The Distribution of Poly-DADMAC Additive in the Inkjet Coating Layer and its Influence on the Print Quality

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Maximizing the functionality of cationic polydiallyldimethylammonium chloride (poly-DADMAC) additive with minimal dosage in inkjet coatings can contribute to both an improvement in quality and a cost reduction. To do this, it is essential to understand how the cationic additive is distributed in the coating layer and how it affects the print quality. This study presents a three-step investigation to enhance the understanding of the distribution of poly-DADMAC, as well as its effect on inkjet print quality. First, the adsorption behavior of poly-DADMAC on silica pigments was investigated by measuring the surface charge of silica and the adsorption of the cationic additive. Second, the influence of poly-DADMAC on the printability of binder films was investigated. Finally, the effect of poly-DADMAC on the print quality of inkjet paper was examined. The addition of poly-DADMAC improved the print quality of the inkjet paper because of the improved holdout of the negatively charged colorants in the ink.

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INTRODUCTION

Inkjet printing is a technology with a wide range of application possibilities ranging from conventional printing to various fields including electronics (Nayak *et al*. 2019), pharmaceutical applications (Scoutaris *et al*. 2016), and tissue engineering (Li *et al*. 2020). In the case of the conventional printing sector, inkjet printing has been used for small office and home office (SOHO) applications. However, its use in industrial production printing has been growing and has replaced some of the roles occupied by traditional analog printing (Pira 2019). When print quality issues arise, printer makers often want to have a better quality substrate to meet quality requirements. Although there is a lot of room for improvement in printer design and printing inks, it is true that the interaction between the printing ink and substrate is one of the decisive factors in improving the final print quality (Svanholm 2007; Kettle *et al*. 2010; Mielonen 2015; Chen *et al*. 2021).

The primary components of inkjet ink are colorants and dispersion medium. After a droplet of ink hits the substrate, ink setting occurs *via* various mechanisms, depending on the substrate type (Svanholm 2007; Kettle *et al*. 2010; Mielonen 2015; Chen *et al*. 2021). Quick drying with good holdout of the ink colorant is an ideal situation for ink setting in inkjet printing. Coated inkjet paper with a microporous coating layer is widely used as a substrate when high print quality is required, because it allows rapid penetration of the liquid phase of inks and good holdout of the colorants. The ink setting process of microporous inkjet coating layers involves various types of ink-substrate interactions, *e.g.*, wetting, capillary penetration, separation of colorants and dispersion media, adsorption, diffusion, and polymerization (Kettle *et al*. 2010). The structural and chemical properties of the coating, *e.g.*, surface morphology, pore structure, and surface energy, have a considerable impact on print quality (Kettle *et al*. 2010; Lamminmäki *et al*. 2011; Moutinho *et al*. 2011; Sousa *et al*. 2014). To address these issues, the types of coating pigments, binders, and additives have some limitations that differ from the conventional paper coating formulation for offset printing.

One concern with microporous coatings is the holdout of ink colorants. If a large portion of the colorant penetrates and is absorbed into the coating layer, then the ink density will be lowered, resulting in poor printing quality. To increase the holdout of the ink colorants in the inkjet coating layer, cationically charged additives are often used in the inkjet coating color preparation, which imparts a cationic charge to the dried coating layer that attracts and holds the anionic colorants on the coating surface. In other words, cationic additives prevent the penetration of anionic colorants into the coating layer, which hampers the color density. Divalent metal salts are often used in commercial products, *e.g.* HP ColorLok® papers, to increase the ink holdout (Varnell 2001; Veverka *et al.* 2013). Cationic polymers, *e.g.*, poly-DADMAC, ethyleneimine, or polyvinylamine, are also widely used for this purpose (Svanholm 2007; Mielonen 2015). These additives attract the negatively charged colorants *via* electrostatic forces and are often referred to as dye fixatives. Typical colorants of inkjet inks are anionic. The major components of inkjet coatings, including silica and polyvinyl acetate emulsion (PVAc), also are anionic.

The production cost of inkjet coating has always been an issue because the expensive silica must be used as the primary pigment. A large surface area and great affinity to water of silica pigments also leads to high binder requirements and increased drying cost (Svanholm 2007). Various strategies have been proposed to reduce the production cost, *e.g.*, lowering the cost of silica pigments or replacing silica pigments with specialty pigments (Malla and Devisetti 2005; Ridgway and Gane 2006; Kenttä *et al.* 2013). Reducing the use of cationic additives is also an alternative that contributes to lowering the production cost for inkjet coatings (Svanholm 2007). By maximizing the function of the cationic additive, an overdose of the cationic additive can be prevented. To achieve this, it is essential to understand how cationic additives become distributed in the coating layer and how they affect print quality.

Cationic additives can be present on the surface of the pigment or inside of the binder film of inkjet coatings. The use of a cationic additive changes the anionic nature of the silica pigment. The additives adsorbed to the pigment surface are often covered with a binder film because the addition rate of the binder is quite high in order to form a continuous film on the pigment surface, which may limit the role of cationic additives as colorant fixatives. The properties of the binder film influence the ink-paper interaction (Lamminmäki *et al*. 2010; Zhang *et al*. 2015). Obviously, the addition of an additive to the binder film will affect the ink setting. Furthermore, the effect of the binder and additive on the ink-paper interaction will be more pronounced when the amount of binder or additives is large. It has been shown that poly(vinyl alcohol) (PVOH) forms a film on the top surface of the inkjet coating layer, thereby keeping the dye on the surface (Svanholm 2007). During the consolidation process, water-soluble polymer binders, *e.g.*, starches, tend to migrate to the surface, and also to the base substrate if the substrate is permeable (Groves *et al*. 2001;

Zang *et al.* 2010). Water-soluble polymer binders, *e.g.*, starches, tend to migrate to the surface during the consolidation process (Groves *et al*. 2001). Therefore, understanding the change of properties of binder films containing cationic additives is important for understanding the effect of their use.

