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Role of Paper Coating Pigments and Additives in Darkfastness of Ink Jet Prints

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Abstract. *The article discusses changes that occur when paper samples printed with an office ink jet printer HP 5550 are exposed to elevated temperature and relative humidity without the presence of light for a prolonged time period according to accelerated artificial aging standard EN ISO 5630-3. The effects of paper coating pigment type (pyrogenic silica, precipitated and ground calcium carbonate) and the amounts of binder and dye fixative on print quality and darkfastness are examined. Results show that both color chroma and print density of prints made with the dye-based inks—cyan, magenta and yellow—on calcium carbonate coatings are higher compared to coatings where silica is used. The opposite is true for the black pigment-based ink where silica clearly outperforms calcium carbonate coating pigments. Accelerated artificial aging deteriorates color vividness much more severely with ground* $CaCO_3$ *than with precipitated* $CaCO_3$ *coatings, while prints on silica coatings exhibit the highest darkfastness. © 2008 Society for Imaging Science and Technology.*
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INTRODUCTION

Ink jet (IJ) printing has witnessed an enormous expansion in the last few decades. In 2003, for the first time digital cameras outsold traditional analog cameras.[1] IJ printing has been not only gradually replacing analog silver-halide photography but also competes more and more successfully with traditional printing techniques, such as offset or gravure. This article is concerned with drop-on-demand IJ printing technology, in particular with the small-office-home-office (SOHO) market segment.

On one hand, the quality of IJ prints is influenced by the characteristics of the inks and on the other hand the medium, e.g., paper, on which images and/or text are printed.[2,3] Printing inks typically consist of water-soluble dyes, predominantly anionic or carboxylated, sulphonated species with conjugated aromatic ring systems,[4] and minor amounts of other ingredients such as binders, surfactants, buffers and cosolvents.[5] Some SOHO printers, e.g., those of Hewlett–Packard, apply black pigment-based inks that differ from the dye-based ones in that they generally have better water- and light-fastness properties but lower color gamut.[6,7]

Paper and its ink-receptive coating are designed to hold the ink dye or pigment at the surface while at the same time rapidly removing ink carrier, which is normally water. Swellable, or nonporous coating[8] consists of a synthetic or natural polymer, which swells when in contact with water or ink. On the other hand, porous coating contains microscopic pigment particles traditionally based on silica or, more recently, precipitated calcium carbonate (PCC)[9] and ground calcium carbonate (GCC), which provide rapid ink carrier absorption and therefore fast drying. Regarding the PCC pigment-ink fixation mechanism, Donigian et al.[10] proposed that PCC holds the dye and allows the fluid phase to pass through, compared to silica coating which holds the fluid phase including the dye of IJ ink. In addition to pigments, such porous and inherently weak coatings need to include a high-strength synthetic binder such as polyvinyl alcohol (PVOH)[11] and often a cationic dye fixative to improve both the attachment of anionic ink colorant and waterfastness of the print. Other ingredients may be added to provide high quality of IJ prints: high optical density, color intensity, dot regularity, low print-through, wicking, and bleeding.

Stability of IJ prints

The importance of the stability of IJ prints, e.g., those with archival role, large-format posters or digital photographs, on a natural polymer substrate, such as paper has become a major focus in recent years. In regard to the substrate paper itself, two related but noninterchangable terms have been identified and studied in detail: permanence and durability. The permanence of paper[12] denotes its ability to remain chemically and physically stable over long periods of time and depends on the chemical resistance of its components to

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Table I. Paper coating formulations

