

Applications of aqueous nano-sericite to a dual-chemical papermaking retention system

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Abstract

A widely used dual-chemical retention system in the paper industry containing polyacrylamide (P) at 300 ppm and globular colloidal silica (GS) at 430 ppm (calculated to actual solid content) was examined. GS was substituted with an indigenous mica mineral, nano-sericite (NS), and effects at different doses on the paper properties were investigated. Concept of electrical charge balance was included in the experimental design to obtain optimal NS doses. A set of experiment was carried out by fixing dose of P and maintaining fiber surface charge at -40 ± 2 mV, which comply with the commonly used GS condition. Another set added NS at doses of 200, 400, 600, 800, and 1000 ppm (calculated to actual solid content). Pulp furnishes were then formed into standard handsheets for paper properties examination. Optimal conditions entailed a P: NS ratio of 300: 600 ppm. Afterward, a 2² factorial design was applied, and paper physical properties were analyzed with ANOVA to delineate the effects of polymer and microparticle interactions on the dual-chemical system. In addition, NS was observed with TEM to provide clues to its roles in the paper forming process. The results indicated that substituting NS for GS enhanced mechanical strengths of the handsheets. Under the optimal conditions, tensile index of handsheets increased from 35.8 ± 1.02 to 41.1 ± 1.26 N·m/g; burst index increased from 2.20 ± 0.08 to 2.43 ± 0.04 kPa·m²/g; elongation rate increased from 3.33% to 4.57%; folding endurance (MIT) increased from 8 to 13. In addition, there was no significant change in the first pass retention, ash content and brightness due to the substitution, which were 84.01%, 16.67%, and 82.67% ISO, respectively. Along with increased NS doses, ash content of the handsheets increased from 15.94% to 17.03%; brightness increased from 82.99 to 83.11% ISO. Tensile index, first-pass retention, and rate of elongation showed an increasing and then decreasing trend. Tensile index and first-pass retention reached maximum at 400 ppm NS charge, at 41.14 ± 1.21 N·m/g, and 84.71%, respectively; burst index and rate of elongation reached maximum at 6000 ppm NS charge, at 2.43 ± 0.04 kPa·m²/g, and 4.57%, respectively; tear index, smoothness, and air permeability didn't change much with varying NS doses. Results of the ANOVA indicated that at a 95% confidence interval, NS dose affected all paper physical properties significantly; while P dose affected first-pass

retention, tensile index, rate of elongation, folding endurance and ash retention significantly.

Key words: Nano-sericite, dual-chemical retention system, tensile index, first-pass retention, charge balance.

1. INTRODUCTION

Dual-chemical retention system has been used in papermaking for decades. Presently, several commercial systems supplied by BASF, Eka, Nalco, Ashland etc. are well-known. The main concept of the system is to apply a relatively low-molecular weight polyacrylamide or similar polymers with high cationic charges to the pulp stock in conjunction with a microparticle of micro- or nanometer scale, such as spherical colloidal silica or a high molecular weight and branched anionic polymers, to increase flocculation of the fibers and paper strengths. Mechanism of the dual-chemical retention system is through both physical and chemical interactions to reactivate the active sites along the cationic polymer chains in the form of loops and ends which tend to flatten onto fiber surface and lose their opportunity to interact with the negative charged particles in pulp suspension. Microparticles, with their negative charges, are proposed to cause the flattened loops and tails of the positively charged polymer to detach from fibers and again allow adsorption of dissolved and colloidal particles onto the polymer chains and eventually onto fibers[1-4] noted that by proper control of various conditions of a retention system, the first pass retention, drainage rate, and paper strengths were increased. In addition, paper formation, printability and the two-sidedness of paper were also improved. Because the minute fines and filler particles are effectively attached to paper web, drainage improves; paper machine operates faster, material loss decreases. And the productivity and economy gains while wire life and steam consumption also benefited[5, 6].

Flocculation mechanisms of fines and filler particles are thought to include: 1) physical or chemical adsorption; and 2) filtration entrainment upon formation of paper web. The coalescing and agglomeration actions cause particles or fines to form microflocs which eventually retained optimally onto paper web and achieved optimal retention control[7-9].

Factors affecting the performance of a dual-chemical retention system are manifold, including molecular weight and charge density of the polymer, the size, shape, charge density of the microparticle and the attractive force between cationic polymer and microparticle all exert their influences on the results. In addition, fiber dimensions, ionic strength and pH of pulp suspension, as well as the concentrations of cationic polymer and microparticle all affected flocculation of the pulp and overall retention [4, 9, 10]

In this study, a preparation of aqueous nanosericite (NS) was used to substitute globular colloidal silica (GS) as the microparticle a dual-chemical retention system. The NS was prepared using a hydrothermal process. Sericite is an mica mineral produced in a mine in eastern Taiwan. It's a platy mineral with 3-layered unit cells of SiO_2 (tetrahedral)- Al_2O_3 (Octahedral)- SiO_2 (tetrahedral). The octahedral is formed by 2 strata of hydrogen atoms coordinating an aluminum atom and form extended planes by sharing common edges with neighboring aluminum atoms. The tetrahedron is formed by a silicone atom with 4 oxygen atoms, which connects together by sharing corners with its neighbors. Because silicone atoms are substituted en masse with aluminum atoms, there are high charge densities between the plate layers. The sericite crystal lattice contains numerous potassium ions to balance the charge.

