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Models for the Charging of Particles in Liquid Toners: A Summary

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Liquid toners have been used in the development of electrostatic images in digital printing since the mid-fifties. In this article, several models are discussed that were introduced to explain the processes responsible for charging toner particles. The acid-base models had to be extended by equilibrium models describing dynamic dissociation, adsorption and de-sorption. The important role of water traces in non-aqueous media for the dissociation of molecules and stabilization of ions was investigated. Later, electrical conditions were considered to explain specific high field effects by additional charge generation and relaxation processes. The focus of this article is on the models developed for the charging mechanisms. The enormous progress in measurement techniques that was necessary for improving and, in some cases, correcting the models is not described in detail here nor are models concerning practical issues in printing.

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Introduction

For the development of digital printing technologies, liquid toner is of high interest. One of the main reasons is the small particle size, in the range below 1 μm, which allows higher resolution compared to common dry toner. During nearly 50 years of research in this field, several attempts have been published to describe the mechanisms resulting in a charged toner particle.

Metcalfe[1] and Mayer[2] independently published that electrostatic images can be developed using a special ink called liquid toner. In the presence of an electrostatic field, charged toner particles dispersed in a dielectric liquid carrier, show electrophoretic mobility. They neutralize opposite charges of the image on a dielectric paper or an image carrier and so this latent image is developed.

Dispersions of toner can be characterized by the size and form of the particles and colloidal stability. However, achievable image quality depends basically on the charge of the particles and their consequent mobility. Thus for the improvement in the quality and the speed of toner based digital printing processes it is necessary to understand the processes of charging the particles and the parameters by which the resulting charge is influenced.

The toner particles typically consist of pigments to enable the human eye to recognize the image, and polymers, supporting the stabilization of the toner dispersion and responsible for fixing the developed image. The polymers often surround the pigment completely, but it is known that even in this case the history of the pigment and small traces of impurity can cause completely different charging behavior in the particle. The dynamic processes of flocculation are controlled by the size and electrostatic charge of the particles. To define and enhance the charge of the toner particle, charging agents are used. In this article, as in the literature, a distinction is made between the charge control agents, which are not soluble in the liquid carrier and/or are bound to the toner particle, and the charge directors, typically ionic surface active molecules dissolved in the carrier fluid. These play different roles in the charging mechanisms. The fact that surfactants may form micelles is important for understanding the sources of electric charge in non-aqueous media.

Several fluids have been tried as a carrier for the toner particles. Most common and in everyday use are mixtures of aliphatic hydrocarbons, as isoparaffins with trade names like Isopar™, Shellsol™ and others. Studies have been published with the results of experiments using perfluorinated hydrocarbons[3] or even cosmetic baby oil. This matter will not be discussed in detail here, but only will be referred to insofar as it is of importance to the charging mechanisms.

Principal Ideas

The main theoretical fundamentals concerning charged particles in fluids were known very early on.[4] These includes the existence of electrochemical double layers at the interface between two phases (Fig. 1), and the displacement of these layers under external forces as the reason for electrokinetic phenomenas. In the case of the

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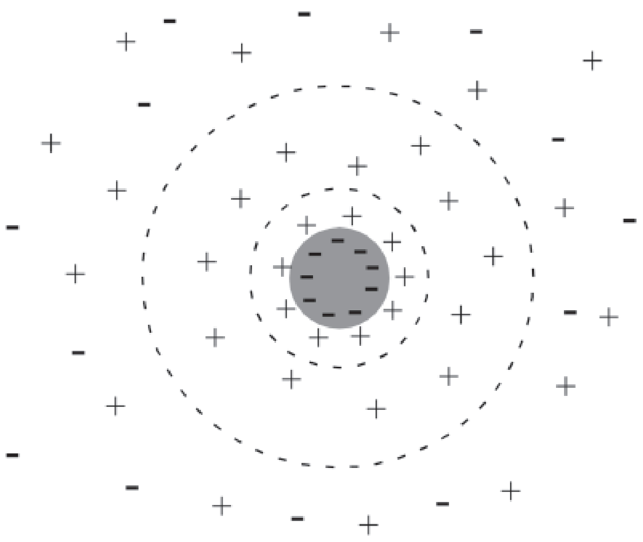

Figure 1. Model of a simple toner particle with the rigid and diffuse double layers.

movement of solid particles in a fluid due to an external electric field, this is called electrophoresis and is the process mainly used in the Liquid Immersion Development technologies in digital printing. Following Claus and Mayer,[4] the electrochemical double layer can occur as the effect of contact potential, adsorption potential, or lyoelectric potential. The contact potential is the result of the different work forces of the two interfacing materials, leading to an electron exchange through the interface. The selective adsorption of particles with a certain electrical sign results in adsorption potential, and the specific orientation of neutral polar molecules is the reason for the lyoelectric potential.