This study consists of three parts with the goal of enhancing understanding of the distribution and effectiveness of cationic additives in inkjet coatings. First, the adsorption behavior of cationic poly-DADMAC on silica pigment was investigated. This was performed by measuring the change in the surface charge of the silica pigment and the change in the amount of poly-DADMAC adsorption with the change of its dosage. This investigation was performed more thoroughly in the range of the typical dosage of the additive, which is less than 3 parts per hundred (pph) of dry pigment. Second, the effect of cationic poly-DADMAC in the binder film on ink jet printing was investigated. Binder films with different amounts of cationic poly-DADMAC were prepared on polyethylene terephthalate (PET) films, and its influence on print quality was examined by measuring the ink density and optical microscopy. Finally, the effect of poly-DADMAC on the printing quality of inkjet paper was investigated. Coated inkjet papers were prepared with three different binder systems and the effect of cationic poly-DADMAC on the print quality was examined by measuring the ink density and water fastness.

EXPERIMENTAL

Materials

A commercially available gel-type silica with an average particle size of 7 μm (ML-381, Dongyang Chem. Co., Incheon, Korea) was used as a pigment. Polyvinyl alcohol (PVOH), provided by Hansol Co. Ltd. (Daejeon, Korea), polyvinyl acetate (PVAc), provided by Hansol Co. Ltd. (Daejeon, Korea), and cationic starch (C-starch) provided by Samyang Co. Ltd. (Incheon, Korea) were used as binders. First, a 10 wt% PVOH solution and 5 wt% cationic starch solution were prepared and used. The water soluble binders were cooked at 95 °C for 30 min, and they were cooled to 50 °C before usage. In addition, poly-DADMAC was used as a cationic additive. Table 1 describes the properties of the coating ingredients used.

Table 1. Properties of Coating Ingredients

<table>
<tr><td rowspan="2">Pigment</td><td>Ingredient</td><td colspan="2">Mean Particle Size (μm)</td><td colspan="2">Surface Area (m^2/g)</td><td>Brightness</td></tr>
<tr><td>Silica</td><td colspan="2">7</td><td colspan="2">300</td><td>98</td></tr>
<tr><td rowspan="4">Binder</td><td>Ingredient</td><td colspan="2">Type</td><td colspan="2">Solids Content (%)</td><td>Viscosity at 25 °C (cPs)</td></tr>
<tr><td>PVA</td><td colspan="2">Solution</td><td colspan="2">10.0</td><td>810</td></tr>
<tr><td>PVAc</td><td colspan="2">Emulsion</td><td colspan="2">45.0</td><td>140</td></tr>
<tr><td>C-starch</td><td colspan="2">Solution</td><td colspan="2">5.0</td><td>900</td></tr>
<tr><td rowspan="2">Cationic Additive</td><td>Ingredient</td><td>Charge Density (meq/g)</td><td colspan="2">Solids Content (%)</td><td>Viscosity at 25 °C (cPs)</td><td>pH</td></tr>
<tr><td>Poly-DADMAC</td><td>5.2</td><td colspan="2">36.5</td><td>30</td><td>3.8</td></tr>
<tr><td colspan="7">Note: The viscosity was measured with a Brookfield viscometer</td></tr>
</table>

A PET film was used as a substrate for preparing the binder film. The grammage and thickness of the PET film were 5.2 g/m^2 and 36 µm, respectively. Inkjet base paper (provided by Hansol Co. Ltd., Daejeon, Korea) was used as a substrate for coating. The grammage and thickness of the base paper were 93.0 g/m^2 and 108 µm, respectively. The opacity, gloss, and brightness of base paper were 87%, 7.9%, and 85%, respectively.

An HP deskjet 5550 printer and its standard inks (H56, H57) were used for inkjet printing. H56 is a pigment-based black ink and H57 are dye-based color inks with cyan, magenta, and yellow colors. Table 2 describes the properties of the magenta and black ink. The average particle size of the black ink, as measured by dynamic light scattering (DLS) technique (Malvern Mastersizer 2000), was 0.064 µm.

Table 2. Properties of the Inkjet Ink

Ink	Type	Solution Charge (meq/L)	Solids Content (%)
Magenta	Dye-based	-46.2	3.2
Black	Pigment-based	-75.3	11.3

Methods

Adsorption behavior of the cationic additives to silica pigments

The adsorption behavior of the poly-DADMAC on silica pigment was investigated using a 10.0 wt% silica suspension. Different dosages of the cationic additive were added to the silica suspension and dispersed for 30 min. The suspension was then centrifuged at 3000 g and the supernatant was collected. The adsorption of cationic poly-DADMAC was measured by comparing the dosage and the amount of the additive remaining in the supernatant. A chemical oxygen demand (COD) test was performed to determine the amount of poly-DADMAC in the supernatant. Before the measurement, a calibration line was made between the amount of poly-DADMAC solution and the COD (as shown in Fig. 1). The COD testing was performed over a range of typical dosages of the additive, *i.e.*, in the range of 0.1 pph to 3.0 pph. The surface charge of the adsorbed silica pigment was also investigated by measuring the ζ potential.

