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Formulation of drop on demand soy inkjet inks

Alexandra Pekarovicova, Zahra Mashhadi Khodabakhsh, Paul D. Fleming III

Western Michigan University, Center for Ink and Printability, 4601 Campus Drive, A-213 Parkview, Kalamazoo, MI 49008-5462, USA

E-mail: a.pekarovicova@wmich.edu

Abstract

Soy protein has a complex 3-D shape and contains 19 different amino acids, which are held together in a coiled structure by peptide bonds. The basic application of industrial-grade soy protein is for a use as a binder in paper coatings. In this work, soy polymer Pro-Cote 4610E was used for formulation of drop on demand piezoelectric inkjet inks. Soy drop on demand inks performance was compared to ones of inks formulated with acrylic polymer Joncryl 678 and commercial inkjet ink. During formulation, sodium hydroxide was more successful in solubilizing of soy polymer than ammonium hydroxide. Preliminary soy ink formulations were made, and based on these results, a design of experiments (DOE) was carried out to optimize ink formulations. The DOE was executed with another aim to compare soy and acrylic based chemistry performance. A combination measure of ink density, surface tension and viscosity was used to assess inkjet printability. Drop behavior was controlled by Reynolds, Weber and Ohnesorge numbers. Reciprocal value of Ohnesorge number (Z number) was calculated, and used for prediction of soy protein inks and acrylic inks jettability. The print design for Dimatix Material Printer DMP-2800 consisted of solid patches and lines of varied width and orientation. At first, even inks with proper Z number were creating satellite drops and were puddling, which was removed by increasing the drop velocity from 0.2 $m \cdot s^{-1}$ to around 4 $m \cdot s^{-1}$. Thus, it was found that higher voltage is crucial for proper drop formation. The quality of printed lines was evaluated by measuring their width and raggedness using image analysis. Print mottle was evaluated on solid patches. All inks, soy, acrylic and commercial, exhibited printed line widths greater than their nominal widths, which was expected, due to printing on plain inkjet paper. There were no significant differences in quality of print lines between soy and acrylic inks and commercial ink. Soy ink achieved the best print uniformity of solids, when compared to formulated acrylic or commercial inkjet ink.

Keywords: soy polymer, acrylic ink, ink formulation, jetting, Z number

1. Introduction

Acrylic polymers are used in the automotive industry, medical devices, coatings, paints, and adhesive industries. With the development of disposable diapers, much of acrylic chemistry is consumed in diaper manufacture. Often times, the ink industry competes for acrylic polymers, which inspired this work on alternative inks based on renewable soy based raw material.

Soybeans, the material used to extract soy protein from, are composed of about 40 % proteins and 20 % oil (Xu et al., 2011). Soy oils have been employed in ink manufacture for a long time. They are used mainly in offset litho newsprint inks, as well as heatset offset inks. Recently, eco-friendly pigment and soy oil were applied in formulating heat resistant soy inks (Liu et al., 2014). The paper industry continues to use soy protein for paper coatings. Currently, soy protein is intensely researched for viability for green packaging application alone or in blends with other green polymers (Garrido et al., 2014; Ciannamea, Stefani and Ruseckaite, 2014; Leceta et al., 2014).

Soy protein features a complex 3-D shape which contains 19 different amino acids. These acids are held together in a coiled structure by peptide bonds (Kinsella, 1979). The major functional groups found in soy protein consist of amino, carboxyl, hydroxyl, phenyl and sulfhydryl ones. Soy proteins can be obtained from soybeans through the extraction of soybean oil (Xu et al., 2011; Kinsella, 1979). It is an excellent raw material for formulating environmentally friendly adhesives, asphalts, resins, cleaning materials, cosmetics, paints, plastics, polyesters and textile fibers (Smith, 1996). The basic application of industrial-grade soy protein is for a use as a binder in paper coatings (Graham and Krinski, 1983; Zhou et al., 2013; Merrifield, 1998). Soy protein is extracted from soy meal under neutral or alkaline conditions, because its water solubility is very closely related to pH, and it drops with decreasing pH, reaching minimum at pH around 4.2–4.6 (Kumar et al., 2002). Fractionation of soy polymer can be also done by ultra centrifugation based on sedimentation constants of proteins. The principal soy protein is glycinin with molecular weight around 320–360 kDa (Kumar et al., 2002). Glycinin contains mainly acidic amino acids and their amides. Soy protein is manufactured from concentrates, which contain denatured insoluble proteins, and isolates, which are water soluble, and useful for industrial application, such as plastic films, composite materials or adhesives.

