Colloid Polym Sci 276:420–426 (1998)
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ORIGINAL CONTRIBUTION

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The charging mechanism of organic colorants in metal soap solutions

Received: 30 September 1997
Accepted: 15 December 1997

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Abstract The mechanism governing the generation of electrical charges on organic colorants dispersed in a metal soap solution is investigated. The system constitutes the basic ingredients of liquid developers used as color printing inks. The variation of surface charge density, σ_s, as a function of the metallic soap concentration in the apolar medium is determined from the electrophoretic mobility measurements. The experiments were conducted with different amounts of moisture in the non-conducting medium and physisorbed binder resins on the particle surface. A site-binding (SB) model is proposed which elucidates the surface complexation reactions occurring with the reactive groups at the particle–solvent interface. An equilibrium approach is adopted which focuses on the molecular description of the surface charge generation. The model results demonstrate good agreement with the measured σ_s. The equilibrium constants are determined from a non-linear, least-square curve-fit analysis.

Key words Electrophoretic mobility – copper phthalocyanine – surface charging mechanism – site-binding model

Introduction

The stability of organic pigment particles in a hydrocarbon medium is of utmost importance in the proper operation of electrophotographic and ink-jet printing processes. In its simplest formulation the liquid developer, used as color printing inks, is composed of organic colorants dispersed in a low permittivity medium. Particle aggregation due to coagulation or flocculation can hamper the flow properties and the final image quality. Metallic soaps are commonly added to the system to improve the wettability or dispersibility of the particles in the non-conducting phase. The surfactant adsorbs onto the particle surface and subsequently enhances the suspension stability in the apolar medium.

The adsorbed metallic soap develops resistance of the dispersed particles to agglomeration in the non-aqueous medium in many ways. They can either impart electrical charge onto the particle surface and, subsequently, stabilize the suspension through an electrostatic mechanism or physically prevent the two surfaces from coming into adhesive contact through steric hindrance. Alternatively, the hydrophobicity induced by the exposed hydrocarbon tails of the adsorbed surfactant molecules will render the particles more compatible with the non-aqueous phase and enhance the suspension stability. The principle of hydrophobicity and steric hindrance to disperse the particles in both aqueous and non-aqueous phases is similar and, therefore, clearly understood. The fundamentals of charging in the non-aqueous medium are lacking, primarily because the generation of stable ionic species in a low dielectric constant medium is not clearly understood [1–3].

There is ample evidence of the existence of electrical charges in non-conducting media in the petroleum

processing and electric power industries [4, 5]. However, a limited number of studies have been conducted in this area. Furthermore, the variety of non-aqueous systems that have been investigated are comparatively few. The mechanisms that have been proposed to date have been inconclusive. Therefore, predicting the electrical charge generation in terms of a physical model remains elusive.

Most investigations of the mechanism of charge generation in a non-aqueous medium have been based on electrophoresis measurements. van der Minne and Hermanie [6] conducted one of the earliest studies to examine the stability of non-aqueous dispersions. Many studies have been reported since which characterize the behavior of charged colloidal particles in a low-polarity medium [7–18]. Most studies are focused on better understanding the effects of ionic surfactants and polar additivies on the surface potentials of a variety of colloidal dispersions in non-aqueous liquids. The zeta potential curves either monotonically decrease [19, 20] or display a maximum [6, 7, 10, 16, 18, 19] as a function in the surfactant concentration. Charge reversal has been observed in some specific systems, which was explained in terms of the crystal structure of the organic particles [13, 18]. These investigations reveal that the electrical behavior of non-aqueous dispersions is complex and depends on a variety of physicochemical factors.

Resin, a polymeric material, is routinely added to the printing inks. It plays a dual role of promoting the dispersion stability and enhancing the image quality. Resin is a non-ionic pigment derivative that act as a deflocculating or steric stabilization agent through adsorption onto the organic colorants. The adsorbed polymeric material reduces cohesion between the pigment particles and optimizes adhesion or binding properties with the output document and other machine parts. This facilitates the formation of stable colloidal dispersions of optimum image quality. A detailed review of its role, and how it affects the performance efficiency of the liquid developers, is presented elsewhere [21, 22].