Sample	Pigment (parts)			Binder (pph)	Dye fixative (pph)
	GCC	PCC	Silica	PVOH	polyDADMAC
1	100	0	0	7	0
2	0	100	0	7	0
3	100	0	0	10	0
4	0	100	0	10	0
5	100	0	0	13	0
6	0	100	0	13	0
7	100	0	0	7	3
8	0	100	0	7	3
9	100	0	0	10	3
10	0	100	0	10	3
11	100	0	0	13	3
12	0	100	0	13	3
13	0	0	100	20	0
14	0	0	100	30	0
15	0	0	100	40	0
16	0	0	100	20	3
17	0	0	100	30	3
18	0	0	100	40	3

pph=parts per 100 parts of pigment

the influence of external factors. Paper durability depends mainly on physical and mechanical characteristics of the principal raw materials: fibers, fillers, sizing agents, and additives and on contamination of these by ions by the environment, the action of light, heat, humidity and microorganisms. Decrease in both phenomena are a consequence of paper aging, resulting particularly in deteriorated mechanical strength, chemical stability, and optical characteristics of paper.[13,14] For evaluation of durability according to ISO 9706, accelerated artificial aging procedure according to the EN ISO 5630-3 standard—elevated temperature (80 °C) and relative humidity (65%) but without the presence of light for 24 days is recommended.

Regarding the ink-receptive layer, several researchers[15,16] found swellable coatings to be less susceptible to air fading compared to the microporous coatings, since the dye colorant is, after drying, protected from detrimental atmospheric factors. As Bugner et al.[17] pointed out in their extensive review on the history of "gas fading," ozone is the air component that is largely responsible for the fading effect. When speaking of IJ prints, one has to distinguish between the lightfastness,[18] which denotes stability against visible and UV light, and the darkfastness, which relates to stability of the print to heat, humidity, ozone, and other environmental factors (see, for example, Ref. 19) or, in case of digital photographs, contact with album materials.

One of the most frequently cited methods for measuring lightfastness of photographic prints has been ANSI IT9.9[20] (recently replaced by ISO 18909) specifying the procedures and equipment needed to characterize the density loss from a test target that has been treated under one of several recommended exposure conditions (illumination, temperature, and humidity). In our study darkfastness of prints was examined using the previously mentioned accelerated aging standard for paper and board ISO 5630-3.[21]

The goal of our research was to examine individual contributions of paper coating components—pigment type, amounts of binder, and dye fixative—and HP DeskJet 5550 printer's cyan, magenta, yellow, and black (CMYK) water-based IJ printing inks to print quality and darkfastness, i.e., stability without a presence of light. Although ozone and other pollutant gases can have an important impact on darkfastness, the scope of this study was limited to a single accelerated aging condition—80 °C and 65% relative humidity—as described next. In particular, we wanted to investigate print performance and degree of color deterioration using different coating pigment-printing ink combinations.

EXPERIMENTAL

Materials and Methods

Paper Coating and Printing

A 70 g base paper for IJ printing from a Slovenian paper mill was coated using a laboratory wire-wound rod coater (RK Print-Coat Instruments, UK) with various coating colors (Table I) differing in pigment type, binder amount, and presence or absence of dye fixative. Three specialty pigments designed for high-end IJ printing were tested: GCC (ND 5890, Imerys), PCC (Jetcoat 30, Imerys), and pyrogenic silica (Aerosil MOX 170, Degussa). Binder and dye fixative were PVOH (Mowiol 28-99, Clariant) and polyDADMAC (Cartafix VXT, Clariant), respectively. Micrographs of paper

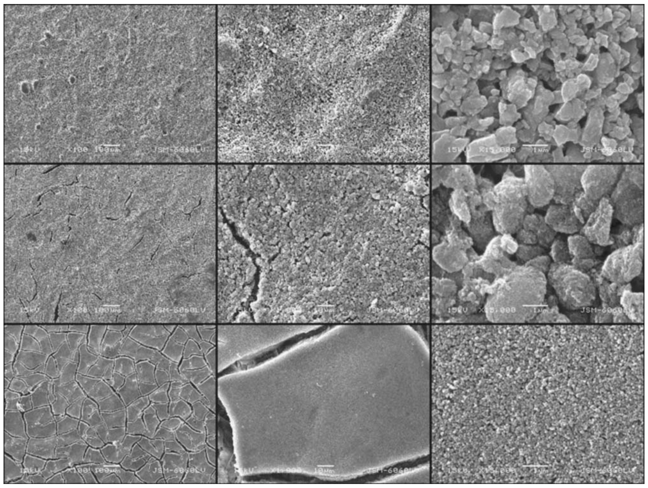

Figure 1. SEM micrographs of unaged coated paper surfaces containing different pigments: GCC (top row), PCC (middle row), silica (bottom row) at 100-X (left column), 1000-X (middle row), and 15000-X (right column) magnification.