So already in 1965 it was assumed that interface related processes, selective adsorption and/or orientation of neutral molecules, may lead to a potential difference and hence to charged particles in dispersions. Furthermore, the definition of zeta-potential, ζ, was known as the potential difference at the shear layer between the fluid and a moving particle. Attempts to measure the zeta potential, and the principal relations between ζ and the electrophoretic mobility, have been described in classical studies, e.g., by Helmholtz and Smoluchowski.

Principal reasons for spontaneous charge separation between phases were in contrast described by Hunter in 1981,[5] characterized by differences in electron affinity, differences in cation/anion affinity, ionization of surface groups, and physical trapping of fixed charges in one phase. Hunter explains that the second and third mechanisms are dominant with respect to the charging of solid toner particles in dielectric solvents.

Acid-Base Theory

Based on much experimental data a theory was developed that toner particle charge is the result of the reaction between acid and base. Lyklema[6] analyzed the sign of the particle charge in toner/fluid systems when both had proton donor and acceptor properties (Brønsted acid and base, respectively). To explain the sign of the charge he proposed a relationship containing basically two parts:

$$PH + SH \rightleftarrows PH_2^+ + S^- \qquad (1)$$

$$PH + SH \rightleftarrows PH^- + SH_2^+ \qquad (2)$$

with *PH* the surface group of the particle and *SH* the solvent molecule.

These relationships describe how a basic particle in an acidic solvent will accept a proton resulting in a positive charge [Eq. (1)], while an acidic particle in a basic solvent will donate a proton resulting in a negative charge [Eq. (2)].

In 1984, Kitahara discusses the acid-base theory for aprotic systems,[7] and refers to the experimental results of Tamaribuchi and Smith.[8] Following this model, water molecules, present even in non-aqueous media in trace amounts, will dissociate and protons will be bound to the more basic phase in the system. This leads to equations like:

$$P + S + H^+ + OH^- \rightleftarrows PH^+ + S + OH^- \qquad (3)$$

if the particle surface *P* is more basic than the solvent molecules *S*, and for the opposite case

$$P + S + H^+ + OH^- \rightleftarrows POH^- + S + H^+ . \qquad (4)$$

The main commonality between Eq. (3) and Eq. (4) is the virtually unchanged solvent molecule, which however plays an important role in the process, since its character determines the “direction” of the process.

For several particle/solvent systems, the utility of the acid-base-model was proved by Fowkes et al.,[9] mainly based on experiments with carbon-black dispersions. The dynamic processes of formation and re-formation of micelles of the dispersants were found to be necessary for the exchange of charge within the cores of these micelles.

Fowkes’ model implies three steps for the charge generation as a dynamic adsorption/desorption process:

1. Adsorption of the dispersant or charge director molecules at surface sites of the toner particle owing to acid-base interaction
2. Proton or electron transfer between adsorbed molecules and particle surface
3. Desorption of charged species (counter ions)

Using *M* for the dispersant and charge director micelles, this process can be written as

$$PH + M \rightleftarrows P^- + MH^+ \qquad (5)$$

$$P + MH \rightleftarrows PH^+ + M^- \qquad (6)$$

with Eq. (5) for an acidic particle, resulting in a negatively charged particle and Eq. (6) for a basic particle, resulting in a positively charged particle, respectively. In both cases, the reason for charging is the transfer of a proton or electron at the particle surface.

The importance of the third step, the desorption, became clear after further experiments,[10] including microcalorimetric characterization of the interaction between toner particle and charge control molecules, determination of adsorption isotherms and measurement of zeta-potential. Small values for the zeta-potential, found in systems with strong acid-base interaction between particle and charging agents, were explained by hindered desorption due to the stronger interaction between the particle surface and the (ionized) micelles.