The concentration dependence of ionic surfactants on ζ-potential in non-aqueous dispersion has been examined [20]. The aim of this paper is to provide a molecular description of the surface charging of organic colorants in a metal soap solution. A detailed fundamental study has been conducted where the surface charge density dependency on the combined effect of surfactant and moisture is investigated in terms of an equilibrium site-binding theory. The proposed theory incorporates the protonation and the association of the mobile ions in solution with the surface ionizable groups of the organic particles. The model results are compared with experimental data of surface charge vs. surfactant concentration. The surface charge of the organic colorants is inferred from the electrophoretic mobility measurements using the PALS technique [23].

Materials

The system consists of 1 wt% copper phthalocyanine (CuPc) particles (the organic colorants) dispersed in a isoparaffinic hydrocarbon (the carrier liquid). The CuPc was obtained from the Irgalite blue range of the chemical compound. The hydrocarbon liquid used in the experiments was obtained from Exxon, Inc. under the trade name Isopar G. Zirconium octanoate (ZrOn) is the oil-soluble ionic surfactant and acts as the dispersing agent. The ZrOn was obtained from Neudex Chemicals, Inc. dissolved in mineral turpentine and used as received.

The water content in the Isopar G was varied between 10 ppm (dry) and 65 ppm (wet). The "as supplied" (a.s.) Isopar G contained 31–32 ppm water. The moisture content in the Isopar G was reduced to a minimum (at 10 ppm) by storing it over 3 Å molecular sieves in a Teflon-capped serum bottle for a few days. "Wet" Isopar G was prepared by mixing equal amounts of water and hydrocarbon and vigorously shaking the resulting mixture. After equilibration, the Isopar G was collected in clean Pyrex bottles. The Karl Fisher potentiometric coulometry was employed to determine the water concentration (in ppm) in the hydrocarbon phase.

Experimental

The phase-analysis light scattering (PALS) technique [23], was used to measure electrophoretic mobilities of the non-aqueous dispersion. The experiments were conducted at Bristol Colloid Centre (BCC), University of Bristol, Bristol, U.K. The PALS technique allows us to measure small electrophoretic mobilities in insulating liquids. Smaller electric field strengths can be applied, which is particularly convenient for measuring electrophoretic mobilities in non-aqueous phases. This circumvents the problem of heating and non-uniform electric field generation that occur when a large electric field is applied across a medium of low polarity.

The electrophoretic mobility, U, was determined by measuring the velocity of the particles in suspension under the influence of a uniform electric field across the ink sample. The zeta potential was computed using the Hückel equation (for $\kappa a \gg 1$)

$$\zeta = \frac{3\eta U}{2\varepsilon_r \varepsilon_0}, \tag{1}$$

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where η is the viscosity of the liquid phase, ε_0 is the permittivity of free space, and ε_r is the dielectric constant of the liquid medium.

Various dispersions were prepared to examine the role played by zirconium octanoate and the effects of water and adsorbed binder resin on the zeta potentials of the CuPc particles. The evidence of uptake of water onto the surface of the CuPc particles has been demonstrated from FT-IR ATR spectroscopy [24]. Both adsorbed water and resin significantly affect the electrophoretic mobility of CuPc particles. The surface charge density and the governing surface complexation reactions were inferred from the mobility curves.

Electrophoretic mobility measurements

Electrophoresis experiments were performed with CuPc dispersions at different ZrOn concentrations in Isopar G using different amounts of moisture and resin. The resin is a vinyl acetate-based polymer derivative which stabilizes the colloidal dispersion by a build up of polymer adsorption layers. In addition, the resin has a strong influence on the adhesion of the CuPc particle to various surfaces such as other toner particles, machine parts, and the paper substrate.

Figures 1 and 2 illustrate the effect of ZrOn on the electrophoretic mobility of the pigment particle as a function of moisture content in Isopar G and physisorbed resin. The role of water on the surface potential is displayed in Fig. 1. All curves reveal a maximum between 1300 and 1700 ppm ZrOn. The electrophoretic mobility of ink particles increases with increasing amount of water in the dispersions. The increase in zeta potential or surface charge with water concentration can be explained in terms of the protonation reactions occurring at reactive groups on the pigment surface. The adsorption of H^+ ions of water results in a higher surface charge density and, therefore, higher zeta potential.

