Visualizing fountain solution droplets in an ink vehicle-fountain solution emulsion by confocal microscopy and 3D image reconstruction

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Keywords: Offset ink, ink-fountain solution emulsion, confocal microscopy, 3D image reconstruction.

Abstract: Using a confocal microscope, a suitable fluorescent dye and 3D reconstruction software, 3D images were produced of fountain solution droplets in an ink vehicle. These 3D images showed the shape of emulsion droplets and they were analyzed to produce the average drop volume and the drop size distribution.

The technique developed was used to demonstrate the effect of fountain solution concentration, shear rate and instruments used for emulsification on the characteristics of the droplets. Problems associated with the technique are considered and potential applications of the technique in future studies discussed.

Introduction

During conventional lithographic printing, a significant amount of fountain solution (FS) is added to the press. The FS (sometimes is simply known as water) plays a critical role in maintaining trouble free press running [1, 2]. Most FS added to the press is evaporated by heat from the rolls and plate; a small amount is transferred to the substrate in the form of “surface water”, while the rest is emulsified into the ink by the shear force between the plate and ink rolls and forms a water-in-oil (W/O) emulsion [3, 4].

The way in which water and ink interact determines the success of lithographic printing. Generally the well-performed inks show a wider "FS uptake window". The wider the window, the greater is the range in the amount of FS the ink can handle effectively (under different printing conditions), while not varying too much in tack and other transfer properties. In contrast, inks that have poor press runnability often show a poor ink/water balance [5]. Various laboratory tests therefore have been designed to measure the water take-up behavior of inks in order to predict their performance on the press [5, 6]. Many factors such as the surface tension of ink and FS, ink composition, press configuration and temperature, are found to influence the performance of an ink on the press [2].

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It is generally believed that the ink/water balance is closely related to the formation and properties of the ink emulsion. Studying the ink emulsion is seen as a key to unlocking the underlying mechanism of "why the ink/water balance controls press performance". This topic has therefore attracted much attention from researchers [2]. Some studies have linked the variables in the pressroom with the appearance and properties of the ink emulsion. Others have connected the characteristics of the emulsion with the press runnability and print quality. Thus, the importance of faithfully describing the appearance and properties of ink emulsions cannot be over emphasized.

We have developed a 2D technique to visualize fountain solution droplets in a lithographic ink vehicle using the confocal laser scanning microscope (CLSM) [7]. A number of other methods have also been developed by other researchers to characterize ink emulsions and these have been reviewed in the first part of our investigation [7]. It was concluded that none of these methods could provide a faithful visual observation of emulsion droplets in their natural states and at the same time provide quantitative drop size distribution information on the emulsion. The motivation for this research is to further develop the 2D method into a more powerful 3D method for studying FS droplet distribution in an ink-FS emulsion and to apply this method to investigating a wider range of ink-plate and ink-paper interactions in lithographic printing.

Experimental

Chemical reagents and materials

A pigmentless ink-vehicle was selected for this work. This ink vehicle was made using a commercial cold set black news ink formulation by Sicpa Australia; it had a strong dark brown colour. The only component not in this ink vehicle was the carbon black pigment. The reason for using ink vehicle was to enable the confocal microscopy to be employed to observe the FS droplets. An ink with pigment would strongly attenuate the fluorescent light emitted from the FS droplets (see below).

FS concentrate (EuroFount – N) was obtained from DS Chemport and was diluted to 2% (v/v) with MilliQ water. The measured pH of the diluted FS was 5.9 and its conductivity was 1000 μs/cm.

Relying on a pinhole to cut off unfocused light, the confocal microscope is capable of optically sectioning a specimen in a physically non-destructive way. The best way to visualize the 3D structure of a translucent object is to stain it with a fluorescent dye. The dye must absorb at the wavelength of the confocal microscope laser. A filter is then used to allow the fluorescent light to pass and reach the detector, but block the scattered light at the laser wavelength.

When using the confocal technique to study the FS droplets in an ink emulsion, a further constraint on the selection of dye to be used for the fountain solution must be overcome. The dye must be very hydrophilic so that it does not dissolve in an oil phase and emitting fluorescent light from there. The dye selected, which almost meet these criteria, was a strongly water soluble dye, 8-hydroxypyrene-1,3,6-trisulfonic acid

(HPTS, pyranine). It was supplied by Molecular Probes and was found in the first part of our study to be suitable for the purpose [8]. Its molecular structure is shown below.