In general, a base inkjet ink is prepared with four main raw materials: resins, colorants, solvents, and additives (Magdassi, 2010; Kipphan, 2001). Resins, solvents, and additives create a varnish, which can be mixed with a pigment dispersion to make the final ink system. Resin is usually added to provide cohesive forces between materials and adhesive forces between materials and substrate. It enhances abrasion resistance, gloss, rub resistance, and also the durability of the ink. Colorant is either a soluble dye or insoluble pigment. Dyes provide transparent and vivid colors. They have lower cost in comparison to pigments, and at the same time, they do not need to be ground or filtered. Dyes also provide a larger color gamut than pigments. Their disadvantage is their low light stability and permanence (Chovancova et al., 2005). Pigments on the other hand are fade resistant, thus they are gaining popularity for inkjet ink formulations (Chovancova et al., 2005). Due to their chemical nature, pigments are inorganic or organic. They are ground into submicron particles in the process of pigment dispersion preparation. Solvent is used in printing ink to adjust the viscosity. Water is the main solvent in water-based inks. Additives are used to give special properties to printing inks and they should be added to ink in amounts not exceeding 5 %. There are several types of additives that have different functions in a printing ink. The main groups of additives are waxes, defoamers, surfactants, humectants, biocides, pH adjusters, cross linkers, and anti-corrosion agents (Leach et al., 1993). To prevent ink contamination, the use of deionized water in the ink formulation is essential. The finished ink should be able to keep its properties such as ink color stability, stable viscosity, surface tension, pH, pigment content, and particle size for a prolonged time (Kipphan, 2001), but also during printing (Frimova et al., 2005). Information on formulating inkjet inks can be found (Magdassi, 2010), but there has not been much work found on formulating inks based on soy protein, thus it became the main goal of this work.

2. Methods

2.1 Ink formulations

Resins Pro-Cote 4610E soy polymer with glass transition temperature of Tg > 160 °C (DuPont), and acrylic polymer Joncryl 678 (BASF); Tg = 101 °C, molecular weight MW = 800, acid number 216 were used to formulate soy and acrylic based inks. NaOH and NH_4OH were used to solubilize soy polymer and keep alkaline pH of solubilized resins. The Joncryl 678 acrylic resin was in powdered form, and a liquid resin needed to be made. The process to make Joncryl 678 resin solution was as follows (total weight 100 g): add 59 ml 50 °C water into a beaker; add 9 g ammonia (27 % as NH_3), then add Joncryl 678 solid resin in small increments (total 32 g) into solution while dispersing.

Surfynol CT-231 and Carbowet 300 (Air Products) were used as ink surfactants. Soy based inks were formulated with the addition of a biocide. Ethylene glycol was applied as humectant for inkjet inks. Commercial cyan pigment dispersion (Hostajet PT, Clariant) was employed to formulate both acrylic and soy based inks. Ink formulating was done similarly for soy and acrylic polymers. First, resins were solubilized in alkali, surfactants and water, then the pigment dispersion and further additives were blended in.

2.2 Printing

A Dimatix Material Printer DMP-2800 was used to print inkjet inks. The print head permits users to fill cartridges with any jettable fluid and print directly with the DMP-2800. The cartridge reservoir has a capacity of 1.5 ml and each single-use cartridge has 16 piezoelectric nozzles linearly spaced at 254 μm with 21.5 μm openings to produce typical drop sizes of 10 pl. Plain inkjet paper was used for printing.

2.3 Analytical

A combination measure of density, surface tension and viscosity was used to assess inkjet printability. Thus, drop behavior is controlled by Reynolds, Weber and Ohnesorge numbers. Ohnesorge number (Oh) can be calculated as follows:

$$\mathrm{Oh} = \mathrm{We}^{1/2} / \mathrm{Re} = \eta \,/\, (\gamma \cdot \varrho \cdot a)^{1/2} \qquad [1]$$

Where ϱ is the density, η is the dynamic viscosity, γ the surface tension, and a is the characteristic length (nozzle diameter).

Reciprocal value of Ohnesorge number is then calculated (Fromm, 1984) and designated as the Z number:

$$\mathrm{Z} = 1 \,/\, \mathrm{Oh} \qquad [2]$$

The density, surface tension and viscosity were measured in order to calculate the Z number. Density was determined using a 25 ml pycnometer and analytical balance. Contact angle and surface tension measurements were performed using a FTA200 dynamic contact angle measurement device. The change in contact angle with time was measured for DI water and methylene iodide on the paper substrate and printed ink films to enable their surface energies to be determined by use of the Owens-Wendt (1969) method. The equilibrium contact angle for each liquid (contact angle where no further change with time was observed) was used in these calculations (Owens and Wendt, 1969). Viscosity of inks was determined with RA 2000 dynamic stress rheometer (TA Instruments, DE) (Yumeizhi, Pekarovicova and Fleming, 2013). Cone–plate couette geometry was used. Viscosity measurements were performed at a fixed temperature of 25 °C while increasing the shear rate from 0 to 1 500 s^{-1}. The solids content of inks was measured by a Smart Turbo-Moisture/Solids Analyzer.