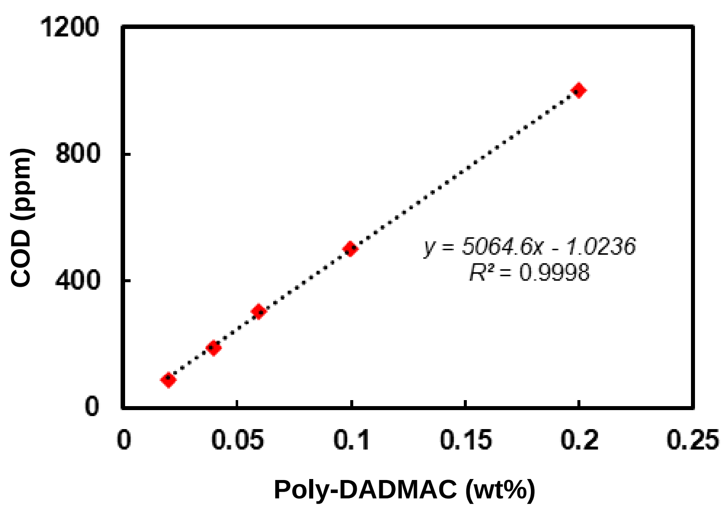

Fig. 1. Calibration of the COD to the amount of poly-DADMAC

The adsorption behavior at high dosages of the cationic additive was also investigated. The COD test did not work when the dosage of poly-DADMAC was high. In this case, the supernatant was dried, and the weight of the cationic additive was determined. This was performed when the dosages of poly-DADMAC were 13 pph and 20 pph. The concentration of the silica suspension was adjusted to 8.3 wt% for the experiments at high dosage rates of the cationic additive.

Preparation of the binder films and print quality measurement

Three different binder systems, including PVOH/PVAc, PVOH, and C-starch, were prepared and used. These binders are widely used binders for inkjet coating. The PVOH and C-starch were nonionic and cationic binders, respectively. The PVAc exhibits hydrophobic properties and is used as a mixture in PVOH/PVAc binder systems. Binder films with three different binder systems were prepared on PET films by using an automatic laboratory rod coater (PI-1210, Sangyo Co. Ltd., Saitama, Japan). After coating, the films were dried in a hot-air drying oven at a temperature of 100 °C for 2.5 min. The coat weight of the binder film was adjusted to 4 to 6 g/m^2.

Inkjet printing was performed using an HP Deskjet 5550 printer. The printed binder films were conditioned at a constant temperature and relative humidity (23 °C and 50% RH) for at least 24 h prior to use in experiments. The ink density of the printed binder films were measured using a densitometer (MacBeth Ink densitometer RD918, New Windsor, NY, USA) and ultraviolet-visible (UV-Vis) spectroscopy. Both dye-based and pigment-based inks were used. Magenta ink, which showed the most distinct peak among the dye-based inks, was used to measure the ink density (as shown in Fig. 2). The surface of the printed binder films was also observed under an optical microscope (CAMSCOPE ICSL-305, Sometech vision, Seoul, Korea).

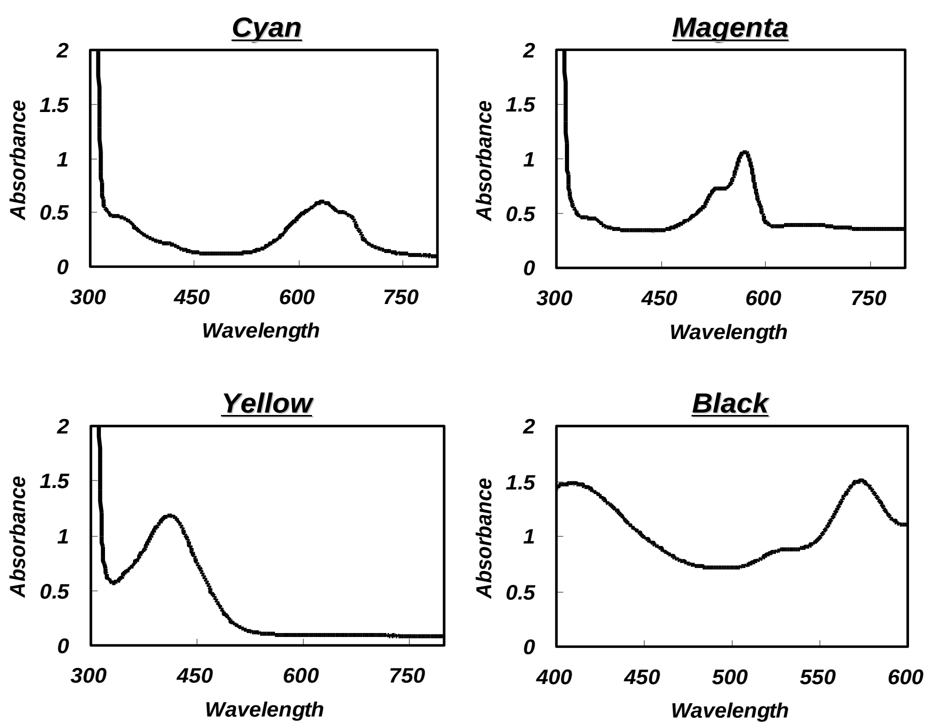

Fig. 2. Absorbance spectra of inkjet inks

Preparation of the coated paper and p*rint quality measurements*

Table 3 shows the coating formulation. The silica pigment was dispersed in distilled water at 2000 rpm for 30 min, and other ingredients were added in the order of a binder and cationic poly-DADMAC. The solids content and pH of the coating color were 15.0 wt% and 8.5, respectively. The coating color was applied to the inkjet base paper using an automatic laboratory rod coater and dried in a hot-air drying oven at a temperature of 105 °C for 3 min. The coated papers were then calendered twice in a soft-nip calender at a line pressure of 114 kg_f/cm and a temperature of 40 °C. The coated paper was conditioned in a constant temperature and relative humidity room (23 °C and 50% RH) for at least 24 h. All inks, *i.e.*, cyan, magenta, yellow and black, were printed using an HP Deskjet 5550 printer. The ink density was measured using a densitometer (MacBeth Ink densitometer RD918, New Windsor, NY). The printed surface was also observed using an optical microscope (CAMSCOPE ICSL-305, Sometech vision, Seoul, Korea).