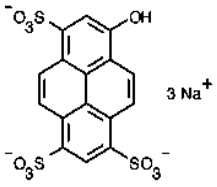


Figure 1 (a). The molecular structure of 8-hydroxypyrene-1,3,6-trisulfonic acid (HPTS, pyranine; H-348).

The tri-sulphonate groups render the molecule strongly hydrophilic, confining the dye almost exclusively to the water phase. A limitation of HPTS is that its absorbance spectrum is pH dependent, with absorbance being very low in the acidic FS at 488nm, the wavelength of the Argon ion laser, (Fig 1). Little fluorescent light could be detected with a standard HPTS concentration. Instead of adjusting the pH value of the FS, which would have affected the formation of the emulsion, the HPTS concentration was increased to 0.2% (w/w) in order to increase the intensity of the fluorescence to an acceptable level.

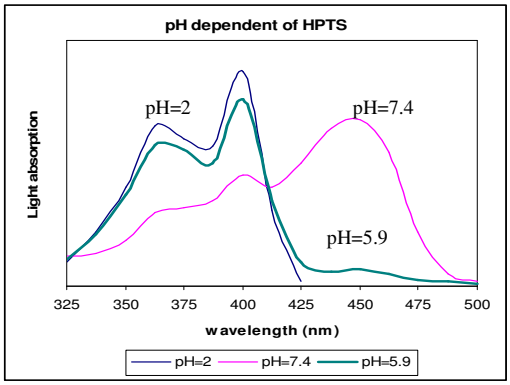


Figure 1 (b). The pH-dependent absorption spectra of HPTS [8].

The optical properties of HPTS are given in Table 1.

MW*	As*** (nm)		Ec**	Em***(nm)
	pH=2	pH=7.4		
524.37	403	454	20,000	511

Table 1. The optical characteristic of HPTS [8]

* Molecular weight; ** Emission coefficient; *** Approximate absorbance and fluorescence emission maxima, in nm.

Sample preparation and image processing

The FS-ink vehicle emulsion samples were prepared using two different instruments, a high speed Dispermat CV manufactured by VMA-Getzmann, and , a roller mixer, a Hydroscope from TestPrint. With the Dispermat, 40ml of ink vehicle was pre-weighed into a 125ml water-cool, double-walled stainless steel container and the container was clamped firmly under the rotor. The temperature of the inner wall of the container was maintained at 25 ^{o}C with water supplied from a thermostat water bath. At 300 rpm impeller speed, a pre-determined amount of FS containing HPTS dye was trickled into the ink vehicle at a rate of 4ml/min. After all the fountain solution had been added, the rotation speed was increased gradually to the pre-determined level and was kept at this speed for 10 minutes. This procedure was recommended by the Dispermat CV user's manual for general ink/paint mixing application [9].

When the Hydroscope was used to generate the emulsion, the system was pre-conditioned with 25 ºC circulating water. Ten grams of ink vehicle was delivered to the rollers and was distributed evenly on the roller surface by rotating them at slow and constant speed. The roller speed was then increased to 40 m/min and FS was introduced to the nip by two nozzles, either by delivering an aliquot of the required FS in a single addition, or by slowly adding the same amount of the FS into the nip at a constant rate of 0.5ml/min.

After preparation, all FS-ink vehicle emulsion samples were transferred to different containers and kept still for 48 hours to let entrapped air bubbles rise to the surface. This is not essential for obtaining the 3D images, as the method can distinguish air bubbles from water droplets [7]. However, removing air bubble is helpful in reducing the scattering loss of the fluorescent signals and is therefore critical for obtaining clear image and facilitating the subsequent measurements.

A small amount of emulsion was taken from the centre of the sample and was transferred into a specially designed microscope container. The design of the container is illustrated in Figure 2. The dimensions of the blind hole were carefully selected so that it was large enough to contain sufficient emulsion sample to avoid sampling error, and at the same time small enough to prevent any undesired movement of the dispersed phase which may be generated by the high magnification objective lens. In other words, the major benefit of this container is to maintain the emulsion sample in its natural state whilst under microscope observation.

