



Role of surfactants on stability of iron oxide yellow pigment dispersions

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ABSTRACT

Iron oxide yellow pigment (PY42) dispersion was prepared using the mixture of dispersing agents (surfactants) alkyl aryl sulphonates (S1), lecithin (mixture of phospholipids) (S2), alkoxyated fatty alcohols (S3), and polyoxyethylene tri-decyl ether phosphate (S4). Evaluation of this pigment dispersion in an aqueous paint resulted in acceptable coloristic properties. However, stability of the dispersion with respect to a significant increase in viscosity and settling behaviors when subjected to accelerated stability test (at 55 °C for 30 days) was observed. Rheology profiles have been used to characterize the pigment dispersions. Role of various combinations of surfactants on rheology of pigment dispersion was studied. It was found that, certain surfactants played a crucial role on stability of pigment dispersions during accelerated stability. Elimination of surfactant S3 or S4 resulted in an acceptable viscosity at ambient conditions. However, in the accelerated stability test pigment dispersion without S3 showed significantly high viscosity whereas, dispersion without S4 showed no increase in viscosity. From the physical examination, viscosity measurements and analysis of rheology profiles it can be concluded that polyoxyethylene tri-decyl ether phosphate (S4) is the key contributor for the viscosity pickup and settling behavior during the accelerated stability test. Moreover, the rheology study is of great help in predicting the relative stability of the samples.

1. Introduction

Role of surfactants is vast, and it generally carries various nomenclatures based on its role. To name a few, surfactants are termed as emulsifiers, wetting agents, dispersing agents, soaps, detergents etc [1]. Emulsifiers are used to emulsify/stabilize the oil droplets against the coalescence of the droplets. Wetting and dispersing agents are the integral part of the dispersion of solid particles e.g. pigments. Pigment dispersions have found place in the field of inks, paints, and coatings etc. to aid color and many performance properties [2,3]. Wetting of surface of pigment particles is crucial in dispersion of pigment particles which ease the process of de-agglomeration. The ratio of meniscus of the liquid to pigment particle size, and liquid to solid surface tension greatly influence the wetting speed of inorganic pigment TiO_2 [4]. To improve the performance properties such as resistance to bleeding, color etc. the pigment particles are modified by organic or inorganic materials [5–7]. Besides enhancing the performance properties, the modification of pigment particles provide active sites for dispersing agents to adsorb. Dispersing agents provide stability by anchoring on the pigment particles. Dispersing agents/surfactants act not just as emulsifiers/wetting/dispersing agents, they also influence some of the properties such as water sensitivity, scrub resistance, control on the rheology etc. adversely. Addition of pigment dispersions (carbon black)

to water based latex paint decreases viscosity of the paint due to the presence of surfactant in pigment dispersion and its interactions with the associative thickener present in the paint [8]. Dispersants with long chain and significantly higher ethylene oxide groups dramatically alter the viscosity of the pigment dispersions. Use of such surfactant decreases the viscosity in presence of hydroxyethyl cellulose (HEC), and increases the viscosity in presence of hydrophobically modified ethylene oxide urethane (HEUR) [9].

The stability of pigment dispersion plays an important role in inks, paints and coating applications. The pigment particle size, shape, and nature of wetting/dispersing agents are the key contributors to the stability of the dispersion [10,11]. For example, pigment dispersion of iron oxide yellow pigment of spherulitic morphology resulted in lower viscosity over the acicular morphology [10]. Inorganic pigments such as iron oxides due to their high specific gravity have greater tendency to flocculate and settle down, and are challenge to disperse and stabilize. Paint systems where flocculation/agglomeration of pigments (of pigment dispersions in paint) occurs, results in poor color, reduction in gloss, change in rheology, sedimentation/separation etc. [10,12]. The aggregation tendency is quite high in non-aqueous colloidal dispersions where large amount of van der Waals forces exist which results in high viscosity and poor stability [13]. Shareep et al. reported that pigment yellow iron oxide-alkyd system showed a thixotropic behavior, and the

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degree of thixotropy depends on pigment quantity, nature of solvents etc. [14]. The flow behavior/rheology of the dispersions largely depends on the surfactant or their combinations and their interactions with other ingredients [15]. Sis et al. have shown that, the contact angle of the carbon black pigment was significantly reduced with the use of highly ethoxylated dispersants over less ethoxylated dispersants [16].

Since, for paints and coating applications the pigment dispersion is generally prepared using ionic and non-ionic dispersants, one has to be careful in selecting appropriate surfactant or their combinations. The ionic dispersants adsorb on the pigment particles and provide electrostatic stabilization, and non-ionic dispersants provide the steric stabilization to the pigment dispersions. Moreover, the pigment dispersions particularly for coatings should be useful for water based and solvent based systems, and hence compatibility in both the system is a demand. Therefore combinations of dispersants are being used to deliver the better compatibility and stability to the pigment dispersions. In summary, pigment dispersions and paints are very complicated systems where various interactions occur between dispersants, pigments, binders, thickeners, solvents etc [17].

Here we report the effect of various surfactant types on the rheology, and stability of the iron oxide yellow pigment dispersions. The rheology profiles have been generated and used to analyze and identify the dispersant causing the instability to the dispersant during the accelerated stability test.

