Particle Size of Commercial Inks and Their Deinkability

Alexandra Pekarovicova*, Jan Pekarovic*, and Andrea Frimova*

Keywords: ink, particle size, deinking, deinkability, color difference

ABSTRACT

The particle size analysis of commercially available process color inks for major printing processes was performed using Submicron 370 NICOMP Particle Sizer. Uncoated toluene based publication gravure ink had slightly smaller particle size than coated toluene based publication gravure ones. Flexo water based inks exhibited smaller particles than publication gravure ones. No relationship was found between colors and their particle sizes within inks for gravure and flexo printing processes. Deinkability was measured up to four weeks after printing as a color difference ΔE of handsheets before and after deinking. Flotation deinkability of gravure inks slightly decreased with increasing particle size of liquid inks and rapidly decreased with the particle size of flexo inks. Washing deinkability for both processes also decreased with decreasing particle size of liquid inks.

INTRODUCTION

Importance of Particle Size in Printing Inks

Controlled pigment distribution in printing ink is essential for the high quality of printing. The effective pigment dispersion method differs depending on the pigment color: high concentration flushing method for yellow and red; and high performance milling method for blue and black pigment. The key factors which control the pigment dispersion are flushing and surface treatment of pigment particles for the former, and mechanical milling efficiency and beads size for the latter (Noguchi, 2000). Each type of ink requires a very careful balance of formulation to meet the requirements of the printing process and the end use properties. The majority of these properties are controlled by the particle size of the pigments. The small particles provide excellent color strength, saturation,

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*Western Michigan University, Department of Paper and Printing Science and Engineering, Welborn Hall, Kalamazoo, MI 49008-5362

gloss (Desjumaux, 1997), hiding power, dispersion (Watanabe, 1990), and flow (Thompson B., 1998; Bayliss, 1974). On the other hand, large particles tend to exhibit poor dispersion (plate or cylinder wear, poor ink/water balance, hickeys, printability problems), poor flow, hiding power, color strength, color fluctuation, and gloss. Measuring of the particle size of the pigments can assess the degree of pigment dispersion.

The important characteristics affecting the color of the printing ink are the color strength (the effective concentration of coloring materials per unit weight or volume of ink) and the opacity (the ability of a pigment to cover or obscure the surface to which it was applied) (Nobuoka, 1982). To achieve the desired visual characteristics, inks need to have varying degrees of opacity and transparency (Noguchi, 2000). This can be accomplished by the choice of colorant and its degree of dispersion in the ink (Bermel, 1999). Different colorants behave differently towards light. The degree of reflectance, absorption and a colorant's refractive index determine its opacity and transparency (William, 1985). The properties of both color strength and opacity are determined by particle size.

Scattering increases with the pigment particle size increase, until the wavelength of incident light is reached. At higher pigment particle size, the opacity decreases again. Thus, the opacity of an ink pigment is maximum around 350-500 nm. If scattering is so intense that no light passes through the material, the ink is opaque. The smaller the particles in the ink, the more transparent the ink is. The transparent inks require smaller particles than opaque inks. Color strength increases with decreasing particle size because of the larger surface area and better bonding among the particles (Ferguson, 1992).

A pigment hides or tints by means of its ability to scatter and absorb light. The ability to scatter light depends upon several factors, but the primary one for pigments is index of refraction (n). The ability to absorb light depends upon the pigment’s absorption coefficient (k). For colored pigments, light absorption is more important than light scattering in developing opacity, although the latter is not negligible. Consequently, the primary property for colored pigments is the complex index of refraction, m and $m = (n-i*k)$; (Patton, 1973), as a function of wavelength and particle size.

For colored pigments, the optimum particle size for hiding power is larger than that required for optimum tinting strength since the former property involves both scattering and absorption, whereas the latter is primarily due to absorption. Also, the hiding power and tinting strength are dependent upon such other factors as the particle size distribution, particle shape, and degree of dispersions (Parikh, 1982).

Methods for Particle Size Measurements

There are many methods available for determination of the particle size and particle size distribution. In general, these techniques can be divided into three basic categories: ensemble methods, counting methods, and separation methods (Lines, 1991). An ensemble method takes a snap shot of a sample and then elucidates the distribution of particle sizes based on the physical properties revealed by the measurement. A counting method characterizes the sample one particle at a time in a more direct manner. The accumulative account of the particles provides the size distribution information. A separation method, on the other hand, first physically separates the samples according to their particle size. Then, various techniques are applied to determine the relative amount of each fraction. Each technique measures a particular dimension that is dependent on the measurement principle.

