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**GAS FADING OF INKJET PRINTS WITH OZONE**

Michal VESELÝ<sup>1</sup>, Petr DZIK, Silvia KÁČEROVÁ,  
Eva ŠTĚPÁNKOVÁ and Klára PASEČNÁ  
Faculty of Chemistry,  
Brno University of Technology, CZ-612 00 Brno

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*Dedicated to Professor Marie Kaplanova of the University of Pardubice in honour of her jubilee*

*Ground-level ozone is a common part of the air atmosphere and has a strong oxidative effect, mainly on inkjet dyes. The resistance of inkjet prints to ozone is mainly given by gas absorption capacity of the ink receiving layer and by the sensitivity of dyes to oxidation. The ground-level ozone concentration is usually low, from 20 to 80 mg m<sup>-3</sup> of air. In spite of such a low concentration, it can cause a total print deterioration in the course of several weeks when displayed. A test target was designed in PhotoShop and printed using Epson printer R220, MIS Dyebase inks (ESC-R200-4-C, M, Y) and Quad Tone RIP. The target contains patches of pure inks with dot area 5, 10, 20, 30, 40 a 100 % and their overprints C+Y, C+M a Y+M and composite black. Ilford Gallery Smooth Gloss (290 g m<sup>-2</sup>) and Hahnemühle-Photo Rag Baryta (315 g m<sup>-2</sup>) photo paper were used as the ink-receiving layer. Gas fading of prints was performed in glass chambers flowed with ozone, generated by discharge way from a mixture of nitrogen and oxygen. Reflective VIS spectra of each patch on the target were recorded and colorimetric*

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<sup>1</sup> To whom correspondence should be addressed.

quantities were calculated. An accelerated ozone test with approx. 1000 times higher ozone concentration compared to the natural conditions was performed. Effect of ozone is very complex and ambiguous, moreover no ISO standard describes process of ozone ageing and its evaluation. Therefore, several methods for ozone ageing evaluation were proposed. Dye fading was evaluated by three methods: 1) optical density decrease, 2) dye concentration calculation from spectral data and 3) quantity accepted by many users — total colour difference value. The dye concentration calculation of pure ink patches and overprinted inks patches enables to judge the catalytic fading. That means one dye fading is accelerated by the products of other dye degradation. The principles of all evaluation procedures used in this research are fully described.

## Introduction

Ground-level ozone is generated by solar radiation in the bottom layers of atmosphere containing nitrogen oxides, volatile organic compounds (VOCs) and low humidity. Its concentration varies from 20 to 120  $\mu\text{g m}^{-3}$  in summer and drops to 30–60  $\mu\text{g m}^{-3}$  in winter. Ozone concentration varies also locally and is closely related to fossil fuel combustion which accounts for its presence even in rural highlands. Ozone is a typical secondary product. It is not directly emitted into the environment and certain conditions must be fulfilled for its generation: solar radiation, temperature, humidity, air circulation. The optimum conditions for ozone-generating reactions take place when there is an equilibrium between nitrogen oxide and VOCs (from 4:1 to 10:1 ratio).

Ozone generating precursors originate mainly from combustion processes and transportation plays a dominant role. Ground level ozone is generated by a series of chemical reactions, initiated by the decomposition of nitrogen dioxide upon its solar irradiation. In this way nitrogen monoxide is created and an oxygen atom is liberated, which immediately reacts with oxygen molecule yielding an ozone molecule. At normal conditions the generated ozone oxidizes nitrogen monoxide back to nitrogen dioxide and ozone is reduced to harmless oxygen. However, if VOCs and their radicals are present, they are able to substitute ozone in the reaction with nitrogen monoxide. Nitrogen dioxide is thus produced anyway but ozone is not consumed and accumulates. The whole process can be summarized by the following equations





Ozone concentration is generally higher in rural and highland areas, but its concentration is even and local concentration peaks are rare. Ozone is created there by natural photochemical cycle in the troposphere. The higher level of solar irradiation in such areas also contributes to ozone generation.

On the other hand, ozone concentration in urban agglomerations is lower due to frequent reactions with nitrogen oxides and, therefore, long-term average values are generally lower. However, an “ozone episode” creating anomalous increase in the ozone concentration can occur in the case of suitable weather conditions. It can last up to several days, and ozone concentration may reach over  $200 \mu\text{g m}^{-3}$  [1].

Office equipment has been found to be a source of ozone, particulate matter, volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs). VOC, SVOC and particles can also be emitted by the paper processed during printing and copying [2]. Emissions of VOCs from printers and copiers are generally higher than from computers, particularly for styrene, toluene, xylene and other alkylbenzenes. For ozone, although the emission rates are unclear, even the low levels of ozone emitted by printers and copier machines can react with other indoor pollutants, resulting in secondary pollutants and generation of ultrafine aerosol particles [3,19].

Ozone is relatively instable gas. It decomposes quickly into oxygen molecule and elemental oxygen upon contact with many materials or reacts with trace amount of water to produce hydroxyl radicals. Both elemental oxygen and hydroxyl radical are very powerful oxidizing agents readily attacking dye molecules of inkjet printouts.

Gas fading becomes very important issue with respect to the stability of dye-based inkjet prints displayed without framing or any other protection. Many authors described the effect of airborne pollutants like ozone,  $\text{NO}_2$  or  $\text{SO}_2$  on digital prints deterioration [2]. Ozone is well known as an aggressive substance, a very powerful oxidant and it is a significantly stronger oxidizer than the other gases mentioned [5,6]. It reacts very easily with conjugated structures present in many chromophoric systems of dyes and pigments [20-24].

