero valent metal particles and particle suspensions by depositing metal onto the surface of a host particle. The particles typically offer a surface area of greater than 5 m²/g, and can be inexpensively produced on a large scale. Moreover the particles have reactivity superior to currently available microscale metal particles.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1(a) depicts a host particle (010) prior to mixing with metal.

FIG. 1(b) depicts a host particle (010) with a metal surface layer (011).

FIG. 2 is a scanning electron microscope (SEM) picture of particles produced by mechanical attrition of coarse iron particles.

FIG. 3 is a scanning electron microscope (SEM) picture of particles produced according to the methods disclosed herein.

FIG. 4 is a scanning electron microscope (SEM) picture of particles produced by thermal reduction.

DETAILED DESCRIPTION

Throughout this specification, the terms "a" and "an" and variations thereof represent the phrase "at least one." In all cases, the terms "comprising", "comprises" and any variations thereof should not be interpreted as being limiting to the elements listed thereafter. Unless otherwise specified in the description, all words used herein carry their common meaning as understood by a person having ordinary skill in the art. In cases where examples are listed, it is to be understood that combinations of any of the alternative examples are also envisioned. The scope of the invention is not to be limited to the particular embodiments disclosed herein, which serve merely as examples representative of the limitations recited in the issued claims resulting from this application, and the equivalents of those limitations.

Zero valent metals supply electrons that enable and sustain chemical reactions (reduction-oxidation, redox). Equation (6) depicts the corrosion of a zero valent metal (M) to produce a metal ion (M⁺, valence of x) and x electrons (e⁻).

\[ \text{M}^{0} \rightarrow \text{M}^{+x} + xe^- \quad (6) \]

The electrons supplied by the metal corrosion can then be transferred to another chemical compound to enable electrochemical reduction. Compounds that can be chemically reduced include halogenated compounds (RX) in an aqueous environment as depicted in equation (7) where RH is the reduced product.

\[ \text{RX} + xe^- + \text{H}_2\text{O} \rightarrow \text{RH} \text{H}_2\text{O} + x\text{H}^+ \quad (7) \]

The net equation resulting from the foregoing oxidation and reduction reactions is depicted in equation (8).

\[ \text{M}^{+} + \text{H}_2\text{O} + x\text{H}_2 \rightarrow \text{M}^{+x} \text{X}^{-} + 2\text{H}_2\text{O} \quad (8) \]

Equations (9) through (11) illustrate the transfer of electrons from a zero valent metal to an aqueous solution containing an ion with hexavalent chromium (Cr⁶⁺). The net product includes the less toxic Cr³⁺ ion and the oxidized metal ion (M⁴⁺).

\[ \text{Oxidation: } \text{M} \rightarrow \text{M}^{+} + xe^- \quad (9) \]

\[ \text{Reduction } \text{Cr}_2\text{O}_7^{2-} + 14\text{H}^+ + 6xe^- \rightarrow 2\text{Cr}^{3+} + 7\text{H}_2\text{O} \quad (10) \]

\[ \text{Net equation: } \text{M}^{+} + 14\text{H}_2\text{O} + 6\text{Cr}_2\text{O}_7^{2-} \rightarrow 3\text{M}^{4+} + 6\text{Cr}^{3+} + 28\text{H}^+ + 12\text{H}_2\text{O} \quad (11) \]

Electrons supplied by the reduction of zero valent metals can accomplish the electrochemical reduction of not only halogenated and metal containing substances but also other chemical substances that include but are not limited to arsenates, phosphates, sulfates, chromates, mercurates, perchlorates, and nitrates.

In the field of remediation, the targeted compounds are often located underground. Commercially available zero valent metal varies in particle size from about 1 micrometer to about 1 millimeter or larger. The large particle size and coarse morphology make it difficult and expensive to transport the zero valent metal particles through the sub-surface as the particles are strained or filtered by soil. Smaller particles can more easily fit within and pass through the interstices of the soil particles. The ability to make sub-micrometer (primary particle dimension less than about 1 micrometer) and nanocrystalline (primary particle dimension less than about 100 nm) particles can help alleviate the problems associated with transporting the zero valent particles to the targeted compounds.

In the field of remediation, the targeted chemical substances are often located underground. Making small, high surface area particles is an important but not sufficient
condition for accomplishing sub-surface particle mobility. Despite their small particle size and high surface area, aggregated or agglomerated groups of smaller particles provide limited underground mobility. Discreted or loosely aggregated particles offer better underground mobility and are preferred for these applications.

[0028] In remediation and catalysis operations, the targeted reactions often take place on the particle surface. Reaction rates generally scale with surface area that is inversely proportional to particle size. Because of the high surface area, smaller sub-micrometer and nanoscale particles are typically preferred for applications where reactivity and catalytic activity are required.

[0029] Aqueous phase halogenated compounds are electrochemically reduced by zero valent metals. Reaction products vary and depend upon several variables that include metal composition and particle size. Considering the electrochemical reduction of carbon tetrachloride for example, reaction products can include partially halogenated substances (i.e., chloroform and dichloromethane), halogen-free hydrocarbons (i.e., methane), and carbon dioxide gas. The partially dehalogenated hydrocarbons are still toxic and usually are more thermodynamically stable than the parent compound. Current zero valent metal particles often provide reaction pathways that produce these partially dehalogenated hydrocarbons. Zero valent metal particles that minimize or eliminate the formation of partially dehalogenated hydrocarbons are instead preferred.

[0030] In the methods disclosed herein, host particles and metal source particles are combined with a dispersant and solvent and the mixture is then mechanically agitated such that the metal is deposited onto the host material.