It was proven that acid-base theory may provide qualitative information about particle charge; however it is not sufficient to explain all the charge; generation processes completely. In reality, these processes are even more complex and other steps and parameters have to be taken into account. One of the phenomena, which cannot be explained by acid-base theory alone, is the higher effectiviness of aminoalcohols as charge control agents compared to unsubstituted amines, though both have similar basicity. Other, very effective charging agents, such as metal salts of beta-diketones, also do not follow this theory.[11] Especially during the last decade several new models were published, which extend the model of acid-base interaction through quantitative description of equilibria in the various stages of charging a particle.

The Concept of Simultaneous Equilibria

Already in 1977, an equilibrium theory was proposed for non-aqueous dispersions containing surfactants.[12] It was assumed by Kitahara et al. that ionic surfactants added to the dispersion will dissociate partially and resulting ions will be stabilized in micelles due to solubilization. The preferential adsorption of ions with a specific sign, e.g., positive, at the particle surface is described by an adsorption equilibrium

$$S + P^{+} \rightleftarrows (SP)^{+}. \qquad (7)$$

Here, S are the adsorption sites at the surface, P^{+} positive ions in the solvent, and $(SP)^{+}$ the adsorbed positive ions. Ions of opposite sign, in this example negative, can interact electrostatically with adsorbed (positive) ions. Due to the relative large radii of solubilized ions, this interaction is weak. The equation

$$(SP)^{+} + N^{-} \rightleftarrows (SPN)^{0} \qquad (8)$$

describes this second, simultaneously existing equilibrium, with the negative ions N^{-} and neutral ion pairs at the particle surface $(SPN)^{0}$.

Using this equilibrium theory, the relationship between zeta-potential and surfactant concentration was described, especially the phenomenon of a maximum of the zeta-potential in several dispersion systems. The equilibrium constants were calculated as well as the initial concentration of adsorption sites at particle surface, based on the experimental relationships between surfactant and ion concentration. The influence of small traces of water always contained in non-aqueous suspensions was recognized, but not included in discussions and developed models.

Another equilibirum model was proposed by Larson et al. in 1992,[13] based on the idea that the ionization of micelles to form charged species limits the charge the particles can attain. The influence of charge director concentration and temperature on ionization of micelles and particle charge was investigated.

One of the two simultaneously existing equilibria is the ionization of the charge director. Following this model, charge director micelles (M^{0}) can ionize resulting in positive (M^{+}) and negative micelles (M^{-}). Because of the linear relation between concentration of charge director and conductivity of the solution and due to the fact that only a small fraction of micelles will be ionized, Larson[13] assumes a reversible reaction of second order:

$$M^{0} + M^{0} \rightleftarrows M^{+} + M^{-} \qquad (9)$$

with the equilibirium constant for the ionization of micelles K_i and generally $[x]$ representing the number of corresponding species x per dispersion volume

$$K_i = \frac{\left[M^{+}\right]\left[M^{-}\right]}{\left[M^{0}\right]\left[M^{0}\right]}. \qquad (10)$$

This model is useful for explaining the increase in conductivity of certain charge director solutions with increasing temperature, which can not be accounted for by viscosity changes in the solvent alone.[14]

Toner particle charging is proposed to be a reversible second order process, as well. A charging site on the particle S^{0} and a neutral micelle produce a charged particle, e.g., a negative one S^{-}:

$$S^{0} + M^{0} \rightleftarrows M^{+} + S^{-} \quad \text{with } K_q = \frac{\left[S^{-}\right]\left[M^{+}\right]}{\left[S^{0}\right]\left[M^{0}\right]} \qquad (11)$$

In contrast to other published works, here the micelle is neutral for charging the particle, meaning that dissociation and hence ionization of micelles takes place but limits the charging instead of being the precondition, due to the two connected equilibria proposed in the system.

Without any particle there is $[M^{+}] = [M^{-}]$, while in the case discussed for negative particles $[M^{+}] > [M^{-}]$. Increasing M^{0} will lead to more charged sites on the particles, but there will be a limit. For large $[M^{0}]$ it is assumed that $[M^{+}] \cong [M^{-}]$, hence the fraction of charged particle sites can be calculated as

$$\frac{\left[S^{-}\right]}{\left[S^{0}\right]} = \frac{K_q}{\sqrt{K_i}}. \qquad (12)$$

In addition to the equilibrium constants and to allow a quantitative interpretation of this equation, the thermodynamic functions ΔG^{0}, ΔH^{0}, and ΔS^{0}, which are the changes in the standard state Gibbs free energy, enthalpy, and entropy, respectively, were determined. Thus it was possible to explain the low values of micellar charge when using neutral barium petronate as a charge director, owing to substantial decreases in the entropy of the micelle when it picks up an ion.