The quality of inkjet printouts has been rising with the development of printers, inks and printing media. There are several kinds of print media used for inkjet print applications that differ in properties, quality and price. Conventional media consist of wood-free paper and surface coating containing porous pigment with high ink absorptivity [7]. More expensive alternative includes media with an inert base and special absorptive coatings. Especially media with instant-dry porous receiving layers are the popular choice for a wide group of consumers. The biggest disadvantage of this type of print medium is the fact that inkjet dyes adsorbed to the pores are relatively strongly exposed to air and may fade

unacceptably in a short time [8]. The open nature and high porosity of these layers enable the air pollutants to penetrate and decompose the dye chromophores [10].

Thus ozone and other airborne pollutants are readily absorbed into the sorbent pores when prints are displayed. In this way they approach a close proximity of the dye molecules and a degradation process by elemental oxygen gets started. The rate of inkjet dye degradation is subject to many influences: ozone concentration, dye chemical structure, sorbent type in the ink receiving layer, humidity, relative humidity etc.

The position and nature of substituents in the azo dye molecule plays a role in determining the state of the tautomeric equilibrium [11]. Since the azo group (N=N) is an electron acceptor, electron donating substituents, especially OCH<sub>3</sub>, stabilize the azo tautomer. However, the imino group (NH) is an electron donor, and the hydrazone tautomer is favoured by electron withdrawing substituents, especially NO<sub>2</sub>. Intramolecular hydrogen bonding can also provide additional stability to the hydrazone tautomer with certain structures [12]. Typically, this occurs between an OH group at the ortho position and the further nitrogen of the azo group. Since oxygen is more electronegative than nitrogen, it forms a stronger hydrogen bond, and thus the hydrazone tautomer is stabilized more. Intermolecular hydrogen bonding with the media and components in the ink can have similar effects, and generally the more polar materials favour the hydrazone tautomer. Of course, the media restrict free tautomeric interchange. Unfortunately, the hydrazone tautomer is less resistant to attack of singlet oxygen formed from ozone [8].

Studies focused on the degradation of azo dyes in aqueous solution have shown that the decay kinetics mainly depends upon the initial dye concentration, pH and O<sub>3</sub> dose present in the aqueous solution [13,14]. Formic, acetic and oxalic acids were detected as the final product of degradation. A possible degradation pathway involving a OH-radical reaction mechanism was proposed without further quantitative analyses.

The process mechanism can be described by the following equations [15]



Indirect reaction occurs with the reactions



In order to protect the images produced by inkjet printing against harmful effect of ozone, they are framed behind glass, laminated with plastic foils or coated with protecting varnishes.

## Experimental

A dedicated test chard containing patches of pure inks (dot area 5, 10, 20, 30, 40 and 100 %), their binary overprints combining C+Y, C+M and Y+M, triple overprints of equal dot area (e.g., 5 % C + 5 % M + 5 % Y), patches of optical density close to 1 for each primary colour and a white unprinted patch (Table I). Separations for all primary colours were generated in Adobe Photoshop. They were printed by QuadTone RIP and Epson Stylus Photo R220 printer onto Ilford Gallery Smooth Gloss (290 g m<sup>-2</sup>) and Hahnemühle-Photo Rag Baryta (315 g m<sup>-2</sup>) papers by subsequent overprinting of the primary inks (ESC-R200-4 ink set by MIS Associates Inc., USA). Colour separations were printed in the sequence cyan, magenta and yellow. A 24-hour time lag for ink drying between the overprints was maintained. Spectral data and  $L^*a^*b^*$  coordinates of printouts were measured before its exposing to ozone accelerated ageing in the closed glass chambers. Gretag Macbeth™ Spectroscan device (D50, 2° observer, no polarizing filter) and Gretag Macbeth™ Measure Tool 5.0.5 were used for spectral data measurements.

The optical densities of each ink were calculated according to the following formulas

$$D_C = -\log \left( \frac{\sum_{580}^{760} R(\lambda) I(\lambda)}{\sum_{580}^{760} I(\lambda)} \right) \quad (8)$$

$$D_M = -\log \left( \frac{\sum_{480}^{610} R(\lambda) I(\lambda)}{\sum_{480}^{610} I(\lambda)} \right) \quad (9)$$

$$D_Y = -\log \left( \frac{\sum_{400}^{510} R(\lambda) I(\lambda)}{\sum_{400}^{510} I(\lambda)} \right) \quad (10)$$

where  $R(\lambda)$  is reflectance and  $I(\lambda)$  is spectral product of densitometric status A [17].

Figure 2 describes the experimental set-up for the ozone accelerated ageing test. The ozone was generated by discharge way from oxygen and nitrogen 4:1

mixture at the temperature of 22.5 °C and air flow of 77.65 dm<sup>3</sup> h<sup>-1</sup>. The generated ozone flowed through a glass chamber with the sample in dark and was absorbed in 100 ml of 0.2M potassium iodide solution, used for analytical assessment using the iodometric method with thiosulphate. It was determined that 80 ppm of ozone flowed through glass chamber. The samples were exposed to ozone up to total ozone dose 960 ppm h.

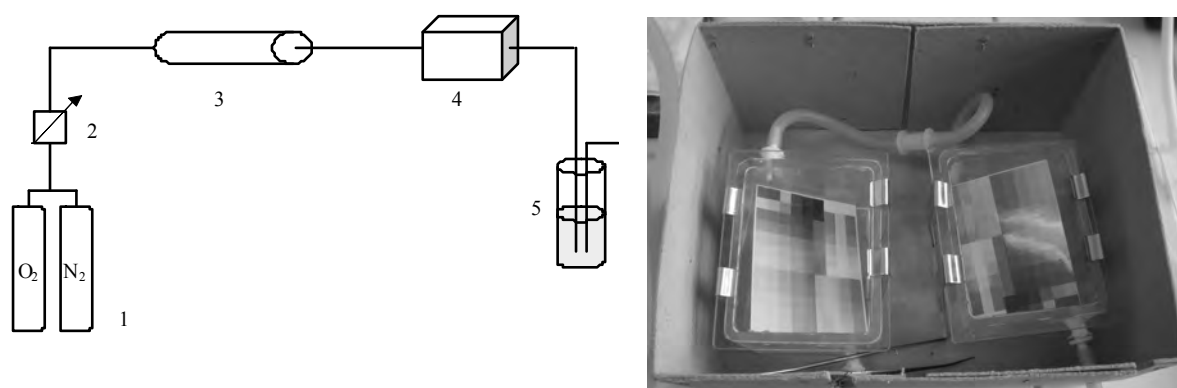


Fig. 1 The experimental setup for ozone generation and sample exposure: 1 – compressed air, 2 – flow meter, 3 – discharge tube, 4 – glass chamber with sample, 5 – absorption solution. Glass chambers on the right hand side)