[0031] Host materials, also referred to as "core" materials or "carrier" particles, may be nanoscale to microscale particles and include oxide ceramics, or mixtures of one or more oxide ceramics, such as aluminum oxide, iron oxide, titanium oxide, silicon oxide, titinates, zircon, tricalcium aluminate, and silicate, for example. Also useful as host materials are non-oxide ceramics and mixtures thereof, such as silicon carbide, calcium carbide, boron carbide, aluminum carbide, silicon nitride. Other useful materials are phosphates, sulfates, and carbonates, for example. Suitable host materials also include clays and minerals such as aluminosilicates, for example, and mixtures thereof. Other suitable host materials include, but are not limited to polymers and plastics such as polycarbonates and nylon for example. Other suitable host materials include intermetallic compounds such as TiAl, and metals and metal alloys, for example. Host materials may be comprised of combinations of any of the foregoing exemplary compounds. There are few restrictions on host material composition other than it should not react with or change the targeted properties or function of the metal surface layer. The carrier particle is present in the range of about 25 to about 99 volume percent, more preferably about 80 to about 99 volume percent of the composite particle.

[0032] Most metals are thermodynamically unstable when exposed to oxygen and other reactive substances. These reactions can be violently exothermic particularly when reactive, high surface area metals are considered. Even moderately reactive compositions such as iron, for example, can react violently when sub-micrometer or nanoscale particles are exposed to air. The compositions produced according to the methods of the invention reduce the propensity for rapid exothermic reactions since a lesser quantity of reactive metal is present and the metal is stored in a non-reactive medium. Another advantage of these methods is the ability to produce a lightweight material, since in most cases the specific gravity of the host particle is lower than that of the metal particle. Lightweight materials result in compositions having lower overall product mass and more stable suspensions, as the particle settling rates are proportional to the solid particle’s specific gravity.

[0033] Suitable metals include Li, Al, Na, K, Si, Mg, Ca, Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, Ge, K, Sr, Y, Zr, Nb, Mo, Pd, Ag, In, Sn, Sb, Cs, Ba, La, Ta, W, Pt, Au, Bi, Ce, and U and alloys and combinations thereof. The metal material is present in the range of about 1 to about 75 volume percent, more preferably about 1 to about 20 volume percent of the composite particle.

[0034] There are literally hundreds of candidate solvents but it has been discovered that only a narrow few are compatible with the process disclosed herein. Water is typically the first choice of solvent due to its low cost and low toxicity. However, water is unsuitable for processing some of the reactive metals, including lithium and sodium, as they react explosively when combined with water. Iron and other more noble metals are somewhat stable in water and initial trials employed water as the solvent. These trials were successful on the laboratory scale; however, in a 300 lb. pilot test this material reacted strongly with water giving off of hydrogen gas and producing a foamy mixture with a pH in excess of 10, thereby producing iron hydroxide instead of the desired metallic product.

[0035] Other non-aqueous solvents have therefore been considered. It has been determined that the solvent preferably should exhibit a relatively high flash point and autoignition temperature (flash point greater than about 75° C., and autoignition temperature greater than about 150° C., for example) and a National Fire Protection Agency (NFPA) flammability rating of 0 or 1. Existing processes using mechanical agitation to produce metals or metal containing-composites use flammable substances. For example, U.S. Pat. Nos. 6,200,547 and 6,482,436 disclose the use of ethanol as a solvent for making magnetic iron/carbon composite particles for spray delivery applications. The explosive nature of ethanol (flash point=13° C., NFPA Flammability Rating=2) limits this process to making small powder quantities or using environments where ignition sources are strictly excluded. Other processes employ mechanical comminution, as disclosed in U.S. Patent Publication Nos. 20020151602 and 20040245187, to process metal powder containing slurries by using potentially explosive butyl acetate (flash point=26° C., NFPA flammability rating=3), propylene glycol monomethyl ether acetate (flash point=42° C., NFPA flammability rating=2), and dodecane (flash point=74° C., NFPA flammability rating=2). When flammable solvents are used, scaling the process to produce a low-cost commodity product presents serious risks. These risks may be mitigated with expensive equipment modifications that add substantial cost and still do not fully guarantee operator safety. Accordingly, any solvent with explosive characteristics is not preferable for the process according to the invention.
Preferably, the solvent system should offer low toxicity and be biodegradable, particularly when used in situ remediation applications where some of the solvent is introduced into the subsurface and can enter aquifers. Generally, solvents with a NFPA health rating of greater than about 1 are considered unsuitable. Non-aqueous solvents having a low flammability and low toxicity satisfying these criteria include, for example, glycerol, glycol ethers, vegetable oils such as diethylene glycol monoethyl ether (NFPA health rating=1, flammability rating=1, reactivity rating=0), propylene glycol (NFPA health rating=0, flammability rating=1 reactivity rating=0), polyethylene glycol (NFPA health rating=0, flammability rating=1 reactivity rating=0), and soybean oils (NFPA health rating=0, flammability rating=1 reactivity rating=0), and combinations thereof.

To assist in attaining low viscosity and high powder fraction slurries that enable high product throughput and low cost, the solvent system should offer a low viscosity of less than about 100 centipoise, for example. Examples of solvents having suitable viscosities include but are not limited to diethylene glycol monoethyl ether having a room temperature viscosity about 4 centipoise (water=1 centipoise), propylene glycol having a room temperature viscosity of about 46 centipoise, polyethylene glycol having a room temperature viscosity of about 50 centipoise, vegetable oil typically having a viscosity of about less than 100 centipoise depending on composition, and combinations thereof. The glycol ethers offer the lowest viscosity and therefore result in the most fluid suspensions.