In the printing ink industry, where size is an important issue both in the terms of cost and quality, the methods of choice are mainly ensemble methods because of their speed, reproducibility, and accuracy. These are some steps away from direct physical interactions (e.g., the sieve with the particles) or arduous, intensive observation (e.g., microscopy), and instead they use fundamental scientific principles to determine the particle size.

Inks and Deinkability

The deinkability of printed (waste) papers depends on many factors, notably the substrate (coated vs. uncoated papers), the ink formulation (hydrophilic vs. hydrophobic pigments, polar vs. nonpolar binders or resins, alkali solubility vs. alkali resistance, wettability, and particle size), the printing history (oxidative drying, age of print), and the printing process (letterpress, offset, gravure, flexography). Most coated papers are readily deinkable because the ink particles are detached with the coating. The deinkability of uncoated papers depends on their ink-absorption rate and depth. Deinking involves pulping in an alkaline environment to detach the ink from the paper, and separating the ink particles by screening, cleaning, flotation, and washing, depending on particle size (Thompson R.C., 1998). Ink vehicles comprising mineral oils and hydrocarbon resins can be satisfactorily deinked. Water based flexo and gravure inks pose difficulties (the particle size produced by pulping being too small for flotation) resulting in insufficient brightness deinked pulp (Galland, 1997). Adding a neutral or acid during repulping precipitates the acrylic ink resin in flotatable sized particles. Ultraviolet curing inks and coatings form very tough, crosslinked films, not entirely removed by screening and cleaning. UV varnished printed waste paper produces large ink particles that are insufficiently removed during the recycling process, leading to specks in the deinked pulp (Galland, 1997). They can be broken down to flotatable size by ultrasonic cavitation. Office waste contains non-impact printed material, from photocopiers, laser printers, and digital presses. Modern flotation cells are able of efficient removal of printing ink, stickies, plastics, fillers and other hydrophobic contaminants in a wide particle size range of 5 to 500 micrometres (Britz, 1998).

Due to their disseminated environmental advantages, water based inks have gained increased acceptance, having found a special niche in flexography (Pelach, 2002). For many years, flexography has remained as a technology to print flexible and rigid packaging products. More recently, flexography has expanded into magazine printing. Water-based flexographic newspapers cause brightness losses in the deinked pulp of more than 20% as compared to offset newspapers. As is widely known today, the poor deinkability of water-based inks is caused by the small size and hydrophilic behavior of their particles after ink detachment (Hanecker, 2000). A considerable amount of research has been devoted to developing and modifying suitable deinking chemicals, and to introducing further stages into the deinking process. Ink manufacturers have made efforts to develop deinkable water-based flexographic inks. One solution to this problem was to increase flotation by reducing the pulping time and increasing the hardness of the medium, but the drawback was a worsening of the effect of washing. Now deinking plants and the water treatment stations are adapting themselves to cope with the increased amounts of water-based inks. One of the alternatives to reduce the greater water contamination caused by water soluble inks is to add microfiltration and even ultrafiltration.

The deinkability of offset-printed paper decreases with the age of the printed product. This effect depends on the content of binder components that dry by oxidation. Alkyd resins in particular cause crosslinking in the ink film, thus rendering the removal of ink particles more difficult. In offset-printed newspapers, this deinkability variation is most relevant during the first few weeks after printing (Grossmann, 1992).

Model studies were performed on the floatability of an offset ink suspension (without fibres), using a Hallimond tube and a standard calcium chloride-sodium oleate collector system (Johansson, 1999). The average ink particle size was determined as 1 μm, indicating poor floatability of untreated primary ink particles. This suspension was mixed with deinking chemicals, coating components and dissolved paper chemicals and the results combined with data on agglomeration kinetics. Findings confirmed that the agglomeration of small ink particles fully controls their floatability. Parameters that increase the agglomeration rate increase the flotation efficiency, while surface-active chemicals that stabilize ink particles decrease flotation efficiency. Apparently, some particles neither do agglomerate nor flotate (Johansson, 1999).

The deinkability of recovered paper describes the printing ink removal by a deinking process that is measured first by the improvement of optical parameters (Gottsching, 2000). These can include luminance and brightness of the undeinked and deinked pulp. Besides the printing ink, the printing process, the print substrate, and the aging of the printed product, the conditions and the parameters of the deinking process in terms of the chemical, mechanical and hydraulic regimes influence the deinkability of printed material. When disintegration conditions of waste-paper are different, flotation steps behave differently, mainly due to the ink particle size distribution and the physicochemical environment. An alkaline environment promotes an increase in the electrostatic and dispersive energy of bubble-ink particle interactions, decreasing the flotation removal efficiency. Particles smaller than 15 µm are usually not removed from the wastepaper suspension (Pelach, 2002).