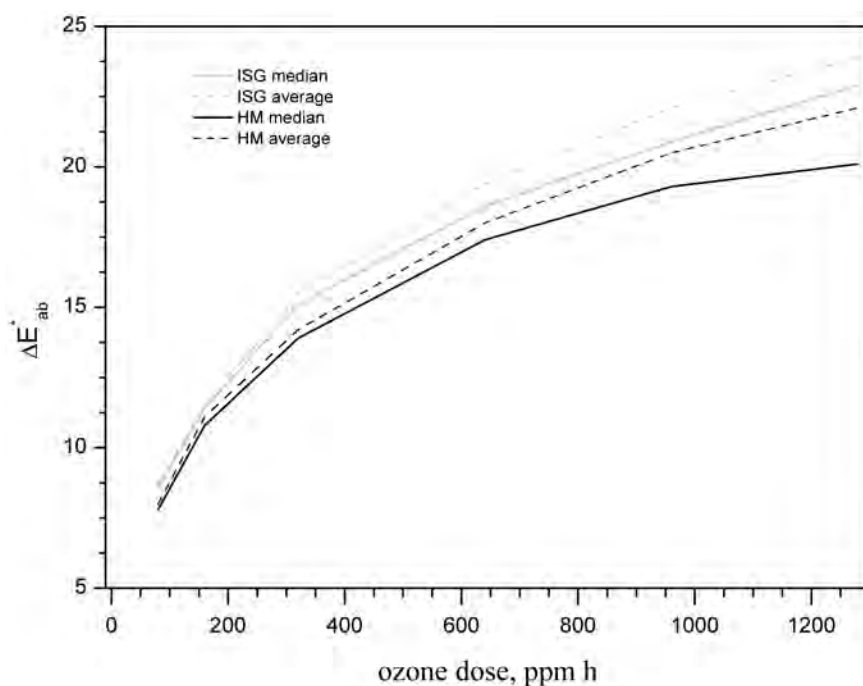


Fig. 2 Average and median values of colour differences on test target

Table I Dot area values (%) of printed patches

|        |         |         |         |         |        |         |         |         |         |                |           |
|--------|---------|---------|---------|---------|--------|---------|---------|---------|---------|----------------|-----------|
| 5C+5Y  | 5C+10Y  | 5C+20Y  | 5C+30Y  | 5C+40Y  | 5C+5M  | 5C+10M  | 5C+20M  | 5C+30M  | 5C+40M  | 5C+5M+5Y       | 100C      |
| 10C+5Y | 10C+10Y | 10C+20Y | 10C+30Y | 10C+40Y | 10C+5M | 10C+10M | 10C+20M | 10C+30M | 10C+40M | 10C+10M+10Y    | 100M      |
| 20C+5Y | 20C+10Y | 20C+20Y | 20C+30Y | 20C+40Y | 20C+5M | 20C+10M | 20C+20M | 20C+30M | 20C+40M | 20C+20M+20Y    | 100Y      |
| 30C+5Y | 30C+10Y | 30C+20Y | 30C+30Y | 30C+40Y | 30C+5M | 30C+10M | 30C+20M | 30C+30M | 30C+40M | 30C+30M+30Y    | 100M+100Y |
| 40C+5Y | 40C+10Y | 40C+20Y | 40C+30Y | 40C+40Y | 40C+5M | 40C+10M | 40C+20M | 40C+30M | 40C+40M | 40C+40M+40Y    | 100C+100Y |
| 5M+5Y  | 5M+10Y  | 5M+20Y  | 5M+30Y  | 5M+40Y  | 40M+5Y | 40M+10Y | 40M+20Y | 40M+30Y | 40M+40Y | 100C+100M+100Y | 100C+100M |
| 10M+5Y | 10M+10Y | 10M+20Y | 10M+30Y | 10M+40Y | 5C     | 10C     | 20C     | 30C     | 40C     | 50C            |           |
| 20M+5Y | 20M+10Y | 20M+20Y | 20M+30Y | 20M+40Y | 5Y     | 10Y     | 20Y     | 30Y     | 40Y     | 50Y            |           |
| 30M+5Y | 30M+10Y | 30M+20Y | 30M+30Y | 30M+40Y | 5M     | 10M     | 20M     | 30M     | 40M     | 50M            |           |

## Results and Discussion

The results were evaluated in three different ways. The first one included a calculation of total colour difference in accordance with Eq. (11). The second one used optical densities evaluation. The patches with original optical density  $1.0 \pm 0.05$  above the minimum density of pure ink were used for this purpose. It is a procedure similar to the lightfastness evaluation precisely defined by ISO standard 18909. The third type of evaluation is based on a calculation of ink concentrations at chosen patches of the test chart.