Table 3. Coating Formulation

Ingredient		Parts per Dry Pigment	Solids Content (%)	pH
Pigment	Silica	100	15.0	8.5
Binder	PVA/PVAc	70 (35 + 35 each)		
	PVA	40		
	C-starch	40		
Cationic additive	Poly-DADMAC	15		

Water fastness was measured to determine the ability of coated papers to retain the ink density after a period of immersion into water. Water fastness is an important characteristic for dye-based ink since dye-based inks are susceptible to water (Mielonen 2015). Yellow ink was used because it showed the most pronounced change in terms of the water fastness. Printed coated papers were cut into 1.5 cm × 1.2 cm pieces and immersed in distilled water at a temperature of 25 °C for 5 min, 30 min, 2 h, and 7 h. Then, the immersed paper was completely dried in a hot-air drying oven at a temperature of 105 °C, and the ink density was measured after drying. Water fastness was calculated according to Eq. 1,

$$\text{Water fastness (\%)} = \frac{\textit{Ink density after immersion to water}}{\textit{Ink density before immersion to water}} \times 100 \qquad (1)$$

RESULTS AND DISCUSSION

Adsorption Behavior of the Cationic Additives to Silica Pigment

Figure 3 shows the change in the zeta potential of the silica pigment. The zeta potential of the silica pigment increased when more cationic additive was added. The anionic surface of the silica pigment was neutralized in the dosage poly-DADMAC range between 0.3 and 0.4 wt%. From this point on, the increase rate of the zeta potential slowed down. When the amount of poly-DADMAC used was small, the additive was adsorbed to the silica surface *via* electrostatic attraction, and the zeta potential rapidly increased.

However, after the neutralization point, the electrostatic attraction decreased and the rate of increase of the zeta potential decreased.

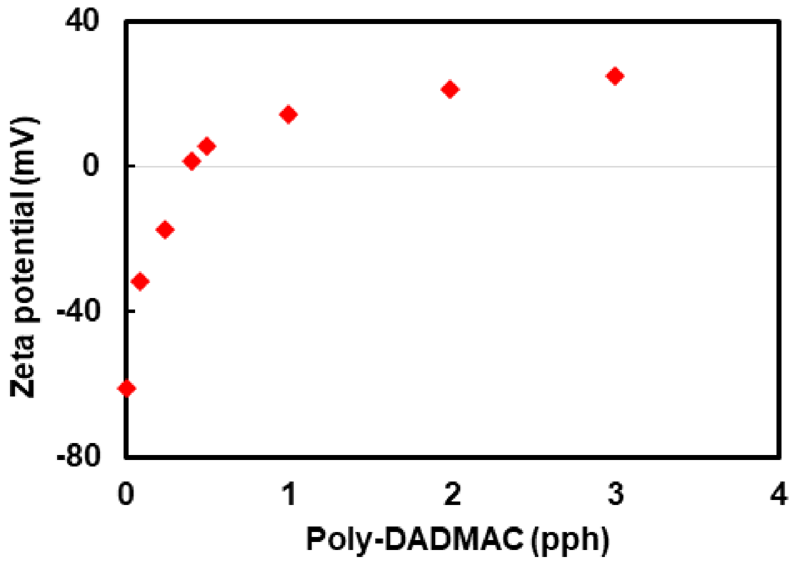

Fig. 3. Zeta potential of the silica suspension with the addition of poly-DADMAC

Figure 4 shows the amount of the cationic additive adsorbed to the silica pigment. Almost all of the cationic poly-DADMAC was adsorbed to the silica surface when the dosage was less than 1 wt%. The adsorption ratio in this range was approximately 95%. However, it decreased as the dosage increased. The adsorption ratio of poly-DADMAC decreased to 80% at a 3 wt% dosage (as shown in Fig. 4a). When the dosage increased further, the adsorption ratio decreased to less than 37% (as shown in Fig. 4b). Obviously, the unadsorbed cationic poly-DADMAC will be present in the binder film after drying.

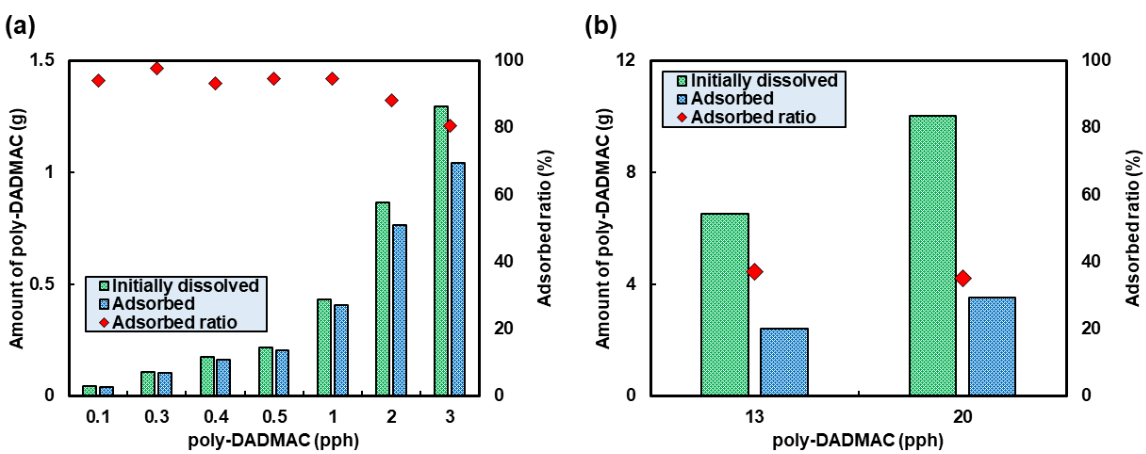

Fig. 4. The adsorbed amount of poly-DADMAC to silica pigments at (a) low dosage and (b) high dosage of poly-DADMAC. Adsorbed ratio = Adsorbed/Initially dissolved. (Note: the measuring methods for low and high dosages were different.)