The solvent system should be water miscible, particularly when used for in-situ remediation applications where the material is mixed with water and introduced into groundwater. The solvent system additionally should be inexpensive and available in quantity.

Initial processing trials employed a mixture according to the foregoing criteria containing aluminum oxide powder, 2 mm diameter spherical steel ball bearings and diethylene glycol monoethyl ether as the solvent. The trials involved adding the solvent and ball bearings into a small media disc mill. After starting the mill, the powder was added incrementally. Additions up to about 15 volume percent powder produced fluid slurries, but greater volume fraction slurries unexpectedly produced a very viscous sludge or paste-like product. After mixing for a few hours, the slurry required dilution to about 10 volume percent powder to lessen the viscosity enough for the composite particle product to be separated from the steel ball bearings. The presence of metal was verified by adding a few drops of dilute hydrochloric acid to the slurry. This induced the rapid evolution of hydrogen gas bubbles and a lightening of the slurry color as the metal was depleted and the white aluminum oxide particle surface was exposed. Reactivity was evaluated by mixing the aluminum oxide/iron composite particles with an aqueous solution containing chlorinated hydrocarbons. The chlorinated hydrocarbon concentrations were reduced, evidencing the ability of the composite particles to accomplish electrochemical reduction reactions.

These experiments revealed a fundamental problem with the process, namely the unexpected difficulty in obtaining fluid, high volume fraction slurries. Sufficiently fluid, low powder fraction slurries could be fabricated only when diluted at the expense of substantially reduced throughput and at substantially increased product cost. While potentially suitable for very small scale synthesis, this process was not commercially viable on a large scale.

High viscosity may be indicative of the presence of powder agglomerates or aggregates. Particles in a slurry or suspension are subjected to ubiquitous, attractive interparticle forces that cause them to flocculate, agglomerate, or aggregate. Agglomerated particles are undesirable for several reasons. Foremost is the high viscosity of aggregated systems, even at relatively low solid fractions.

It is now known that making fluid, high particle fraction slurries by this process therefore requires using chemical dispersants. Dispersants (also known as surfactants) are chemical substances, often polymers or oligomers that adhere to particle surfaces. The presence of the dispersant provides repulsive forces (electrostatic or steric) that maintain interparticle separation and prevent agglomeration thereby dramatically reducing slurry viscosity.

The dispersant also plays another important role in the manufacturing processes described herein. Typically the metal particles are substantially larger than the composite particles of the finished product, which comprise metal particles deposited on a carrier particle support. Accordingly, the metal source particles must be separated from the product. Settling velocity has been found to be inversely proportional to viscosity and roughly proportional to the square of particle size. The dispersant reduces slurry viscosity, often dramatically, and this allows for the larger metal particles to be separated from product by settling for a short time period.

Hundreds of dispersants having widely different characteristics are commercially available. Most, however, will not work for a randomly chosen solvent/particle system and will have little if any positive influence on reducing agglomeration and the resulting deleterious consequences. Similar to the solvent selection process previously described herein, a precise set of process-specific criteria results in a narrow window of dispersants useful for the particular solvent/particle systems according to the invention, and identifying those dispersants is a non-trivial process.

Realizing the challenges involved in finding a suitable dispersant system, a systematic approach has been developed to identify and screen dispersants for use with the previously identified solvents/particle systems. An important variable to consider is miscibility with the identified solvents. The hydrophile-lipophile balance (HLB) often characterizes dispersants/surfactants. A surfactant with a relatively low HLB, typically below about 10, is usually soluble in water and other polar solvents. Conversely, a surfactant with a relatively high HLB, typically above about 10, is typically soluble in oils or other non-polar solvents. It has been determined that surfactants with a relatively low HLB are compatible with the solvent/particle systems useful according to the processes as described herein. As some chemical dispersants have no published HLB, experiments or published data readily apparent to any person having ordinary skill in the art may be used to determine relative solubility.

Dispersant solubility may be tested in the selected solvent by any appropriate routine experimentation readily
apparent to any person having ordinary skill in the art. For example, 2 mass percent dispersant additions may be mixed with solvent and poured into glass vials. Where the solvent is transparent, dispersant containing solutions that maintain a transparent appearance may be considered soluble with the solvent. Dispersants producing an opaque or translucent solution may be eliminated from consideration.

[0047] To operate effectively, dispersants should adhere to the particle surfaces. Particle surfaces have both positive and negative point charges. A negatively charged dispersant molecule, typically an anionic dispersant, will adhere to the particle surface at the location of a positive point charge. Similarly, a positively charged dispersant molecule, typically a cationic dispersant, will adhere to the particle surface at the location of a negative point charge. Although inferences can be made upon what dispersant type (i.e., anionic or cationic) will be effective for a particular particle system, in practice performing experiments is the most pragmatic and best way to identify and screen candidate dispersants.

[0048] Realizing the need to screen candidate dispersants, a procedure has now been developed that is effective for determining dispersants that will work with an identified particle/solvent system. This procedure involves making dilute particle suspensions that are poured into vials and then observing the suspension characteristics. For example, about 1 to about 10 mass percent carrier particle suspensions with about 0.0015 to about 0.0045 g dispersant per square meter of total particle surface area may be used, for example. This concentration typically ensures saturation of dispersant molecules on the particle surfaces. Although typically suitable for screening dilute suspensions, it should be noted that this concentration would most likely not be optimized for making the targeted concentrated suspensions where further optimization is typically required. After mixing the solvent, dispersant, and powder, ultrasonic agitation, for example, is then used to provide energy to break up powder agglomerates. This allows for the dispersant molecules to adhere to most or the entire available particle surfaces.