The particle size of ink and ink ingredients was measured by Submicron Particle Sizer Model 370 Nicomp (Particle Sizing Systems, Santa Barbara, California) based on photon correlation spectroscopy. The ImageXpert (KDY Inc.) image analysis system was used for measurement of average width and raggedness of printed lines. ImageXpert image analysis system consisted of a motion table for sample positioning, two calibrated cameras for image capture and ImageXpert image analysis software (IX 10.0b63). Five readings were taken for every feature measured on each sample. For color and optical density characterization, an X-Rite 530 SpectroDensitometer was employed. Specular gloss was measured by Novo-Gloss 20°/60°/75° meter at 60 degree geometry.

3. Results and discussion

Commercial soy polymer was implemented into alkaline water based slurries. Different alkalis, such as Na_2CO_3, NaOH and NH_4OH were tested for soy powder solubilization. It was found that soy polymer dissolves to different extent in various alkalis, resulting in particle size ranging from 800 to 400 nm. Moreover, it dissolves best in ammonium hydroxide, in which the particle size reached 400 nm (Khodabakhsh, 2013). Dispersed soy protein was then blended with pigment dispersion and surfactants. The particle size of such polymer dispersion decreased even further after mixing with pigment dispersion to 150−160 nm depending on ink formulation and pH environment. Rheology, surface tension and density of these inks were measured. These data were used to calculate Z numbers, combining influence of density, surface tension and viscosity on ink jettability (Owens and Wendt, 1969; Yumeizhi, Pekarovicova and Fleming, 2013; Derby, 2011). Applying Weber and Ohnesorge calculations, Fromm used the reciprocal value of Oh number and came to conclusion that the Z number should be in the range $2 \leq Z \leq 14$, in order for ink formulations to be suitable for ink jetting (Fromm, 1984; Derby, 2011; Reis, Ainsley and Derby, 2005).

According to preliminary formulations with appropriate Z number (data not shown), a design of experiments was employed to optimize formulation of soy and acrylic based inks (Table 1). Print design is illustrated in Figure 1.

Commercial inkjet ink extracted from a cartridge, and formulated soy and acrylic inks were printed using a Dimatix Material Printer DMP-2800. Even with inks with Z number in the optimum range, it took formulation changes and surfactant amount optimization as well as Dimatix waveform modification to reach proper jetting. At first, inks created satellite drops and puddled (Figure 2). Also, it seemed like the drop velocity was very slow, thus, it was decided to measure the velocity

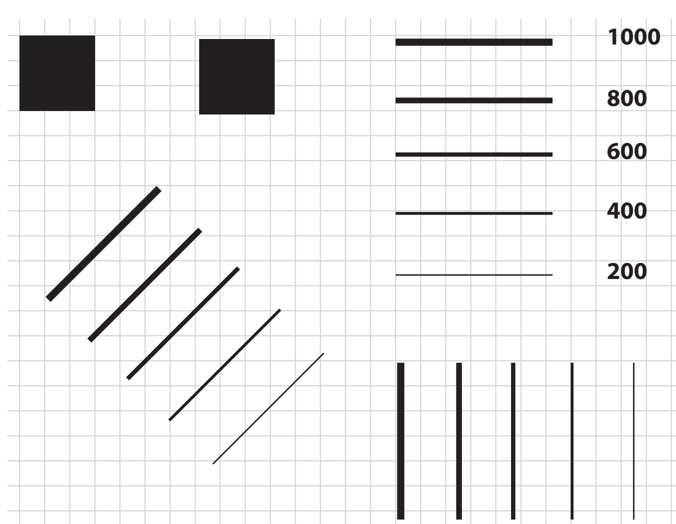


Figure 1: Design for printing acrylic and soy inks on Dimatix printer

of the drops and then compare with required velocity of ink for a proper ejection and without breakup. The drop velocity was calculated using the video system on Dimatix (data not shown). From these data the Weber number We and $We_{critical}$ (Pilch and Erdman, 1987) were calculated:

$$We_{critical} = 12 \cdot (1 + 1.077 \cdot Oh^{1.6}) \qquad [3]$$

It was shown previously that, in the case where the We number is smaller than the $We_{critical}$, there would not be any breakup nor satellite drop formation (Lim et al., 2013). Also, it was found that the required velocity to achieve proper drop formation was around $4\ m \cdot s^{-1}$, while the velocity of inkjet ink jetted as shown in Figure 2 was $0.2\ m \cdot s^{-1}$. Thus, it is clear that ink needs to be jetted at higher voltages to get higher drop velocity. Another challenge was to avoid nozzle clogging (Figure 3). Nozzle clogging reduction was enabled using humectant, such as ethylene glycol up to 10 % wt., in the ink formulation. Appropriate jetting of acrylic and soy based ink is illustrated in Figure 4 and Figure 5.