The aim of this work was to describe some of the relationships between ink particle size and their deinkability.

EXPERIMENTAL

Inks

The commercial water-based and solvent-based (toluene) process colors, cyan, magenta, yellow and black and solvent-based (isopropylacetate) spot colors, produced for rotogravure printing process, and water-based flexo inks were used for determination of the particle size. Also, the particle size of extenders (polymers) was measured.

Gravure Printing

A Cerutti pilot-plant rotogravure web printing press (Cerutti Model 118, Casale Monferrato, Italy) was used to print test samples. Four color process was running at the speed of 305 m/min (1000ft/min). The inks selected were commercial toluene – based inks. Their efflux time (“printing viscosity”) was 20 s on a Shell #2 efflux cup for yellow, magenta and cyan inks and 18 s for black ink. Solids content of inks was 34. 0 % at 20 s efflux time and 32.8 % at 18 s efflux time. Oven dryers were set to 60 ^{o}C at 9000 cfm nozzle velocity. Electrostatic assist (ESA) was applied at 4 kV and 1.4 mA. Impression pressure was 125 psi at 3/8 nip flat with an 85 durometer (Shore A) roller.

Flexo Printing

Comco Comander 3-color web flexo press was employed for printing web width 10" at 700 ft/min speed. Conventional hot air dryers were set up to 105 ^{o}C. Anilox rolls 550 lpi were used for yellow and magenta print stations with 175 lpi plates. Cyan anilox had 350 lpi. Two different water based ink systems from different manufacturers were used to determine how the ink quality affects the show-through. The inks are referred to as “A” and “B”. The viscosity of inks was measured as “efflux time” using a Zahn #2 cup. All the inks were adjusted to efflux time of 28 seconds which corresponds to 62 cP according to the Dietzgen conversion chart (Dietzgen Co., U.S.A.).

Substrates

Commercially manufactured publication grades - light weight coated (LWC), and coated freesheet stock were used for printing.

Particle Size Analysis

The Particle Sizer Submicron 370 NICOMP based on DLS (dynamic light scattering) was employed in this experiment.

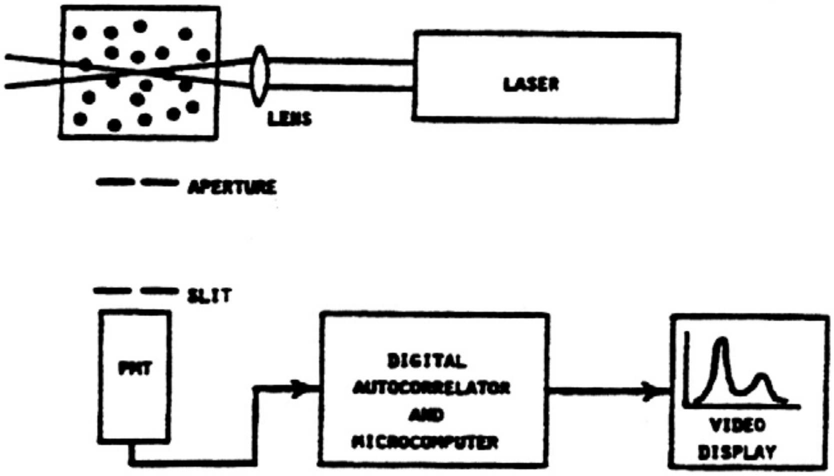

Figure 1: Simplified Block diagram – NICOMP DLS Instrument

The operating principle of dynamic light scattering is illustrated in the **Fig. 1**. Light from a laser is focused into glass tube or cuvette containing a diluted suspension of particles. The temperature of this scattering cell is held constant, for reasons, which will soon become apparent. Each of the particles illuminated by the incident laser beam scatters light in all directions. The intensity of light scattered by a single, isolated particle depends on its molecular weight and overall size and shape, and also difference in refractive indices of the particle and the surrounding solvent. The incident light wave can be thought of as consisting of a very rapidly oscillating electric field, of amplitude E_o. The arrival of this alternating field in the vicinity of a particle causes all of the electrons, which are free to be influenced (polarizable electrons), to oscillate at the same frequency. These oscillating electrons give rise to a new oscillating electric field, which radiates in all directions as the scattered light wave. The quantity of interest in a scattering measurement is the intensity of the scattered wave, I_s, rather than amplitude, E_s. The intensity is given by the square of the amplitude: $Is = (E_s)^2$.