[0049] Once the dilute suspensions are mixed and agitated, they are poured into capped glass vials and a subjective analysis is used to determine dispersant compatibility with the solvent/particle system. A good suspension exhibits distinct characteristics. One favorable characteristic is the presence of a bluish tint or Tyndall Effect that is indicative of the scattering or reflection of light provided by only small, dispersed particles. Another favorable characteristic is the formation of a thin, uniform sheet of liquid suspension that cascades down the inner surface of the capped glass vial surface when shaken. Conversely, poorly dispersed systems do not exhibit a bluish appearance and exhibit clumping or a non-uniform distribution of particles on the glass vial surface. Dispersants meeting the foregoing criteria are exemplified by but not limited to Disperbyk® 111 and Disperbyk® 180, proprietary polymeric dispersants made by Altana Chemie (Wessle, Germany). Similar compatibility tests may be used in addition and/or in the alternative to the foregoing example, such tests being readily apparent to any person having ordinary skill in the art.

[0050] Subsequently, tests are performed to determine whether the candidate dispersants can produce the required high particle fraction slurries. When high particle fraction surfaces are formulated, an objective measure of a dispersant’s effectiveness is relatively low viscosity. One test, for example, uses approximately 30 volume percent carrier particle/solvent slurries with a series of dispersant concentrations ranging from about 0.5 to about 3.0 mg per square meter of total surface area. Solvent and dispersant are added to an attritor mill with 2 mm spherical zirconia media. Metal powder is not added to the agitated system in this evaluation. Carrier particle powder is added incrementally to help maintain fluid suspensions and mixing is continued for about 60 minutes at about 600 RPM. Viscosity is measured and the concentration that provides the minimum viscosity for candidate dispersants is identified. Concentrations between about 2.0 and about 3.0 mg dispersant per square meter of surface area provide a suitably low viscosity with a minimum at about 2.5 mg dispersant per meter square. Similar tests may be used in addition and/or in the alternative to the foregoing example, such tests being readily apparent to any person having ordinary skill in the art.

[0051] Further evaluations are performed to test the system with metal particles added to the agitated formulation. For example, a procedure similar to that described in the preceding paragraph may be used to make approximately 30 volume percent carrier/solvent slurries, but with the addition of metal particles. A dispersant concentration of about 2.5 mg per square meter powder is used, for example. The mixture is agitated, for example in a small media disc mill (HDDM-01 disc mill, Union Process, Akron, Ohio) at 2500 RPM in a nitrogen atmosphere. The initially white slurry should become dark as the metal particles contact the carrier particles. After an appropriate time, for example, about three hours, the fluid suspension may be decanted and the metal particles separated by settling. Similar evaluation techniques may be employed in addition and/or in the alternative, such techniques being readily apparent to any person having ordinary skill in the art.

[0052] In one set of experiments, aluminum oxide (P10F grade, Alnatis, Pittsburgh, Pa.) was used as the carrier particle, diethylene glycol monoethyl ether was used as the solvent, steel shot (UltraSof® U280, Peerless Metal, Detroit, Mich.) was used as the metal source, and either Disperbyk® 111 or Disperbyk® 180 was used as the dispersant. Each of the compositions comprised components meeting all of the criteria described herein. The compositions were stored, as typical, in closed plastic containers.

[0053] The Disperbyk® 180 containing slurry maintained the initial gray color of the harvested material. However, the Disperbyk® 111 containing slurry experienced a color change and had assumed a brownish, rust-colored appearance after about 3 days. It was suspected that the Disperbyk® 111 possessed acidic characteristics that initiated a reaction with the metal and caused corrosion. This is unacceptable if metallic characteristics are desired in the product, as is the case for enabling electrochemical reactions, for example. This was an unexpected circumstance that further enhanced the criteria required for selection of suitable dispersants. That is, the dispersant must have a compatible pH.

[0054] The Disperbyk® 180 containing slurry was tested for presence of metal by adding a few drops of dilute hydrochloric acid. Unexpectedly, few hydrogen gas bubbles were evolved when compared to prior material that was made without dispersant. This was despite the fact that
metallic powder color clearly indicated the presence of a metal fraction. Perplexed by this observation, the ability of this material to accomplish the electrochemical reduction of aqueous phase chlorinated hydrocarbons was tested. Unlike prior material made without dispersant, the material accomplished little or no electrochemical reduction despite its metallic color.

[0055] Explanations were sought to account for the non-reactive nature of the dispersant-containing particles. While several theories accounting for this observation have been set forth, none are determinative of the problem. Ultimately this unforeseen problem has now been solved using a surprisingly unique processing approach.

[0056] All materials are still selected based upon the same foregoing criteria. The solvent, carrier particles, and source metal particle mixture are initially agitated at an approximately 10 to 60 volume percent powder fraction slurry without added dispersant. When mixed in a small media mill, this mixture became very dark but was extremely viscous and unusable, in part because the carrier supported metal particles could not be separated from the metal particle source. After mixing for an appropriate time, for example, three hours, dispersant is added to the sludge to reduce viscosity to an acceptable level. In contrast to prior work, a minimum viscosity was attained using a much lower dispersant concentration of about 0.25 to about 1.50 mg per square meter of total surface area. Using this procedure, the metal source particles are still easily separated from the powder fraction slurry product by settling.

[0057] The powder produced using this process may be evaluated by adding a few drops of dilute hydrochloric acid to the slurry; metal content is evidenced by the rapid evolution of hydrogen gas bubbles. The ability to enable electrochemical reduction may be evidenced by the rapid elimination of halogenated hydrocarbons in an aqueous solution.