Figure 2. A schematic of the microscope container used for observing the emulsion under the confocal microscope

The ink emulsion was held in the microscope container for about 10 to 15 minutes to allow trapped air bubbles to float to the surface. The excess emulsion was then scraped

off carefully and a cover glass was placed firmly over the blind hole. The sample was then examined under the microscope.

A two-channel Optiscan F900e confocal laser scanning microscope, incorporating an Olympus BX50 microscope fitted with a 60X/1.40 NA oil immersion lens, was used to acquire confocal images of the emulsion. This microscope is attached to a laser launch and detection unit (LDU) that supplies 488 nm Argon-Ion laser light at variable intensity to the microscope through a 450 nm acrylate coated optical fiber. This fiber is also used to transmit the fluorescent light back to the LDU.

The light returning from the sample passes through a 515 nm long pass filter which removes the scattered laser light and then the intensity of the remaining fluorescent light is measured by a photo- multiplier tube (PMT). The PMT converts light into electrical signals and, with scanning of the laser beam across the observed region of the sample, these signals are translated into a live image by the electronics in the master control unit (MCU). The live images are captured by the computer work station through an image grabber card and are stored in the work station as a tiff image file.

Image Pro Plus version 5.0 (developed by Media Cybernetics), with the 3-D Constructor plug-in installed, was used for 3-D reconstruction and image analysis.

Results and Discussions

Reconstructing 3D emulsion images

The selection of appropriate settings for the confocal microscope during image acquisition is critical for the quality and accuracy of the 3D images. Three critical settings are the z-direction step size between images, the gain level of the photo multiplier tube and the black level of the live images.

In order to collect adequate information to reconstruct accurately all the details of objects, the step size (i.e. the z-direction distance between two subsequent images) must be set small to avoid affecting the z-direction resolution. If the step size is chosen appropriately, the resolution of the optical system, principally the objective lens as given by its NA value, is likely to limit the resolution of the system [10]. The step size used in this experiment was 0.17 μm.

The gain level of the detector circuitry is changed by adjusting the accelerating voltage of the PMT [10]. A higher gain level generates a brighter image. When it is properly adjusted, the pixels in the brightest areas of the image should be given the maximum gray scale value, which is white. This ensures the use of the full gray scale range of the data handling system. However, one must also be careful not to saturate the live image, as this will have the effect of enlarging the size of the image area. If the gain level is set too low, low intensity image areas will not be clearly distinguished from the dark background, particularly if they are small, resulting in tiny objects being missed. The gain level should be adjusted in conjunction with the laser power to obtain maximum contrast in the image, whilst keeping the laser light intensity below the level which would cause photo bleaching of the fluorescent dye.

The black level of the PMT amplifier should be adjusted so that the electronic signal caused by dark current from the PMT is eliminated. In practice the black level can be set by blocking all light from entering the PMT and adjusting the black level until the scanned area becomes just invisible against the unused part of the monitor screen. The black level should not be set too high to avoid eliminating weak signals generated from the low intensity image areas. If the image on the display has insufficient contrast, image processing software could be used to enhance the contrast.

Contrast enhancement and the application of various filter functions, such as a Median or Gaussian filter, using image software, are very useful for enhancing images. They are widely used to enhance biological images where no accurate measurements are required from analysis of the images. Image enhancement works by manipulating the gray scale values of pixels of an image by such operations as setting a threshold below which the grey scale values are all set to zero, or applying a factor which is a function of the gray scale value. These operations will normally change the appearance of the objects, modify their size and therefore change the measured dimensions. Thus, they should be used with caution if measurements to be made. The best possible 2D images should be obtained from the confocal microscope using minimal image enhancement before using software to reconstruct the 3D structure and obtain quantitative information on the emulsion.

After obtaining a satisfactory 2D images sequence, the reconstruction of 3D images of the emulsion was carried out using the software, '3D Constructor'. One parameter that needs to be carefully selected is the level of sub-sampling. As the name implies, it is a technique used by the software to reduce the amount of data to be manipulated in reconstructing the 3D structure, in order to reduce computing time. Since there are many tiny droplets that may be represented by only a few pixels, it would be ideal if there was no sub-sampling, especially when accurate measurements are required. Unfortunately, due to the large size of the file (around several hundred megabyte) and the capacity of the computers being used, a small degree of sub-sampling was required.