The dependence of the scattered light intensity I_s on the molecular weight (MW) or volume (V) of the particle is particularly simple when the particle diameter is much smaller than the laser wavelength λ (Rayleigh region). In this case, all of polarizable electrons within a particle oscillate together in phase, because at any given time they all experience the same incident electric field. Hence, the scattered wave amplitude E_s is simply proportional to the number of polarizable electrons, times the incident wave amplitude, E_o. The former quantity is essentially proportional to the overall molecular weight of the particle or its volume. The constants of proportionality, which connect these various physical quantities, depend on the indices of refraction of the particle n_p and solvent n_o. That is, how well a given particle scatters light depends not only on MW or V but also on the polarizability of the particle (related to n_p) relative to that of the solvent (related to n_o). For the very small particles in the Rayleigh region, we arrive at simple expression for the scattered intensity I_s:

$Is = f\ (n_p,n_s)*(MW)^2*I_o$
$Is = g\ (n_p,n_s)*V^2*\ I_o$

where: I_o is the incident laser intensity, and g (n_p,n_s) are functions of the indices of refraction of the particle and solvent, which are fixed for a given system composition. For these small particles in the Rayleigh region, there is negligible angular dependence in the scattered intensity T. (5).

After setting up the appropriate conditions, different for water-based inks and for the solvent (toluene)-based inks, the particle size of each sample was measured.

- **Refractive index**: 1.333 (water), 1.494 (toluene), 1.3728 (isopropylacetate)

This parameter establishes the index of refraction of the solvent, in which the particles are suspended, assuming a dilute dispersion. The inks particle sizes were measured in this case using water and toluene as solvents.

- **Viscosity**: 1.002 cP (water), 0.590 cP (toluene), 0.53 cP (isopropylacetate)

The viscosity of the sample suspension is expressed in units of centipoises (cP). The particle suspension must be very dilute for measurements based on dynamic light scattering, in order to avoid errors due to interparticle interactions and multiple scattering. Therefore, the viscosity is by default as a viscosity of the pure solvent in which the sample particles are suspended.

- **Intensity**: 200-300 kHz

The average scattered intensity or photopulse rate, expressed in kHz, which is desired for a measurement, can be established by setting this parameter. The default value is set 200-300 kHz. This value is typically recommended for most samples, which scatter adequately. It is designed to optimize the efficiency of the autocorrelation process and thereby minimize the time needed to obtain reliable, accurate results for most samples.

- **Temperature**: 20°C

After measurements, the results obtained from NICOMP® Particle Sizing Systems - CW380 Version 1.51a Software were evaluated.

- **Calibration:**

Prior to actual inks measurement, the instrument was calibrated using following particle size
20.00±0.10μm (Duke Scientific Co.).

Deinking

The samples (pieces ca 5x5 cm) were soaked in the water (50 °C) for 10 min. Samples were put into a laboratory mixer (Waring Model 47 BL84 CB6 Heavy Duty Blender). The repulping was done at a temperature of 50 °C at 2.5% consistency at the lowest RPM. After 1 minute of repulping the pH was adjusted with NaOH to the value of pH = 10.0. The repulping was continued for 4 minutes at the same pH. The final temperature was 50 °C, due to the heat generation during the agitation process.

The flotation cell (Adirondack, Format ™ Flotation Deinking Cell, 29.1 L) was used at a consistency of pulp slurry of approximately 0.7%. The temperature was adjusted with direct steam to 40°C. The slurry circulated at 57 L/min. The feed sample was taken. Non-ionic surfactant was added (Lionsurf 729, 0.125% on dry solids) and the slurry was allowed to circulate for 5 minutes. After 5 minutes of circulation, the airflow was opened with the rate of 225 L/min. The foam from the center of the flotation cell was collected and weighed. After 10 minutes of flotation, the air input was closed and the accept sample was taken.

Washing was done in TAPPI handsheet mold at laboratory temperature with the same concentration of nonionic surfactant (Lionsurf 729, 0.125% on dry solids). Water to pulp ratio was 3500:1. Pulp slurry was washed for four times.

Analysis of Deinked Pulp

Handsheets from slurry before and after flotation were made in accordance to TAPPI Standard T 205. Brightness was measured in accordance to TAPPI Standard T 452. Rejects were not diluted. The pads from rejects were formed by filtering the samples (cca 75 mL) using a Buchner funnel (Whatman 42 Ashless Filter Paper). The solids were determined by drying the samples in a laboratory CEM Lab Wave 900 microwave.

