

USING CATIONIC POLYMERS TO IMPROVE ALKENYL SUCCINIC ANHYDRIDE (ASA) SIZING EFFICIENCY IN HIGH-YIELD PULP CONTAINING FURNISH

Qijie Chen,^{a,*} Yonghao Ni,^{b,*} and Zhibin He^b

High-yield pulp (HYP) such as bleached chemi-thermo-mechanical pulp (BCTMP) from aspen wood, is now being used in the production of high-quality fine papers, and this is particularly true in China. In this study, the effect of using cationic polymers such as poly-aluminum chloride (PAC), polyethylenimine (PEI), cationic polyacrylamide (CPAM), and poly-(diallyldimethylammonium chloride) (PDADMAC) on Alkenyl Succinic Anhydride (ASA) sizing efficiency in HYP-containing pulp furnish was investigated. The results showed that the addition of only ASA emulsion to the HYP-containing furnish, without any cationic additives, did not yield an appreciable degree of sizing in the paper sheet. PAC (or alum) could improve the ASA sizing efficiency. PEI, PDADMAC, CPAM, and NaHCO₃ all improved ASA sizing efficiency in the presence of PAC. The charge density of PEI was important for improving the ASA sizing performance. Also, the sequence of adding the cationic polymer affected the ASA sizing performance.

Keywords: ASA sizing; Fine paper; High-yield pulp (HYP); PAC; PEI; PDADMAC

Contact information: a: College of Chemical and Biological Engineering, Changsha University of Science and Technology, Changsha, Hunan Province, 410004, People's Republic of China; b: Limerick Pulp and Paper Centre, University of New Brunswick, Fredericton, New Brunswick, E3B 5A3, Canada;

**Corresponding authors:* chenqijie@126.com (Qijie Chen); yonghao@unb.ca (Yonghao Ni)

INTRODUCTION

In recent years there has been increasing interest in using high-brightness, high-yield pulp (HYP) such as aspen bleached chemi-thermomechanical pulp (BCTMP), also known as HYP, for the partial replacement of hardwood bleached kraft pulp (HWBKP) in the production of high-quality fine paper. HYP has a low furnish cost, but high bulk, opacity, and stiffness (Connell and Cockram 2000; Yuan *et al.* 2006; Hu *et al.* 2004, 2006, 2007; Zhou 2004). In general, HYPs have less total pore volumes than the chemical pulps from the same wood species, although there are more micro-pores in HYPs; in addition, HYP fines have much more pore volume than their long fiber counterparts (Hui *et al.* 2009). HYP has a higher specific surface area and surface charge density than the bleached kraft pulp (Hu *et al.* 2004), which can affect the papermaking wet-end operations. At the same time, the presence of dissolved and colloidal substances (DCS) in HYP can also influence the sizing efficiency (Li *et al.* 2002a). Cationic polymers such as poly-aluminum chloride (PAC), polyethylenimine (PEI), and poly diallyl-dimethyl ammonium chloride (PDADMAC) are often used at the wet-end to

improve the running ability of the paper machine and the paper product quality (Wågberg and Ödberg 1991; Kekkonen *et al.* 2002).

Alkenyl succinic anhydride (ASA) is commonly used to improve the water resistance characteristics of printing and writing paper in the neutral/alkaline paper-making process. It is a highly reactive sizing agent that can impart liquid repellence to paper by decreasing the wettability of the cellulose fibres (Hodgson 1994; Isogai and Morimoto 2004; Gess and Rende 2005; Hubbe 2006; Martorana *et al.* 2008, 2011). ASA develops sizing in the paper sheet faster than alkylketene dimer (AKD), which is another widely used sizing agent (Lindström and Larsson 2008; Martorana *et al.* 2010). However, in the presence of water, or if the furnish conditions are not carefully controlled, ASA can undergo hydrolysis, which is detrimental to the ASA sizing efficiency (Hubbe 2006). When HYP is used for the partial replacement of HWBKP in the production of fine paper grades, there are a number of technical challenges (*e.g.* optical properties and sizing behaviour), which need to be overcome. Significant efforts have been made to improve the optical properties of HYP and HYP-containing papers (Li *et al.* 2002; Zhang *et al.* 2007; Liu *et al.* 2007, 2008; He *et al.* 2009). The effects of substituting HYP for hardwood kraft pulp on AKD sizing or rosin sizing have been studied (Hu *et al.* 2004; Li *et al.* 2002a,b; Zhang *et al.* 2007). In a previous study (Chen *et al.* 2012), the substitution of HYP for HWBKP in the production of fine papers and its effect on ASA sizing performance were investigated. Results showed that at a HYP substitution of less than 20%, the ASA sizing efficiency could be increased by using HYP in such a system. In the present study, further efforts were made to optimize the ASA sizing efficiency through investigation of the effects of cationic additives such as polyaluminum chloride (PAC), cationic polyacrylamide (CPAM), polyethyleneimine (PEI), and poly-dimethylammonium chloride (PDADMAC) on ASA sizing efficiency in the HYP-containing pulp system.