Performing calculations on 3D images

The 3D images reconstructed by '3D Constructor' from the sequence of images acquired by the confocal microscope are only a step away from the derivation of useful data. The FS content in the emulsion, the average FS droplet size, and the size distribution should be easily obtainable, provided that suitable parameters are given to instruct the software. Unfortunately, '3D Constructor' is not specifically designed for analyzing a multitude of tiny objects such as FS droplets in the ink vehicle emulsion. Therefore, even with a proper selection of the parameters, there are still some uncertainties about the quantitative results.

If the calculation errors are minimized, these sorts of measurements will provide strong support for studies related to emulsion properties, and be a convincing tool for checking measurements made using other methods [7]. The images of the 3D structure

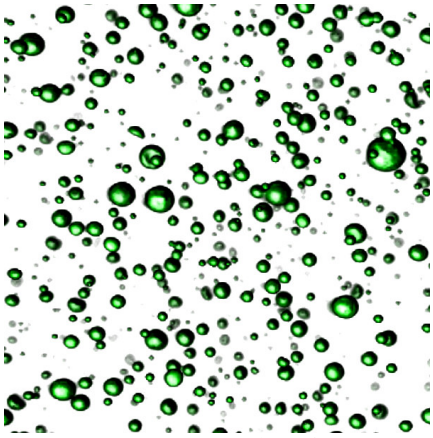
not only give the measured data, but also the shape of the droplets, which is not accessible with any other method.

Applications of the method

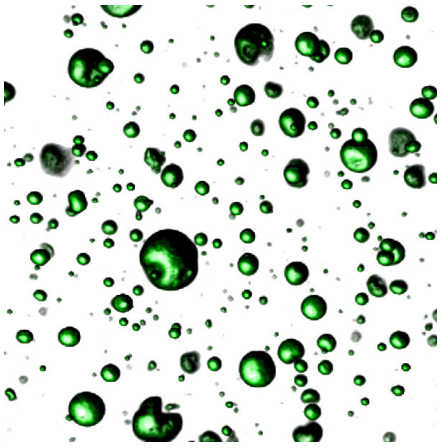
To show the application of this method in studying the properties of ink vehicle emulsions, some experiments have been performed and results are presented below.

1. Emulsions of different FS concentration

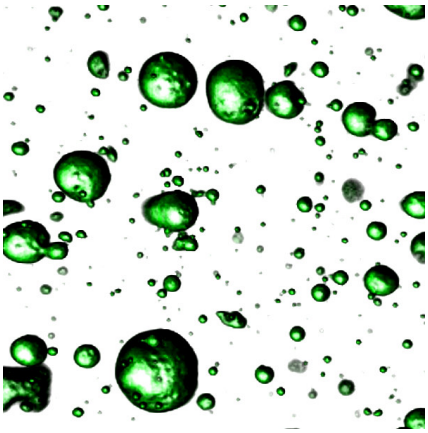
Following the experimental procedure described above, emulsions of different FS concentrations (5%, 10% and 15% w/w respectively), were prepared with the Dispermat using a rotor speed of 500 rpm at 25^{o}C. The 3D images of these emulsion samples are given in Figures 3(a) to 3(c).



a. FS concentration: 5%



b. FS concentration: 10%



c. FS concentration: 15%

Figure 3. FS-ink vehicle emulsions prepared using the Dispermat at rotor speed of 500 rpm, with different amount of FS added (field of view 100×100 μm^2, Z depth about 30 μm)