surfaces (Figure 1) showing coatings consisting of a single coating pigment and middle level of PVOH amount (i.e., samples No. 3, 4, and 14 in Table I) were recorded with the low-vacuum scanning electron microscope JSM-6060LV by JEOL at different magnifications.

After coating and conditioning for 24 hours at 23 °C and 50% relative humidity according to ISO 187,[22] the samples were printed with office printer HP DJ 5550 using IJ print test chart (Figure 2) and print quality settings set to standard. No attempt was made to check whether the printer actually printed pure, i.e., 100% CMYK patches or the prints contained small amounts of the other primary colors according to the printer driver's internal color management algorithms.

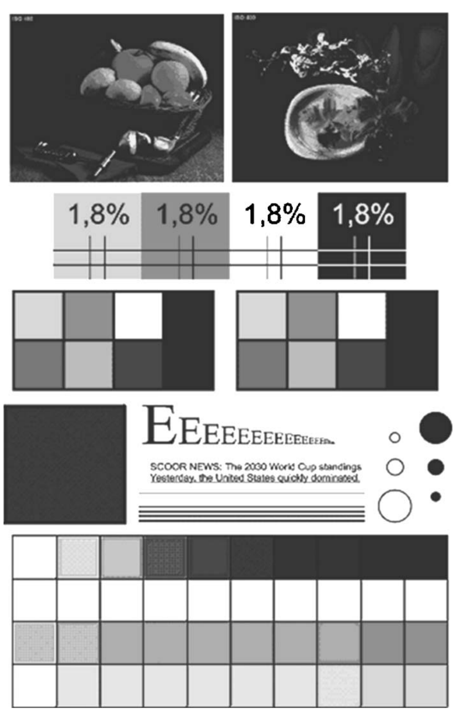

Figure 2. Ink jet test chart used for the analysis of prints.

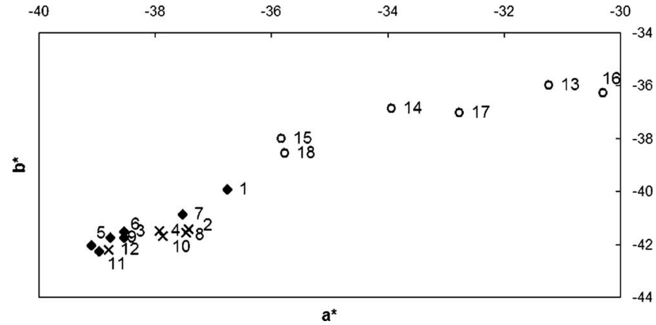

Figure 3. CIE a* and b* color coordinates of unaged cyan prints.

Accelerated Artificial Aging

IJ prints were put into the climate chamber where they were exposed to the accelerated aging conditions specified in ISO 5630-3: 24 days at 80 °C and 65% relative humidity, according to ISO 9706 standard for permanent paper. After the treatment, the same set of measurements was performed as with the unaged samples.

Testing and Analysis

Several methods were applied to determine the quality of both untreated and treated IJ prints: for each of the four colors, CMYK, optical density was measured by densitometer D185 and CIE $L^*a^*b^*$ color coordinates by color spectrophotometer Eye-One, both by Gretag–Macbeth. From color measurements on the aged and unaged prints (differences designated as Δ) total color change (ΔE) values for CMYK print areas were calculated using the following equation:[23] $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{0.5}$. Yellow-to-black ink bleeding and ink wicking indices were determined on scans produced by Microtek flatbed scanner ScanMaker 5900 (resolution 600 dpi) by implementing image analysis routines in a public-domain Java image processing program ImageJ.[24] Differences in measured properties after and before the accelerated aging treatment were then linearly transformed using the principal components analysis (PCA), a statistical multivariate technique used to detect trends in the measured data and visualize relationships among the variables as well as similarities and differences among the tested samples. PCA reduces data volume by projecting into lower dimensions; it identifies directions with greatest data variation and discards directions with least variation.[25]