[0058] In addition to the host particle, metal constituents, solvent and dispersant, a wide variety of materials may optionally be included, the benefits of which are readily apparent to anyone having ordinary skill in the art. Particularly beneficial optional ingredients include, but are not limited to metals more noble than the host metal such as Pd, Pt, Au, Cu, and Ni for example. More noble metals are easily added to the host metal surface using an electrochemical reaction. Other additives can include micronutrients such as phosphorus, for example, and non-metals such as carbon and sulfur, for example. Depending upon the particular field and application for the particles, any person having ordinary skill in the art would be capable of determining the types of additives that would be beneficial.

[0059] Agitation may be performed using one or more suitable devices, which are readily apparent to anyone having ordinary skill in the art. Suitable devices include, but are not limited to stirred media mills having a vertical mixing chamber such as an attritor mill for example. Other devices include stirred media mills having a horizontal mixing chamber such as a sand mill for example. The stirred media mill can be operated in batch or continuous mode. Similarly tumbling mills such as jars mills and ball mills, for example, are suitable. Additional examples include vibratory mills such as a SWECOR mill, ultrasound, and lapping. If a smaller or larger mixing chamber is used, the mass of each constituent is proportionally increased or decreased.

[0060] The mixtures are mechanically agitated until the slurry assumes the desired metallic color. Water may be pumped through a cooling jacket, for example, in order to maintain a constant temperature of about 30°C, for example. The time required to produce the desired composite powder is typically a few hours although this is dependent upon material composition and agitation rate. Generally, relatively higher agitation speeds are preferred for this process; the stirred media mills operate at speeds of 500 to 3500 RPM. Any person having ordinary skill in the art can easily determine when a suitable amount of metal has been deposited onto the host particles. The mixtures are agitated in air, nitrogen, oxygen, forming gas, vacuum, or other suitable atmospheres that are readily apparent to anyone having ordinary skill in the art. The processing temperature should be maintained between the freezing and boiling temperatures of the selected solvent system.

[0061] The resulting slurry is typically decanted into another vessel. The coarse and high density metal source particles rapidly settle to the bottom of the vessel. The suspended product may be pumped or decanted, for example, into another container.

[0062] The particles are typically configured of a host material with metal deposited thereon, such as a core and shell. Some free, non-supported metal and non-metal particles may also be produced by the process and mixed within the core and shell particles.

[0063] The resulting particles typically resemble the initial size of the host particles. In some cases, reduction of the initial particle size may occur as the metal particles can act as media to break down the host particles. The process is capable of producing very small particles, in the nanoparticle regime. The process is also capable of producing larger microscale particles. This versatility is enabling since the particle size can be tailored to meet the requirements of the targeted application. The preferred particle size is typically small if reactivity and catalytic activity are desired; for other applications larger particles may be preferred. For in-situ remediation applications, sub-micrometer particles are often preferable to nanoparticles. As the particle size is decreased the particles are more susceptible to agglomeration. Accordingly, using slightly larger but discreet sub-micrometer particles can provide better underground mobility.

[0064] The resulting particles typically resemble the shape of the initial host particles. In some cases shape change may occur if the host particles are broken down during the synthesis process. Therefore, several particle morphologies including but not limited to equiaxed, acicular, and platelet shapes are possible if host particles of these geometries can be obtained.

[0065] After synthesis, the composite particle suspensions are typically stored in closed airtight containers or in an inert atmosphere. Stable suspensions can be maintained for days or weeks using the solvent and dispersant used for particle synthesis.

[0066] While the foregoing examples have focused primarily on remediation and catalysis applications, these particles have utility in any field involving the use of non-aggregating, micrometer and nanocrystalline zero valent metal particles and particle suspensions. Their value is readily apparent to anyone having skill in the relevant arts.
EXAMPLE 1
Aluminum Oxide with Spherical Carbon Steel Ball Bearings

[0067] 1240 g of 0.5 mm diameter spherical case hardened steel ball bearings (Union Process, Akron, Ohio), 230 g of solvent (diethylene glycol monoethyl ether), and 2.2 g dispersant (Disperbyk® 180, ALTANA Chemie, Wessling, Germany) were added to a disc mill with a 750 ml. vertical mixing chamber (HDDM-01 disc mill, Union Process, Akron, Ohio). The mill speed was set to 2500 RPM and was operated at a constant temperature of 30°C. in a nitrogen atmosphere. Subsequently about 490 g of roughly spherical aluminum oxide particles (RCHP, Biskowski Malakoff, Malakoff, Tex.) were added in stages to help maintain a fluid suspension. After the iron contact, most of the alumina particles, metal was deposited onto exposed surfaces. The particle slurry, initially white, became darker with increasing time. This indicated that iron was deposited onto the particle surface. After 3 hr. slurry was harvested by decanting the mixture into a vessel. The coarse and dense iron particles quickly settled to the bottom and the suspended aluminum oxide particle supported metal suspended to 1500 RPM was decanted. Shiny color was light metallic gray and the surface area was measured at about 10 m²/g using a surface area analyzer. This translated to an equivalent spherical diameter of about 0.1 micrometer using an equivalent spherical diameter assumption and specific gravity of 5.

EXAMPLE 2
Aluminum Oxide with UltraSoft® Steel Shot

[0068] About 1060 g of roughly spherical steel blasting grit (UltraSoft® US-280, Peerless Metal Detroit, Mich.), 250 g of solvent (diethylene glycol monoethyl ether), and 2.3 g dispersant (Disperbyk® 180) were added to the HDDM-01 disc mill with a 750 ml. vertical mixing chamber. The mill speed was set to 2500 RPM and was operated at a constant temperature of 30°C. in a nitrogen atmosphere. Subsequently about 425 g of roughly spherical aluminum oxide particles (P730, Alnatis, Pittsburgh, Pa.) was added in stages to help maintain a fluid suspension. After 3 hr. slurry color was dark metallic gray and the metal source particles were separated by settling. Powder surface area was measured at about 12 m²/g.