RESULTS AND DISCUSSION

The particle size of rotogravure publication printing inks formulated for the coated (**Fig. 2**) and uncoated substrates (**Fig. 3**) was compared. For the coated gravure inks, the particle size was found in the range from 245 nm (process

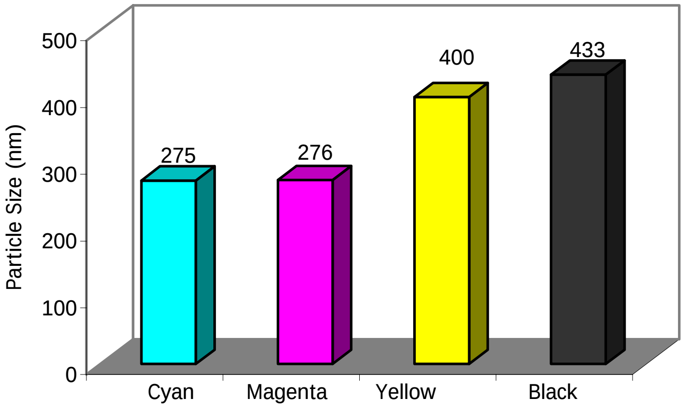

Figure 2: The Particle Size of Gravure Coated Inks

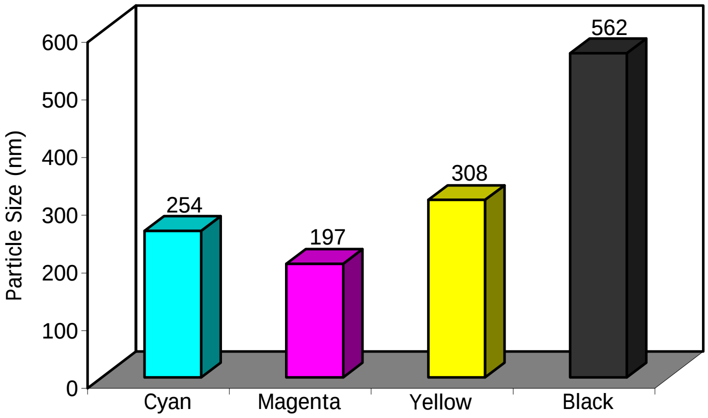

Figure 3: The Particle Size of Gravure Uncoated Inks

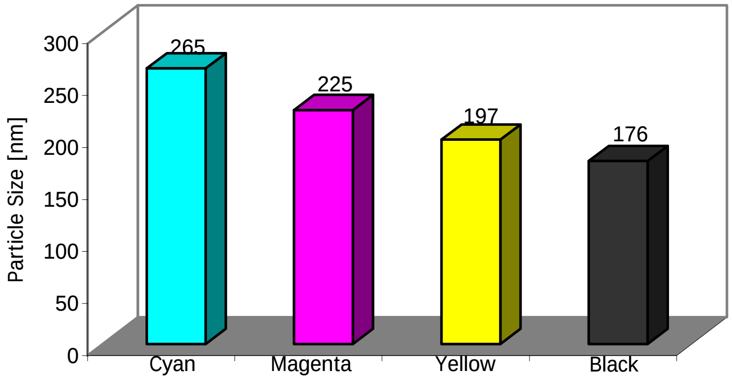

Figure 4: The Particle Size of Water-Based Flexographic Inks

cyan) to 433 nm (process black). The uncoated inks exhibited smaller particles than coated inks (**Fig. 3**). The average value of the particle size of gravure extender was around 179 nm. For both gravure inks, the agglomeration was found only in the process black color ink. Both coated and uncoated process black inks had the highest particle size of all gravure process inks. Unusual particle size of 1900 nm was present in amounts of 3.5 % in gravure uncoated ink and 1100 nm in 20 % of gravure uncoated process black ink.

Water-based flexographic inks exhibited the smallest particles compared to all other fluid inks (**Fig.4**). The particle size was found in the interval from 176 nm for process black color to 265 nm for process cyan (**Fig.4**). No presence of agglomerates or flocculates were observed.

The process inks were used for rotogravure and flexo printing. Flotation deinking was done for each process color solid print separately to find out whether there exists any correlation between particle size of liquid ink and printed ink removal in the flotation deinking process. The deinking was done within four weeks from printing and no testing of ink aging on deinking was studied at this point.