Figure 2 displays ζ-potential curves obtained with bare and surface-treated CuPc particles. The surface additive alters the compatibility and the charging ability of the CuPc particles in the hydrocarbon phase. Comparing Figs. 1 and 2 the qualitative trends in the σ_s remain the same. A pronounced maximum in the ζ-potential curve is observed in a similar range of ZrOn concentration compared to those obtained as a function of water content in Isopar G.

CuPc surfaces modified with adsorbed resin display a decrease in the surface potentials. Perhaps the polymeric material conceals the exposed surface sites on the CuPc particles and reduces the total number of reactive surface groups available for protonation and association reactions. This reduction in the accessible free-surface sites for ionic association/dissociation equilibrium reduces the surface potential. This hypothesis is somewhat reinforced by the decrease in ζ-potential at a higher resin content measured in a separate set of experiments not reported in this paper. It appears that the resin does not significantly change the physico-chemical characteristics of the interfaces since the qualitative trends in the measured data remain the same.

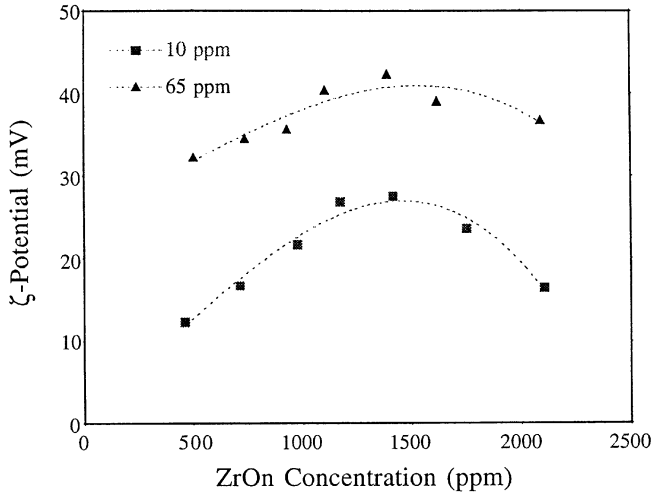

Fig. 1 Concentration effect of ZrOn on the variation in ζ-potentials in dry (10 ppm) and wet (65 ppm) isopar G

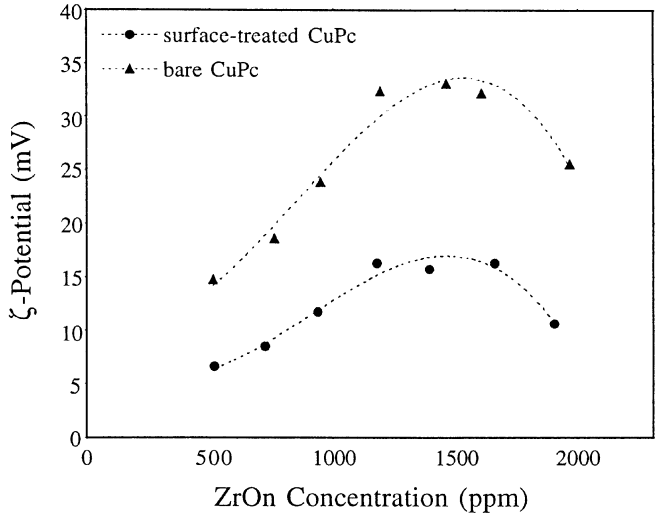

Fig. 2 Concentration effect of ZrOn on the variation in ζ-potentials with bare and surface-treated CuPc particles in isopar G

The pronounced maximum in the ζ-potential curves has been observed in a variety of other non-aqueous dispersions. Several attempts have been made to adequately explain the existence of a maximum in the ζ-potential curves [19, 25]. In this work we have considered the screening of the existing positive charges on the particle surface resulting in the reduction in the mobility curves. The mobile Oct^- ions in solution electrostatically interact with the positive ions already adsorbed on the surface. The positive ions include the H^+ ions of water and the ZrO^{2+} of the metallic soap. The ion pairs formed on the surface result in the reduction of the ζ-potential. These mechanisms have been incorporated in the site-binding model to provide a molecular representation of the charging mechanism.