Table 1: Design of Experiment for soy inkjet inks optimization (AWoutS – acrylic without surfactant, AWS – Acrylic w/surfactant, SWoutS – Soy without surfactant; SWS – Soy with surfactant)

Formula No.	**AWoutS (1)**	**AWS (2)**	**SWoutS (3)**	**SWS (4)**	**AWoutS (5)**	**AWS (6)**	**SWoutS (7)**	**SWS (8)**
Acrylic Joncryl 678 (g)	12	12	–	–	12	12	–	–
Soy polymer solution (g)	–	–	12	12	–	–	12	12
Pigment dispersion (g)	15	15	15	15	10	10	10	10
Surfactant (Carbowet 300) (g)	–	0.1	–	0.1	–	0.1	–	0.1
Humectant (Ethylene Glycol) (g)	10	10	10	10	10	10	10	10
DI water (g)	63	62.9	63	62.9	63	62.9	63	62.9

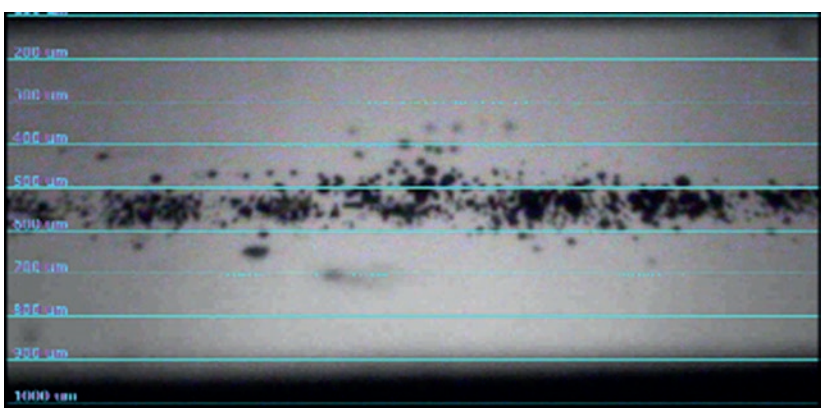


Figure 2: Soy ink puddling while printed on Dimatix Material Printer DMP-2800

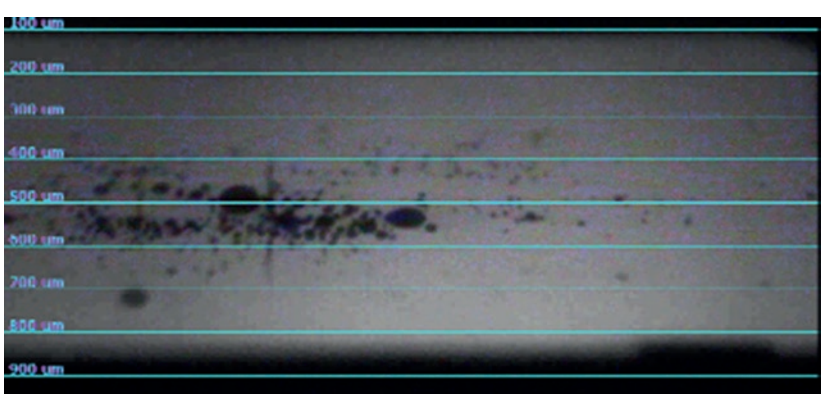


Figure 3: Soy ink clogging nozzles on Dimatix Material Printer DMP-2800

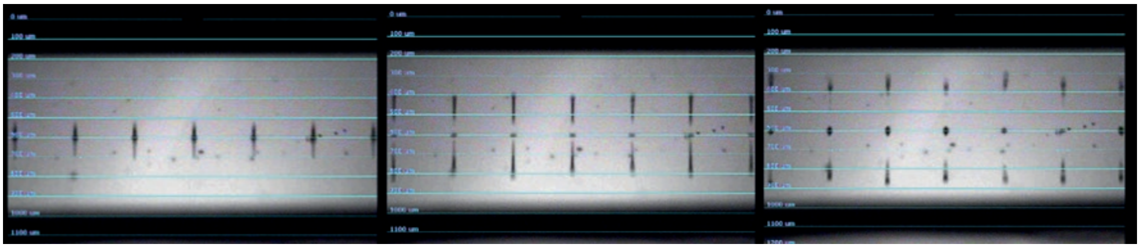


Figure 4: Jetting of acrylic based inks on Dimatix

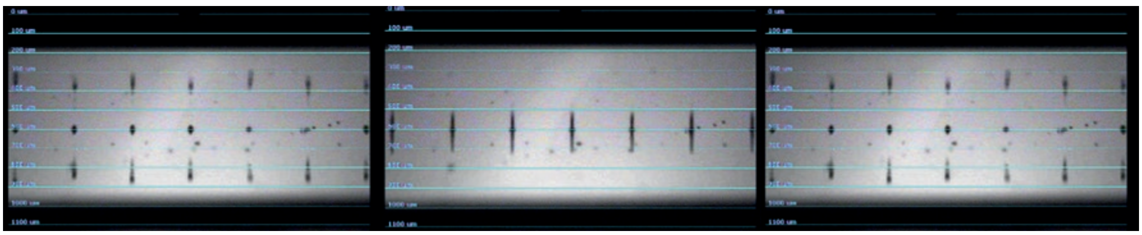


Figure 5: Jetting of soy based inks on Dimatix

The viscosity versus shear rate for acrylic-based inkjet ink and soy-based inkjet ink was studied. The viscosity curves suffer from noise in the region 0.1–4 s^{-1} and then again from 300 s^{-1} (acrylic) and 400 s^{-1} (soy), data not shown. Both soy and acrylic inks behave as Newtonian fluids in the region of shear rate 4–300 s^{-1} (acrylic AWS) and 4–400 s^{-1} (soy SWS). It is obvious that the acrylic ink has slightly lower viscosity (2.4 mPa · s) than the soy ink (3.9 mPa · s). Similarly, viscosities of all formulated inks were measured. Figure 6 shows comparison of viscosity of all inks with/without surfactant and with varying amount of pigment dispersion. As expected, the ones with lower pigment load had lower viscosity (Figure 6).