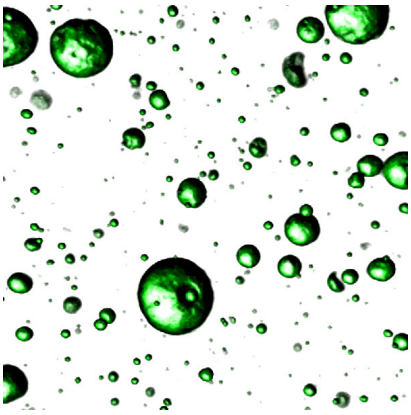
From Figure 3, it can be seen that a higher FS concentration doesn't necessarily produce more FS droplets in the emulsion, but leads to larger droplets. This is probably because at a higher FS concentrations, the probability of droplet collision is higher, so will be the coalescence and Ostwald ripening (processes of two drops collide and form one larger drop), which happen during the degassing process. These processes lead to fewer droplets and larger volume per droplet. In addition, the Ostwald ripening is self-accelerated when some droplets become larger (the Kelvin effect). Table 2 lists the number of droplets detected in each volume and average drop volume.

FS content	Number of droplets in image	Average droplet volume ($um^{3)}$
5%	294	11.9
10%	177	23.9
15%	126	69.1

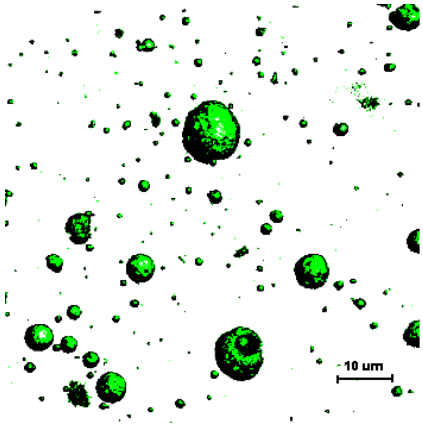
Table 2. Properties of emulsions of different FS concentration.

2. Emulsions made at different rotation speeds

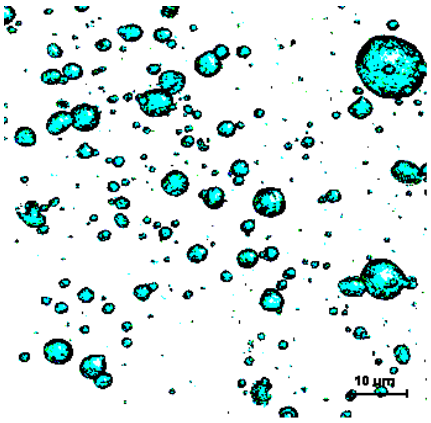
Following the same procedure, emulsions containing 10% FS were prepared using the Dispermat at different rotor speeds; 5000 rpm, 10000 rpm and 15000 rpm. The 3D images of the resulting emulsions are displayed in Figures 4(a) – 4(c).



a. 5000 rpm



b. 10000 rpm



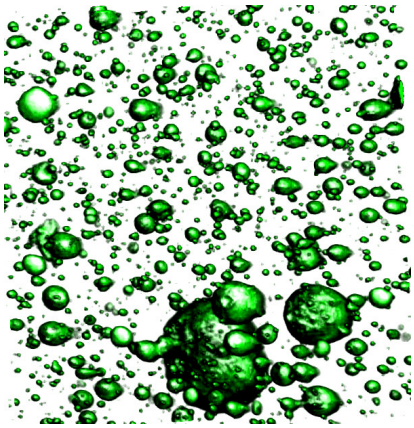
c. 15000 rpm

Figure 4. FS-ink vehicle emulsion samples prepared using the Dispermat at different rotor speed. (Field of view 100×100 μm^2 and sampling depth of about 40 μm).

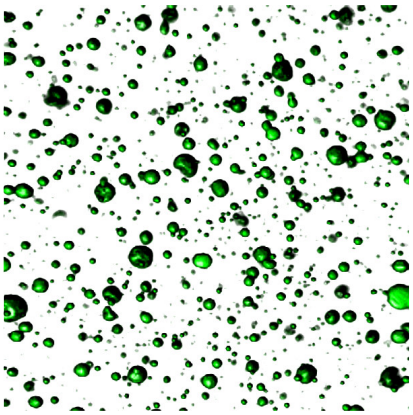
Compare Figure 3(b) and Figures 4(a) – 4(c), it seems that a higher rotor speed does not necessarily produce more uniform FS droplets. Probably the coalescence and Ostwald ripening which happen during the degassing process reduce the differences between emulsions prepared at different speeds.