RESULTS AND DISCUSSION

Colorimetric Properties of Prints

Plots of CIE a^* versus b^* values for CMYK solid print areas at a given lightness on unaged samples are displayed in Figures 3–6 and can be used to estimate each color's saturation (vividness or chroma). Cyan prints (Fig. 3) are evidently more vivid when using calcium carbonate, either PCC or GCC, rather than silica as a paper coating pigment as indicated by a bottom left position of samples 1–12. This can be explained by the fact that although silica pigment produces microcracks as a result of the coating layer shrinkage that are responsible for quick water penetration and fast drying,[26] calcium carbonate pigments form coating layers with high permeability and an even more open pore structure (see Fig. 1 at 15.000 x magnification) that enables rapid dye fixation

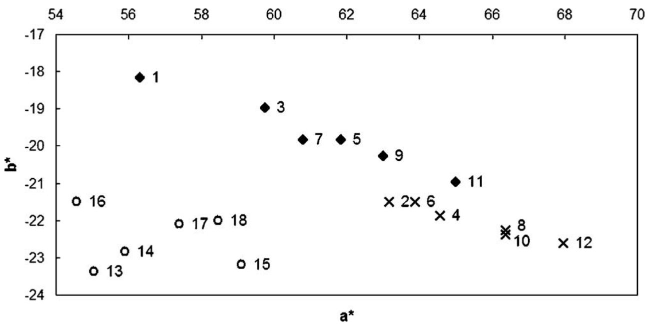

Figure 4. CIE a* and b* color coordinates of unaged magenta prints.

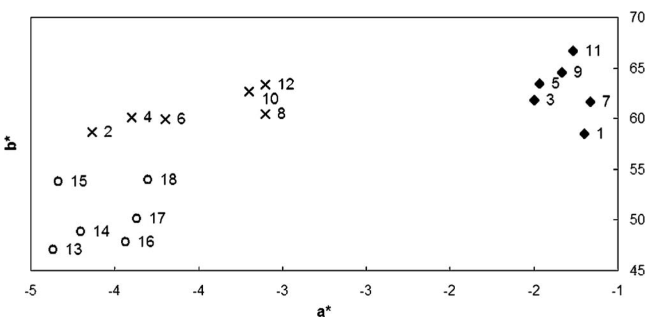

Figure 5. CIE a* and b* color coordinates of unaged yellow prints.

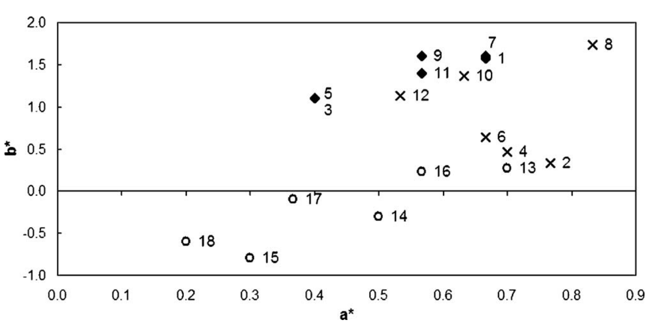

Figure 6. CIE a* and b* color coordinates of unaged black prints.

on the paper coating surface in addition to a quick water penetration into the coating structure. Moreover, several studies, e.g., Ref. 27, have shown that with cationic PCC pigment, binders are not necessarily needed to attach colorants and cationic PCC particles were able to fix the soluble dyes to their surfaces independently from the binder. Higher amounts of a water soluble hydrophilic polymer, PVOH, regardless of the pigment type being used (compare e.g., samples 1, 3, and 5 or 13, 14, and 15) as well as the presence of a cationic polymer, polyDADMAC, in calcium carbonate-based coating formulations (e.g., 1 compared to 7, 2 to 8, etc.) are, as can be expected, beneficial to cyan chroma. Similar trends can be observed when examining the prints made with the other two dye-based inks magenta (Fig. 4) and yellow (Fig. 5). Silica in both cases yields duller, less vivid images when compared to those produced on substrates coated with PCC or GCC. Also, the same positive effects of PVOH and polyDADMAC on the colors' chroma can be found as those described previously for cyan prints.