EXAMPLE 3
Aluminum Oxide with AT40.29 Iron Particles

[0069] About 1050 g of roughly spherical iron particles (AT40.29, North American Hoganas, Hollsopple, Pa.) and 250 g of solvent (diethylene glycol monoethyl ether) were added to the HDDM-01 disc mill with a 750 ml. vertical mixing chamber. The mill speed was set to 3000 RPM and was operated at a constant temperature of 30°C. in a nitrogen atmosphere. Subsequently about 425 grams of roughly spherical aluminum oxide particles (P10 Feinst, Alnatis) was added in stages. After 3 hr. slurry color was dark metallic gray. The mill speed was reduced to 1500 RPM and 4.2 g of dispersant (Disperbyk® 180) was added to reduce viscosity and enable separation of iron source particles by settling. Powder surface area was measured at about 19 m²/g. FIG. 3 is a scanning electron microscope (SEM) picture showing the discrete sub-micrometer particles, generally free of agglomerates and aggregates.

EXAMPLE 4
Aluminum Oxide with Cut Wire Zinc Shot

[0070] About 1260 g of unconditioned cylindrical cut wire zinc shot (30U, Premier Shot, Cleveland Ohio) and 260 g solvent (diethylene glycol monoethyl ether) were added to the HDDM-01 mill with a 750 ml vertical mixing chamber. The mill speed was set to 3200 RPM and was operated at a constant temperature of 20°C. in a nitrogen atmosphere. About 450 g of roughly spherical aluminum oxide (P730) was added in stages. After 3 hr., slurry color was metallic gray. The mill speed was reduced to 1500 RPM and 4.2 g of dispersant (Disperbyk® 180) was added to reduce viscosity and enable separation of zinc source particles by settling.

EXAMPLE 5
Magnetite with AT40.29 Iron Particles

[0071] About 1400 g of roughly spherical iron particles (AT40.29), 225 g solvent (diethylene glycol monoethyl ether) and 1.3 g dispersant (Disperbyk® 180) were added to the HDDM-01 mill with a 750 ml. vertical mixing chamber. The mill speed was set to 3250 RPM and was operated at a constant temperature of 30°C. in a nitrogen atmosphere. About 560 g of roughly spherical magnetite (Fe₃O₄) (Elector Oxide 20, North American Hoganas) was added in stages to help maintain a fluid suspension. After 4 hr., slurry color (initially black) was metallic gray and settling separated the metal source particles.

EXAMPLE 6
Aluminum Oxide with a Mixture of Solvents and Pd

[0072] About 1060 g of roughly spherical iron (AT40.29), 245 g of mixed solvent (80 m/m% diethylene glycol monooethyl ether, 10 m/m% propylene glycol, 10 m/m% polyethylene glycol 400) were added to the HDDM-01 mill. The mill speed was set to 3000 RPM and was operated at a constant temperature of 30°C. in a nitrogen atmosphere. About 425 g of roughly spherical aluminum oxide (P730) was added in stages to help maintain a low viscosity suspension. After 4 hr., slurry color was metallic gray. The mill speed was reduced to 1500 RPM and 4.0 g of dispersant (Disperbyk® 180) was added to reduce viscosity and enable separation of zinc source particles by settling. The suspension was split to test the performance of palladium as a hydrogenation catalyst. Pd metal was electroplated onto the metal particle surface by stirring 0.027 g of palladium(II) chloride to 100 g of the suspension (about 65 m/m% solid particles). This provided a material with 0.025 m/m% (solid basis) Pd.

EXAMPLE 7
Scale Up Using Larger Horizontal Mill

[0073] About 250 kg of solvent was added to a stirred mixing tank along with 3.4 kg of dispersant (Disperbyk® 180). The solvent was a mixture of diethylene glycol monoethyl ether (80 m/m%), propylene glycol (10 m/m%) and polyethylene glycol 400 (10 m/m%). In recirculation mode the solvent/ dispersant mixture was pumped through a disc mill (Union Process DM-20 Delta Mill, Akron, Ohio) with a 20 liter volume horizontal mixing chamber. The water-cooled mixing chamber was filled to 85% of net volume with
US-280 Ultrasoft Shot steel particles. As the solvent/dispersant mixture was pumped through the Delta Mill, 350 kg of the aluminum oxide (Almatis P10F) was added incrementally to maintain a fluid suspension. The slurry was re-circulated through the mill operating at a tip speed of 2500 feet per minute and back into the stirring tank. The initially white slurry darkened into a metallic color with increasing time. After 8 hours the slurry was pumped into the mixing tank and the remaining iron remained in the mixing chamber and can be used for another run.

EXAMPLE 8

Reactivity Studies

The ability of the particles to accomplish the electrochemical reduction of an aqueous halogenated hydrocarbon solution was tested. This experiment tested:

[0074] Aluminum oxide/iron composite particles without Pd

[0075] Aluminum oxide/iron composite particles with 0.025% m/m electroplated Pd

[0076] Aluminum oxide/zinc composite particles without Pd

[0077] Coarse reference powders (electrolytic iron North American Iognas AC325 grade and carbonyl iron BASF OM grade, Mount Olive, N.J.) were also evaluated. The work used an 8.4 pH buffered 200 μM carbon tetrachloride solution. About 175 g of the carbon tetrachloride solution was transferred to a 240 mL bottle to which 2.2 g (1.5 g powder basis) of the composite particle slurry was added. The reference materials used 1.5 g of dry powders. These powders were tested straight out of the container and no attempt was made to remove any oxide surface layer. The bottles were not shaken or agitated during the testing period. Chemical composition was measured by injecting headspace gas into a gas chromatograph over a 2 day period. Table 1 shows the measured carbon tetrachloride concentration for each compound. Table 2 shows the measured chloroform concentration for each compound.