According to rule of thumb, the surface tension of inks should be at least 10 $mN{\cdot}m^{-1}$ lower than the surface energy of the substrate, in order to achieve proper print quality and adhesion. The substrate surface energy is defined as the sum of the excess energies at the surface of the substrate compared to the bulk (Whiting et al., 2011). In inkjet printing, it is very important to obtain an accurate drop placement and a uniform dried film, which can be controlled by adjusting the surface energy of the substrate (Whiting et al., 2011). It was found that acrylic-based inkjet inks have larger surface tension than the soy-based inkjet inks, maybe because soy protein is a natural surfactant. It was also found that the surface tension values of inks with 15 g of pigment dispersion are lower than of inks with 10 g pigment dispersion. It is most likely because commercial pigment dispersion contains wetting agent blended in, which may reduce the surface tension. Also, surfactant has a slight effect on surface tension of acrylic-based inkjet inks, while it has no effect on soy-based inkjet inks. It was also shown that the surface tension of soy-based inkjet inks with 10 g pigment dispersion with or without surfactant was very close to that of commercial inkjet ink (Figure 7). Soy inks had slightly lower surface tension 32.5–34.8 $mN \cdot m^{-1}$ than acrylic inks 35.3–39.3 $mN \cdot m^{-1}$ as compared to commercial ink having surface tension 34.0 $mN \cdot m^{-1}$. Soy inks particle size was slightly larger