3. Emulsions made using different instruments

An emulsion produced in the Hydroscope roller mixer by adding the FS slowly (0.5 ml/min) is shown in Figure 5(a), and all at once in Figur 5(b).



a. FS added slowly at 0.5 ml/min



b. FS added in a single shot (2ml) at the beginning of mixing

Figure 5. Emulsions made at 25°C with a Hydroscope and at a roller speed of 40 meter/minute.

As expected, when all the fountain solution was added in a single shot at the beginning of mixing, the emulsion formed is much more uniform than that made by continuous addition of FS. This is simply because when adding the FS all at once, the FS has more time to mix up with the ink vehicle. However, FS is more likely to be added continuously in a commercial press.

Comparing Figure 6 with images of other emulsions made with using the Dispermat, it is reasonable to conclude that roller mixing produces more uniform emulsions than the high shear instrument like a disperser, if conditions are the same, e.g. temperature, liner speed, amount and means of FS addition. This can be seen from Figures 6 and 7, where the average drop volume and size distribution of differently prepared emulsions are compared. Though using a laboratory disperser is an easy way of obtaining ink emulsions, it is not necessarily the best way to do the job.

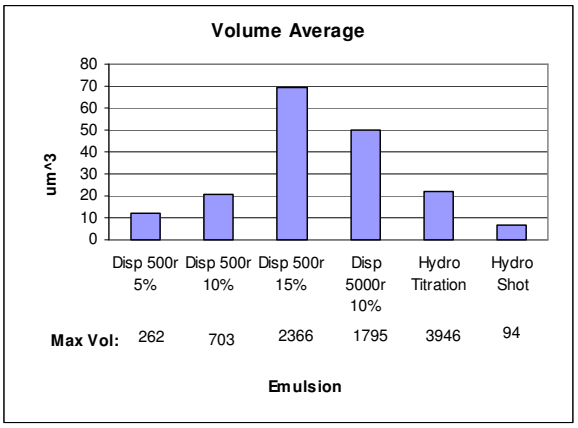
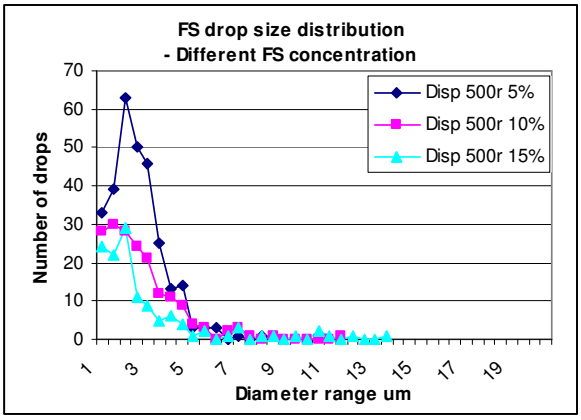
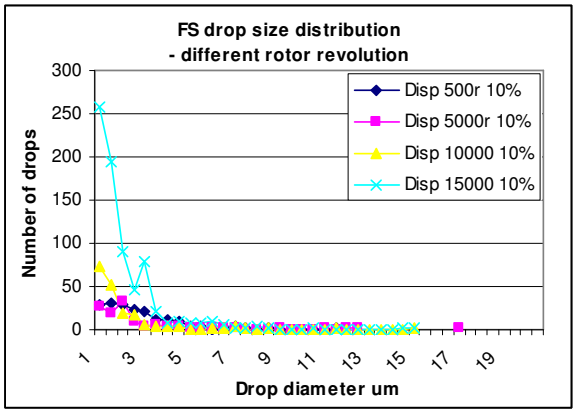


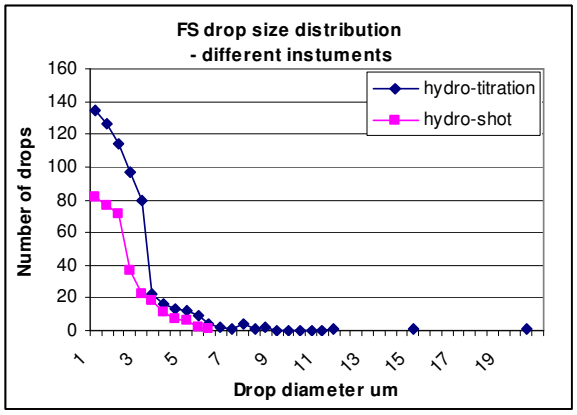
Fig 6. The average volume of FS droplets in emulsions made under different conditions



a. emulsions with different FS concentration



b. emulsions made at different rotor speed in a Dispermat



c. emulsions made with a Hydroscope with FS added in different ways

Fig 7. The size distributions of FS droplets in emulsions prepared under different conditions

Some problems with confocal microscopy method

Three major problems of the confocal microscopy method were noticed which can cause error in FS droplet size analysis.