For the actual coating formulation containing the binder, the adsorption of poly-DADMAC to silica will be lower than the results in Fig. 4. This is because the affinity of the cationic additive to the silica surface would decrease with the presence of a binder. In the initial stage of adsorption, poly-DADMAC will preferentially adsorb to the silica pigment because silica has a stronger electrostatic attraction to the cationic additive due to its higher polarity of Si-O bond than the polymer binder. As the surface charge of the silica pigment increases with the adsorption of the cationic additive, the electrostatic attraction of the silica pigment becomes weaker, and the cationic additive remains in the aqueous phase until it dries in the binder film. This phenomenon is expected to prevail when the dosage of poly-DADMAC is greater than the amount required for the neutralization of the silica.

Influence of poly-DADMAC in the Binder Film

Figure 5 shows the ink density of the pigment-based ink printed on the dried binder film. The results of the densitometer and UV-Vis spectroscopy were in good agreement with each other. Therefore, UV-Vis spectroscopy was used to evaluate the ink density results of the dye-based inks. The results of the pigment-based ink showed that the ink density increased with the addition of cationic additives, regardless of the binder system. Cationic additives are commonly used as dye fixatives. However, it was also effective in fixing the pigment-based colorants to the binder film.

Swelling is the primary mechanism of ink penetration into the binder film (Svanholm 2007; Chen *et al.* 2021). Because cationic additives prevent the ink colorants from penetrating into the binder film, despite swelling of the binder film, they show a positive improvement in the print ink density. It is clear that the additive increased the holdout of the anionic colorants by changing the nature of PVOH/PVAc and PVOH to cationic. It is interesting to note that for the cationic starch binder, the ink density also increased. This was because the higher charge density of the cationic additive increased the electrostatic attraction to the ink colorants.

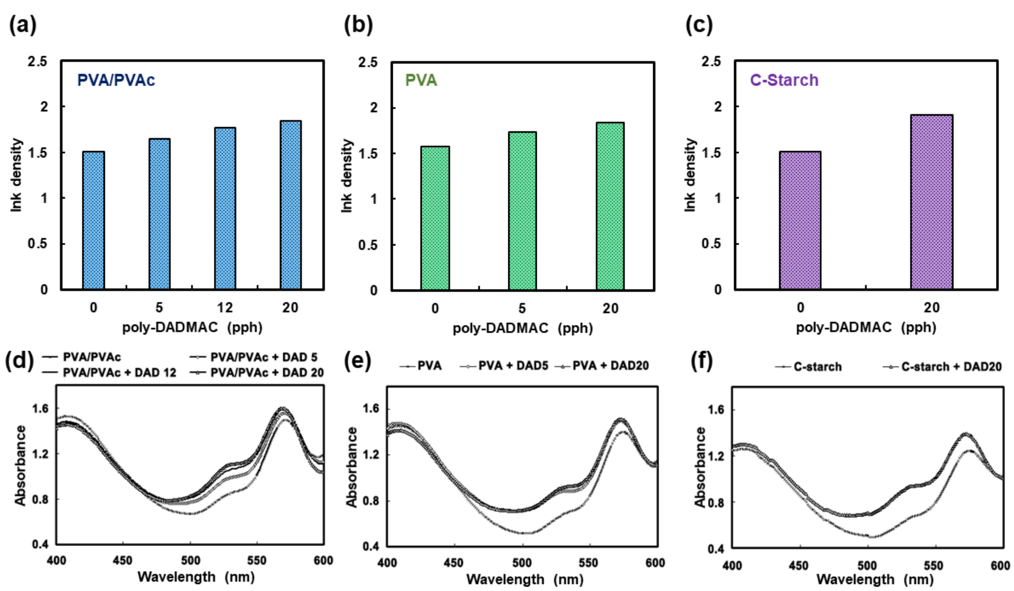

Fig. 5. The ink density results of the binder films where pigment-based ink was printed. Results from the densitometer of binder films consisted of (a) PVOH/PVAc, (b) PVOH, and (c) C-starch. Results from UV-Vis spectroscopy of binder films consisted of (a) PVOH/PVAc, (b) PVOH, and (c) C-starch. The ink density increased with the addition of cationic additives, regardless of the binder system.

Figure 6 shows the optical microscope images of the binder film after printing with the pigment-based ink. The ink density increased as more cationic additives were used. The observations from the images were in good agreement with the results in Fig. 5. One difference was that a mottled appearance was observed when the cationic additive was added to the PVOH/PVAc binder. It was speculated that the hydrophobic PVAc locally agglomerated during drying, resulting in a non-uniform binder film. Cationic poly-DADMAC appeared to agglomerate the binder to larger sizes, resulting in nonuniform ink absorption of the coating and a mottled appearance after printing. This is noteworthy because it shows that the cationic additive that increases the holdout of the colorants may cause detrimental effects, *e.g.*, print mottle, for some binder systems.