### Colour Differences Evaluation

The colour difference was calculated as follows

$$\Delta E_{ab}^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (11)$$

The evaluation of colour difference on selected patches, for example of those having 100% dot area or optical density close to 1, is not particularly useful for both practical and formal reasons (lack of appropriate standard). Thus it might seem suitable to evaluate more patches to get more information. But we still face the problem of selecting representative patches. Expressing the total colour difference as mean or median gives a single figure, but it is strongly depending on the number of test chart patches. The following plot (Fig. 3) shows the average colour differences and the median of colour differences of measured 108 test patches. According to the generally accepted properties of colour difference,  $\Delta E$

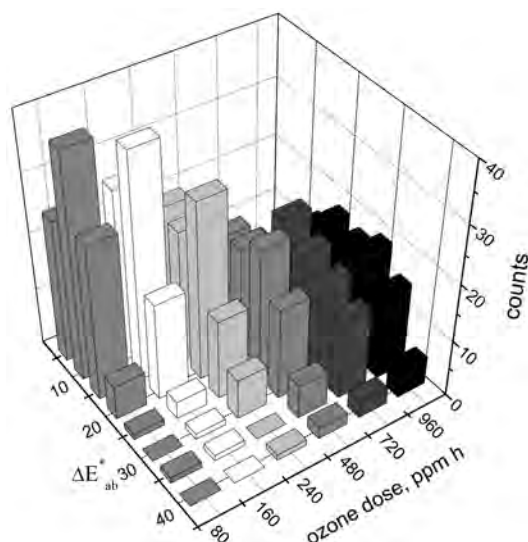


Fig. 3 Colour differences counts as a function of ozone dose. Ilford Gallery Smooth Gloss

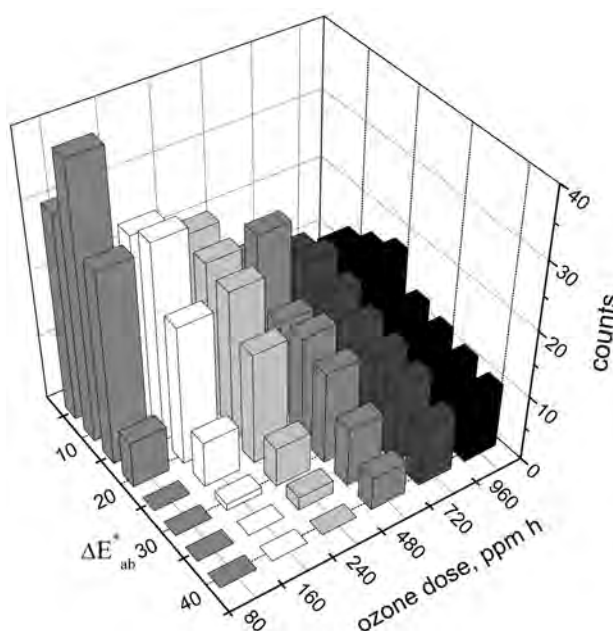


Fig. 4 Colour differences counts as a function of ozone dose. Hahnemühle-Photo Rag Baryta

$\Delta E = 8$  is considered distinctive. Therefore we can expect  $\Delta E = 16$  to be so upsetting that the image can be rated as unacceptable. If we use a test chart with a large number of patches (more than 500) designed to contain the saturated colours close to the hull of gamut, then the median value seems to be a good single criterion. If smaller test chart is used as in this study (108 and less patches), then the mean value seems to be more suitable.

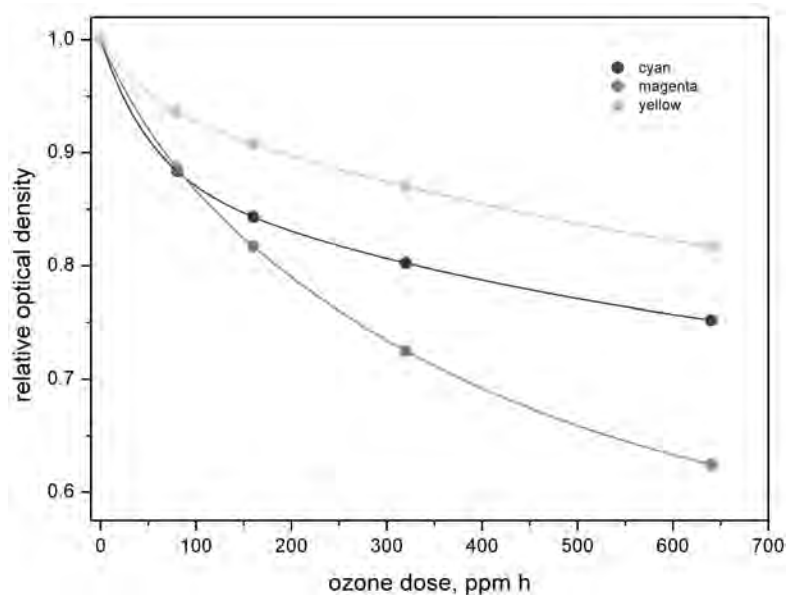


Fig. 5 Relative optical density changes during ozone fading. Patches with original optical density  $1 \pm 0.05$ . Hahnemühle-Photo Rag Baryta

If the test charts contain enough patches, it is possible to express the image fading during ozone-initiated degradation by distribution function of colour differences (i.e. histogram). It seems that this approach brings a large information content about the degradation process, but it is quite difficult to set the endpoint criteria for this type of fading data presentation. Anyway, the histogram presentation can be a highly synoptic way of comparing the fading data of extended test charts. As the fading process goes on, the distribution of colour difference values broadens.

### Optical Density Evaluation

As mentioned above, the fading of prints can be evaluated by the criterion of optical density decrease by 30 % on patches having initial density  $D_0 = 1 \pm 0.05$ . This approach is inspired by ISO 18909, which considers this degree of fading as the maximum allowed, and the prints exceeding this value are thus considered just unacceptable [18]. This standard is valid for silver-halide based materials only. But due to the lack of special inkjet-oriented standard, ISO 18909 is widely used by both inkjet media manufacturers and independent permanence testing labs for inkjet prints as well.