<table>
<thead>
<tr>
<th>Example Number</th>
<th>Description</th>
<th>Initial</th>
<th>2 hr</th>
<th>1 day</th>
<th>2 day</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>Aluminum oxide/iron no Pd</td>
<td>200 μM</td>
<td>139 μM</td>
<td>53 μM</td>
<td>30 μM</td>
</tr>
<tr>
<td>6</td>
<td>Aluminum oxide/iron 0.025% Pd</td>
<td>200 μM</td>
<td>87 μM</td>
<td>36 μM</td>
<td>5 μM</td>
</tr>
<tr>
<td>4</td>
<td>Aluminum oxide/zinc</td>
<td>200 μM</td>
<td>104 μM</td>
<td>37 μM</td>
<td>11 μM</td>
</tr>
<tr>
<td>Reference Electrolytic iron</td>
<td>200 μM</td>
<td>200 μM</td>
<td>193 μM</td>
<td>170 μM</td>
<td></td>
</tr>
<tr>
<td>Reference Carbonyl iron</td>
<td>200 μM</td>
<td>200 μM</td>
<td>193 μM</td>
<td>170 μM</td>
<td></td>
</tr>
</tbody>
</table>

[0078] TABLE 2

<table>
<thead>
<tr>
<th>Chloroform (CHCl₃) concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Example Number</td>
</tr>
<tr>
<td>----------------</td>
</tr>
<tr>
<td>6</td>
</tr>
<tr>
<td>6</td>
</tr>
<tr>
<td>4</td>
</tr>
<tr>
<td>Reference Electrolytic iron</td>
</tr>
<tr>
<td>Reference Carbonyl iron</td>
</tr>
</tbody>
</table>

[0079] The experimental data showed that both the aluminum oxide/iron and aluminum oxide/zinc particles immediately began to convert carbon tetrachloride to chloroform. Chloroform was subsequently eliminated more rapidly so by the aluminum oxide/zinc and aluminum oxide iron/Pd compositions. Terminal reaction products were gaseous hydrocarbons including methane and ethane. The reference iron powders showed negligible reactivity in the time period studied under these processing conditions.

[0080] The increased reactivity of aluminum oxide/iron particles without Pd is presumed to be a result of the high surface area since reaction rates generally scale in proportion with available surface area. Additional factors that may positively influence reactivity include storing the material in a non-reactive solvent in an oxygen-free environment. Conversely, the dry reference powders can experience surface oxidation to produce a non-reactive surface layer. Similarly, powder synthesized or stored in water react to form other non-reactive substances including oxides, hydroxides, and oxyhydroxides.

[0081] The even more increased reactivity of the aluminum oxide/iron particles with Pd is consistent with prior work as disclosed in U.S. Pat. No. 5,534,156 that demonstrated the ability of Pd to enhance reaction kinetics. The even more increased reactivity of the aluminum oxide/zinc particles is consistent with the more reactive nature of zinc in relation to more noble iron. Further increases in reactivity are achievable when using even more reactive metals such as magnesium.

1. A process for creating a composite particle having a size in the range from nanoscale to micrometer, said process comprising:
   a) forming a composition comprising a core material, a metal material and a solvent;
   b) mixing said composition until an amount of said metal material is deposited onto a surface of said core material thereby forming a composite particle; and
   c) adding a dispersant material to said composition after said metal material is deposited onto a surface of said core material; wherein said solvent material is selected based upon characteristics comprising flash point temperature, autoignition temperature and viscosity; and
wherein said dispersant material is selected based upon characteristics comprising estimated hydrophilic-lipophile balance and solvent system compatibility.

2. The process according to claim 1 wherein the core material is selected from the group consisting of oxide ceramics, non-oxide ceramics, phosphates, sulfates, carbonates, clays, minerals, polymers, plastics, intermetallic compounds, metals, metal alloys and combinations thereof.

3. The process according to claim 1 wherein the metal material is selected from the group consisting of Li, Al, Na, K, Si, Mg, Ca, Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, Ge, Rb, Sr, Y, Zr, Nb, Mo, Pd, Ag, In, Sn, Sb, Cs, Ba, La, Ta, W, Pt, Au, Pb, Bi, C, and U and alloys and combinations thereof.

4. The process according to claim 2 wherein said metal is zero valent.

5. The process according to claim 1 wherein the solvent material is selected based upon further characteristics selected from the group consisting of pH, NFPA flammability rating, NFPA health rating, degree of biodegradability, degree of toxicity, degree of miscibility, NFPA reactivity rating and combinations thereof.

6. The process according to claim 1 wherein the solvent material has a flash point temperature of greater than about 75°C.

7. The process according to claim 1 wherein the solvent material has an autoignition temperature of greater than about 150°C.

8. The process according to claim 1 wherein the solvent material has an NFPA flammability rating of ≤1.

9. The process according to claim 1 wherein the solvent material has a viscosity of less than about 100 centipoise.

10. The process according to claim 1 wherein the solvent material has an NFPA health rating of ≤1.

11. The process according to claim 1 wherein the solvent material has an NFPA reactivity rating of ≤1.

12. The process according to claim 1 wherein the solvent is selected from the group consisting of glycols, glycol ethers, vegetable oils and combinations thereof.

