PARTICLES CONTAINING REVERSIBLE COVALENT BONDS WHICH MAY BE SEQUENTIALLY FORMED AND BROKEN MULTIPLE TIMES

New solid particles covalently bonded to polymers that are stable and dispersible in any media and can be recycled multiple times. These particles can be used in a range of fields such as cosmetics, medicine, pharmacology, microelectronic, inks, coatings, pesticides etc...

Context

Small size particles, used in a wide range of domains (cosmetics, medicine, pharmacology, printing, inks, sensors, ceramics, colorants, pesticides etc.), present the disadvantage of aggregating in a medium due to interaction forces. To stabilize the dispersion, the medium must be adapted to the chemical properties of each particle; stability additives can also be added to the medium.

Polymers can be grafted or adsorbed onto the particle surface. This creates a repulsion between the particles which stabilizes the dispersion. Nonetheless, this method is irreversible; bare particles cannot be recovered from polymer grafted particles, nor reused. Moreover, steric and electrostatic interactions can occur only if the polymer is solubilized into a specific dispersing medium.

There is thus a need for particles that provide long term stable dispersions, and that can be recovered and re-used.

Invention description

The invention consists in innovative solid particles, bonded to polymers; it provides a solution to attach polymers (M) to particles (P) by the mean of reversible covalent bond(s) -A---B-. The polymer M can present a degree of polymerization between 5 and 1000 and act as a stabilizing agent to avoid the formation of aggregates. -A---B- can be a disulfide, a boronic ester, an acetal or an imine.

These new particles $P-(A-B-M)_x$ ($x \ge 1$) can be dispersed in any media (solvents, reactive solvents, resins, matrices etc) with stability. Particle dispersion is reversible and controlled; particles can be further aggregated, recovered and reutilized for other applications.

The covalent bond -A---B- can be subsequently broken to recover A-functionalized particles P. This can be induced by an external stimulus (pH, reducing/oxidizing agents) or by adding another molecule C interfering with -A---B-. The reversible covalent bond can thus be broken and reformed several times.

The new particles $P-(A--B-M)_x$ can be dispersed in a medium mechanically or by a mechanochemical method (ultrasonication, ball-milling, grinding).

A-functionalized solid particles P can further be aggregated in a medium by breaking the covalent bond or by decreasing the solubility of the polymer M in the medium. Depending on

the polymer, different methods can be used to decrease its solubility such as changing the temperature, pH, ionic strength of the dispersion or applying light, external magnetic or electric fields.

Added value

The technology's implementation is straightforward. It provides new solid particles that can be covalently bonded to a polymer in a reversible way. The new particles are stable in dispersed media and can be recycled.

The invention may be used with **any kind of solid particles of any size** $(0,005 \text{ to } 1000 \ \mu\text{m})$ *i.e.* **mineral particles** like metal particles (alkaline earth metal, transitional metal, rare earth metal, alloys, aluminium, copper, cadmium, silver, gold, titane, zinc, cerium, nitrides, carbides...), **organic particles** (polystyrene, poly(vinylacetate), poly(-methylstyrène), copolymers of styrene, nylon, poethylene powders...), **pigments** (ink, coating compositions, liquid and solid toners, films, plastics, coloured pigments...), **conductive particles** (carbon nanotubes...), **magnetic particles** (ferrite, magnetite...), **catalysts**.

A large number of polymers that vary in terms of composition, topology and functionality can be used.

Potential market

These new particles can be used in a range of fields like cosmetics, medicine, pharmacology, microelectromechanical systems, printing, inks, inkjet inks, toners, semiconductors, sensors, catalysis, elastomer/polymer reinforcement, coatings, plastic, rubbers, ceramics, colorants, abrasion-resistant polymers, electrophotography, flavor enhancers, pesticides, lubricants.

Intellectual property

JP2016502570 (granted) CN104955902 (pending) EP2912117 (granted) US20150240005 (granted)

Keywords

Functionalized particles; Functionalized polymers; Reversible covalent bond; Stable particle dispersion; Particle recycling

Technology domain

Basic materials chemistry; Macromolecular chemistry; Polymers; Materials; Metallurgy

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