It is worth stressing out that densitometric measurements can give correct information only when the fading rate of all dyes is similar. This is true in the case of silver halide prints, not in the case inkjet media. Dyes used in inkjet inks can fade at significantly different rates. Measurement of optical density of one parti-

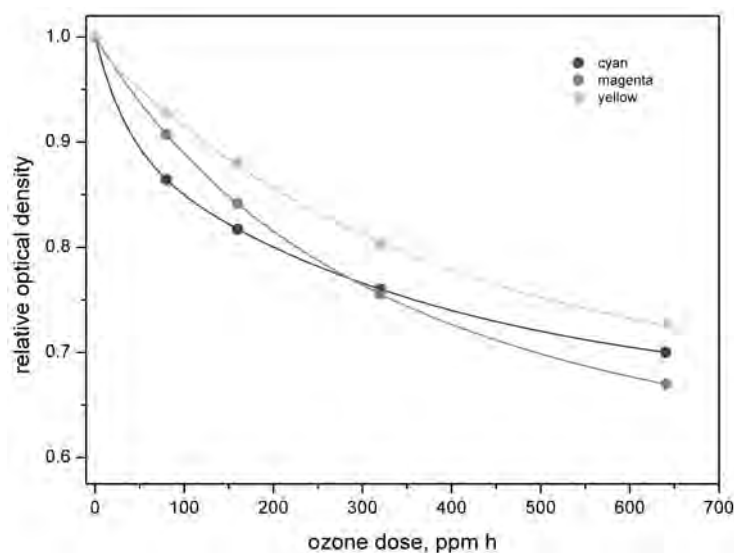


Fig. 6 Relative optical density changes during ozone fading. Patches with original optical density  $1 \pm 0.05$ . Ilford Gallery Smooth Gloss

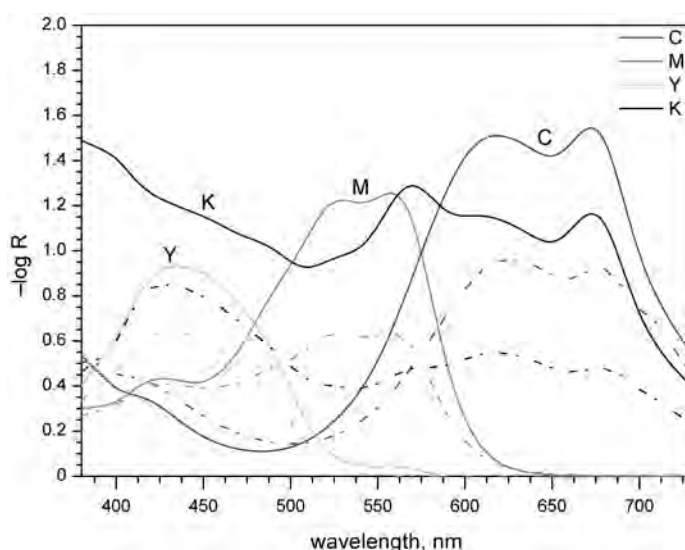


Fig. 7 VIS spectra of pure inks on patches with dot area 40 % before (solid lines) and after ozone exposure (dashed lines). Ilford Gallery Smooth Gloss

cular dye is affected by the spectral properties of other dyes present in the layer. So if, for example, the cyan dye degrades faster than the yellow one, the optical density of the yellow dye will drop faster than the yellow dye concentration because the faster degrading cyan dye has a prominent absorption also in the blue part of the spectrum [16] (Figs 8 and 9).

The plot of optical density decrease measured on patches having initial density  $D_0 = 1 \pm 0.05$  is depicted in Figs 6 and 7. Yellow dye on the Hahnemühle-Photo Rag Baryta photopaper was so stable that the 30 % criterion has not been reached during this experiment. However, the criterion is reached when any of the primary inks drop by 30 %. The presented results indicate that despite the different

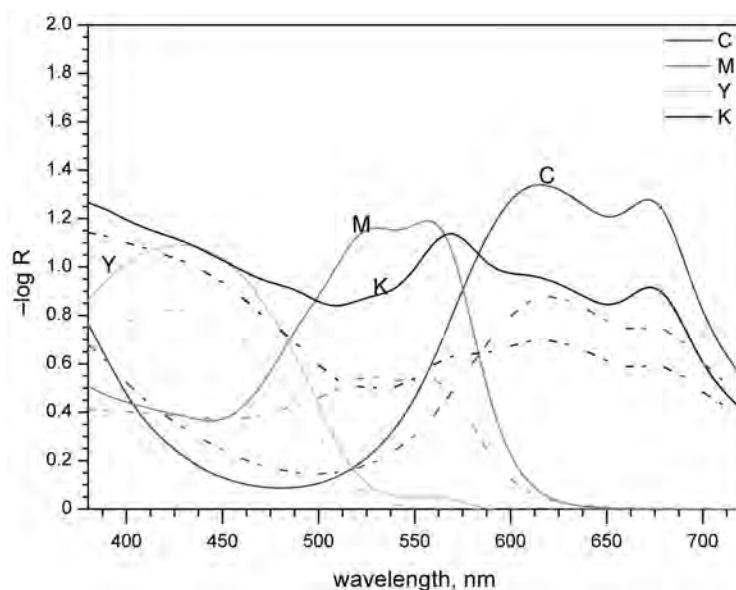


Fig. 8 VIS spectra of pure inks on patches with dot area 40 % before (solid lines) and after ozone exposure (dashed lines). Hahnemühle-Photo Rag Baryta