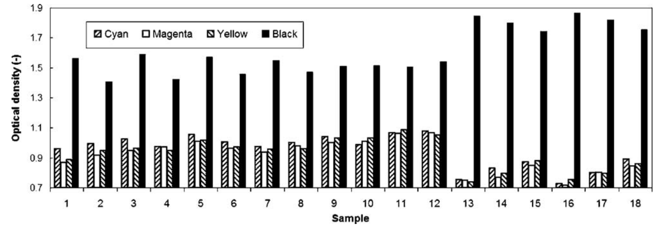

Figure 7. Optical density of unaged prints.

The influence of the type of calcium carbonate type is, however, different for magenta and yellow color: the former favors PCC and the latter GCC. In case of yellow prints, PCC-based coated substrates also produced undesirable green-shaded prints ($-a^*$).

The diagram of color coordinates a^* and b^* for black prints (Fig. 6) shows a different picture. Here paper samples coated with silica exhibit performance that is superior to coatings with calcium carbonate pigments as manifested by a closer proximity of samples 13–18 to the coordinate origin. This finding is in accordance with the results of our previous studies[28] and is due to a different nature of dye-based (cyan, magenta and yellow) versus pigment-based (black) ink chemistry and their interactions with the paper coating pigments. More elaborate discussion on this subject is given elsewhere.[29]

Optical density values of CMYK print areas are displayed in Figure 7. As with color coordinates, cyan, magenta and yellow prints differ in their behavior from that of black ones. In all three dye-based color prints, calcium carbonate outperforms silica while the differences between PCC and GCC are not significant. A higher amount of PVOH, and in case of calcium carbonate-based paper coatings, the presence of polyDADMAC increase optical density and consequently, print quality.

Inspection of the optical density of the black prints reveals that silica yields the best results, followed by GCC and PCC. This finding is in agreement with the previously mentioned effects of different coating pigments on black prints' color coordinates and suggests that silica should be used in IJ paper coatings if one is interested in a high black-and-white print quality. Note that higher PVOH amounts improve the optical density in case of PCC, but deteriorates it where GCC or silica are being used in paper coating formulations. Ink wicking along the fibers and inter-color (yellow to black) bleeding are shown in Figure 8; higher indices correspond to poorer performance. Both negative phenomena of IJ prints are dependent upon the coating pigment type—PCC produces evidently poorer image quality—as well as upon the binder amount and presence of cationic fixative, although here relationships are less evident. Higher PVOH amount in combination with silica pigment (samples 15 and 18) deteriorates print quality while presence of polyDADMAC together with GCC (compare, e.g. 7, 9, and 11 to 1, 3, and 5) increases bleeding but decreases wicking.

Colorimetric Properties of Aged Prints

Effects of accelerated artificial aging on print quality are complex and numerous. Although some of them can be ob-

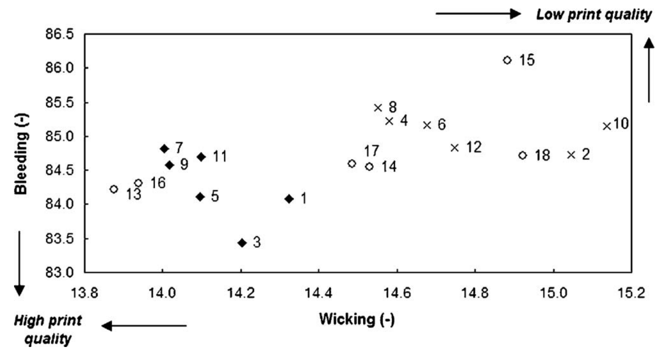

Figure 8. Wicking and bleeding of unaged prints.

