

# Fine Particle Pigment Concentrates for Ink Jet Printing Inks

*H. P. Hauser and N. E. Bühler*  
*Pigments Division, Ciba Specialty Chemicals Inc.,*  
*Basel, Switzerland*

## Abstract

Aqueous, low viscosity, stable pigment concentrates suitable for formulating Ink Jet printing inks have been realized based on selected fine grade pigment preparations. This paper deals with the **prerequisites for successful concentrate technology**, such as:

- fine grade pigment preparation by combining salt milling and kneading technologies
- reaching a high pigment load while maintaining low viscosity
- the three stages of successful dispersion technology (wetting, desagglomeration and dispersion stability)
- the suppression of particle flocculation.

Pigment chromophores usable for Yellow, Cyan and Magenta process colors are discussed.

## Making the Right Choice

Ink jet printers and electro-photographic laser printers have emerged as to-day's leading (and competing) non-impact technologies. Both feature the capability of printing black and color on many surfaces. Whereas electro-photographic (dry or liquid) toner systems rely mostly on pigments finely dispersed in a resin matrix, most ink jet inks still contain soluble dyes. The choice between pigmented and dyed inks depends on the desired print-out profile such as color gamut, transparency, color strength and fastness properties.

Multicolor industrial printing applications (wide-format graphics and photo-finishing in particular) have triggered a growing demand for ink jet inks combining good light-, weather- and waterfastness with a high brightness. A qualitative figure of merit for dyes and pigments is shown in figure 1:

To find suitable Yellow, Magenta and Cyan dyes combining optimal brightness with outstanding fastness is a challenging task; realizing a pigment ink formulation with stable particle sizes < 100 nm and high color strength while still maintaining all the fastness advantages high performance pigment systems normally hold is even more demanding.

Choosing an appropriate pigment chromophore with high light- and weather-stability is the first step to success. Table 1 depicts typical high performance pigments

exhibiting high chroma/hue, high color strength and outstanding light- and weather fastness as well as high product safety.

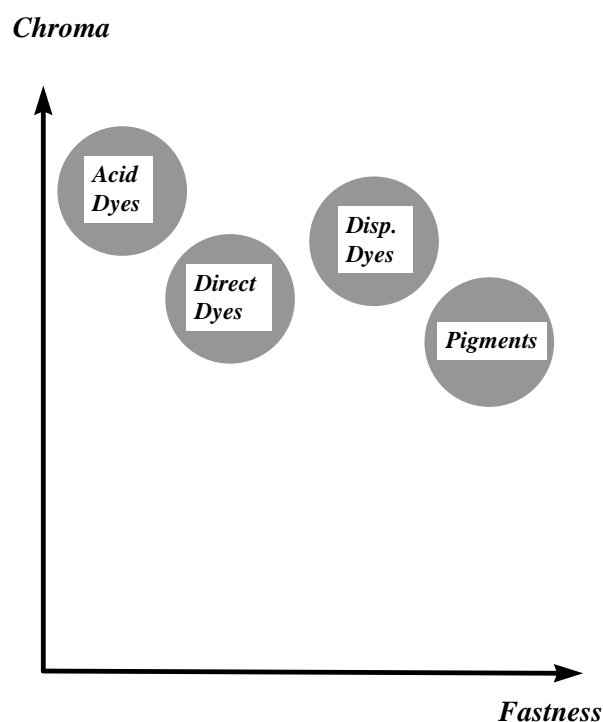


Figure 1

Pigment Yellow 128 is a greenish-shade yellow with excellent colour strength and high light- and weather-fastness.

Magenta shades can be composed by using Pigment Red 122, 254 and 184. Cu-phthalocyanine pigments are standard Cyan shades.

## Fine Particle Pigment Concentrates

Most pigment chromophore systems have been optimized into a special pigment form (crystal modification, particle size, particle size distribution, surface modification) appropriate to the desired use in automotive and industrial

paints, in plastics and in printing inks. Particle size reduction can be achieved either by inhibiting the growth of the pigment crystal during synthesis or by salt-milling and kneading technologies.

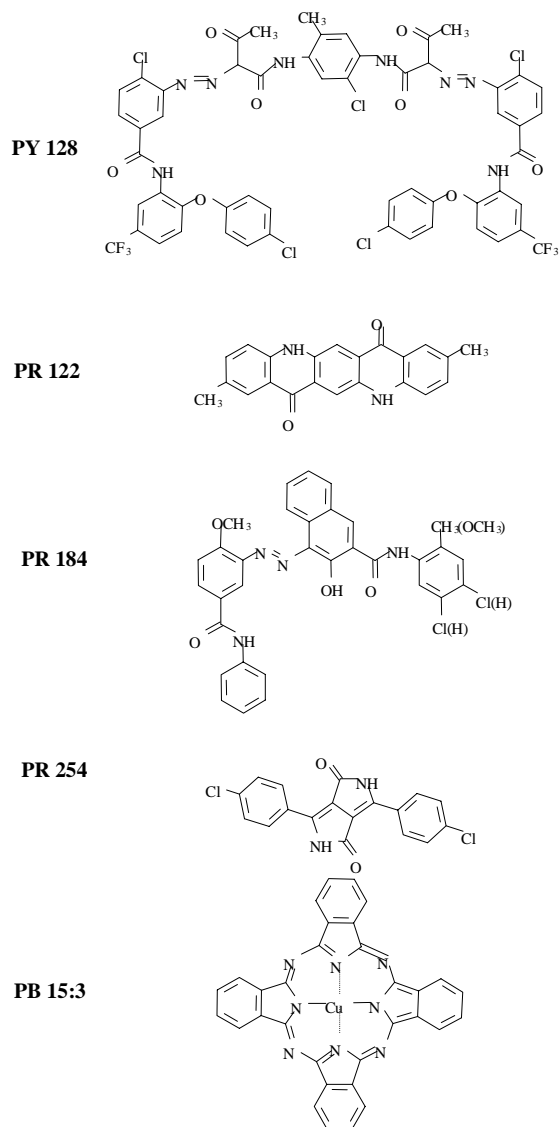


Table 1

Figure 3 visualizes this procedure:

