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Stable TiO_2 dispersions for nanocoating preparation

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ABSTRACT

In this research the preparation and characterization of titanium (TiO_2) coated self-cleaning cellulose materials starting from TiO_2 P25 powder (Degussa, Germany) was studied. The aim of the research was to decrease high aggregation of TiO_2 P25 nanoparticles, using surfactant species as dispersant and/or stabilisers (considering the balance between repulsive and attractive forces), in view of the fact that TiO_2 nanoparticles, when optimally separated into smaller particle populations, present the best properties in the system they are used in (coatings). For this purpose cationic alkanediyl-α,ω-bis-N-dodecyl-N, N′-dimethyl-ammonium bromide (Gemini) and anionic sodium dodecyl sulphate (SDS) surfactants were applied, with concentrations under their CMSs. Size and zeta-potential (ζ-potential) characterization of stable colloidal dispersions were performed. For stable 0.5, 2.5 and 5.0 mg/mL TiO_2 dispersions in the presence of 250×10^{-6} mol/L Gemini surfactant (ζ-potential ~ 40 mV) only two scattering populations were determined, at 78–95 nm and at ~280 nm. As a proof of stabilized TiO_2 P25-surfactant colloidal dispersions uniform coatings were obtained, generated at the fibre surfaces, which were analyzed by scanning electron microscopy (SEM). With the usage of proper amounts of surfactants, homogeneous thin TiO_2 coatings were formed. Superior dense coatings on the fibre surfaces were formed after treatment in 5.0 mg/mL aqueous TiO_2 P25 dispersions, in the presence of 250×10^{-6} mol/L Gemini surfactant in 5.0×10^{-3} mol/LKBr at 25 °C. In addition to that, self-cleaning test was performed. Higher photocatalytic activity was determined for samples with denser coatings. Fibres with thin coatings had a lack of photocatalytic activity.

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1. Introduction

The production of inorganic particles in the form of powder, as well as their application field, has been developed rapidly in the last decade. Inorganic particles are frequently used to obtain materials with special properties such as composites, ceramics and coatings. Self-cleaning applications using semiconducting powders or thin films have become a subject of increasing interest especially in the last 10 years. Self-cleaning materials were developed by coating glass, ceramic tiles, plastics or fibres with highly photoactive semiconducting photocatalyst titanium dioxide (TiO_2) [1–6].

Redispergation and stabilization of powders in liquid media is still a big problem of different technological processes, since numerous properties of the final product depend strongly on colloidal stability of particles and their distribution in a certain volume [7].

One of the most important and controllable properties of such suspensions is their stability. In the macroscopic sense this term expresses an even distribution of components throughout the whole volume and resistance of the phases to separate from each other with time [8]. The sedimentation behaviour is often seen as the crucial stability criterion. According to the Derjaguin, Landau, Verwey and Overbeek theory (DLVO theory), there are two basic forces controlling the stability of colloidal suspensions: the van-der-Waals and the electrostatic forces [9]. If the value of the total potential energy is positive and high enough, particles will repel each other; otherwise they experience a mutual attraction. The van-der-Waals force is attractive between particles of the same kind and can be repulsive or attractive between two different particles. The electrostatic force is related to the particle charge. For multicomponent system it is either attractive or repulsive depending on the sign of charge of the respective particle kinds. The zeta potential (ζ-potential) represents the effective measure of the particle charge. For most one component suspensions high values of this parameter indicate high stability, while low values imply coagulation. One of the most effective ways to affect the properties of colloidal suspensions is the addition of surfactants, which adsorb at the solid–liquid interface. Surfactants are known to play a vital role in many processes of interest in both fundamental and applied science.

In order to obtain self-cleaning coatings, TiO_2 P25 photocatalyst was applied in the present research. TiO_2 P25, containing a mixture of rutile and anatase crystalline forms, is the most widely used photocatalyst and has proven to be the best photocatalyst towards a broad range of organic pollutants [10–15]. Like in other catalytic applications, the surface area which is available for reactions plays a part in photocatalysis also [16]. Since the level of TiO_2 P25 nanoparticles aggregation is rather high

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[16,17], surfactants were introduced into the process to control nanoparticles aggregation.

The adsorption of conventional surfactants on TiO_2 and other surfaces [7,18–28] as well as Gemini adsorption at solid/liquid interfaces and on solid surfaces was already investigated by several researchers [29–31]. However, the aim of our research was to determine the influence of surfactants on the nanocoating formation. Gemini consisting of two surfactant units show significant surface active efficiency and form micelles at much lower concentrations then the corresponding homologues [32,33]. High surfactant efficiency and low critical micelle concentration (CMC) values have suggested the use of Gemini surfactants. They form micelles at concentrations much lower than corresponding monomeric species. Higher effectiveness of Gemini causes more stable dispersions, due to their surface tension at the CMC (γ_{CMC}) [34]. For this purpose Gemini and SDS surfactants were selected with concentrations below the critical micelle concentration (CMC).

2. Experimental

2.1. Materials

In the research TiO_2 P25 nanoparticles were used (kindly provided by Degussa, Germany) with average diameter of 21 nm and specific surface area close to $55 \pm 15\ m^2/g$ and refractive index above 2.5 [35,36], which were dispersed in filtered double-distilled water or in 5.0×10^{-3} mol/L KBr solution.