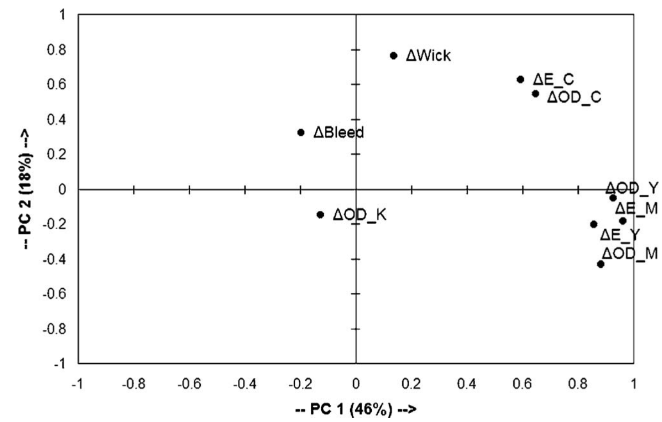

Figure 9. Differences in parameters between the aged and unaged prints located in PC1-PC2 coordinate system.

Table II. Difference between the aged and unaged prints

	Color difference				Optical density differences					
Sample	Δ*E*_Cyan	ΔE_Magenta	ΔE_Yellow	ΔE_Black	ΔOD_Cyan	ΔOD_Magenta	ΔOD_Yellow	ΔOD_Black	ΔWick	ΔBleed
1	9.2	5.9	6.6	0.4	0.16	0.10	0.18	0.01	0.30	−0.65
2	6.9	3.2	1.3	1.2	0.11	0.00	0.04	−0.01	0.77	−0.61
3	9.1	6.6	7.1	0.7	0.15	0.12	0.18	0.00	0.28	−0.93
4	6.3	3.3	1.3	1.2	0.04	0.01	0.05	−0.03	0.03	−0.02
5	8.3	6.4	6.0	1.1	0.14	0.11	0.17	0.00	0.27	−0.33
6	8.2	3.0	1.2	0.5	0.06	0.00	0.07	−0.02	0.18	−0.02
7	8.7	6.7	10.4	0.5	0.16	0.12	0.25	−0.01	0.45	0.25
8	10.0	2.7	1.9	0.9	0.22	−0.06	0.04	0.00	0.51	0.44
9	8.9	7.4	11.1	0.9	0.18	0.14	0.28	0.01	0.44	0.18
10	9.7	2.7	2.6	0.9	0.19	−0.01	0.09	0.00	0.80	0.36
11	9.3	7.4	10.9	1.2	0.21	0.14	0.27	0.00	0.45	−0.06
12	8.0	3.0	2.0	1.6	0.21	0.01	0.05	0.00	0.07	−0.51
13	3.6	2.7	2.8	0.6	0.06	0.04	−0.05	0.05	−0.04	−0.06
14	3.8	2.5	4.9	0.2	0.01	0.01	−0.05	0.00	0.06	−0.46
15	4.1	2.9	4.5	0.3	−0.01	−0.01	−0.05	0.00	0.69	1.20
16	10.9	3.9	4.5	0.4	0.14	−0.01	−0.06	0.04	0.22	0.18
17	9.1	3.5	4.4	0.5	0.13	−0.01	−0.05	0.04	0.41	−0.36
18	8.2	3.6	3.4	0.6	0.12	−0.01	−0.04	0.00	0.67	−0.55