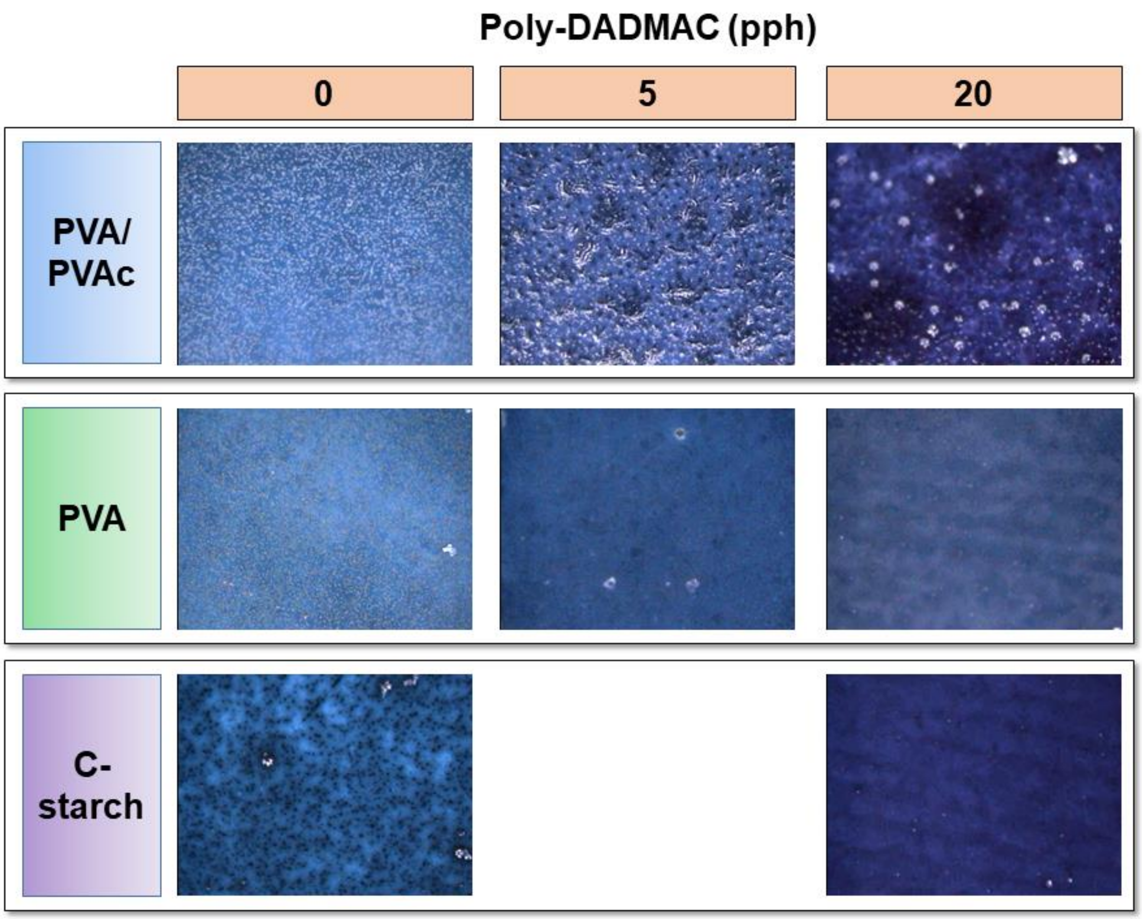

Fig. 6. Optical microscope images (x400) of printed binder films (pigment-based ink)

Figures 7 and 8 show the results of the ink density and optical micrographs after printing with the dye-based ink, respectively. The UV-Vis spectroscopy results showed that the ink density increased with the addition of cationic additive, regardless of the type of binder system. However, the addition of the cationic additive did not show a substantial difference in the visual print density. One interesting point was that the mottled appearance was not observed in the PVOH/PVAc binder system.

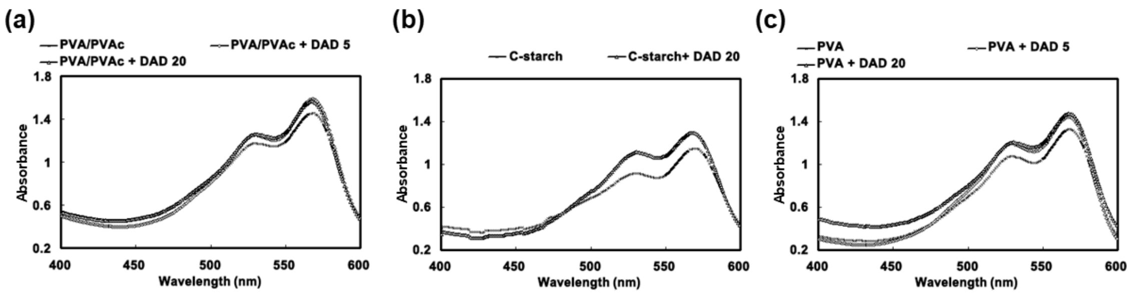

Fig. 7. The ink density results of the binder films where dye-based ink was printed. The UV-Vis spectroscopy of binder films consisting of (a) PVA/PVAc, (b) PVOH, and (c) C-starch.