EXPERIMENTAL

Raw Materials

The softwood bleached kraft pulp (SWBKP) and hardwood bleached kraft pulp (HWBKP) were obtained in dry form from a mill in the northern U.S., while aspen HYP was obtained from a mill in eastern Canada. PEI (MW 25,000; MW 750,000) and PDADMAC were purchased from Aldrich Co. CPAM (Percol®292) was obtained from Ciba Specialty Chemicals. ASA (a C18 product), alum, PAC, cationic potato starch (CS) (degree of substitution, DS 0.03), and NaHCO₃ were all commercial products. The characteristics of the cationic polymers used in this study are shown in Table 1.

Table 1. Characteristics of the Cationic Polymers

Polymer	pH	Charge Density (meq/g)	Average Molecular Weight (g/mol)
PEI -A	7	6.4	750,000
PEI -B	7	6.9	25,000
PDADMAC	7	4.5	100,000
CPAM	7	1.7	5.0×10^6

Preparation of the ASA Emulsions

The ASA aqueous emulsions were freshly prepared using the following procedure. The cationic potato starch slurry at 4% solids was heated to 95°C and held at that temperature for 30 minutes with stirring. The cooked starch was cooled to room temperature, and then ASA was added for emulsification. The amount of liquid ASA in the emulsion was 1%, and the mass ratio of cationic starch to ASA was 4:1. ASA emulsions were prepared in batches with a variable-speed homogenizer (IKA, T25, Germany).

For the emulsification, the rotor speed was set to 20,000 rpm, and the shearing time was 2 minutes. The ASA emulsion prepared was uniform and homogeneous (Chen *et al.* 2012), and the freshly prepared ASA emulsion was used in the experiments.

Determination the Charge Density of Cationic Polymers

The charge density of each cationic polymer at 10 mg/L was determined by titration with 0.5 mN Potassium polyvinyl sulfate (PVSK) using a Particle Charge Detector, Mütek PCD 03 (BTG, Herrsching, Germany facility). Three repetitions were conducted to get an average for the charge density of each cationic polymer. All the charge density data were collected at pH 7.

Handsheet Preparation

The SWBKP and HWBKP were refined in a PFI mill to 400 CSF and 500 CSF, respectively. The aspen HYP was used as received without further refining treatment. To prepare ASA-sized handsheets, a pulp slurry of 0.3% consistency was prepared, which consisted of 30% SWBKP, 50% HWBKP, and 20% HYP. According to the specification, cationic polymers such as PEI or PDADMAC were added first, then PAC and ASA emulsion were added into the mixed pulp slurry. For those trials with the addition of CPAM, the PAC and ASA emulsion were added first, followed by the addition of CPAM to the mixed pulp slurry.

For those trials with the addition of NaHCO₃, the required amount of NaHCO₃ was added first, followed by the addition of PAC and ASA emulsions to the mixed pulp slurry. The pH of the slurry was then adjusted to about 7. The delay time period between addition of successive chemical agents was 45 seconds. The dosage of all the additives was based on the active substance.

Handsheets with a basis weight of 60 g/m² were prepared according to TAPPI method T205 om-88. The number of paper sheets prepared for each condition was six. Sizing testing was then carried out after reconditioning the handsheets at 23°C and 50% relative humidity for more than 12 hours in accordance with TAPPI method T530 om-96 (Hercules Size Tester, HST).

No additional curing was performed. A Hercules Size Tester (Model HST-1F) was used, and the ink was prepared using equal volumes of Hercules Ink-Pak dye solution (2.5%) and 2% formic acid. Six HST results were obtained, and the average was reported.