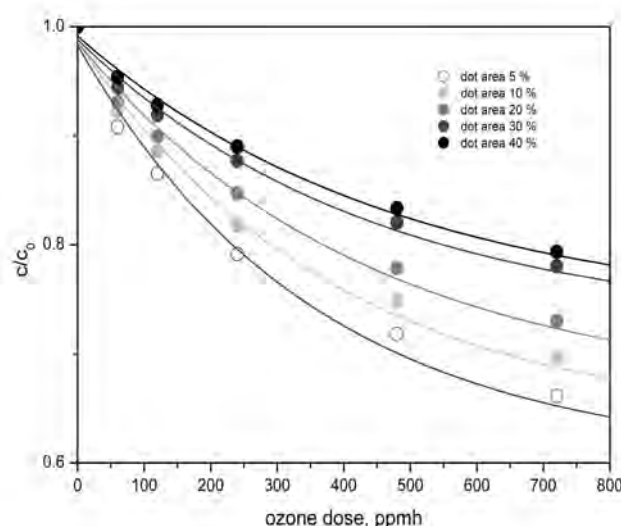


Fig. 9 Concentration decrease of yellow dye in pure yellow patches. Hahnemühle-Photo Rag Baryta paper

shape of the curves especially in the initial stage of the experiment, the end-point criterion of 30 % decrease in the optical density was caused by different ozone doses. These are reported in Table II. If the criterion had not been reached, the corresponding expected dose was found by extrapolation. The figures clearly indicate that with this particular inkset, the prints produced on the Hahnemühle-Photo Rag Baryta paper are much more resistant to ozone than the prints on Ilford Gallery Smooth Gloss. The reason for this behaviour is in the receiving layer composition influencing the reactivity of dyes adsorbed and the ozone uptake.

Table II End-point criterion expressed as ozone dose (ppm h)

| Ink-receiving media                                  | Cyan | Magenta | Yellow |
|------------------------------------------------------|------|---------|--------|
| Ilford Gallery Smooth Gloss (290 g m <sup>-1</sup> ) | 641  | 493     | 925    |
| Hahnemühle/Photo Rag Baryta (315 g m <sup>-1</sup> ) | 1360 | 376     |        |

### Dye Concentration Evaluation

A test chart consisting of patches with well-defined amount of deposited ink can be used for the evaluation of dye concentration, both of patches printed by single dye and of binary overprinted ones.

Lambert–Beer law was used for the concentration calculation (12). Absorbance  $A$  can be approximated by  $-\log R$ , which can be determined by a spectrophotometric measurement and calculation. The product  $c \times l$  can be replaced by surface concentration  $c_p$ . The only unknown remaining is then the molar absorption coefficient  $\varepsilon$ . A set of solutions of different ink concentrations was measured by UV-VIS spectrophotometer to find the molar absorption coefficient  $\varepsilon$  for each ink.

$$A = -\log R = \varepsilon cl, \quad l = \text{const.}, \quad cl = c_p = -\frac{\log R}{\varepsilon} \quad (12)$$

The ink concentrations on overprinted patches (for example 20 C and 20 Y – dot area) were calculated for both inks from the measured reflectance spectra, decomposed by the least squares method. The reflectance spectra of pure inks were known ( $x$  and  $y$ ) as well as the spectrum of these two inks overprinted patch ( $z$ ). Constants  $a$  and  $b$  were calculated by minimizing function (13), where  $x_i$  and  $y_i$  denote the reflectance of pure inks at wavelength  $i$ ,  $z_i$  stands for the reflectance of the overprinted patch at wavelength  $i$  (the wavelengths run from 380 to 730 nm with 10 nm step). Minus logarithm reflectance spectra of pure inks corrected by constants  $a$  and  $b$  were used for ink concentration calculation by means of Eq. (12)

$$A(a, b) = \sum_i (ax_i + by_i - z_i)^2 \quad (13)$$

Sample spectra analyses and the computation of ink concentrations were performed in the MatLab software. Then the  $c/c_0$  ratio was calculated to see the ink degradation on ozone-aged printouts.

The calculated  $c/c_0$  ratio was plotted against ozone dose and fitted by the following function

$$y = A_1 e^{-\frac{x}{t}} + y_0 \quad (14)$$

Then the  $t$  value can be treated as a parameter of ink dye fading. As  $t$  increases, so does the fading rate. The results of kinetic study of dye degradation initiated with ozone are in a good agreement with published results. The dominant mechanism of observed degradation complies with the pseudo first order kinetics [13,24].

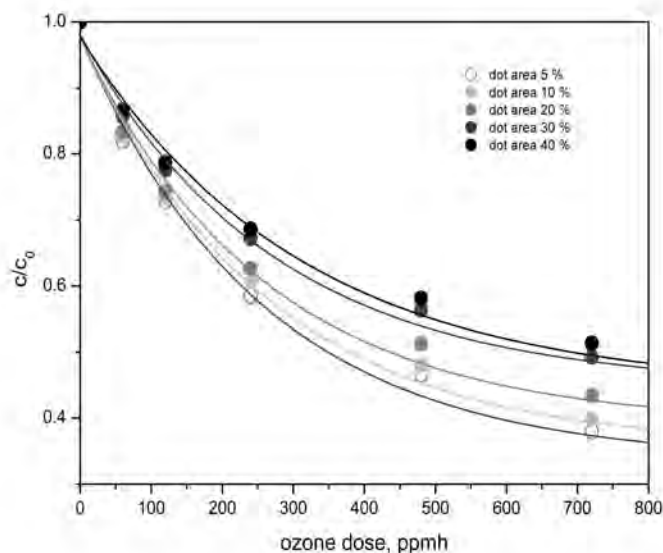


Fig. 10 Concentration decrease of magenta dye in pure magenta patches. Hahnemühle-Photo Rag Baryta paper

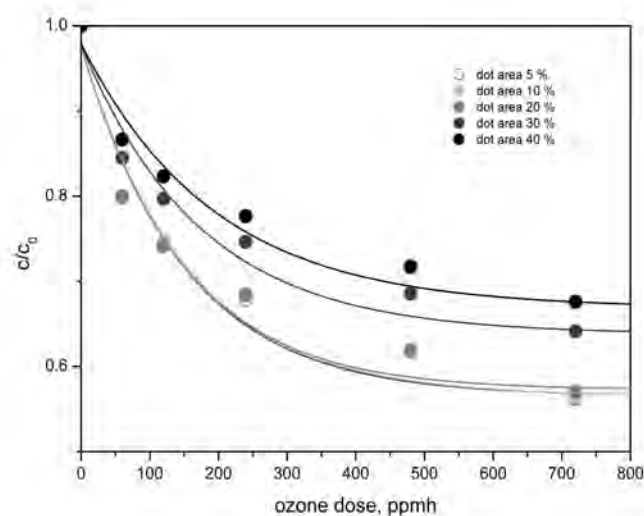


Fig. 11 Concentration decrease of cyan dye in pure cyan patches. Hahnemühle-Photo Rag Baryta paper

The patches printed with pure inks C, M, Y and binary overprinted patches with a constant dot area of 40 % of one ink and variable dot area of the second one, e.g., patches of yellow ink of 5, 10, 20, 30 and 40 % dot area were overprinted by cyan ink at 40 %.