The site-binding model

A site-binding (SB) model provides a general molecular description of the surface charging of CuPc particles in the metal soap solution. The model explains the role of moisture in Isopar G and the effect of physisorbed surface additives on CuPc particles on the surface charge. Both water and resins influence the zeta potential and, therefore, the particle surface charge. A generic ionizable functional group, [AH], on the particle surface is considered to act as a reactive site for acid–base interactions with the H^+ of water and the zirconyl (ZrO^{2+}) and octanoate (Oct^-) ions of the metallic soap. The association–dissociation reactions occurring at the charged surface site which control both the generation and the variation in the surface electrical potential are elucidated.

The ionic association–dissociation equilibria can be represented by the reactions

$$AH + nH^+ \overset{K_1}{\Leftrightarrow} AH^{n+}_{n+1}\,, \tag{2}$$

$$AH + ZrO^{2+} \overset{K_2}{\Leftrightarrow} AHZrO^{2+}\,, \quad \text{and} \tag{3}$$

$$AHZrO^{2+} + 2Oct^- \overset{K_3}{\Leftrightarrow} AHZrO(Oct)_2\,. \tag{4}$$

Equations (2) and (3) account for the interactions of the dissociative H^+ ions of water and ZrO^{2+} ions of ZrOn with the fixed ionizable group [AH] on the CuPc surface. The preferential adsorption of these ions results in the observed increase in the surface potential at low ZrOn concentrations. To develop a generalized site-binding model, n protons are considered to be involved in an association equilibrium with [AH] (Eq. (2)). The Oct^- ion enters into an ion pair formation with the already adsorbed zirconyl ions through Coulombic interactions (Eq. (4)). As a result of this neutralization reaction the surface charge density decreases.

The expressions for the equilibrium constants of the acid–base interactions can be written as

$$K_1 = \frac{[AH^{n+}_{n+1}]}{[AH][H^+]^n_s}\,, \tag{5}$$

$$K_2 = \frac{[AHZrO^{2+}]}{[AH][ZrO^{2+}]_s}\,, \quad \text{and} \tag{6}$$

$$K_3 = \frac{[AHZrO(Oct)_2]}{[AHZrO^{2+}\,\mathrm{I}\,Oct^-]_s}\,. \tag{7}$$

$[I]_s$ represents the molar surface concentration of species i. Since the Debye lengths of electrical double layers in a non-aqueous medium are very large, $[I]_s$ can be assumed to be equal to the bulk concentration or $[I]_s \approx [I]_b$.

The surface charge density, σ_s, of the pigment particles created by ionic reactions (Eqs. (2) and (3)) can be represented as

$$\sigma_s = e\{n[AH^{n+}_{n+1}] + 2[AHZrO^{2+}]\}\,, \tag{8}$$

where e is the electronic charge. The total number of surface sites, N_s, available on the pigment surface is

$$N_s = [AH] + n[AH^{n+}_{n+1}] + 2[AHZrO^{2+}] + 2[AHZrO(Oct)_2]\,. \tag{9}$$

Employing a high-resolution electron microscope, Kirkland et al. [26] presented micrographs showing the structural arrangement of phthalocyanine molecules. The value of N_s determined from these images is $2 \times 10^{18}\,m^{-2}$.

Combining and expressing Eqs. (5)–(9) in terms of the equilibrium constants yields

$$\sigma_s = eN_s\left[\frac{nK_1[H^+]^n_s + 2K_2[ZrO^{2+}]_s}{1 + nK_1[H^+]^n_s + 2K_2[ZrO^{2+}]_s + 2K_2K_3[ZrO^{2+}]_s[Oct^-]^2_s}\right]. \tag{10}$$

The above equation represents the surface complexation reaction dependence on σ_s of CuPc particles in the presence of water and ZrOn in the hydrocarbon phase.

Equations (2)–(4) represent the SB model describing the moisture and metallic soap concentration dependency on the surface charging of the CuPc particles. The equilibrium constants, K_i, and the degree of association, n, are determined from a fitted function of Eq. (10) with the measured σ_s data.