RESULTS AND DISCUSSION

Effect of PAC (or Alum) Dosage on ASA Sizing

As can be seen from Fig. 1, when only the ASA emulsion was added to the HYP-containing pulp furnish, without any retention-promoting chemical at all, the sizing degree of the handsheet was negligible. PAC or alum was effective in improving the ASA sizing efficiency. The sizing degree reached its maximum at the alum and PAC charge of 2 to 2.5%. PAC and alum are widely used in the rosin acid and ASA sizing to improve the sizing efficiency (Hodgson 1994). PAC and alum can increase the ASA size retention and at the same time can anchor the ASA size effectively, thus improving the sizing efficiency (Martorana *et al.* 2010, 2011). In addition, the ASA-alum hydroxide complexes may also contribute to the sizing degree (Hodgson 1994). However, if the dosage is too high, PAC or alum may decrease the sizing degree due to the fact that the surface negative charge of pulp fibres is too low.

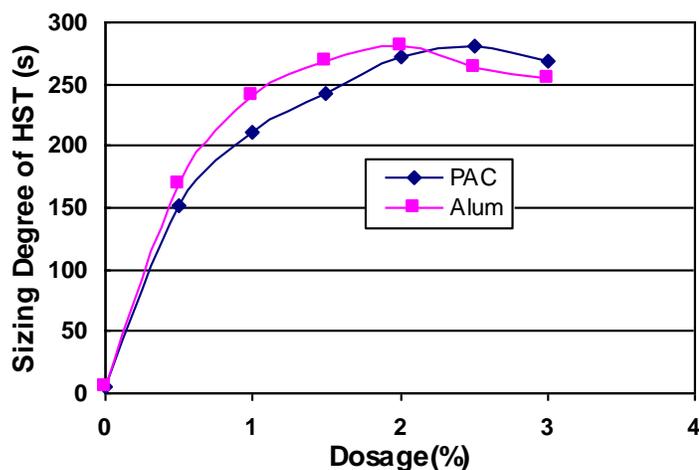


Fig. 1. Effect of PAC (alum) dosage on ASA sizing (0.3% ASA, 20°C)

Effect of PAC (alum) Addition Order on ASA Sizing

Since the PAC and alum have positive charges and the ASA emulsion also has a cationic surface charge (a cationic starch was used during the emulsification process), the influence on the ASA size retention and sizing degree of their order of addition to the pulp suspension was investigated in the absence of CPAM. The results, as shown in Fig. 2, were in the following order, based on decreasing sizing degree:

- 1) Adding first PAC or alum to the mixed pulp furnish, then ASA (denoted as PAC (alum) → ASA);
- 2) Adding ASA first to the mixed pulp furnish, then PAC or alum (denoted as ASA → PAC (alum)).

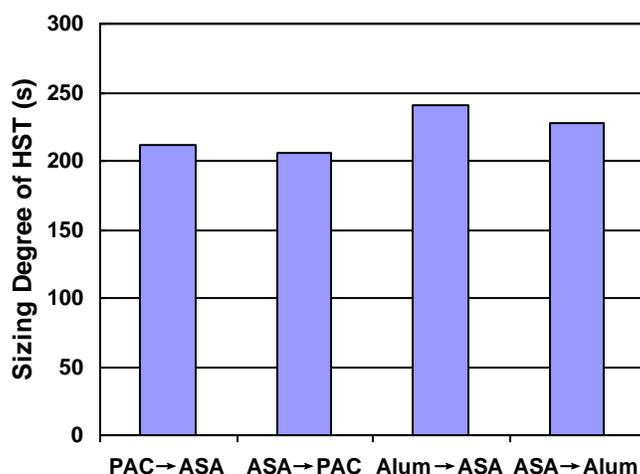


Fig. 2. Effect of the order of addition of PAC (alum) on ASA sizing (1% PAC or alum, 0.3% ASA, 20°C) in the absence of acrylamide-type retention aid

It can be seen that the order of addition PAC (alum)→ASA resulted in a somewhat higher sizing degree than that of ASA→PAC (alum). This can be explained by the fact that the addition of the PAC or alum to the pulp mixture containing HYP neutralized some of the anionic trash that came mainly from the aspen HYP, thus improving the ASA size retention. Also the aluminium products may boost the ASA sizing due to its better anchoring and alignment of ASA or the acid molecules (Martorana *et al.* 2010, 2011).