Step a): Pigment particles are crushed in a high-shear mode by hard salt crystals and yield a finely divided pigment with broad particle size distribution.

Step b): A carefully controlled amount of a suitable solvent dissolves the finest particles and helps to heal (i.e. re-crystallize) the "wounded" pigment particles into a fine grade pigment with narrow particle size distribution.

Step c): Separation from the milling media and drying completes the procedure.

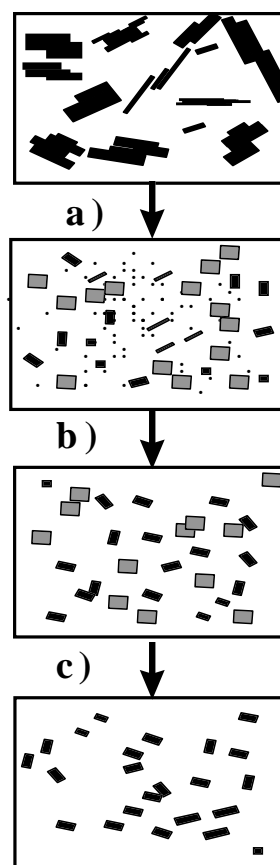


Figure 2

High-shear dispersing of pigments in water yields de-agglomerated particles - but the tendency to re-agglomerate is normally very high with organic pigments. Stabilization of these fine grade pigment particles in an aqueous medium is normally achieved by a combination of electrostatic and steric mechanisms. Polyelectrolytes based on styrenated polyacrylates are used. They wet the pigment surface and then stabilize the de-agglomerated particles.

## Ink Performance

A typical aqueous test ink<sup>1)</sup> consists of 5 % ultra-fine grade pigment dispersed in about the same amount of polyelectrolyte, 10% of a humectant, 0.5% surfactant and 0.1% of an antifoaming agent. The ink is then ultra-filtered and analyzed for particle size distribution.

Accurate measurement of particle size distributions is a delicate affair: such different methods as light diffraction, disc centrifugation or transmission electron microscopy are used, offering different degrees of trustworthiness, comfort and expense. Table 3 compares these methods.

A typical particle size distribution is shown in Table 4 (yellow ink made from a concentrate of salt-kneaded Pigment Yellow 128 and measured by Joyce Loebli disc centrifuge):

**Table 3**

Criteria	Light Diffraction	Disc Centrifuge	TEM
<b>Resolution</b>	about 300 nm	20 nm	few nm
<b>Preparation from ink</b>	much dilution needed	some dilution needed	solid preparation needed
<b>trust in results</b>	critical, extrapolates to small diameters	very good reliable distribution	very good, but sample much different from ink
<b>expense</b>	easy, fast	time consuming	average

**Table 4**

D (0.16)	D (0.50)	D (0.84)
0.083 microns	0.103 microns	0.152 microns

The test inks were loaded into a thermal ink jet printer (HP Quiet Jet) and printed onto a mat, white Ink Jet paper (Canon LC201). Good printability was maintained even after 4 months storage at room temperature.

The print-outs were exposed in an "Atlas"-Fadometer and optical densities determined before and after exposure by a "Macbeth" Densitometer. In contrast to print-outs made with commercially available dye-based inks these pigmented inks retained their initial optical density. In a typical example a full-shade yellow print-out based on an ink containing such an ultra-fine grade Pigment Yellow 128 exhibits an optical density of 1.13 before and after exposure to 30 kJ/cm<sup>2</sup>.

### Summary

Dyed and pigmented inks for ink jet applications strive for different parts of the digital printing market, where they can exploit their key advantages (color strength vs. fastness).

The key success factors of pigment-based inks lie in the optimum composition of an ultra-fine grade, well dispersed pigment system with narrow particle size distribution and a long-term stabilization of the pigment particle dispersion by polyelectrolytes. A balanced formulation of the fine-grade pigment concentrates yields inks with high color strength, while maintaining a lightfastness superior to dye-based alternatives.

### Acknowledgements

The authors wish to thank Dr. Klaus Ruf and Yves Grandidier for their excellent work in the salt-kneading technology and Dr. Werner Sieber for his valuable input concerning measurements and interpretation of particle size distributions, fine particle morphology and handling.

### References

1. US Patent 5,106,417 and EP 0425439A2

### Biographies

**Hans Peter Hauser** (56) has a 40 years experience in ink application and marketing with Ciba Specialty Chemicals. He his head of the Marketing Center High Performance Pigments for Printing Inks in the Pigments Division.

**Niklaus Bühler** (53) holds a Ph.D. degree in synthetic chemistry and photochemistry. He joined the company in 1975 and held different managerial position in Corporate Research and in the Business Unit Electronic Materials. He currently heads the Technological Innovation Group in the Pigments Division.