As cationic surfactants alkanediylα,ω-bis (N-dodecyl-N, N′-dimethylammonium bromides), alkylammonium Geminis, were used, with dodecyl groups linked to both ends of α, ω-N,N′-dimethylamine chains separated by two or six methylene units, which act as spacers between the polar head groups. 12-6-12 Gemini surfactant was used with CMCs below the milimolar range, 5.0×10^{-4} mol/L in 5.0×10^{-3} mol/L KBr at 25 °C.

As anionic surfactant sodium dodecyl sulphate (SDS) was used with CMC 8.0×10^{-3} mol/L in 5.0×10^{-3} mol/L KBr at 25 °C.

TiO_2 P25 concentrations used were 0.5, 2.5 and 5.0 mg/mL, SDS concentration used was 5.0×10^{-3} mol/L. In the meantime cationic Gemini surfactant concentrations used were 1.0 and 250×10^{-6} mol/L.

Regenerated cellulose Lyocell fibres (1.17 dtex; Lenzing, Austria) were coated.

2.2. Preparation of stable colloidal dispersions

2.2.1. Preparation of KBr solution

KBr solution was prepared using 5.0×10^{-3} mol/L KBr and deionized water (pH ~ 7).

2.2.2. Preparation of Gemini solutions

Water solution of 5.0×10^{-4} mol/L Gemini surfactant in deionized water was prepared (pH ~ 6). During a research, diluted solutions with proper surfactant concentrations were prepared from an initial solution. The Gemini surfactant concentrations used were: 1.0 and 250×10^{-6} mol/L.

2.2.3. Preparation of SDS solutions

While stabilization by cationic Gemini surfactants was investigated using different concentrations, SDS was used at a single concentration only. Water solution of 5.0×10^{-3} mol/L SDS surfactant in deionized water was prepared (pH ~ 7).

2.2.4. Preparation of TiO_2 P25 suspensions

0.5, 2.5 and 5.0 mg/mL TiO_2 P25 was mixed with 5.0×10^{-3} mol/L KBr solution (pH ~ 4.5). The suspensions were dispersed for 1 h using ultrasound device Branson 5200 (Branson, Danbury, CT), operating at 47 kHz and 185 W, maintained at ~25–30 °C, to re-disperse large agglomerates before addition of surfactant, to separate big agglomerates into smaller units with the goal to obtain more uniform particle distribution in aqueous suspension. In this way, higher available surface area of TiO_2 P25 for more efficient surfactant adsorption is obtained.

2.2.5. Preparation of TiO_2 P25 dispersions with surfactants

After 1 h of TiO_2 P25 suspension sonication in ultrasound device, drop-wise addition of Gemini/SDS solutions followed. The resulting mixtures were prepared at 25 °C and kept under stirring for two days to ensure equilibration.

Stabilization process began with mixing and stirring of two solutions, Gemini/SDS and TiO_2 P25, which resulted in formation of stable colloid dispersion. Adsorption of surfactant molecules on the TiO_2 P25 nanoparticles surfaces, led to formation of one or more layers, which prevented further TiO_2 P25 aggregation. An expected stabilization process in the case of Gemini surfactant (cationic Gemini

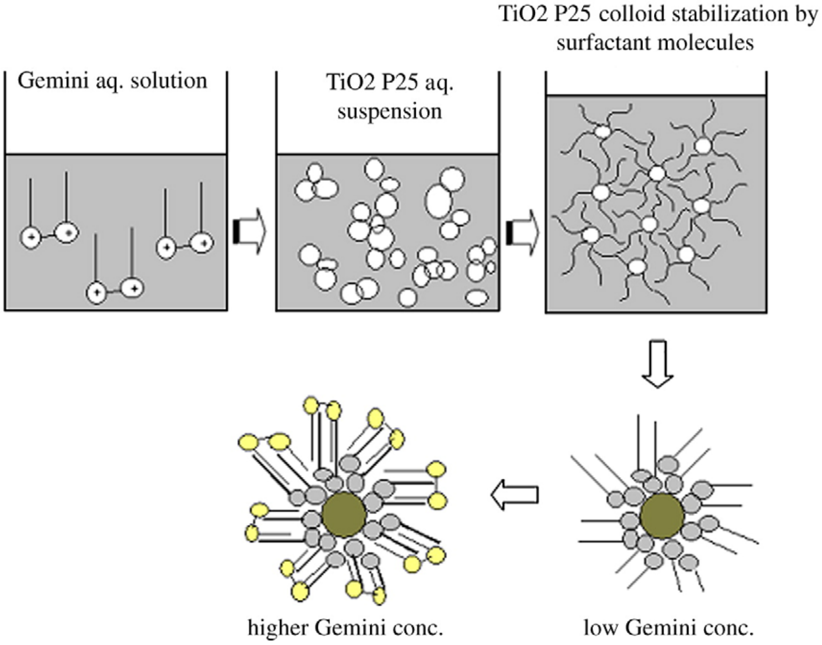

Fig. 1. Stabilization process.

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