Effect of Pulp Temperature on ASA Sizing

As shown in Fig. 3, the pulp temperature had a strong effect on the ASA sizing efficiency.

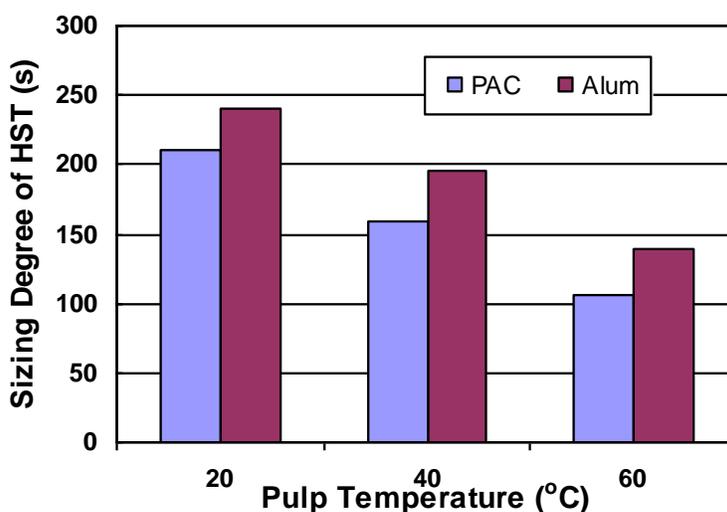


Fig. 3. Effect of pulp temperature on ASA sizing (1% PAC or alum, 0.3% ASA)

When the pulp temperature was increased, the HST sizing degree decreased. When the pulp temperature was 60°C, the ASA sizing degree decreased by 49.8% and 42.3% for the PAC and alum system, respectively, compared to their sizing degree at 20°C. The ASA emulsion could be hydrolyzed to a significant extent. The higher the pulp temperature, the more the ASA emulsion was hydrolyzed. With more and more white water recycling in paper mills, the pulp temperature can be expected to rise, thereby negatively influencing the ASA sizing efficiency.

Effect of PEI on ASA Sizing

Figure 4 shows the effect of PEI on ASA sizing efficiency, still in the absence of CPAM. As can be seen, in the presence of PAC, PEI-A, or PEI-B, the sizing degree improved remarkably. When the PEI dosage was 0.2%, the sizing degree of the handsheets was nearly at 500 s, which is about 137% higher in comparison to the sizing degree without PEI addition. However, when the PEI dosage was more than 0.2%, the sizing degree of the handsheets decreased dramatically. The reason for this is that PEI with a high cationic charge density can function as a charge neutralization agent, neutralizing the anionic trash in the mixed pulp and improving the ASA size retention. However, when too much PEI is added, it makes the pulp system too cationic, thus decreasing the ASA size retention. PEI is also known to improve the AKD and rosin acid sizing in the HYP-containing system by neutralizing the anionic trash and improving the AKD and rosin acid retention (Li and Ni 2001; Li *et al.* 2002b; Hu *et al.* 2004).

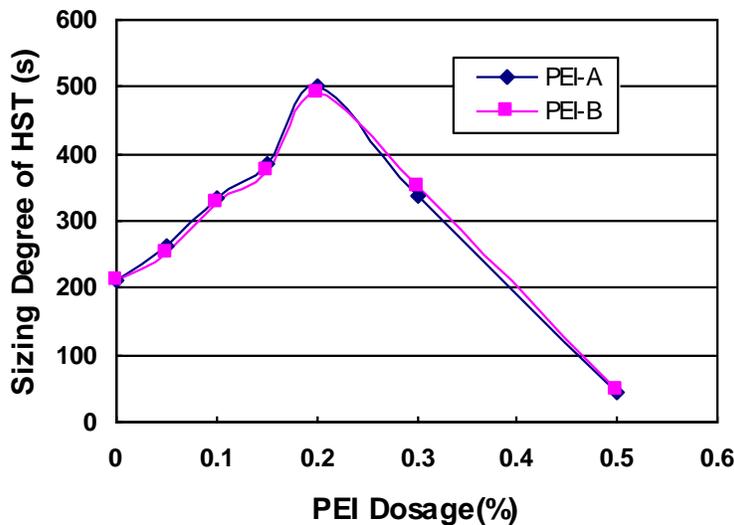


Fig. 4. Effect of PEI on ASA sizing (1% PAC, 0.3% ASA, 20°C)

Figure 4 also shows that PEI-A and PEI-B had similar effects on the ASA sizing efficiency. PEI-A has a similar charge density but higher molecular weight than PEI-B. These results supported the conclusion that the molecular weight of PEI may not be critical for improving the ASA sizing performances in the HYP-containing furnish; rather, its charge density is.