served by studying differences in measured parameters between the treated and the nontreated prints, a greater comprehension of the accompanied changes can be obtained by transforming these differences (Table II) with PCA. With this method it is possible to largely reduce the number of important data dimensions to two or three of the so-called principal components (PCs): PC1, PC2, and PC3. Together these account for nearly 80% of all variability contained in Table II. Note that variable ΔE_Black was not included in PCA, since this parameter contributes little to the overall picture of aging effects. Table II shows that these differences are very small regardless of the paper coating formulation used, which is in accordance with findings from our earlier investigations[30] and literature reports about higher color stability of black pigmented IJ ink.[31] PCA enables visualization of important patterns in the data: dependencies among the parameters, i.e. variables (loading plots: Figures 9 and 10) and (dis)similarities among the IJ prints (score plots: Figures 11 and 12) by plotting corresponding PC1 versus PC2 and PC1 versus PC3 diagrams. Loading plot PC1-PC2 (Fig. 9) shows that, in terms of optical density and total color change (ΔE), accelerated aging shifts magenta, yellow and, to a lesser degree, cyan prints in a similar way, since these parameters are highly correlated and therefore group close to each other. They all have a high orthogonal projection to PC1 and a low one to higher PCs and can be therefore treated together thus representing one "composite" variable as will be discussed later. From PC1-PC2 score plot (Fig. 11) it can be seen that it is the prints on GCC coatings where the biggest differences, i.e., the most severe degradation takes place since the

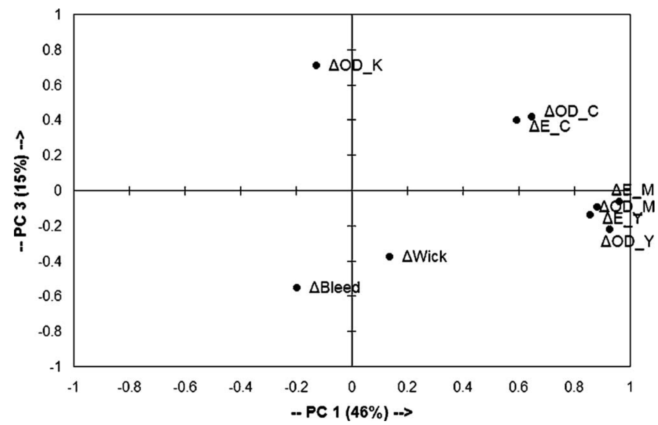

Figure 10. Differences in parameters between the aged and unaged prints located in PC1-PC3 coordinate system.

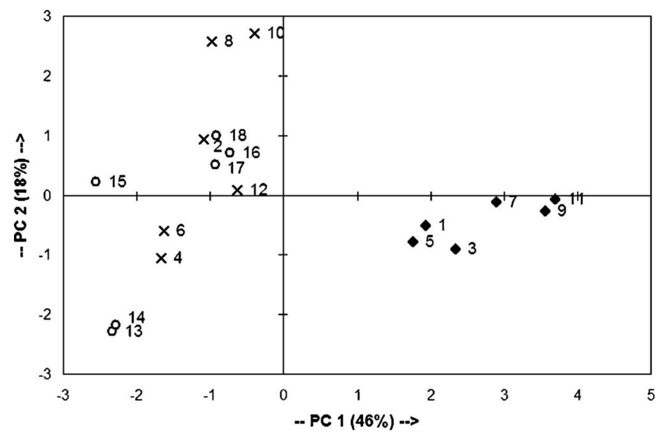

Figure 11. Position of prints in PC1-PC2 coordinate system.

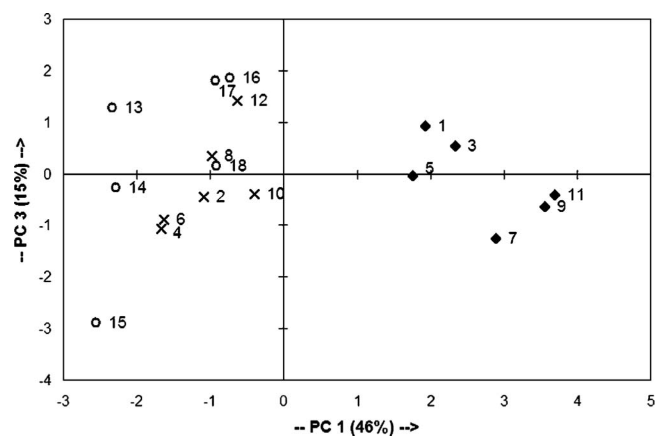

Figure 12. Position of prints in PC1-PC3 coordinate system.

samples 1, 3, 5, 7, 9, and 11 are all located in the far-right areas of both PC1-PC2 plots. While the amount of PVOH does not seem to be important, the presence of cationic dye fixative is evidently detrimental to prints' stability regardless of the coating pigment being used (compare samples 1–7, 6–12, etc.). Shaw–Klein[32] came to similar conclusions when investigating the effect of polyDADMAC on lightfastness.