13. The process according to claim 1 wherein the dispersant material is selected from the group consisting of anionic, cationic, amphoteric, nonionic and combinations thereof.

14. The process according to claim 13 wherein the dispersant material has a hydrophilic-lipophile balance of ≤10.

15. The process according to claim 14 wherein the dispersant is Disperbyk® 180.

16. The process according to claim 1 wherein the core material, metal material and solvent composition is in the range from about 10 to about 60 volume percent powder fraction.

17. The process according to claim 1 wherein the composition comprises additional materials.

18. The process according to claim 17 wherein additional materials are added to the composition after adding the dispersant material.

19. The process according to claim 17 wherein the additional materials are selected from the group consisting of metals, non-metals and micronutrients.

20. The process according to claim 19 wherein the additional materials are selected from the group consisting of Pd, Pt, Au, Cu, Ni and combinations thereof.

21. The process according to claim 20 wherein the additional materials are introduced to the composition by an electrochemical reaction.

22. The process according to claim 1 wherein said core material is added incrementally to the composition.

23. The process according to claim 1 wherein the mixing is performed by at least one mechanism selected from the group consisting of milling, ultrasound, lapping and combinations thereof.

24. The process according to claim 23 wherein milling is performed with a mill selected from the group consisting of attritor, sand, jar, ball, vibratory and combinations thereof.

25. The process according to claim 23 wherein the mixing is performed in modes selected from the group consisting of batch and continuous.

26. The process according to claim 23 wherein the mixing is performed from about 1 to about 5 hours.

27. The process according to claim 23 wherein the mill is operated at speeds in the range of about 500 to about 3500 rpm.

28. The process according to claim 23 wherein the mixing is performed in steps.

29. The process according to claim 23 wherein the mixing is performed in steps to add the dispersant material.

30. The process according to claim 23 wherein the process temperature is maintained between the freezing point and boiling point of the solvent material.

31. The process according to claim 23 wherein the process is performed in a gas selected from the group consisting of air, nitrogen, oxygen, forming gas and combinations thereof.

32. The process according to claim 23 wherein the process is performed in a vacuum.

33. The process according to claim 23 wherein the amount of dispersant is in the range from about 0.25 to about 1.50 mg/m² total surface area of the composite particle.

34. A composition comprising:

a) a composite particle having an inner core material and an outer metal material deposited onto said core material;

b) a solvent; and

c) a dispersant having miscibility in said solvent;

wherein the size of said composite particle is in the range from nanoscale to microscale.

35. The composition according to claim 34 wherein the core material is selected from the group consisting of oxide ceramics, non-oxide ceramics, phosphates, sulfates, carbonates, clays, minerals, polymers, plastics, intermetallic compounds, metals, metal alloys and combinations thereof.

36. The composition according to claim 34 wherein the metal material is selected from the group consisting of Li, Al, Na, K, Si, Mg, Ca, Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, Ge, Rb, Sr, Y, Zr, Nb, Mo, Pd, Ag, In, Sn, Sb, Cs, Ba, La, Ta, W, Pt, Au, Pb, Bi, C, and U and alloys and combinations thereof.

37. The composition according to claim 36 wherein said metal is zero valent.

38. The composition according to claim 34 wherein the solvent is selected from the group consisting of glycols, glycol ethers, vegetable oils and combinations thereof.
39. The composition according to claim 34 wherein the dispersant material is selected from the group consisting of anionic, cationic, amphoteric, nonionic and combinations thereof.

40. The composition according to claim 39 wherein the dispersant material has a hydrophile-lipophile balance of ≦10.

41. The composition according to claim 34 wherein the dispersant is Diaperbyk® 180.

42. The composition according to claim 34 wherein the core material, metal material and solvent powder fraction is in the range from about 10 to about 60 volume percent.

43. The composition according to claim 34 further comprising materials selected from the group consisting of metals, non-metals and micronutrients.

44. The composition according to claim 43 wherein the materials are selected from the group consisting of Pd, Pt, Au, Cu, Ni and combinations thereof.

45. The composition according to claim 34 wherein the amount of dispersant is in the range from about 0.25 to about 1.50 mg/m² total surface area of the composite particle.

46. A method for remediation comprising contacting contaminated material with a composite particle made by the process according to claim 1.

47. A method of remediation comprising contacting contaminated material with the composite particle according to claim 34.

48. In a remediation process, the improvement being use of a particle according to claim 34.

49. A composition produced by the process according to claim 1.

50. A process for selecting a dispersant comprising

a) forming a particle suspension of about 1 to about 10 mass percent, wherein

i) said particle is selected from the group consisting of oxide ceramics, non-oxide ceramics, phosphates, sulfates, carbonates, clays, minerals, polymers, plastics, intermetallic compounds, metals, metal alloys and combinations thereof; and

ii) suspending medium is a solvent selected from the group consisting of glycols, glycol ethers, vegetable oils and combinations thereof;

b) selecting a candidate dispersant, wherein

i) said candidate dispersant is soluble in said solvent; and

ii) said dispersant has a hydrophile-lipophile balance of ≦about 10;

c) introducing said candidate dispersant in an amount of about 0.0015 to about 0.0045 g/m² total particle surface area into said suspension;

d) agitating said suspension; and

e) observing said suspension for evidence of at least one result selected from the group consisting of Tyndall Effect, uniform distribution of particles, and combinations thereof.

51. The process according to claim 50 wherein said agitating is performed by ultrasound.

* * * * *