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Results and discussion

The DLVO theory is employed to determine the surface charge density from the mobility data. The theory has been shown to be applicable to non-aqueous dispersions [2]. The particle surface charge, Q_s, can be computed using the Debye–Hückel approximation for double layers around a sphere. This is represented as

$$Q_s = 4\pi\varepsilon_0\varepsilon_r\psi_s a(1 + \kappa a) , \quad (11)$$

where $\varepsilon(= \varepsilon_0\varepsilon_r)$ is the dielectric permittivity ($1.859 \times 10^{-11}\,C^2\,J^{-1}\,m^{-1}$), a is the radius of the sphere, and κ is the Debye–Huckel parameter which is a function of the ion concentration in the bulk solution and the valency of the electrolyte. The surface charge density, σ_s, is $Q/4\pi a^2$. For large Debye lengths in non-aqueous dispersions, the difference between the surface potential, ψ_s and ζ-potential is small and can be assumed to be equal in magnitude, i.e., $\zeta \approx \psi_s$. Equation (11) can be rewritten and the relation between σ_s and ζ can be expressed as

$$\sigma_s = \varepsilon\varepsilon_0\kappa\zeta . \quad (12)$$

The σ_s curves as a function of water and ZrOn concentrations in Isopar G have been inferred from the results of the electrophoresis experiments, which are presented in Figs. 1 and 2 as ζ vs. ZrOn concentration. Since σ_s is independent of the particle size the polydispersity of the CuPc particles will not affect the model calculations.

The surface charge density is computed from Eq. (12) and shown in Figs. 3 and 4. The pronounced maxima observed in the ζ-potential curves as functions of the ZrOn concentration are clearly suppressed in these figures. This can be explained on the basis of the dependence of σ_s on ζ and ZrOn concentration. The parameter, κ, is directly proportional to the square root of surfactant concentration. Therefore, the decrease in ζ-potential is, in part, compensated by a corresponding increase in the ZrOn concentration. Thus, the variation in the surface charge, computed from the ζ-potential curves, tend to flatten out in the range of surfactant concentration where the maximum occurs.

The theoretical curves of σ_s vs. [ZrOn], (solid lines in Figs. 3 and 4) represented by the SB model (Eq. (10)), are obtained from the curve-fit operation on the measured σ_s data. The Igor Pro software uses the Levenberg–Marquardt algorithm based on the principle of non-linear, least-squares fitting. It performs an iteration of the curve-fit algorithm and searches the equilibrium constant values that provide the minimum value of the chi-square. This parameter is a multi-dimensional error function that indicates the error between the fitted function and the raw data.

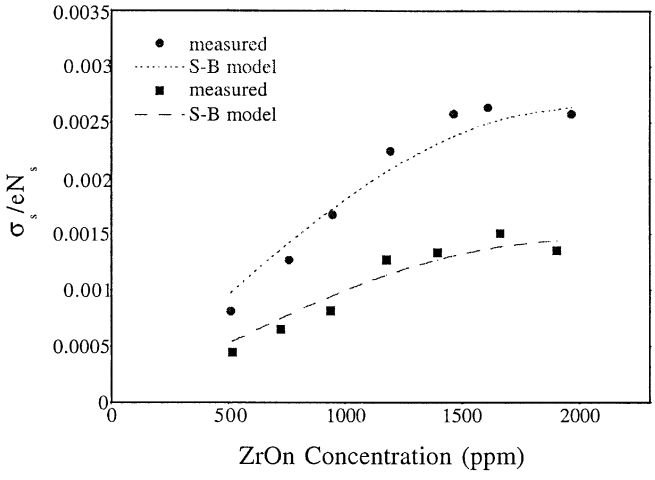

Fig. 3 Comparisons between experiments (Eq. (12)) and theory (Eq. (10)) surface charge densities of CuPc particles in the non-aqueous phase. The round and square filled markers represent the measured σ_s data in "dry" and "wet" Isopar G, respectively. The values of equilibrium constants for the fitted function are provided in Table 1