2. Experimental

2.1. Materials

Iron oxide yellow pigment was procured from Lanxess, Germany. The dispersants alkyl aryl sulphonate [S1], lecithin (mixture of phosphatidylethanolamine (PE), phosphatidylcholine (PC), phosphatidylinositol (PI), and triglycerides) [S2], alkoxyated fatty alcohol [S3], polyoxyethylene tridecyl ether phosphate [S4] are procured from Croda India Company Pvt. Ltd., Cargil India Pvt. Ltd., Huntsman International (India) Pvt. Ltd., and Rhodia specialty Chemicals India Ltd. respectively and are used as received. General chemical structures of the above dispersants are shown in Fig. 1, the chemistries of the major components of the lecithin, phospholipids is shown. Hydrophilic lipophilic balance (HLB) are as follows, S1 has HLB ~ 11.7 , S2 has HLB 2–9, S3 has HLB ~ 13.7 , and S4 has HLB ~ 9.6 . Glycol was procured from India Glycols. De-mineralized water was used in all the experiments.

2.2. Preparation process for pigment dispersion

Pigment dispersions (approximately 50% of pigment) were

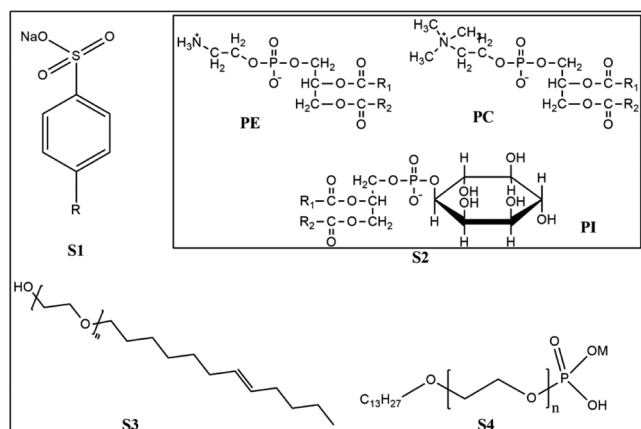


Fig. 1. General structure of the dispersing agents used in the present study. “R” stands for the alkyl chain length, and “M” stands for counter ion like Na, NH_4 , etc.

prepared by using high speed dispermat (VMA Getzmann GmbH, Germany) at ~ 3000 rpm for about 30 min. The pre-mix which contain water, dispersants, were mixed thoroughly for 10–15 min at ~ 600 rpm before pigment was added to it. After completing the grinding, the mixture was added with water and additives.

2.3. Characterization of dispersions

The viscosity was measured using Brookfield viscometer (KU2 Viscometer) using paddle type spindle at 200 rpm at 25°C .

The accelerated stability test was performed by keeping the samples in 300 ml lacquered containers at 55°C for 30 days in accordance to ASTM D 1849 [18]. The samples were kept in duplicate for interim observation at 15 days.

The flow behaviors of the pigment dispersions were characterized using rheometer Physica MCR 301 from Anton-Paar using cup and bob geometry. The rheometer was equipped with EC motor with low friction bearings, normal force sensors, and combination of rotational and oscillatory mode. All rheograms were recorded at 25°C and the samples were used as such without dilution.

The hysteresis profiles were recorded by applying shear ramp in the range of 0.01 s^{-1} – 3500 s^{-1} . The up curve in the hysteresis curve was while increasing shear rate from 0.01 s^{-1} – 3500 s^{-1} , and the down curve in the hysteresis curve was while decreasing shear rate from 3500 s^{-1} – 0.01 s^{-1} .

Using the same rheometer, creep recovery profiles were recorded at applied stress of 0.1 Pa for 600 s and then the recovery (deformation) was monitored for next 900 s. The oscillatory amplitude sweep test was performed by applying the strain amplitude 0.01–100% at constant frequency of 1 Hz. After generating the profiles, linear viscoelastic range (LVR) was calculated (5% deviation in the linearity of storage modulus G'). The frequency sweep test was used to understand the material structure at the LVR. Here, the material structure was perturbed without destroying it. The frequency used in this test was 10 – 0.1 s^{-1} at 25°C . A more detailed description/definition of various terminologies related to the rheology are available elsewhere [19–22]

3. Results and discussions

Iron oxide yellow pigment dispersion prepared using mixture of all dispersants S1 to S4 showed significantly increased viscosity after accelerated stability test, along with that there was hard settling at the bottom of the container. To analyze these observations, initial (at ambient conditions) and accelerated samples were subjected to rheometry to understand the rheological behaviors under applied shear. Fig. 2 shows hysteresis curves (viscosity against shear rate) for the iron oxide dispersion which was subjected to increasing and decreasing shear rate in the range of 0.01 s^{-1} to 3000 s^{-1} .

Rheogram of samples after the accelerated stability test showed higher viscosity when compared to the rheogram of initial sample up to the shear rate of 2000 s^{-1} , whereas, beyond shear rate of 2000 s^{-1} both the samples exhibited similar viscosity behaviors. However, the interesting observation was that, even with initial sample above the shear rate of 400 s^{-1} , there was increase in viscosity (Fig. 2). Such a shear thickening behavior was not observed in rheogram of accelerated sample, such a behavior might be due to the increased viscosity during accelerated stability test. The shear thickening behavior of initial sample might be due to the instability or phase separation or rearrangement at the particle-medium interface or the turbulent flow at high shear condition. Generally, paints and pigment dispersions are pseudo plastic in nature. Shear thickening behavior was generally observed for slurries/high concentrated dispersions [23]. The area between the hysteresis curves corresponds to the viscosity lag. Higher the area, sluggish is the viscosity recovery of the samples when subjected to decreasing shear rate. The sample after accelerated stability test showed more sluggish response to applied shear rate than that of initial

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