The first problem is the non-uniform spatial distribution of the FS droplets in the emulsion samples. This problem negatively affects the validity of any single 3D sampling of the emulsion. This problem is most likely related to the long degassing time used in the sample preparation. During the prolonged stand-still degassing period a mild phase separation of ink vehicle could have occurred in emulsion. Since the ink vehicle is a mixture of many chemical components, its stability might have been compromised when mixed with FS. After degassing, the spatial distribution of FS droplets showed a clear non-uniformity. The only way to overcome this problem seems

to be to increase the number of sampling and the undesirable consequence is that large quantity of data must be processed.

The second problem is the result of a limitation in the confocal microscopy technique and is therefore more difficult to overcome. The fluorescent signal emitted from a deeper position in an emulsion sample suffers from a greater attenuation loss than that from a shallow location. The large number of emulsion droplets in the sample makes the attenuation loss, through scattering and absorption, of the signal from a deeper layer ever more severe. Although the signal intensity and contrast could be enhanced using the technique proposed by Xu and Parker [11], some loss of the signal is unavoidable. As a result, only large droplets (which usually have stronger emission signal) were observed when the focal was moved deeper into the emulsion; small droplets simply could not be detected due to their weak emission intensity. This problem is difficult to overcome but could be reduced by limiting the depth in imaging.

The third problem of this method is that it does not work as well for commercial inks that contain dark pigment as for ink vehicle. Pigments greatly reduce the fluorescent light transmission and thus restrict the depth of scanning. This problem could be overcome by using printable model inks that contain a smaller amount of pigments.

Potential application of this method

Despite the problems and difficulties in reconstructing and analyzing the 3D structure of an ink emulsion, confocal microscopy and image processing still provide a very useful method for characterizing the emulsions. 3D images and quantitative information obtained from them will provide details that may help explain the rheological properties of emulsions and the press performance of inks. The techniques developed could also be very useful for studying the ink film splitting phenomenon as the water droplets and air bubbles can be clearly distinguished.

A more exciting application for this method is that it may be used to answer the question “where does the water go when an ink emulsion is applied to the paper surface?” It provides a simple way to monitor the penetration behavior of water, and ink vehicle (if the latter is also dyed and observed using a two-channel confocal microscope). Figure 8 shows the cross section of a fiber immersed in an ink emulsion. The fiber wall emits fluorescent light because water penetrated into it. When the ink vehicle is also dyed with another fluorescent dye, the location of ink vehicle could also be seen.

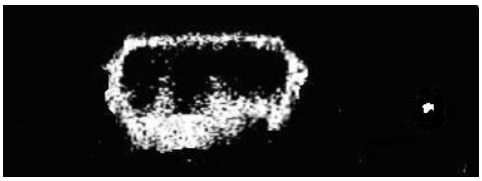


Fig 8. Confocal image of a fiber immersed in an ink emulsion.

Conclusions

3D images of FS-ink vehicle emulsion, and quantitative information from them, can be obtained using a confocal microscope, suitable fluorescent dyes, 3D image reconstruction software and appropriate image analysis.

A specially designed microscope container enabled an ink vehicle emulsion to be observed in its natural state, without distortion. Good 3D images and accurate measurements of droplet size distribution are achievable by the careful preparation of emulsion samples and the appropriate selection of the best parameters for confocal imaging and the optimum selection of parameters in the software in 3D reconstruction and image analysis.

The technique developed shows quantitatively, the effect of FS concentration, shear rate and dispersion instrument on the FS drop volume and size distribution. Potential applications of this technique in other offset printing related studies are discussed.

Acknowledgements

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