Effect of PDADMAC on ASA Sizing

Figure 5 shows the effect of PDADMAC on ASA sizing efficiency (without the use of CPAM). As can be seen, in the presence of PAC, PDADMAC further improved the ASA sizing efficiency. When the dosage of PDADMAC was 0.3%, the sizing degree was about 580 s, which is about a 174% increase from the sizing degree without any PDADMAC. However, when its dosage was 0.3%, the sizing degree reached a plateau, which was different from those of PEI on ASA sizing (Fig. 4). PDADMAC is widely used as an anionic trash catcher in the paper industry. In the present system, PDADMAC effectively neutralized the anionic trash from the HYP. Earlier studies have shown that PDADMAC promotes AKD sizing (Li *et al.* 2002b).

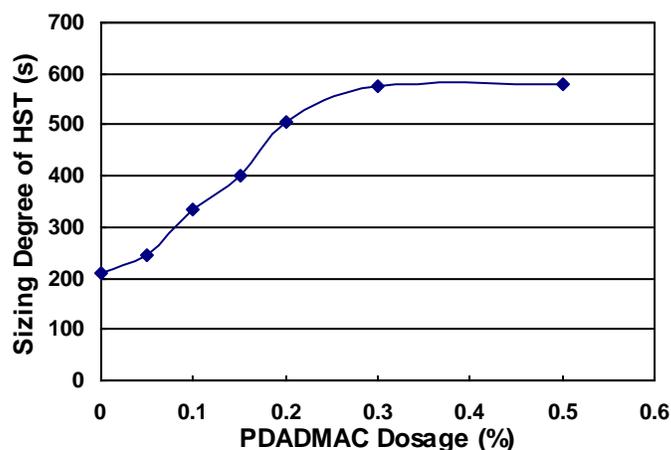


Fig. 5. Effect of PDADMAC on ASA sizing (1% PAC, 0.3% ASA, 20°C)

As shown in Figs. 4 and 5, PEI and PDADMAC had different influences on ASA sizing when the dosage was more than 0.3%. This could be related to the difference in the charge densities of these two cationic polymers. The primary amine and secondary amines are dominant in PEI, while the quaternary amines are dominant in PDADMAC. The charge density of PEI used in the experiments was higher than the PDADMAC (Table 1). Adding too much of a higher charge density polymer of PEI into the pulp system would make the pulp fiber system too cationic and the fibers and fines excessively flocculated, thus negatively influencing the ASA size anchoring and orientation on the surface of fibers and decreasing the ASA sizing efficiency.

Effect of CPAM on ASA Sizing

Figure 6 shows the effect of CPAM on ASA sizing efficiency. As can be seen, in the presence of PAC, CPAM improved the ASA sizing efficiency, and the HST sizing degree reached 300 s when the CPAM dosage was 0.09%, an increase of about 42% from the sizing degree without the CPAM addition. However, when the CPAM dosage was more than 0.09%, the sizing degree of the handsheets decreased. CPAM with high molecular weight and low charge density is widely used as a retention aid in the wet-end operations of papermaking.

The CPAM added into the mixed pulp acts as a retention aid by way of bridge flocculation action and increases fine retention in the pulp system. As a result, the ASA size retention in the pulp, and consequently the sizing degree, can be improved. On the

other hand, the liquid penetration of paper depends not only on the paper chemistry but also on the porous structure of the paper and the diffusion of water into the fiber wall (Hu *et al.* 2004). When CPAM dosage is too high, CPAM with high molecular weight can also induce serious fiber flocculation, increasing the porosity of the handsheets. This means that CPAM may have had a very negative effect on the formation uniformity of the handsheets, consequently decreasing the sizing degree.