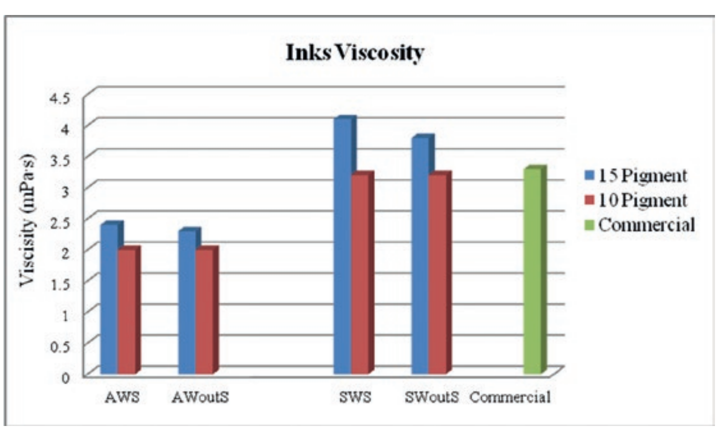


Figure 6: Comparison of viscosity of formulated and commercial inkjet inks

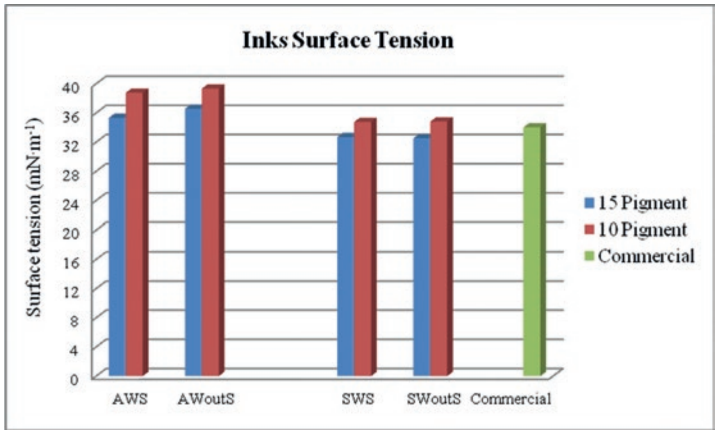


Figure 7: Comparison of surface tension of formulated and commercial inkjet inks

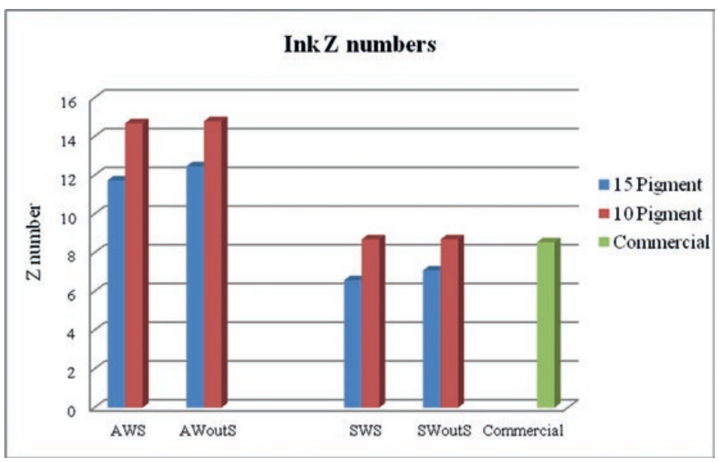


Figure 8: Comparison of Z numbers of formulated and commercial inks

(range of 188–197 nm) than that of the acrylic inks (range of 160–174 nm), data not shown.

Acrylic-based inkjet inks had larger Z numbers than soy-based inkjet inks (Figure 8). Inks with 10 g pigment dispersion had larger Z number than those with 15 g of pigment dispersion. Also, the Z number for soy-based inkjet inks with 10 g pigment dispersion (with and without surfactant) was very close to that of the commercial inkjet ink (Figure 8). It was found that increased level of surfactant in ink does not affect the Z number for both acrylic-based and soy-based inkjet inks.

A print done with one of the successful formulations of soy ink is illustrated in Figure 9 and a print of commercial ink is illustrated in Figure 10. The quality of printed lines was evaluated by measuring their width and raggedness using image analysis. There were five lines with different nominal widths: 200, 400, 600, 800, and 1 000 μm measured for each ink formula. Only lines parallel to print direction were measured. It was found that all line widths are greater than their nominal widths (Figure 11). It is due to ink spreading, which would be observed as tone value increase, or dot gain, if measured on individual printed dots. The type of paper, in this case plain uncoated paper, has a significant effect on spreading. The plain paper pulls liquid into its body or moves it laterally along the surface, while paper with a coating tends to hold the ink at the surface and does not allow adsorbing into or wicking along its surface (Briggs, 2002; Forrest et al., 1998).
The raggedness of both sides of the lines was measured to consider the line quality. Again, only lines parallel to print direction were measured. The more feathering occurs, the more ragged the line is. It was found that some prints suffered from overspray and smearing. Figure 12 illustrates line raggedness for all acrylic and soy inks and commercial ink and Figure 13 shows detail of line raggedness for acrylic, soy and commercial ink and how it was measured.

Overall, Figure 12 shows that acrylic ink with or without surfactant printed less ragged lines than soy ink. Also, ink formulations without surfactant printed less ragged

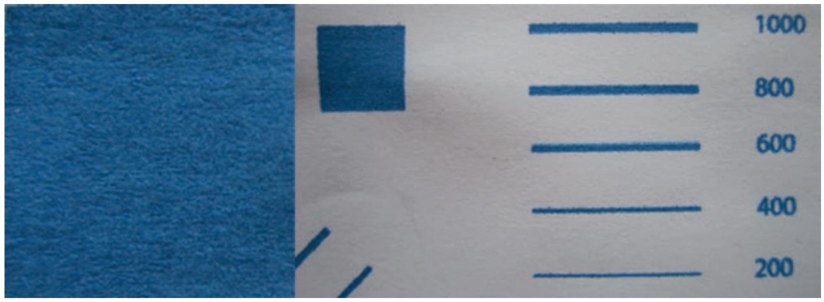


Figure 9: Print of soy based ink with viscosity 4.1 mPa · s, surface tension 32.7 mN · m⁻¹, and Z number 6.58

