



Influence of electrostatic particle interactions on the properties of particulate coatings of titanium dioxide



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ABSTRACT

Hypothesis: Particulate coatings are used in a wide range of technical applications. The application affecting properties of these coatings depend strongly on the structure formation along the production process. Thus, primary and secondary particle size, size distribution, particle morphology as well as the particle–particle and particle–fluid interactions of the used formulation affect the resulting coating properties.

Experiments: In this investigation titanium dioxide particles were dispersed in ethanol with a stirred media mill and stabilised electrostatically. Subsequently, the suspension was destabilised to reach specific pH* values and processed into coatings by dip coating. The influence of the pH* value of the suspension on the suspension's properties such as viscosity, agglomerate size and zeta potential and on its application properties such as coating thickness, micro-mechanical properties, abrasion resistance, gloss, roughness and adhesion was examined.

Findings: The electrostatic particle interactions show a significant influence on the structure formation as well as on the properties of nanoparticulate coatings. The coating properties are affected by the coating structures on micro-, meso- or macroscopic scale. Selective coating properties were related to the coating structure using the theoretical model of Rumpf. Besides other important process and formulation parameters, for the production of homogeneous, functional coatings with the desired properties a precise adjustment of the particle interactions is necessary.

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1. Introduction

The application of particulate coatings to influence macroscopic surface properties are used in a variety of industries [1–3]. Depending on their physical and chemical properties, colloids are used in applications ranging from optics [4–6], electronics [7,8], abrasion resistance [9] and solar energy engineering [10,11] to catalysis [12] and medical technology [13]. Colloidal titanium dioxide exhibits a wide range of technical applications. It is found in food and pharmaceutical industry [14,15] and as a pigment in paints [16], but also for photo catalytic and self-cleaning surfaces [17–19]. Application affecting properties of nanostructured coatings, i.e. roughness [20], porosity [21,22], gloss [23], transparency [24], scratch resistance [25], wear and other mechanical properties [26] are influenced mainly by the raw material properties as well as the final coating structure. This final coating structure depends strongly on the used production process of the raw material and on the process and formulation parameters of each process step during the entire process chain [27]. In Fig. 1 an exemplary process chain with influencing factors and functional properties is shown.

The formation and modification of the structures along the production process and, thus the obtained coating properties have only been investigated partly. To understand the resulting coating structures it is important to conduct a systematic investigation on how process parameters in each step affect the final application properties of coatings. The structure of colloidal systems depends on several factors: Besides the properties of the disperse system, such as agglomerate structure, size distribution, morphology, and stability, the structure formation process is determined by interparticle interactions and transport kinetics [27]. Guenther et al. [20,28,29] demonstrated for the coating process of water based silica-systems the effect of the suspension stability on the structure of thin coatings using roughness and extinction measurements as well as SEM images. The structure of dip coated coatings was observed to vary with the pH value and the ion concentration. It was found that the surface roughness increased and the transparency decreases with increasing electrolyte concentration. With decreasing stability of the suspension the surfaces become rougher indicating a change in the porosity of the coatings. Low repulsive forces acting between the particles lead to more porous films.

In the present study the approach is to fabricate coatings consisting only of TiO₂-nanoparticles from ethanolic suspensions by means of dip coating. The suspensions were manufactured using a stirred media mill in which the suspension was dispersed and

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Nomenclature

| | | | |
|------------------|---|-------------------------|---|
| ζ | zeta potential, mV | h | coating thickness, nm |
| V_{tot} | total interaction potential, J | η | viscosity Pas |
| V_{vdw} | van-der-Waal-potential, J | v | withdrawal velocity, m s^{-1} |
| A | Hamaker constant, J | σ | surface energy, N m^{-1} |
| A_p | Hamaker constant particle (titania), J | g | gravitation acceleration, m s^{-2} |
| A_f | Hamaker constant fluid (ethanol), J | ρ | density, kg m^{-3} |
| a | distance, m | $\dot{\gamma}$ | shear rate, s^{-1} |
| r | particle radius, m | F_{max}/A | maximum indentation force/contact area, $\mu\text{N mm}^{-2}$ |
| v | valence, – | ε | theoretical porosity, – |
| ε | dielectric coefficient, – | k | coordination number, – |
| ε_0 | electric constant, C (Vm)^{-1} | σ_{Rumpf} | aggregate strength, N m^{-1} |
| R | gas constant, J (Kmol)^{-1} | F | adhesion force, N |
| T | temperature, K | χ | primary particle size, nm |
| F | Faraday constant, C mol^{-1} | W | wear/weight loss, mg |
| Ψ_0 | surface potential, V | R_a | arithmetic roughness, nm |
| κ | Debye parameter, m^{-1} | R_m | adhesion, N |
| I | ionic strength mol, m^{-3} | G | gloss GU |
| k_B | Boltzmann constant, J K^{-1} | | |
| $x_{50,3}$ | median value of the volume distribution, nm | | |