The effect of traces of water on the zeta-potential of dispersed particles was investigated e.g., by Jenkins et al.,[15] based on Kitahara's model modified for Cu-phthalocyanine and the charge director $ZrO(Oct)_2$. Zirconiyem octanoate dissociates partially and the positive ions are preferentially adsorbed. Again using S for the binding site of the particle surface, this can be expressed as

$$\mathrm{ZrO(Oct)_2} \rightleftarrows \mathrm{ZrO^{2+}} + 2\mathrm{Oct^{-}} \qquad (13)$$

$$S + \mathrm{ZrO^{2+}} \rightleftarrows S\mathrm{ZrO^{2+}}. \qquad (14)$$

If the charge director concentration increases, negatively charged octanoate ions will be attracted electrostatically by the positive ions, resulting in charge neutralization:

$$S\mathrm{ZrO^{2+}} + 2\mathrm{Oct^{-}} \rightleftarrows S\mathrm{ZrO(Oct)_2}. \qquad (15)$$

The degree of dissociation in Eq. (13) depends on the water content of the solvent and affects the equilibria in Eq. (14) and Eq. (15). Water is not involved in the particle charge directly, but it generally plays a profound role concerning the electrochemical properties of particles in nonaqueous media[16]: On the one hand, water molecules are adsorbed onto the surface of hydrophilic particles, thus changing the acid-base character of the surface and hence the related interaction mechanisms. On the other hand water supports the formation of inverse micelles of the charge director and its later dissociation, so that charged ions can ultimately reach the particle surface.

The idea of particle charging by adsorption of ions is demonstrated by analyzis of adsorption isotherms, showing saturation of adsorption at concentrations similar to those where a maximum in the measured mobility, zeta-potential, and surface charge density occurred.

This principle was extended.[17] Considering a 2:1 surfactant denoting as AB_2, and, as above, a surface site, S, the charging and neutralization processes can be described by:

$$AB_2 \rightleftarrows AB^+ + B^- \tag{16}$$

$$S + AB^+ \rightleftarrows SAB^+ \tag{17}$$

$$SAB^+ + B^- \rightleftarrows SAB_2 \tag{18}$$

Despite the fact that the equilibrium constants for these equations could not be calculated directly from the measured mobilities, the model described fit experimental results rather well, even without invoking the presence of charged micelles. Important are the dependence of the electrophoretic mobility on the concentration of surfactant, and the influence of the kind and amount of polymers used as a particle stabilizing agents.

Effects of External Electric Fields

With all the models briefly described above it is rather difficult to explain why the electrophoretic mobility, and hence the charge of particles, depends on the external electric field. This phenomenon was first discussed by Stotz[18] for polystyrene spheres dispersed in an aliphatic hydrocarbon. He explained the increase in the particle charge and mobility in ranges of electric field between 5×10^2 and 5×10^3 V/cm by the partial "stripping off" of counter-ions from the electrochemical double layer. Due to the complete stripping off of the double layer, the mobility of the charged particle becomes field independent for high field conditions. The range of field dependence is determined by the particle radius and the conductivity of the solvent.

Caruthers et al.[19] extended Larson's model, described in the previous section, in order to find a minimum set of reactions which adequately explain the field dependence as well as other experimental results, especially the relations between the charge director concentration and zeta-potential,[20,21] Q/M data,[22] and mobility,[23] respectively, as reported previously. The idea of the self-ionization of micelles was retained, but Eq. (11) was separated into two parts: The neutral micelles can form complexes with chargeable sites on a toner particle surface, which can afterwards dissociate resulting in positive micelles and negatively charged sites on the toner particle surface. This leads to the following equations:

$$S^0 + M^0 \rightleftarrows S^-M^+ \quad \text{with } K_f = \frac{\left[S^-M^+\right]}{\left[S^0\right]\left[M^0\right]} \tag{19}$$

for the formation of complexes and

$$S^-M^+ \rightleftarrows S^- + M^+ \quad \text{with } K_d = \frac{\left[S^-\right]\left[M^+\right]}{\left[S^-M^+\right]} \tag{20}$$

for the dissociation of complexes with significance of the symbols similar to the usage in Eqs. (9) to (11).