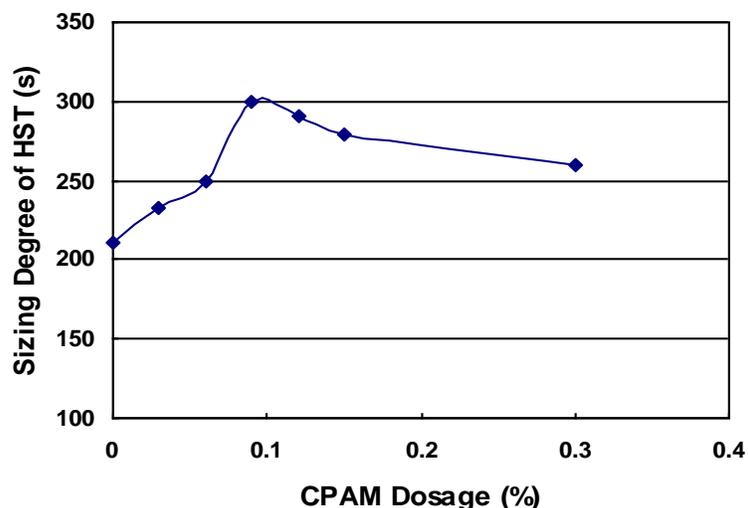


Fig. 6. Effect of CPAM on ASA sizing (1% PAC, 0.3% ASA, 20°C)

Effect of NaHCO₃ on ASA Sizing

It can be seen in Fig. 7 that, in the presence of PAC, sodium bicarbonate improved the sizing degree of the handsheets.

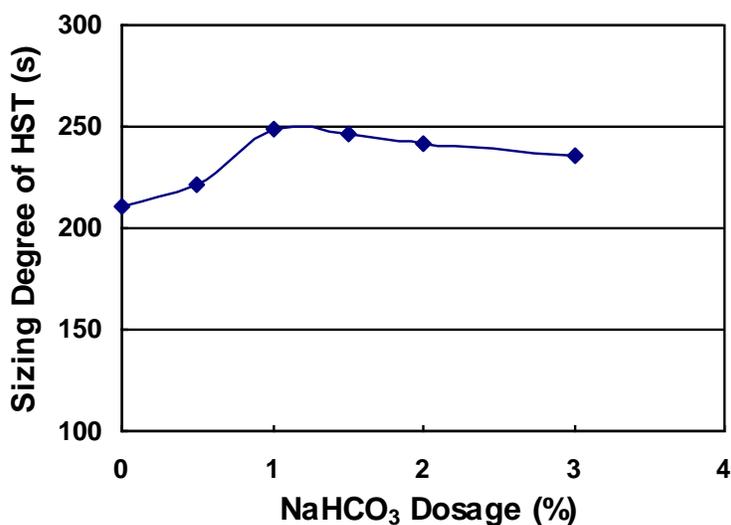


Fig. 7. Effect of NaHCO₃ on ASA sizing (1% PAC, 0.3% ASA, 20°C)

When the dosage of NaHCO_3 was 1%, the sizing degree of HST was about 250 s, which is an increase of about 18% from the sizing degree without the NaHCO_3 addition. When the dosage was more than 1%, the sizing degree decreased slightly, but it was still higher than it was without the NaHCO_3 addition. Sodium bicarbonate can increase the swelling of pulp fibers (Hodgson 1994), which facilitates the interactions between the ASA emulsion and the fibers. Also, the NaHCO_3 addition yielded the appropriate alkalinity of the system, thus improving the ASA sizing efficiency.

Effect of Cationic Additive Addition Sequence on ASA Sizing

Two different addition sequences were investigated for their ability to decrease the negative effects of anionic trash: one was to add the ATC (PEI-A, PEI-B, or PDADMAC) to the mixed pulp furnish (30% softwood kraft, 50% hardwood kraft, and 20% HYP), then to add PAC and ASA emulsion, and lastly to add CPAM; the other was to add the ATC (PEI-A, PEI-B, or PDADMAC) to the HYP (20%) first, then to mix it with the kraft pulp (30% softwood kraft, and 50% hardwood kraft), and then to add PAC and ASA emulsion, and lastly to add CPAM. The results are shown in Fig. 8. As can be seen, when CPAM was the final additive, adding the ATC to the HYP was more effective in increasing the sizing degree than the other option of adding it to the mixed furnish. The results can be explained as follows: PEI and PDADMAC acted mainly as the anionic trash catcher in the present system. By adding ATC to the HYP, it was able to neutralize the anionic trash more effectively than that of adding the ATC to the mixed furnish.