The graphs of ozone degradation (Figs 9-14) indicate that the order of remaining dye concentration is M, C, Y after the ozone dose of 960 ppm h. The ra-

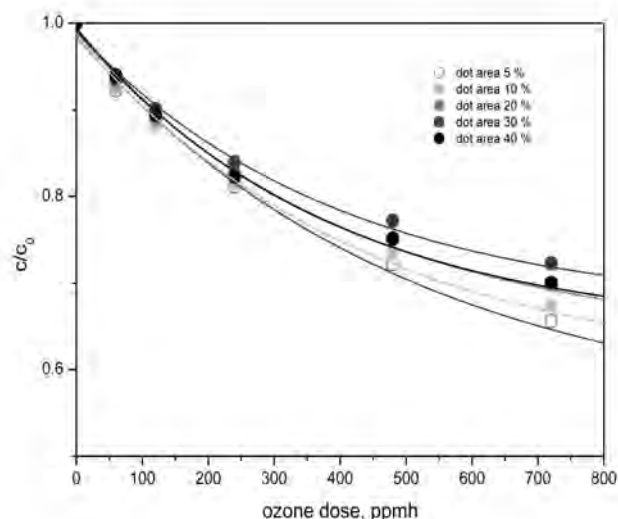


Fig. 12 Concentration decrease of yellow dye in pure yellow patches. Ilford Gallery Smooth Gloss paper

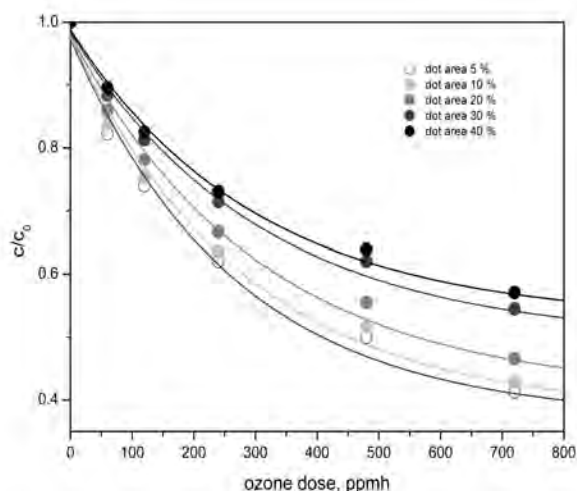


Fig. 13 Concentration decrease of magenta dye in pure magenta patches. Ilford Gallery Smooth Gloss paper

te of degradation expressed according to relation (14) by the fading coefficient is changing in the order C, M, Y. The fading coefficient is influenced by the initial degradation rate in particular.

By comparing the surface concentration and fading rate coefficients of single-ink and overprinted patches it is possible to evaluate if the catalytic fading process takes place. Catalytic fading means that the degradation products of one dye would promote the fading of another dye. The presented graphs (Figs 15 and 16) clearly show that the yellow dye at binary overprinted patches containing any other dye (cyan or magenta) fades faster than the yellow dye deposited at purely yellow patches. So we can assume that the degradation products of both magenta or cyan dye accelerate the degradation of the yellow dye. Similarly, magenta ink degrades faster on the overprinted patches containing magenta and cyan. On the contrary, cyan ink is not susceptible to catalytic fading, although the rate of fading

found from these experiments and expressed as the above defined fading coefficient  $t$  is the greatest for cyan dye for both sorts of papers.

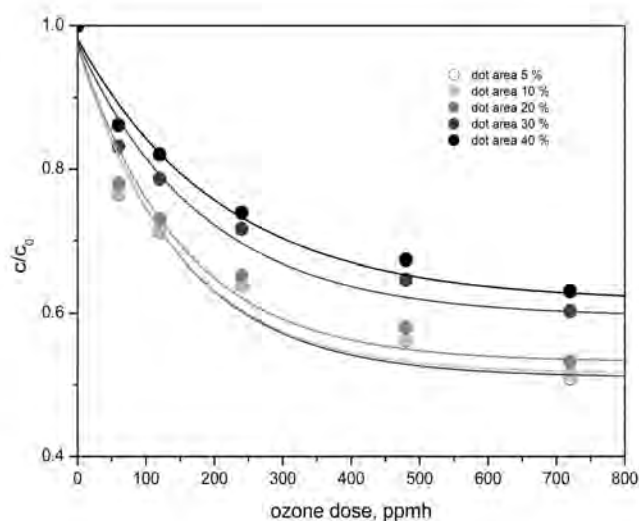


Fig. 14 Concentration decrease of cyan dye in pure cyan patches. Ilford Gallery Smooth Gloss paper