Aging affects ink wicking in a different manner as indicated by its high PC2 loading (Fig. 9). Prints with the most pronounced wicking differences (Fig. 11) are 10, 8, 18, 2, and also 15 (see Table II). Samples located at the opposite end of PC2—13, 14, and 4—exhibit the most stable behavior. Impact of accelerated aging on the other two parameters, bleeding and optical density, is more difficult to explain. Their proximity to PC1-PC2 coordinate origin indicates that they are related to higher PCs (see Figs. 10 and 12), which account for a much smaller percentage of variability in the data (PC3: 15%, PC4: 11%) than PC1 and PC2 so PCA cannot interpret effects of coating components so accurately. Table II shows that fixative-free (with exceptions of samples 11 and 12) coatings based on calcium carbonate produce prints where there is a decrease, rather than a normal increase, in bleeding index after the aging treatment, a fact that will have to be studied more closely in the future. Regarding the optical density of black prints it can be noted that the differences between the treated and nontreated samples are extremely small and that neither effect of the studied coating components could be proved to be statistically significant.

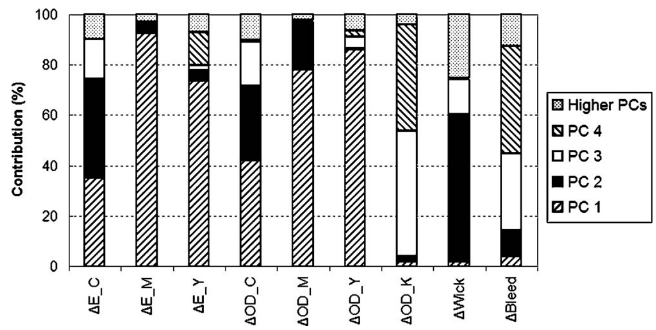

Figure 13. Contribution of differences in parameters between the aged and unaged prints to PCs.

Figure 13 shows breakdown of PCA variables by the first four PCs. As stated previously, differences in optical density and colorimetric properties of cyan, magenta, and yellow prints show similar behavior in terms of the effect of coating components—predominantly pigment type—and these six variables contribute to a high degree to PC1. PC2 is, on the other hand, mainly determined by wicking differences. It can therefore be proposed that the physical meaning of PC1 is related primarily to the changes in optical and colorimetrical properties of IJ colored (i.e., cyan, magenta, and yellow) prints, which are governed by physical interactions of a printed substrate with light (opacity, light scattering, and absorption), i.e., by different structural properties of coating. PC2, on the other hand, seems to correspond more to chemical phenomena on the surface (substrate surface tension and ink surface energy, etc.) leading to differences in wicking behavior between aged and unaged prints.

In the end it should be mentioned that optical density and $L^*a^*b^*$ color measurements were performed on solid print patches only. Possible nonlinear effects of accelerated aging with respect to starting optical density were not investigated in this study.

CONCLUSIONS

Our study showed that the choice of paper coating pigment decisively affects IJ printability and also influences the stability of prints under the absence of light. Both color vividness and print density on untreated samples using colored dye-based inks are much higher on calcium carbonate coatings compared to coatings where silica is used. In the case of black pigment-based ink silica performs much better than PCC or GCC. As for the treated samples, it was determined that accelerated artificial aging decreases color vividness much more severely with GCC than does with PCC while prints on silica coatings exhibit highest darkfastness. Presence of cationic dye fixative—polyDADMAC—in coating color has a negative impact on image stability.

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