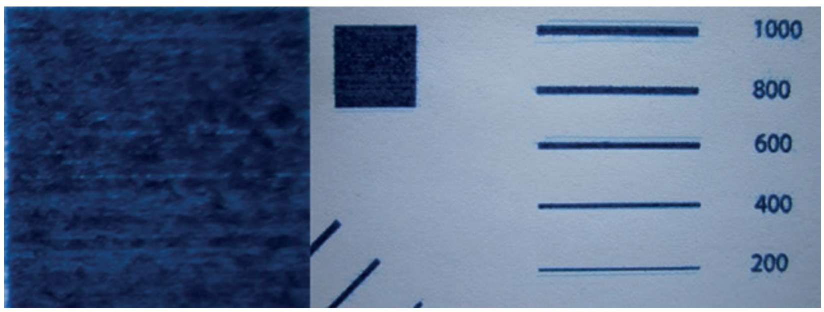


Figure 10: Print of commercial ink with viscosity 3.3 mPa · s; surface tension 34.0 mN · m⁻¹ and Z number 8.54

lines than the formulations with surfactant. Also, more viscous inks with 15 g of pigment dispersion were less ragged than those with 10 g of pigment dispersion. It may indicate that lower viscosity encourages spreading and raggedness. Overspray and smearing were observed with all of these three types (acrylic, soy and commercial) of inkjet inks (Figure 13), thus these problems can be also related to selection of ink viscosity and printing conditions of Dimatix printer as well as quality of the paper surface. It can be concluded that line raggedness of commercial and formulated acrylic and soy inks are comparable, which pinpoints the need of further optimization of Dimatix waveform printing conditions.

Optical properties and print quality were then analyzed. Both soy and acrylic inks achieved low specular gloss around 2–3 %, (data not shown), maybe due to surface roughening because of alkaline pH of inks and low quality of paper substrate. Optical densities of all soy inks were lower (1.11–1.21) than that of commercial ink (1.36). However, optical density can be modified with pigment dispersion addition. Soy ink achieved the best print uniformity of solids, when compared to formulated acrylic or commercial ink (Khodabakhsh, 2013).

CIELAB data of all inks are shown in Figure 14. All inks with 15 g pigment dispersion had lower L^* value than

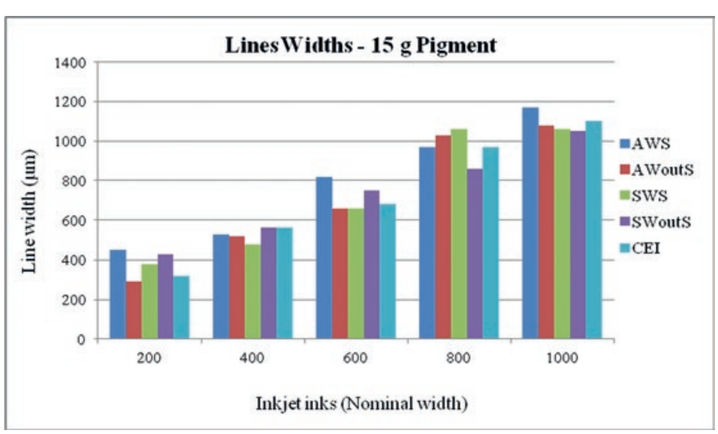


Figure 11: Line width of inkjet inks with 15 g of pigment dispersion (CEI – commercial)

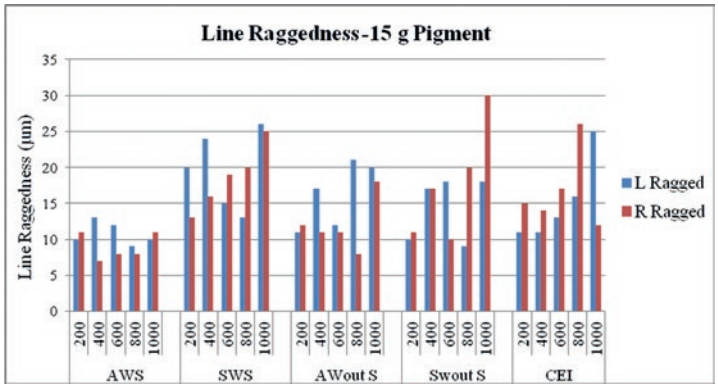
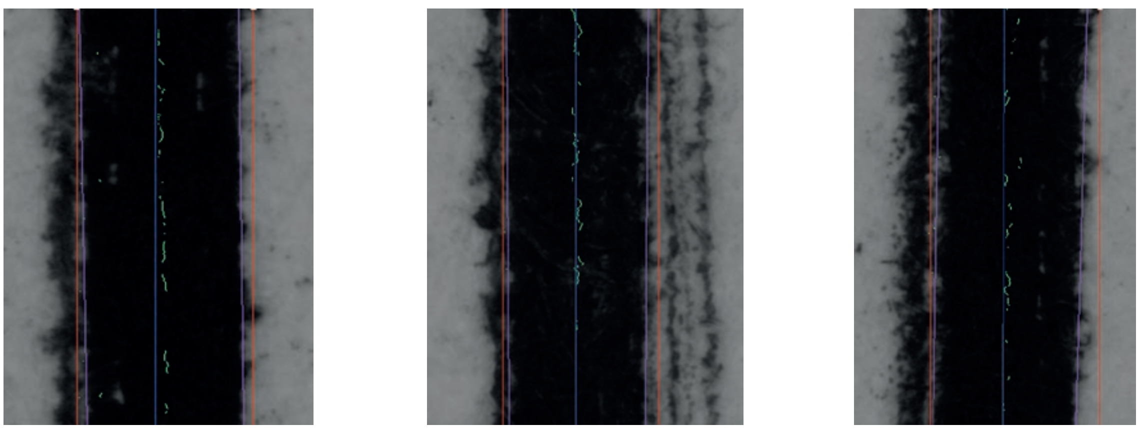