stabilised electrostatically. In order to investigate the influence of the suspension's stability, represented by the agglomerate and aggregate size, the viscosity and the total interaction potential, on the resulting coating structure and properties such as coating thickness, surface roughness, abrasion resistance, micromechanical properties, gloss and adhesion the stable suspension was destabilised by adding nitric acid to vary the pH^{*}-value. Differently stabilised thin particulate coatings were produced by dip coating from the suspensions. The particulate coatings were then analysed for coating thickness, mechanical properties, abrasion resistance, surface roughness, adhesion and gloss.

2. Materials and methods

2.1. Materials

As coating substrate electro polished stainless steel 1.4301 (X5CrNi1810) of 1 mm thickness was used. Coatings on steel have a high level of technical significance as they are used to significantly increase scratch resistance, wear resistance and corrosion

protection. As feed material the pyrogenic titania AEROXIDE[®] P25 from Evonik Industries exhibiting an average primary particle size of 21 nm and a specific surface area of $50 \pm 15 \text{ m}^2/\text{g}$, according to datasheets [30], was dispersed in ethanol and stabilised with nitric acid at pH^{*} 4. Pre-tests considering the viscosity, the agglomerate size as well as the zeta potential with different stabilisers at different concentrations respectively pH^{*} values exhibit a well stabilised suspension using nitric acid at pH^{*} value of 4. P25 consist of the crystal modifications anatase and rutile. Literature reports different compositions in the same P25 sample from 73–85% anatase, 14–17% rutile and 0–18% amorphous TiO_2 [31]. In Fig. 2 TEM-pictures from very different structures are depicted, showing that the raw material exists out of aggregates and agglomerates.

As ethanol is also subjected to an auto proteolysis reaction, the dissociation constant of ethanol is $10^{-19.1} \text{ mol}^2/\text{l}^2$, therefore the pH scale ranges to 19.1 [32,33]. The dissociated protons result in a positively charged particle surface similar to the effect in an aqueous medium. For the experiments no specific pH sensor for organic solvents was available. For this reason, a sensor for aqueous systems was used (compare [34,35]). Since reference buffer solutions based on ethanol were not available either to adjust the scale of the

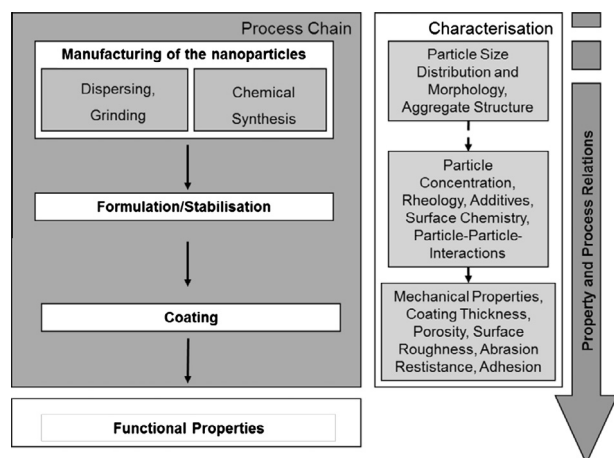


Fig. 1. Process chain of nanoparticle containing coatings and properties that are connected to each process step.

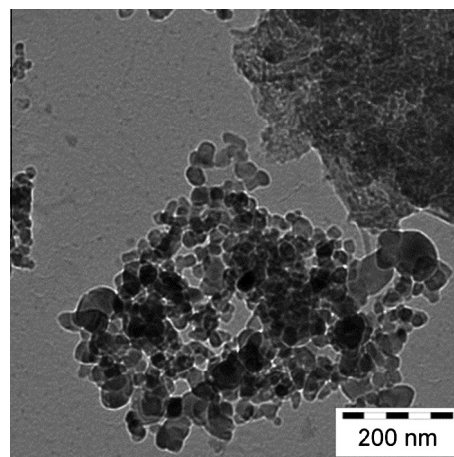


Fig. 2. TEM-picture of non-dispersed P25.

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