The color difference ΔE was calculated from CIE L*a*b* values measured on handsheets made from slushed fibers before and after flotation **(Fig.5)**. Calculation was done according to equation [1]:

$$\Delta E = \sqrt{\left(L_2^* - L_1^*\right)^2 + \left(a_2^* - a_1^*\right)^2 + \left(b_2^* - b_1^*\right)^2} \qquad [1]$$

where:

L_1^*, a_1^*, b_1^* – CIELAB coordinates of color of handsheets after flotation and

L_2^*, a_2^*, b_2^* – CIELAB coordinates of color of handsheets before flotation

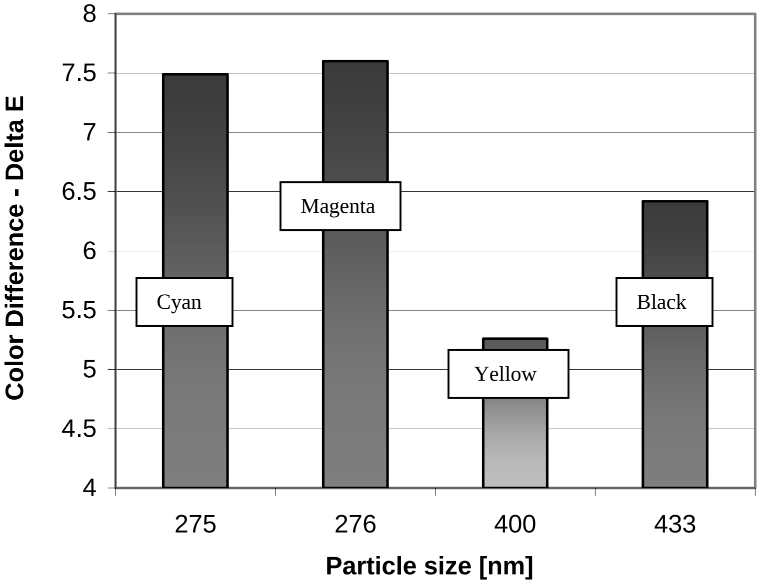

Figure 5: Particle size of coated publication gravure (toluene-based) inks and their flotation deinkability (Control: Color difference for unprinted paper was ΔE = 0.64)

For the gravure printing deinkability, the largest color difference, thus flotation deinkability, was found for magenta ink (ΔE= 7.60), followed by cyan (ΔE=7.49). These inks had quite similar particle size (275.0 nm magenta, 274.7nm cyan). The lowest ΔE (ΔE = 5.26) exhibited yellow gravure ink with particle size 400 nm **(Fig. 5)**. Slightly better flotation deinkability than yellow was found at black gravure ink (particle size 433 nm and ΔE = 6.42). The flotation deinkability in this experiment slightly increases with decreasing ink particle size of ink in the fluid state. A similar trend was found at water based flexo inks- deinkability slightly increased with decreasing particle size of fluid ink (**Fig. 8**).

Washing deinking was less efficient for gravure inks than flotation **(Fig. 6).** Two- step washing was done with the same nonionic surfactant Lionsurf 729 as used in the flotation experiment. Color difference ΔE for first wash was from 1.51 to 3.1- lowest for black ink with the largest particle size, ΔE =1.51, and largest for cyan, ΔE = 3.1. Cyan had the smallest particle size from all gravure inks. Extensive washing resulted in further ink removal, largest ΔE drop was found at magenta print ΔE = 7.02, followed by cyan ΔE = 6.57 and black ΔE = 5.06. The worst washing efficiency was found at yellow print ΔE = 2.57 **(Fig. 6)**. Brightness and color of nonprinted paper was degraded due to removal of high brightness coating colors, which left darker mechanical fibers (Data not shown).

Deinkability of individual gravure inks by combination of flotation and washing was also done **(Fig.7)**. The resulting color difference for combination treatment was higher than that found at individual steps. However, it was lower than the cumulative effect of individual flotation and washing steps. ΔE from 10.91 (cyan) to 5.45 (yellow) was found. Obviously, the deinkability increased with decreasing particle size of fluid gravure ink, but there are probably other effects equally important, such as effects of individual functional groups on pigment molecule, ionization, hydrophobicity or hydrophilicity, causing poor deinkability of yellow gravure print.

Flotation deinkability of water- based flexo inks **(Fig. 8)** was worse than that of toluene-based gravure inks. This was expected because of known problems with flotation of hydrophilic water-based flexo acrylic resin chemistry. Flotation deinkability was best for yellow ink and worst for cyan inks, decreasing with increasing particle size of fluid inks.

Table 1: Reject after flotation deinking at gravure and flexo print

Reject	**Gravure [g]**	**Flexo [g]**
Magenta	64.6	52.7
Yellow	63.2	54.2
Cyan	68.2	51.4
Black	65.6	N/A
Control	61.2	50.6

However, the comparison of flotation deinkability for gravure and flexo process is difficult to make, because of very different chemistry of resins for both systems. The difficulties with deinkability of hydrophilic flexo resins were reported earlier [Gottsching, 2000]. The gravure LWC substrate was behaving differently during flotation deinking than the flexo substrate (Coated freesheet), which is demonstrated also by a higher amount of reject in control flotation for nonprinted gravure than for nonprinted flexo sheet **(Tab.1)**. Reject amount in gravure print deinking was largest for cyan ink and smallest for yellow. This order also corresponds to color difference of deinked sheet for these colors. Reject amount in flexo print deinking **(Tab.1)** was largest for yellow print, which corresponds to largest color difference, thus deinkability, found for yellow ink. Smallest reject was found for cyan flotation deinking, which corresponds well to smallest color difference found for cyan ink.