Based on the equilibrium constants K_f and K_d the model enables quantitative calculation of the charge and mobility of the toner particles. The variation of particle mobility with the concentration of charge director is determined by the stability of the intermediate complex S^-M^+. If these complexes are stable, chargeable sites on the particle surface become more and more neutralized. Because of the ionization equilibrium of the charge director, the dissociation of the complexes is reduced at high charge director concentrations, leading to lower toner charge, zeta-potential, and mobility. But unstable complexes or easy dissociation cause an increase of these parameters with increasing charge director concentration.

Stable intermediates could consist of inverse micelles chemisorbed onto toner particles, or of a double layer of charged micelles electrically held to the particles. The latter explanation is considered to be more plausible. The stability depends on the charge director and the chargeable sites of the particle, determined by the pigment used and the charge control agents in connection with the polymer.

The field dependence of the mobility of charged toner particles is explained by the field dependence of the dissociation of the intermediate complexes. A higher electric field would support the strip-off of micelles leaving charged sites on the particle.

Evidence for the field induced effects of particle charging was also published by Chen et al.[24] introducing a serial capacitor configuration for analyzing toner behavior in the electric field. This method allows the direct determination of electrophoretic mobility and charge density without conductivity measurement. The calculated values for conductivity are about two orders of magnitude higher compared to values from other measurements., which is explained by additional charge generation at high field.

A simple model describing the field dependence was presented by Jin[25] based on investigations of the mobility of mono-dispersed CN-terminated silica particles in di-2-ethylhexylsulfosuccinate (AOT)/Isopar solutions using ELS (Electrophoretic Light Scattering). Quite analogous to Stotz's descriptions, the model assumes a field induced strip-off of counter ions out of the electrochemical double layer. Weak fields do not disturb the space charge surrounding the particles, and hence in this range mobility is constant. If the external field exceeds the field inside the double layer, approximately ζK, strip-off starts and the effective particle charge increases. This leads to increased mobility, until all counter ions are removed. In the high field region, mobility is constant again, but on a higher level than at the lower electric field.

The counter-ions are proposed to be located in an area with a distance d to the (effective) particle surface and are held there weakly. For a negative particle charge, this interaction can be written as

$$SM^- + M^+ \rightleftarrows SM^-M^+ \tag{21}$$

with SM^- a site on the surface of the particle with negative charge, M^+ a positive counter ion and SM^-M^+ a weak bound pair of surface site and counter ion.

These ideas were continued by Keir et al.[26] who investigated field dependence of electrophoretic mobility in copper phthalocyanine dispersions in Isopar containing zircon octanoate as a charge director. As already described in the previous chapter, a 2:1 surfactant is assumed and the charging and neutralization processes can be written as in Eq. (16) to Eq. (18). The weak binding of neutralizing counter ions is introduced as the equilibrium,

$$AB^+ + B^+ \rightleftarrows SAB^+ ......B^- \tag{22}$$

which can be disrupted by an external field, and a quantitative expression is given for the transition from low to high field mobility. The resulting formula combines the equilibrium constant for Eq. (22), Boltzmann's equation describing the distribution of ions in the solvent due to thermal movement and the electric forces between the charged particle and ions, and a term describing the effect of the external field. The expression was used for fitting the experimental data and was found to work quite well, again without invoking micelles and considering ions only.

Conclusions

Several models have been developed by researchers describing the origin of charge on solid particles in non-aqueous media. These models can be used to predict the behavior of liquid toners with respect to Q/M data and mobility. The acid-base theory is widely accepted and has been extended by equilibrium models. They explain the dissociation and the interactions between surfactants used as charge directors, polymers, and pigments, such as adsorption, charge transfer and desorption. The preferential adsorption of ions or micelles due to the properties of pigment or polymer is demonstrated by several experiments. Using these models it is now even possible to explain the field dependence of mobility.

Further work is necessary to correlate electrochemical data like zeta-potential and mobility to practical issues in printing, including developed mass per area and quality aspects. A few studies have been published in this field, however there are so many possible combinations of toner systems and image carriers that a systematic approach is still missed. Finally, new toners adapted to the specifications of the system should be investigated, based on the deeper understanding of toner charging mechanisms, as well as the relationships between process parameters, toner behavior, and quality issues.

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