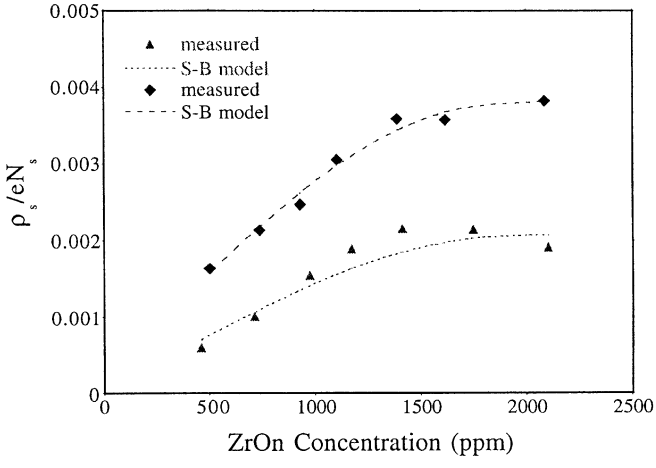

Fig. 4 Comparisons between experiments (Eq. (12)) and theory (Eq. (10)) surface charge densities for bare (diamond filled markers) and surface-treated (triangular filled markers) CuPc particles in a.s. Isopar G. The values of equilibrium constants for the fitted function are provided in Table 2

Figure 3 shows the curve-fit analysis performed on measured surface charge density curves in surfactant-bearing Isopar G at different water concentration levels. The curve-fit operation was executed for different values of n which relates to the number of H^+ ions associating with the surface functional group. A higher degree of convergence, i.e., minimum value of chi-squared, was obtained for $n = 1$. The good agreement between the experimental data and model results is clearly illustrated in Figs. 3 and 4.

The comparison was inexact for $n = 2$. For $n \geq 3$, the fitted function failed to converge during the curve-fitting operation.

It is clear from Figs. 3 and 4 that the SB model provides a good overall fit with the σ_s vs. ZrOn curves. Table 1 presents the values of the equilibrium constants (K_1, K_2, and K_3) that are obtained from the fitted function acquired from the curve-fit operation at low (10 ppm) and high (65 ppm) moisture content in Isopar G. The presented K_i values reveal consistency over a small range. Ideally these values should remain constant. However, a small variation in the measured values of K_i, clearly within the experimental error, is very much expected.

The agreement between the experiments and the theory illustrates that the SB model is a realistic and effective representation of the acid–base interactions occurring between the surface functional groups of the organic pigment and the mobile ions in solution. The increase in the positive surface electrical potential is caused by the preferential adsorption of the H^+ and ZrO^{2+} ions with the functional group, [AH]. The adsorption mechanism is represented in the SB model through Eqs. (2) and (3). At higher ZrOn concentrations the negative Oct^- ions interact electrostatically with the positive zirconyl ions already adsorbed on the particle surface (Eq. (4)). As a result, ions pairs are formed on the surface. A maximum in the surface potential is induced which screens the exposed positive surface electrical charges.

It can be argued that the decrease in the surface electrical charges can also result from the screening of the $[AH_{n+1}^{n+}]$ surface groups. This alternative hypothesis was tested by reconstructing the existing SB model. In the modified equilibrium theory, the Oct^- ion interacts with the adsorbed H^+ ions (protonated site) instead of forming an ion-pair with the adsorbed ZrO^{2+} ions (Eq. (4)). This exercise was conducted to investigate whether the modified theory better explains the surface charge dependency on the surfactant concentration. The ionic equilibrium replacing Eq. (4) of the site-binding interactions is expressed as

$$AH_{n+1}^{n+} + nOct^- \overset{K_4}{\Leftrightarrow} AH_{n+1}(Oct)_n. \tag{13}$$

The SB model represented by Eqs. (2), (3) and (13) was tested with a curve-fit operation on the surface charge density data. The modified theory was unable to accurately predict the trends in the particle charging in ZrOn-bearing Isopar G. This reinforced the postulate that the Oct^- ion forms an ion pair with the adsorbed zirconyl ions instead of associating with the adsorbed H^+ ions and suppresses the σ_s at high ZrOn concentrations.

