Cosmetic formulations comprising ZnO nanoparticles

By BASF

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The invention relates to the formation of surface treated zinc oxide and titania particles, and in particular zinc oxide and titania nanoparticles, with a silo xane star-graft copolymer coating, comprising a looped and/or linear polymeric structure on a star-graft copolymer coating, on a particle surface to control the interfacial surface interactions between the particle and the oil phase of the cosmetic skin formulation.

The surface treated particles are used in personal care formulations defined as cosmetic or dermatological preparations for skin care, hair care, foot care, sun care, oral care, baby care, toiletries, color cosmetics, personal cleaning, and topical human sunscreens.

General Batch Process for Surface Treating Zinc Oxide and/or Titania Particles

The method comprises introducing zinc oxide and/or titania particles comprising a plurality of nanoparticles into a surface treatment vessel that is capable of mixing and heating its contents under a controlled environment. Example of suitable surface treatment vessels comprise a Buchi Rotovap (small scale available from Brinkmann Instruments), V-blender (commercial scale available from Patterson-Kelley), ribbon-blender (commercial scale available from Jaygo), rotary oven (commercial scale available from Thermal Processing Solutions), and a fluidized bed (commercial scale available from Littleford Day). a) The particles are introduced into the surface treatment vessel using methods known to those skilled in the art. Oxygen is removed from the vessel, typically by vacuum followed by inert gas flush, and the plurality of nanoparticles is mixed by methods such as, but not limited to, rotating the vessel or by rotating elements within the vessel. The particles are substantially spherical nanocrystalline nanoparticles and readily flow using standard unit operation methodologies. Particle mixing is carried out at a temperature, in an environment, and for a time that is effective at exposing particulate surface area to the environment of the surface treatment vessel enabling conditioning of the particle surface. Mixing may occur continuously, or at programmed intervals, and at a range of mixing rates. Mixing may occur at room temperature or at temperatures above or below room temperature depending on the chemistry of the surface treated particles. The degree of mixing may be used to control the bulk density of the final product--greater mixing yields a higher bulk density particulate product. b) Particle surface conditioning comprises, but is not limited to, removing material sorbed to the particle surface, adding dopants to the particle surface, or a combination of conditioning steps. Particle surface conditioning may be accomplished by, but is not limited to, the following unit operations: vacuum treatment, plasma treatment, washing or flushing or fluidizing with a gas, fluid washing, reactive gas or fluid treatment, etc. In all instances reactive by-products and residues are removed prior to the application of surface treatment precursors. c) Subsequent to particle surface conditioning, the particles are mixed with surface treatment precursors and heated in an environment, to a pre-determined temperature, and for a time effective for the star-graft copolymer to coat the nanocrystalline zinc oxide and/or titania particle and the difunctional precursors to polymerize to form the looped and/or linear chains on the star-graft copolymer. During surface treatment particulate mixing enables continuous surface exposure and promotes application of a uniform surface treatment to the plurality of particles. The nanoparticles and the coating precursor are added in relative quantities effective to enable a personal care application. The amount of coating precursor used is directly related to the particle surface area or the particle size. d) Surface treatment sequences may include, but are not limited by, the followings process sequences: particle conditioning followed by surface treatment as in b) and c) above, multiple particle conditioning steps followed by surface treatment, particle conditioning followed by multiple surface treatment steps, sequential particle conditioning--surface treatment--particle conditioning--surface treatment steps, and others imagined by those skilled in the art. e) The particles may comprise a single composition or multiple compositions. f) Methods of introducing the surface treatment precursors may

include, but are not limited by, fluid spray or vapor flow, employing any metered technique known to those skilled in the art. g) The surface treatment precursors may be introduced as a precursor mixture, as a precursor mixture followed by a single precursor, or by sequential single precursor additions. h) The surface treated particles are dried, if wet, cooled to room temperature, if reaction occurs at elevated temperature, and removed from the surface treatment vessel.

The surface treatment on the zinc oxide product, when added to cosmetic formulation, prevents ion leakage and renders the surface treated zinc oxide compatible with charged organic moieties, such as acrylate-based polymers, .alpha.- and .beta.-hydroxy acids. No other known commercial product or known surface treatment on zinc oxide particles enables the observed chemically passive behavior.

Cosmetic Formulation:

Surface treated zinc oxide nanoparticles, produced by methods disclosed above, were incorporated into the oil in water formulations using a homogenizer.

Sunscreen Formulation

Item No	Ingredient Name	Weight %
	Phase A	
1	Steareth-2	1.50
2	Steareth-21	0.50
3	Cetearyl Alcohol	3.00
4	Bees wax	0.20
5	Cetearyl Ethylhexanoate	20.00
6	Surface Treated Zinc Oxide	5.00
	Phase B	
7	Acrylates/C10 - C30 Alkyl Acrylate Crosspolymer	0.30
8	Deionized Water	qs
9	Triethanolamine	0.04

Procedure: 1. Heat Phase A ingredients to 80oC. 2. Stir Phase A at 11000 rpm using a homogenizer for 3 minutes 3. Mix Phase B ingredients and heat them to 80.degree. C. 4. Add Phase B to Phase A to form Mixture 1 5. Stir Mixture 1 at 11000 rpm using a homogenizer for 30 sec 1 min 6. Cool Mixture 1 to room temperature under gentle planetary mixing 7. Stir Mixture 1 at 11000 rpm using a homogenizer for 30 sec 1 min.