



Wetting and rheological characteristics of hydrophobic organic pigments in water in the presence of non-ionic surfactants



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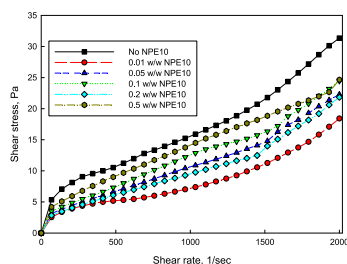
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HIGHLIGHTS

- Non-ionic surfactants could increase the wettability of pigments in aqueous medium.
- Non-ionic surfactants could decrease the viscosity of pigments in aqueous medium.
- Ethoxylation degree of surfactants affects the dispersion properties of pigments.
- The suspensions of CB pigments exhibited Bingham plastic behaviour.
- The suspensions of CuPc pigments exhibited shear thickening (dilatant) behaviour.

GRAPHICAL ABSTRACT

Rheological properties of carbon black (CB) suspensions were remarkably influenced by nonylphenol ethoxylated surfactant (NPE10) in aqueous medium.



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ABSTRACT

Wettability and rheological characteristics of highly hydrophobic organic pigments were determined in the absence and presence of surfactants. Two widely used pigments, carbon black (CB) and copper phthalocyanine blue (CuPc), were selected as model pigments. As wetting and dispersing agents, a range of non-ionic nonylphenol ethoxylated (NPE) surfactants with varying hydrophilic chain length was used in the tests. Wetting properties of the pigment powders were investigated by the contact angle measurements based on capillary rise method. Rheology experiments were carried out by a controlled shear stress rheometer. Rheological properties of pigment suspensions were determined by applying common rheological models to shear stress (τ) versus shear rate ($\dot{\gamma}$) plots. The experimental results demonstrated that, highly ethoxylated surfactants were more efficient than less ethoxylated surfactants in reducing contact angle of pigment powders. Finally, it was inferred that CB and CuPc suspensions behaved virtually as Bingham plastic and shear thickening (dilatant) type suspensions, respectively.

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1. Introduction

Organic pigments have gained considerable part in the colouration industry due to favourable properties such as brilliance, light and weather fastness, temperature stability, low density, economy, non-toxicity and high colour strength [1]. In general, they

are not readily biodegradable, and not expected to be toxic to environment because of their negligible solubility in water and in most solvents. Fine particle size, high specific surface area and oil absorption capacity of organic pigments makes them very difficult to be dispersed in the media. Therefore, they tend to aggregate during industrial applications. Primary carbon black particles of 10–100 nm form aggregates with dimensions ranging from tens to a few hundred nm through collision or fusing in the combustion zone of the reactor during manufacturing. These aggregates have a strong tendency towards forming 2–5 μm size of

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agglomerates characterized by weak bonding between aggregates in the medium.

Presence of pigment aggregates gives rise to rheological structure formation, which further thickens the dispersions and makes high pigment loadings unmanageable [2]. In addition, extensive flocculation can result in the formation of pigment network and can give rise to a yield value and to thixotropic behaviour [3]. These aggregates cannot be dispersed uniformly solely by mechanical mixing, and for fine and homogenous dispersion appropriate type and amount of the dispersant is needed [4–10]. The interaction between dispersant and pigment is of major importance, and its optimization permits to ensure a good quality of the finished products. Before applications, basic information on the rheological and dispersion properties is essential to assess the product quality. For successful results, dispersions should have good rheological properties, among them viscosity is perhaps the most important property [11]. Pigment wetting can be described as the interaction between the pigment and binder or dispersing agent that helps to prevent the aggregation. In order to obtain a good product, the difficulty of wetting the pigment in grinding resin should be overcome. Wettability studies often rely on the use of contact angle data, the significant concept being that the lower the contact angle, the greater the wetting. Direct measurement of contact angles of a liquid droplet on a smooth and clean solid surface is not applicable to powder particles. Thus, another method, called Washburn method, based on determination of the kinetics of liquid rise (or penetration) in a packed bed column of powders, was developed [12]. Alternatively, instead of measuring liquid penetration rate as height in the column, the rate has been measured by recording the weight gain of the powder column over time.

Wettability of CB and CuPc in aqueous and non-aqueous medium was investigated by some investigators who utilized different contact angle measurement techniques [10,13–18]. Investigations were mostly carried out by applying sessile drop technique on the pelletized powders [13,14,16,18,19], yet wetting studies in powder form are very limited [15,17]. Measuring contact angle of powder by recording the wicking (or penetration) rate of a liquid through a packed column of powder offers an advantage over alternative methods by accounting for both absorption and wetting properties simultaneously without spoiling the originality of the powder during preparation [17,20].