Figure 4 illustrates the comparisons between the experiment and theory for bare and surface-treated pigment particles in a.s. Isopar G. The equilibrium constants obtained from the curve-fit analysis of the σ_s/eN_s vs. [ZrOn] curves for bare CuPc dispersions are provided in Table 2. The values are clearly within the range of those obtained with "dry" and "wet" Isopar G. This is expected, based on thermodynamic principles, where the K_i values must be independent of the moisture content. This is telling evidence that the equilibrium theory of the SB model is a reasonable representation of the charging mechanism of the colorants in the metal soap solutions.

Inexact values of K_i, i.e., values different from those provided in Table 2 for bare CuPc, were obtained from the curve-fit analysis of the σ_s plot of surface-treated CuPc particles. A reasonably good fit of the data was obtained using the K_i values obtained in the previous case with the parameter N_s in Eq. (10) reduced to 1.1×10^{18} sites m^{-2} (45% reduction), as shown in Fig. 4. The equilibrium constants of the fitted function are presented in Table 2. The number density of fixed surface functional groups, accessible for acid–base interactions, was reduced because of the adsorption of stabilizing polymer layers on the pigment particles. This result indicate that the resin conceals the ionizable surface groups involved in the acid–base interactions with the hydroxyl ions. However, the surface chemistry of the uncovered regions of the CuPc particles is not altered because the equilibrium constant values remain independent of the adsorbed polymeric material.

Thus, the equilibrium theory considers three site-binding equilibrium reactions dictating the variation in surface charge in the CuPc dispersion. The maximum observed in the ζ-potential curves (Fig. 2) is explained in terms of the preferential adsorption of positive ions (H^+ and ZrO^{2+}) in solution at low ZrOn concentration that are subsequently neutralized by electrostatically interacting with negative ions (Oct^-) at higher ZrOn concentration. The former and the latter adsorption equilibrium represent the increase

Table 1 Equilibrium constants, K_i, of the best-fitted function with measured σ_s data in "dry" and "wet" Isopar G

Moisture content (ppm)	K_1	K_2	K_3
10	1.92×10^5	1.41×10^6	0.60
65	1.81×10^5	1.19×10^6	0.69

Table 2 Equilibrium constants, K_i, and total surface sites, N_s, of the best-fitted function with measured σ_s data for bare and surface-treated CuPc particle in a.s. Isopar G

Pigment type	N_s	K_1	K_2	K_3
Bare CuPc	2.0×10^{18}	1.83×10^5	1.35×10^6	0.64
Surface-treated	1.1×10^{18}	1.77×10^5	1.27×10^9	0.62

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and subsequent decrease in the electrical surface charges, respectively, with ZrOn concentration.

Conclusions

The charging mechanism of organic pigments in an insulating liquid is characterized in terms of a predictive model. The combined effect of moisture and ionic surfactants on the variation of surface charge density is probed using an equilibrium SB model. The variation in σ_s is represented in terms of the surface complexation reactions to precisely establish the generation of electrostatic charges in the non-aqueous dispersion. The predictions of the model were verified from comparisons with the measured σ_s for bare pigment particles at two different water concentration levels in Isopar G (Fig. 3). The initial rise in the σ_s vs. ZrOn concentration plot is postulated based upon the adsorption of H^+ and ZrO^{2+} ions on the particle surface. The subsequent association of the Oct^- ions with already adsorbed zirconyl ions at higher surfactant concentrations neutralizes the exposed positive sites. As a result, a maximum in σ_s vs. [ZrOn] plot is observed. Good agreement between the theory and experiments for similar values of equilibrium constants (Table 1) reinforces this hypothesis.

The model results were tested for bare and surface-treated CuPc particles in a.s. Isopar G (Fig. 4). The σ_s curves for both kinds of CuPc provided remarkable fit with the theory in the range of K_i values obtained from the fitted function in Fig. 3. However, N_s was reduced by 45% to obtain a reasonable curve-fit for surface-modified CuPc particles. This clearly indicates that the exposed surface sites available for ionic interactions and subsequent electrical charging, are screened by the adsorbed resin. Reasonably constant K_i values (Tables 1 and 2) obtained with varying amounts of water and binder resin validate the proposed SB model as an effective molecular representation of charging mechanism of the organic colorants.

Acknowledgements The author thanks Prof. John Ralston and Dr. Paul Jenkins for many valuable discussions. This work was supported by the Collaborative Research Grants Scheme of the Australian Research Council.

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