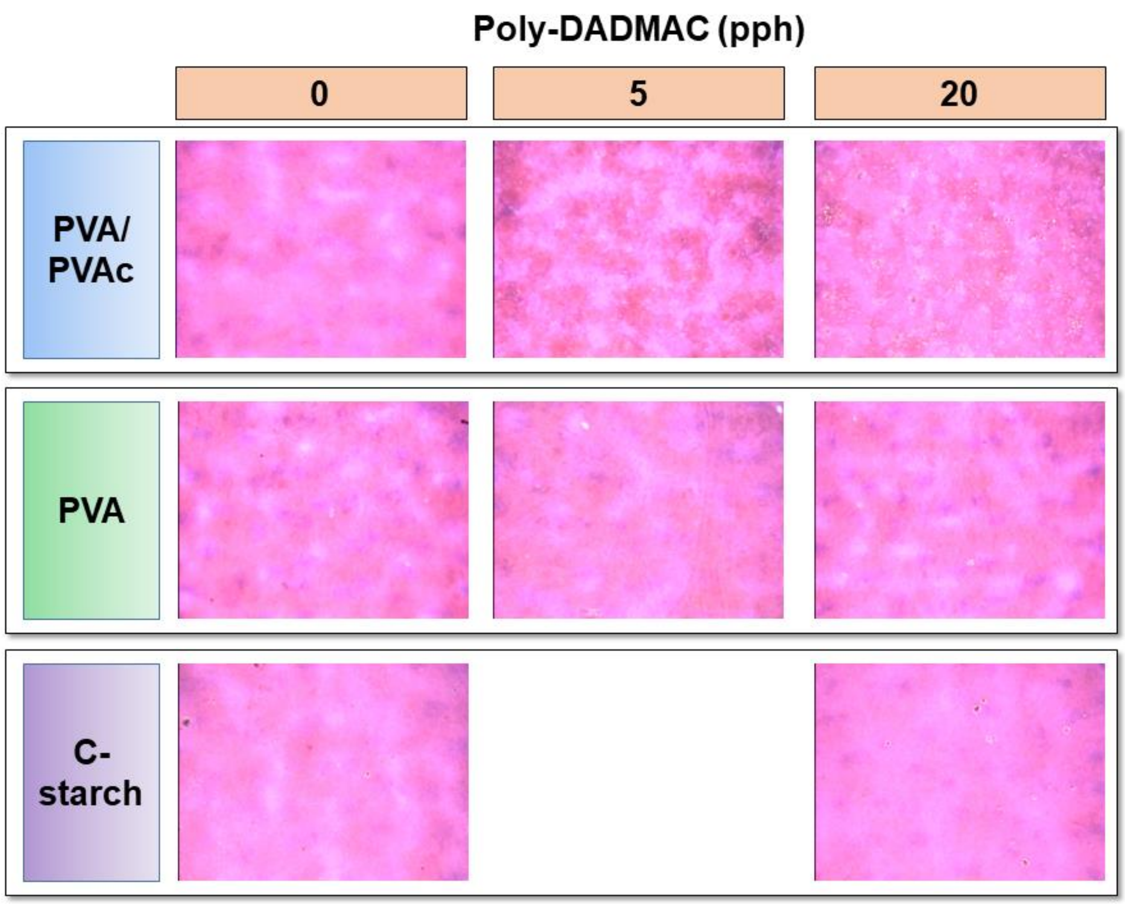

Fig. 8. Optical microscopy images (x400) of printed binder films with a dye-based ink

Influence of poly-DADMAC in Coated Paper

Figure 9a shows the ink density of the coated paper after printing. Note that 15 pph of the cationic additive, which was an excess amount for surface saturation of the silica pigment, was used. When cationic poly-DADMAC was used, dye-based inks, *i.e.*, cyan, magenta, and yellow, showed higher ink density except when PVA was used as a binder. On the other hand, the ink density of the pigment-based black ink did not change or even decreased when poly-DADMAC was used. This is because the sizes of the dye molecules are so small that they are more strongly affected by the electrostatic attraction within the coating layer. On the other hand, pigment particles, typically 20 times larger than the dyes, may be less susceptible to electrostatic attraction. Another noticeable point was that the addition of the poly-DADMAC in the PVOH/PVAc binder system (as shown in Fig. 9a), as in the binder film in Fig. 6, resulted in print mottle. This shows that the addition of cationic additives can have a detrimental effect on the printing of pigment-based ink in this coating formulations. It is known that the primary mechanism of colorant fixation for pigment-based inks is filter cake formation (Kettle *et al*. 2010). Therefore, adjusting the pore structure of the coating rather than adjusting the coating surface chemistry would be an adequate strategy for controlling the ink setting for pigment-based inks. Although the coated papers used in this study showed that cationic additives rarely improve the print quality of pigment-based inks, they may be effective for bond papers, where the swelling of the binder is the dominant mechanism of ink setting (Svanholm 2007; Chen *et al.* 2021). This can be speculated from the results of the binder films shown in Figs. 6 through 8.