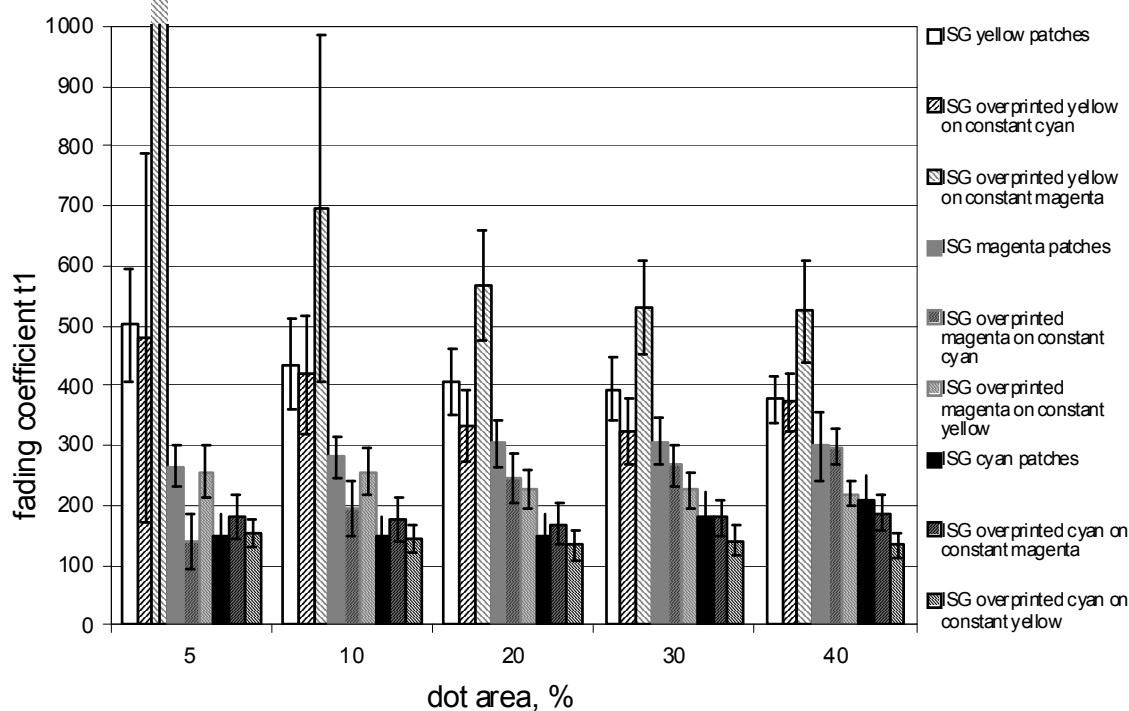


Fig. 15 Values of fading coefficient  $t$  of fitting equation. Ilford Gallery Smooth Gloss paper

## Conclusion

As there is no generally accepted standard, 3 different approaches were used for the evaluation of print degradation by ozone. The simplest method utilizes densitometric measurements of patches having initial density  $D_0 = 1 \pm 0.05$ . This method is not suitable for the estimation of inkjet media lifetime based on the extrapolation of accelerated fading experiments, because densitometric filters might not match the spectral absorption curves of various inkjet dyes. Errors of measurements would be further pronounced by extrapolation, and significant errors would result.

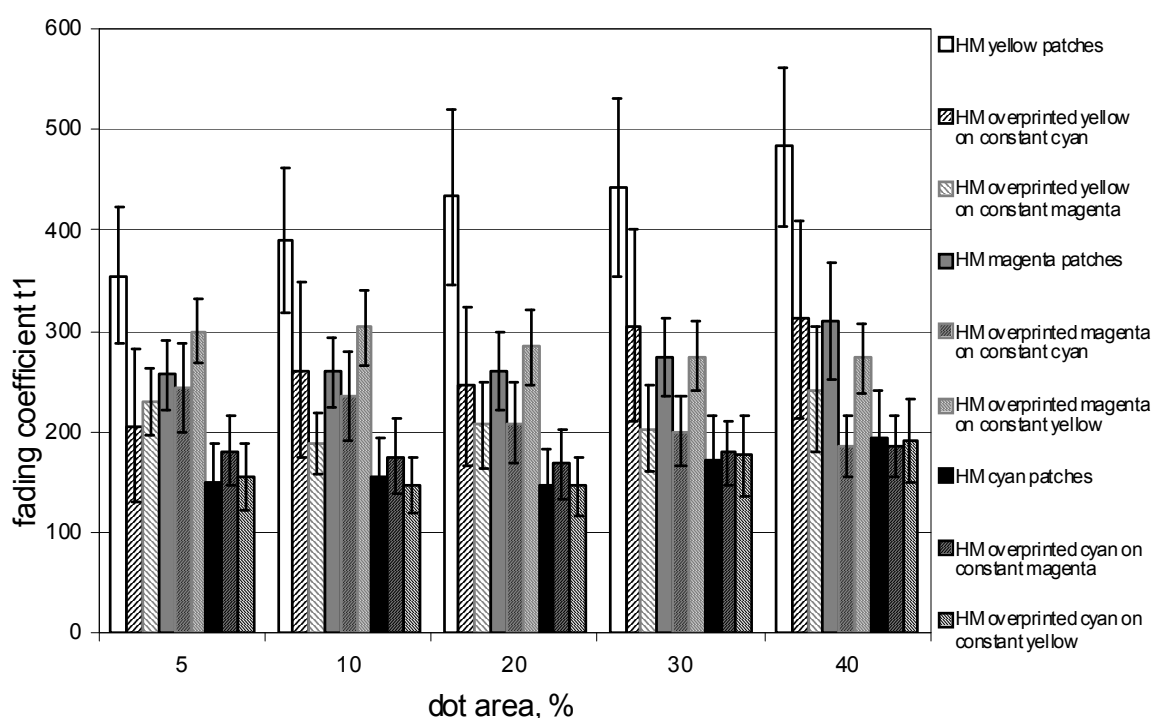


Fig. 16 Values of fading coefficient  $t$  of fitting equation. Hahnemühle-Photo Rag Baryta paper

The colorimetric method based on the mean colour difference is strongly dependent on the test chart design and/or patch selection. The method requires more sophisticated instruments (spectral colorimeter preferably) but is still reasonably simple. It certainly needs to be further developed to identify suitable endpoint criteria.

The third method is based on reflection spectrometry of selected test patches and calculation of the surface concentration of printed dyes. This approach is by far the most complicated and time consuming. However, it is capable of delivering not only information about the dye degradation rate, but also about their mutual interactions.

## Acknowledgements

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