Figure 12: Raggedness of both left and right sides of line of all inkjet inks with 15 g of pigment dispersion



a) b) c)

Figure 13: Line raggedness of acrylic, soy and commercial inks (15 g of pigment dispersion); a) AWoutS (#1), b) SWoutS (#3), c) CEI (#9)

those with 10 g pigment dispersion. Formulated acrylic inks had lower CIE L^* values (L^* range 28 to 31) than soy inks (L^* range 35 to 37). Commercial ink had lowest CIE L^* value of 25.8. Commercial ink was also redder (CIE $a^* = 16.2$), while our formulated inks were greener (CIE a^* in range of -0.5 to -10.6), which indicated that different crystalline structure cyan pigment was used. CIE b^* value of commercial ink was -41.5, while formulated inks had CIE b^* value in the range of -44.6 to -52.4; acrylic had lower b^* values, than soy inks, which indicates that acrylic resin developed bluer cyan than the soy resin.

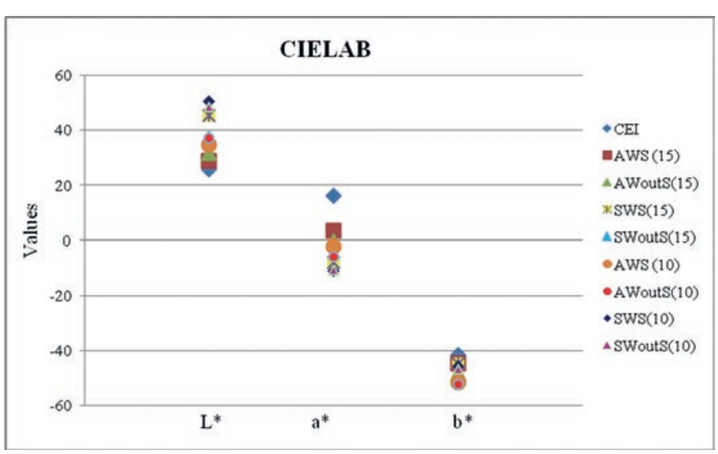


Figure 14: CIELAB values of commercial and formulated cyan inks

4. Conclusion

The aim of this work was to determine if the soy polymer can be used to formulate drop on demand inkjet inks. First, preliminary formulations were made and printing on the Dimatix Material Printer DMP-2800 was tested. Based on these preliminary results, a design of experiment was prepared for formulation of soy protein inks. Inks were designed so that their combined performance regarding viscosity, density and surface tension would give a certain value of Z number, predicted to be jettable. All inks formulated according to design of experiment were successfully jetted using the Dimatix printer. It was confirmed that both soy and acrylic inks behaved as Newtonian fluids in the region of shear rate 4–300 s^{-1} (acrylic AWS) and 4–400 s^{-1} (soy SWS). Acrylic ink had slightly lower viscosity (2.4 $mPa \cdot s$) and particle size (range of 160–174 nm) than the soy ink (3.9 $mPa \cdot s$ and 188–197 nm). However, soy inks exhibited slightly lower surface tension (32.5–34.8 $mN \cdot m^{-1}$) than acrylic inks (35.3–39.3 $mN \cdot m^{-1}$) maybe because soy polymer acts as a natural surfactant. Printability of soy ink jet inks was comparable to acrylic based ink jet inks and commercial drop on demand ink. The quality of printed lines was evaluated by measuring their width and raggedness using image analysis. Print mottle was evaluated on solid patches. All inks, soy, acrylic and commercial, exhibited printed line widths greater than their nominal widths, which was expected, due to printing on plain inkjet paper. There were no significant differences in quality of print lines between soy, acrylic inks and commercial ink. Soy ink achieved the best print uniformity of solids, when compared to formulated acrylic or commercial inkjet ink.

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