



Aggregation of inkjet ink components by Ca and Mg ions in relation to colorant pigment distribution in paper



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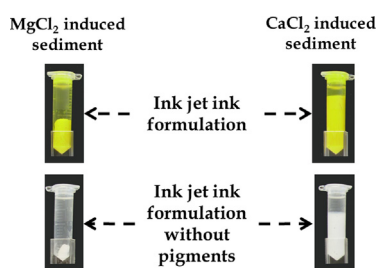
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HIGHLIGHTS

- We study the effect of divalent cations on ink aggregation.
- Dry colorant pigment distribution on papers treated with salt containing Ca²⁺ and Mg²⁺ is similar and close to the surface.
- The aggregation behavior is controlled by the dispersing polymers.
- We link this to specific ion effects.

GRAPHICAL ABSTRACT



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ABSTRACT

Papers coated with salts containing divalent cations exhibit superior inkjet print quality, which has been suggested to be due to fast aggregation of the colorant pigments close to, or even on, the surface of the paper. In this work we show the pivotal role of the carboxylic acid containing dispersing polymer. We report a series of aggregation and sedimentation experiments with commercial inks, generic ink formulations and specific ingredients comprising these formulations, and find differences in their response to the presence of MgCl₂ or CaCl₂. In particular, flocs and sediments formed in the presence of MgCl₂ are denser than those formed in the presence of CaCl₂. These differences are not predicted by the Derjaguin-Landau-Verwey-Overbeek (DLVO) theory. We suggest that ion specific interactions occurring between Mg²⁺ or Ca²⁺, and charged carboxylate groups residing on the dispersing polymers, provoke the observed behavior.

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1. Introduction

Inkjet printing is a digital non-impact printing technology based on the ability to impinge ink drops in the volume range of about

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1–100 pL on desired substrate locations [1]. Today it is used in a large range of applications, but with an origin in industrial graphic inkjet printers in the 1970s and graphic desktop inkjet printers during the 1980s. High speed inkjet presses are considered as a recent technological development, aimed at fulfilling the increasing demand for small printing batches that are too expensive to be produced by any of the traditional techniques, such as flexography, rotogravure and offset lithography [2].

Inkjet print quality is greatly affected by the series of events that an inkjet drop is subjected to once it lands on a paper [3]. The fastest

event is associated with the dissipation of the energy resulting from the impact between the drop and the paper: the inertial spreading, penetration and drop oscillation, which decay in less than 200 μ s from the moment of impact. Imbibition and spreading related to the wetting extent of the ink on the paper substrate may occur right from the moment of impact, but will extend to longer time periods in the order of milliseconds [4,5] until the entire drop is absorbed by the paper. The liquid will continue to spread inside the porous paper structure even after the drop is absorbed, extending the time scale associated with this phenomenon to even longer periods [6]. Evaporation also plays an important role in the drying process [5], and swelling of the paper substrate can become significant as well [7].

Some examples of how liquid spreading and imbibition affect print quality are given in the following. Color to color bleed occurs when drops of different colors coalesce on the surface of the paper due to insufficient drying rate. The situation can be prevented by promoting fast imbibition and/or evaporation. At the same time, fast imbibition and spreading may reduce color intensities (gamut area and volume) since colorants will follow the liquid vehicle as it spreads inside the paper, thereby reducing gamut values. This effect is further enhanced when the ink formulation consists of low amounts of colorants, as in the case of waterborne inkjet inks. Thus, an ink–paper system that is tuned to promote drop imbibition in order to prevent color to color bleed may reduce color gamut. This difficulty can be solved by coating the paper with a layer of high porosity and specific surface area that can quickly absorb the drops [8]. Spreading of the colorant can be prevented by adjusting the surface charge of the coating layer to retain oppositely charged colorants, separating them from the advancing liquid vehicle. Surface charge adjustment can be done by tuning the pH of the stock solution used for coating the paper or applying colorant fixative agents such as polyelectrolytes [3].