Efficiency of washing deinkability **(Fig. 9)** of flexo water based inks was higher than its flotation deinkability and higher than washing deinkability of gravure inks. The color difference from ΔE = 6.8 (Yellow) to ΔE = 2.9 (Cyan) was achieved after first wash. Extended washing resulted in color differences ΔE= 14.3 (Yellow) to ΔE = 7.2 (Cyan). Washing efficiency of water based flexo inks decreased with the increasing particle size of all fluid inks tested **(Fig. 9)**.

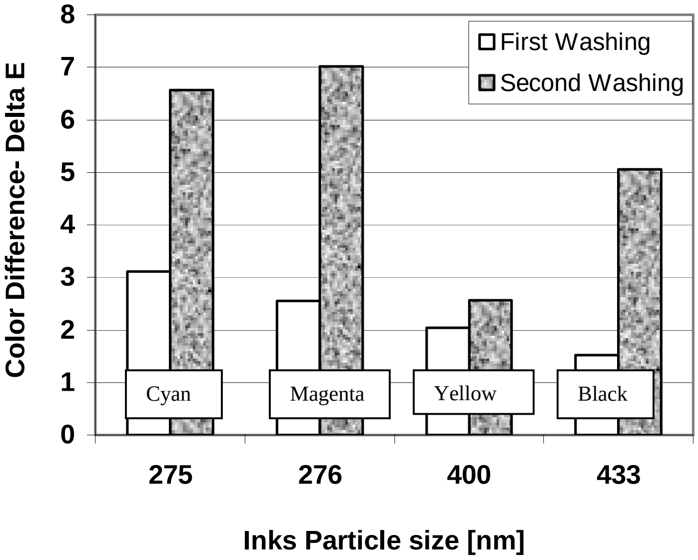

Figure 6: Deinkability of publication gravure inks by washing.
Control: Unprinted paper color difference was ΔE = 4.32

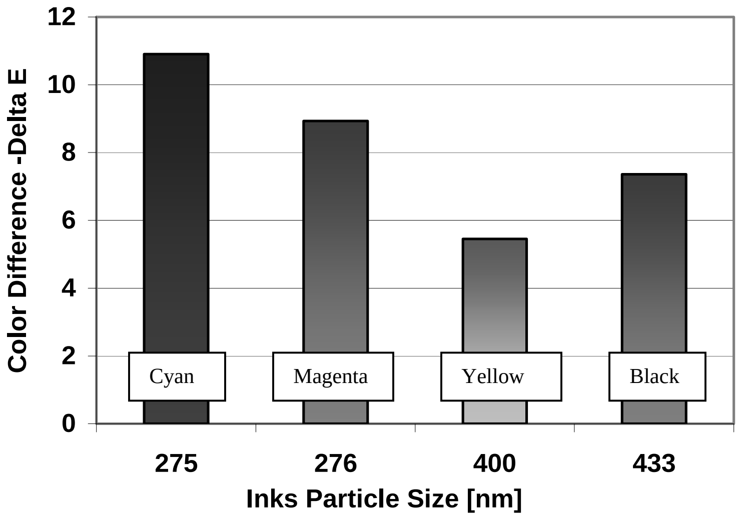

Figure 7: Deinkability of publication gravure inks by combination of flotation and washing. Control- unprinted paper color difference was ΔE = 6.69

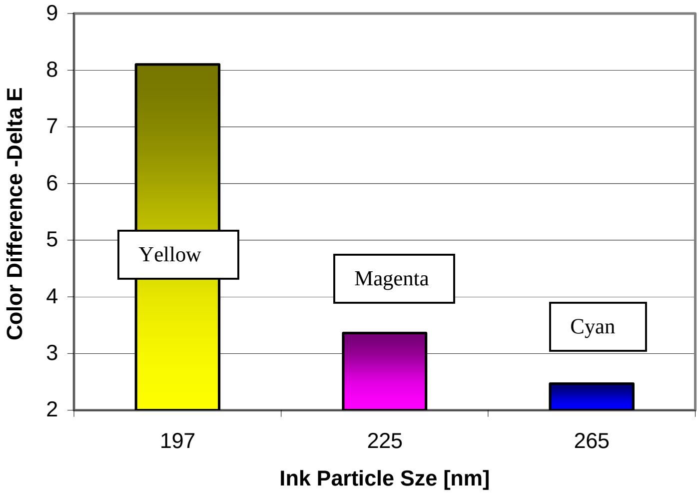

Figure 8: Flotation deinkability and particle size of water based flexo inks (Control - Color difference for unprinted paper was ΔE=1.43)