Effect of a series of poly(ethylene) glycol (PEG) type surfactants on wetting properties of CuPc pigment in aqueous medium was studied. It was found that these surfactants could improve wetting to various extents depending on their concentration and molecular weight [13]. Musselman and Chander [14] studied the wetting and adsorption properties of acetylenic diol based non-ionic surfactants on lampblack and CuPc pigments. They found that, at low surfactant concentrations below critical micelle concentration (CMC), the contact angle increased with an increase in the concentration. As concentration was increased further, the adsorbed surfactant molecules re-oriented to form a monolayer in which the hydrophobic portion of the surfactant adsorbed in a vertical configuration leaving the ethylene oxide chains extended into solution. Gupta and Bhagwat [15] used anionic, cationic, and non-ionic surfactants as dispersant for carbon black powder in aqueous medium. The performance of surfactants was tested by adsorption, wetting and stability experiments. Powder wettability was measured by liquid penetration (capillary rise) method, in which the mass gain of the powder was recorded over time. In order to neutralize the charge on the acidic carbon black surface the investigators pretreated the powder surface with triethanolamine and observed the changes in the adsorption behaviour. They found that the pretreatment had negligible effect on the adsorption behaviour of non-ionic surfactants, but the wettability and stability of the powder improved with pretreatment. In another study, Jiang et al. [10] modified carbon

black powder with a titanate coupling agent to improve its compatibility with poly(ethylene terephthalate) (PET). The contact angle analysis by drop shape (morphology) method showed that the surface modification increased hydrophobicity of the powder pellets. The optimum dosage of coupling agent for dispersion of CB within PET matrix was considered to be 4% by weight.

The aim of the present study is to investigate the wetting and flow properties of highly hydrophobic pigments in aqueous medium in the presence surfactants. The wetting properties of organic pigments in powder form are not studied to any great extent in the literature. This study includes contact angle and rheology experiments in the presence of a series of non-ionic surfactants used in a wide range of concentrations. Eventually, rheological properties of the pigment solutions were discussed in relation to the wettability of pigment powders by surfactants.

2. Materials and methods

2.1. Materials

Two commercial hydrophobic organic pigment powders were tested in the experiments as received. Carbon black powder was purchased from Alfa Aesar GmbH&Co KG (Germany) and Irgalite blue NGA a Cu phthalocyanine blue (β) was kindly obtained from Ciba Specialty Chemicals Co. (Basel, Switzerland). Some important properties of the pigments are given in Table 1, which shows that both are very fine in size.

A series of nonylphenol ethoxylates (NPE) were kindly obtained from Dow Chemicals Company (USA). These surfactants have a constant hydrophobic group composition and varying numbers of ethylene oxide (EO) groups. Solubility of the surfactants increases with increasing EO mol numbers in the groups. Some other properties of the surfactants are given in Table 2. Due to polydispersity of non-ionic surfactants, their concentrations are presented in mass units (mg/L) rather than molar units (mol/L) [14,21].

Hexane, used as completely wetting liquid for the pigments in contact angle measurements, was purchased from Merck KGaA, Darmstadt, Germany. Distilled water produced by GFL 2102 Equipment (GFL Gesellschaft für Labortechnik mbH, Burgwedel, Germany) was used in all experiments.

2.2. Contact angle measurements

Sigma 701 tensiometer from KSV Instruments Ltd. (Helsinki, Finland) was used to perform contact angle measurements. It is supplied with a standard sample cell which is a glass cylinder with 10 mm inner diameter and 25 mm height, and with a standard 50 ml glass container (vessel) in that the surfactant solution is prepared. The sample cell is open at the top from where the sample powder is loaded, and perforated at the bottom, the part brought into contact with the solution during the test. The instrument utilizes Washburn method based on the phenomena that liquid rises into pores of the powder due to capillary action resulting from the difference between the solid/gas and solid/liquid interfacial energies [22]. It is operated by versatile software that grants subtracting out the amount of liquid adsorbed by the cell itself to make accurate contact angle calculations. After calibration and adjusting the operational settings, the sample cell containing powder (CB or CuPc) was brought into contact with the prepared solution in the vessel.

When the powder pack comes into contact with a wetting liquid, the rise of the liquid through the powder pack will obey the Washburn equation (Eq. (1)).

$$m^2 = \left(\frac{C\rho^2\gamma \cos \theta}{\eta} \right) t \quad (1)$$

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