The focus of this work is on a different type of colorant fixative known to affect only waterborne pigment inks, namely salts with divalent cations. Once an aqueous inkjet drop lands on the surface of a paper that is coated or treated with these salts, the ions would quickly diffuse into the liquid drop and destabilize the ink dispersion [9]. This promotes pigment separation from the liquid vehicle and deposition on the paper surface, and therefore enhancement of optical densities and gamut values. We start by illustrating how treatment of a paper with salts containing divalent cations, $MgCl_2$ and $CaCl_2$, influences the distribution of inkjet printed dry colorant pigments. We then determine the key ingredient in the ink formulation that controls the aggregation and sedimentation of the ink. We use a set of pigment-based inks from the two main types of printers, namely an ink from a home and office desktop printer using the drop-on-demand (DOD) technique, and generic ink formulations suited for high-speed continuous inkjet (CIJ) applications. By a series of experiments we are able to elucidate the crucial role of the dispersing polymer in the aggregation process, as well as the nature of the interactions controlling the aggregation and sedimentation of the ink, which is investigated by comparing the response of the inks and the polymer dispersants to the presence of either Mg^{2+} or Ca^{2+} .

2. Materials and methods

2.1. Ink formulations

Information on the inks and solutions used in this work is provided in Table 1. Commercially available water based pigment yellow ink (HP 38 Yellow Pigment Ink Cartridge C9417A, Hewlett-Packard Development Company, L.P., USA), hereafter referred to as DOD-Y, denoting drop-on-demand yellow ink, was withdrawn from an ink cartridge using a needle and a syringe. A pigment free formulation, DOD-Y0, was obtained by centrifugation at 50 000 rpm for 24 h using an Optima Beckman Ultracentrifuge (Beckman Coulter AB, USA), after which the supernatant, having only residual traces of yellow color, was carefully separated from the yellow sediment. The total solids and polymer contents of these inks were measured by weighing the samples before and after drying for several hours at 120 °C. We note that in a separate experiment we did not observe any degradation of the polymers at this temperature, while essentially all water and any co-solvent were evaporated. The pigment content was calculated as the difference in solid content between the DOD-Y ink and the DOD-Y0 ink solution.

Two water based inks were prepared according to generic formulation and supplied by Eastman Kodak Company: CIJ-Y, denoting continuous inkjet yellow ink that contains yellow pigments, and CIJ-Y0 that is free from pigments but otherwise nearly identical to the CIJ-Y formulation. The pigments in the CIJ-Y ink have a volume-weighted median particle diameter of 10–14 nm, measured by a Microtrac Ultrafine Particle Analyzer instrument. The composition of the generic ink formulations, shown in Table 1, is similar, but not identical, to commercial formulations used in high-speed inkjet printing presses. The generic inks were used for simulating the response of real inks when exposed to different salt solutions. The particle dispersion in the CIJ-Y formulation is stabilized by two polymeric dispersing agents. The first, referred to as P1, is a proprietary polymer provided by Eastman Kodak Company that belongs, although not identical, to a class of polymers described in the patent literature [10]. The molecular weight range of P1 is 7000–10 000 Da, and it contains carboxylate acid groups as well as hydrophobic benzyl groups. The acid number of P1 is in the range of 200–230, meaning that 25–40 carboxylic acid groups are present on each polymer molecule. The second polymer stabilizing the pigments, referred to as P2, is a commercial styrene acrylic resin (BASF chemical company, www.basf.com) named JonCryl HPD 696, with a molecular weight of 16 000 Da and acid number of 220, giving an average of about 60 carboxylic acid groups on each polymer. The carboxylic acid groups of both polymers are neutralized with potassium hydroxide. Both polymers were provided as an aqueous solution and used without further purification.

2.2. Sedimentation

Salt stock solutions for the sedimentation experiments were prepared from $CaCl_2$ dihydrate powder (ACS reagent, Merck, Germany) and aqueous 1 M $MgCl_2$ solutions (Bioultra, Fluka,

Table 1
pH, total solids, polymer, pigment and humectant contents in the ink solutions and polymer solutions.

Sample	pH	Total solids [w/w%]	P1 [w/w%]	P2 [w/w%]	Pigment [w/w%]	Humectant [w/w%]
DOD-Y	9.0	10	–	–	~3	–
DOD-Y0	9.0	7	–	–	~0	–
CIJ-Y ^a	8.2	11.3	0.9	0.65	3.0	6
CIJ-Y0 ^a	7.9	8.3	0.9	0.65	0	6
P1 solution	8.8	0.9	0.9	0	0	0
P2 solution	8.2	0.65	0	0.65	0	0
P1/P2 solution	8.7	1.55	0.9	0.65	0	0

^a The remaining components in the ink formulations (0.75 w/w%) are pH and conductivity regulators, surfactants, biocides and anticorrosive additives.

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