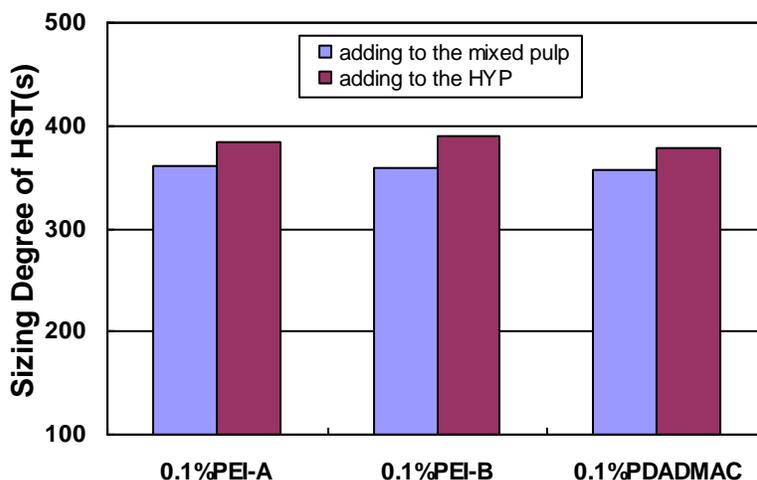


Fig. 8. Effect of PEI (PDADMAC) addition sequence on ASA sizing (1% PAC, 0.3% ASA, CPAM 0.03%, 20°C)

Results pertaining to the effect of different cationic agent addition sequences on ASA sizing are shown in Table 2. The addition sequence was: first adding the ATC (PAC, or PEI-A, or PEI-B, or PDADMAC) to the HYP (20%), then mixing it with the kraft pulp (30% softwood kraft, and 50% hardwood kraft), then adding ASA emulsion, and lastly adding CPAM. As can be seen, in the presence of CPAM, the ATC (PAC, PEI-A, PEI-B, or PDADMAC) further improved the ASA sizing efficiency; the PAC effect was the best, probably because of its higher ASA size retention and the improved anchoring and alignment effect of ASA or the acid molecules (Martorana *et al.* 2010, 2011).

Table 2. Effect of Cationic Additives Addition Sequence on ASA Sizing

Cationic Additives Addition Sequence	Sizing Degree of HST (S)
ASA----CPAM	28
PAC-----ASA-----CPAM	233
PEI-A-----ASA-----CPAM	170
PEI-B-----ASA-----CPAM	192
PDADMAC-----ASA-----CPAM	138

(1% PAC, 0.1% PEI-A, 0.1% PEI-B, 0.1% PDADMAC, 0.3% ASA, CPAM 0.03%, 20°C)

CONCLUSIONS

1. The addition of only ASA emulsion to the HYP-containing furnish, without any cationic additives, did not yield an appreciable degree of sizing in the paper sheet. PAC (or alum) effectively improved the ASA sizing performance in such a system due to improved retention and anchoring. When the dosage of PAC or alum was 1%, the HST sizing degree was more than 200 s. When the pulp temperature increased, the ASA sizing efficiency decreased because of the ASA emulsion hydrolysis.
2. Cationic polyelectrolytes having high density, *i.e.* PAC, PEI, or PDADMAC, were shown to function as anionic trash catchers in the HYP-containing pulp furnish, thereby improving the sizing degree remarkably. The charge density of PEI was important for improving the ASA sizing performance. CPAM with a high molecular weight and low charge density and NaHCO₃ also improved the ASA sizing efficiency in the presence of PAC. The cationic polymer addition sequences can also affect the ASA sizing performance. Adding the ATC to the HYP was somewhat more effective in increasing the sizing degree than was adding it to the mixed furnish. When CPAM was the final additive, the addition of ATC (PAC, PEI-A, PEI-B, or PDADMAC) further improved the ASA sizing efficiency; of the four additives PAC was the most effective.

ACKNOWLEDGMENTS

This work was financially supported by the International Science and Technology Program (ISTP) of China and Canada, and the Canada Research Chairs' Program.

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Article submitted: February 16, 2012; Peer review completed: June 16, 2012; Revised version received: June 25, 2012; Further revisions: July 7, 2012; Accepted: July 8, 2012; Published: July 11, 2012.