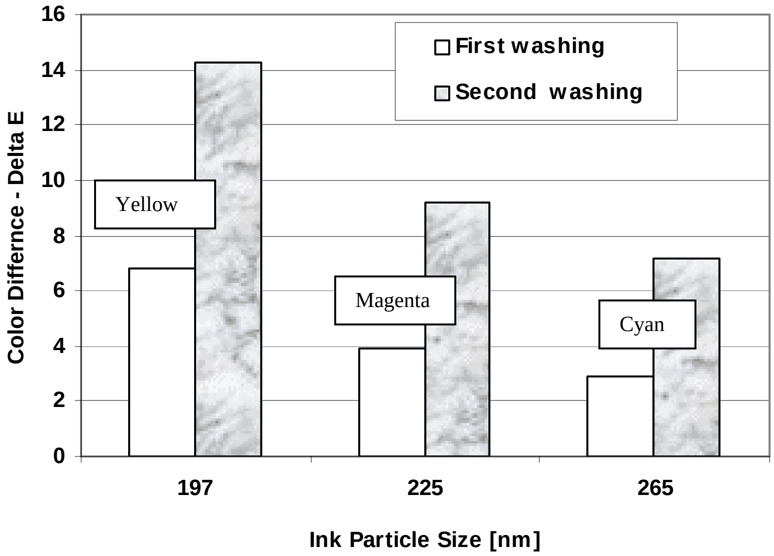

Figure 9: Washing deinkability of water based flexo inks. Color difference for unprinted paper was ΔE = 2.31 for first washing and ΔE = 3.10 for second washing.

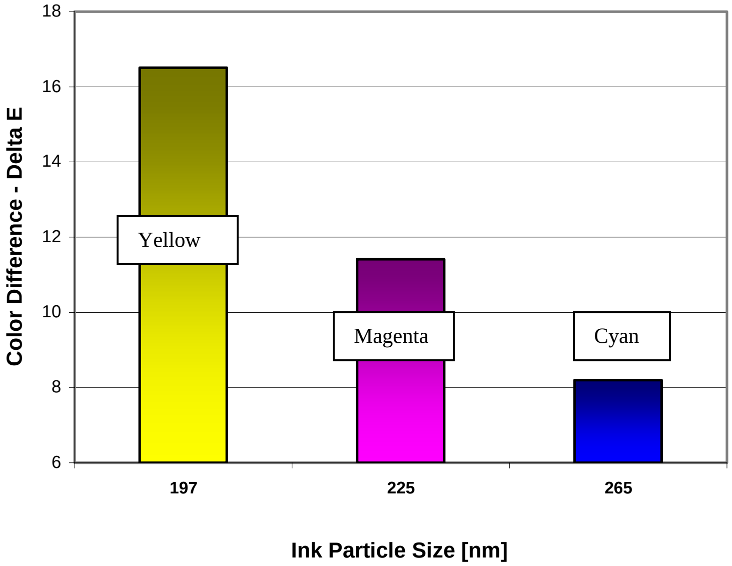

Figure 10: Combination effect of flotation deinking and washing on deinkability Control- unprinted paper color difference was ΔE= 3.10

Combination of flotation and washing deinkability of water based flexo inks **(Fig. 10)** was better than single flotation or single washing steps. However, similarly as in the combination of flotation and washing at the gravure print, deinkability of flexo print was lower than cumulative effect of individual flotation and washing steps. The highest deinkability measured as color difference $\Delta E=16.5$ was found at yellow print, the smallest $\Delta E=8.2$ was measured at cyan ink. Also here applies that the decreasing particle size of fluid ink improves deinkability in combined flotation and washing process.

CONCLUSION

Particle size of commercial fluid ink publication gravure coated and uncoated as well as flexo water based inks was measured. Flexo inks exhibited smaller particle size than gravure inks. The uncoated gravure inks exhibited smaller particles than coated inks. Deinkability of gravure solvent based and flexo water based inks in the relationship to the particle size of fluid inks was studied. Washing deinkability was improving with the decreasing particle size of both flexo water based and gravure solvent based inks. Flotation deinkability was also improving with decreasing fluid ink particle size for both flexo and gravure process. From these results is obvious that the particle size of individual fluid inks affects the deinkability of printed products.

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