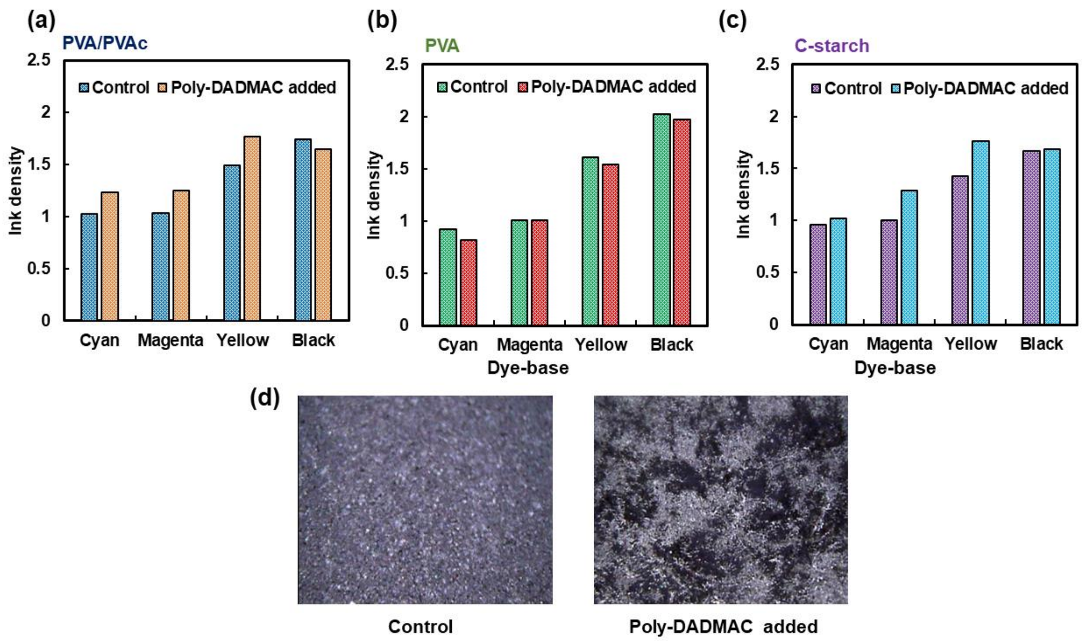

Fig. 9. The ink density of the coated paper after printing. The densitometer results of coated paper consisting of (a) PVA/PVAc, (b) PVA, and (c) C-starch binder system; and (d) optical microscopy images (x400) of printed coated paper consisting of PVA/PVAc binder system (Note: the cyan, magenta, and yellow inks are dye-based and black ink is pigment-based)

Another improvement achieved by the cationic additive was the water fastness of coated paper (as shown in Fig. 10). Coating layers prepared with two binders, *i.e.*, PVOH/PVAc and C-starch, were selected and investigated. Cationic starch is a water-soluble binder. However, half of the binder is hydrophobic in the case of the PVOH/PVAc binder system. Both coatings showed very high levels of water fastness when poly-DADMAC was used as a cationic additive and maintained an ink density of greater than 88% after immersing in water. Even though the immersion time was extended to 7 h, the water fastness did not decrease much (as shown in Fig. 10b). This indicated that poly-DADMAC strongly anchored the dye in the coating layer and provided a high level of water fastness.

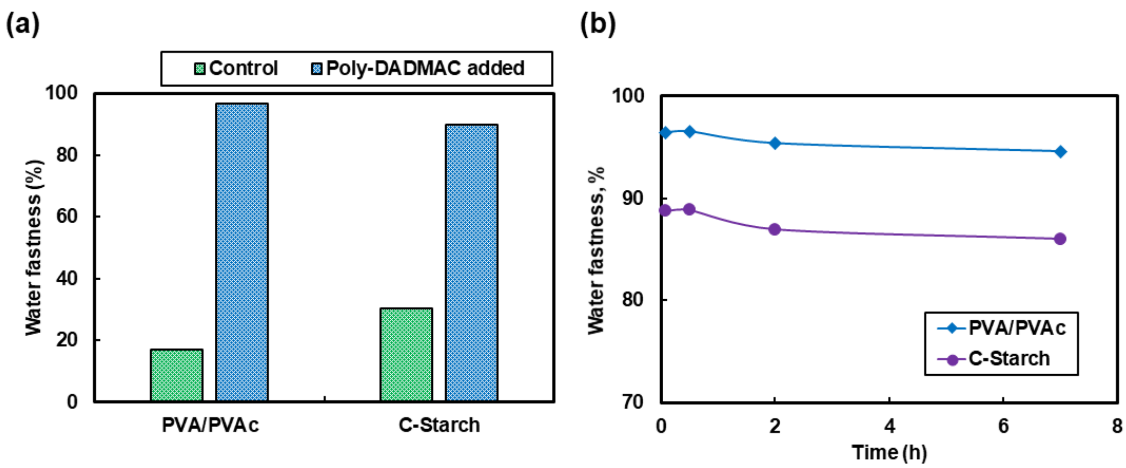

Fig. 10. The water fastness of coated paper after printing (dye-based ink): (a) water fastness of coated papers after 5 min of immersion to water; and (b) time dependent water fastness after the addition of poly-DADMAC

CONCLUSIONS

The distribution and influence of cationic additives on inkjet print quality were investigated. To improve the understanding of the distribution of the cationic additive, the adsorption behavior of poly(diallyldimethylammonium chloride) (poly-DADMAC) on silica pigments was investigated. Conclusions were as follows:

1. The zeta potential of the silica pigment rapidly increased with the addition of the cationic additive and reached the neutralization point in the dosage range between 0.3 to 0.4 wt% of poly-DADMAC. After the isoelectric point was reached, the increase rate of zeta potential was noticeably slowed down.
2. The adsorbed ratio of the cationic additive decreased as the dosage increased. A high adsorbed ratio was maintained in the typical dosage range of cationic additives, *i.e.*, less than 3 pph. Therefore, in the case of the coating formulation with 15 pph of poly-DADMAC, it was assumed that the cationic additive was adsorbed to the silica particles or remained in the aqueous phase until dried and remained in the binder film.
3. The cationic additive increased the holdout of the colorant in the binder film regardless of the ink types. In the case of the binder film consisting of poly(vinyl alcohol)/ poly(vinyl acetate) (PVOH/PVAc) binder, print mottle was observed when printing a pigment-based ink.
4. Poly-DADMAC increased the colorant holdout and water fastness of the dye-based inks, which indicated that it increased the fixing of the dye in the coating layer. However, poly-DADMAC had less effect on the holdout of a pigment-based ink. In the case of coated papers consisting of PVOH/PVAc binder, it caused a detrimental influence on the print quality by inducing print mottle.

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