Nanosericite in aqueous phase is a novel material, there are presently only a few research mentioned about its potential applications to wet end of papermaking and performance [11-13]. Hence, in this study, we conducted transmission electron microscopy (TEM) observations of the self-prepared NS and commercial GS and size distribution measurements. These chemicals were then used in a dual-chemical retention system, using NS to substitute GS as the microparticle and examined their respective effects at different doses on the handsheet paper properties. In addition, an ANOVA analysis was applied to the results of a 22 factorial experimental design

to delineate the effects of different cationic polymer and NS doses.

2. EXPERIMENTAL

Experimental design

We conducted separate particle size distribution measurements of the NS and GS, and their TEM observations. In accordance with the experimental results of Solberg and Wågberg[8], the retention aid polymer and GS were added to pulp stock at optimal conditions. For which the dose condition of P3GS (for designations, see the end of next paragraph) was measured to provide the norm for fiber surface charge. Fiber furnishes with different doses of NS substituting for GS were then titrated to find equivalent fiber surface charges as P3GS. The specific NS dose was regarded as the optimal NS dosing concentration. Furthermore, handsheets prepared from different NS doses were also tested to investigate the effect of maintaining the same cationic polymer dose while varying NS doses on the first pass retention, ash retention, and handsheet physical and optical properties.

Finally, an experimental design of 22 factorial using both cationic polymer (P) and microparticle (NS) doses as independent variables was carried out and the results analyzed using the IBM SPSS statistics 20 software for a one-way ANOVA to examine the benefit of using NS in the microparticle system. Because the original solids content of the 2 microparticle systems were different (GS at 7%, and NS at 10%), for ease of comparisons, all subsequent dosing converted them to actual solids content in ppm. Thus, in the study, we used designations of P_xNS_y , the x and y respectively represented dosing of P and NS. The unit of doses was 100 ppm. Thus, P3NS3 suggested a dual-chemical doses of 300 ppm of P and 300 ppm of NS.

Chemicals

The following chemicals and fiber were used to make handsheets: Flocculent cationic polyacrylamide (P): Eka PL1510, ($M_w=5.5-5.7 \times 10^6$, density = 0.8 g/cm³, 88% solids content); GS: EKa NP882, (15% solids content, average size =

31.68 nm, specific surface area = 700 m²/g, with anionic surface charges, solid content 7 %); and NS.

The NS was self-prepared at our lab using a hydrothermal process. In which sericite: NaOH solution at 1: 15 ratio was mixed in an autoclave and heated to 240°C for 4 h to achieve intercalation of the plate layers of the mineral. Upon cooling, the sericite was acidified with 40% nitric acid to swell the mineral, and then NaOH was used to adjust pH to neutrality. At which time, the sericite became exfoliated and appeared translucent gel-like. The exfoliated NS was then repeatedly washed with ultracentrifugation (10,000 rpm) to remove entrained salts. The exfoliated NS was then freeze-dried. And then epoxy silane was suitably added under pH 6, adjusted by acetic acid, to coat exfoliated sericite plates forming a uniform aqueous dispersion. At 10% solids content, the NS has CEC = 85.9 meq/100 g, and an average particle size = 120.7 nm.

Other chemicals used in the handsheet preparation included: cationic starch, ZMCAT-T305-1, (D.S.=0.01%, Zhi-mao Starch, Taiwan); sizing agent AKD, TD-15, (15% solids content, Ashland, Taiwan); and filler, CaCO₃, Hydrocarb 75F, 15% solids content, pH 9.23, ISO brightness 94.9%, particle diameter 0.4–2.0 μm (Omya, Taiwan). The pulp stock, with 81% ISO brightness, was prepared using a bleached hardwood kraft pulp (BHKP) at 3% consistency and having 650 mL CSF.

Characterization of nanosericite

The aqueous dispersion of NS and GS were caught using a carbonized copper mesh and observed with transmission electron microscopy (JEM-1400, Jeol Co., Japan). A dynamic laser scattering device (DLS, Nano S 90, Malvern Co, UK) was used to determine the particle size distribution.

Stock preparation

Pulp refining in the laboratory was based on simulations of the mill site furnishing 150 principles. A laboratory Valley beater (model 303, Liensheng, Taiwan) was used to refine the BHKP from their original freeness to 430 mL CSF. The pulp then diluted to 0.3% for further experiments.

Addition of wet end chemicals also simulated the on-site practice with the following chemicals addition sequence and the doses were based on the oven-dry weight of the pulp: pulp at 0.3% consistency; sizing agent (AKD TD15, 1 %); cationic starch 1%; filler 25%; and PGS or PNS. For the fiber surface zeta potential determination, after adding the chemicals, 500 mL portion of the stirred pulp suspension was taken and measured with an SZP meter (Mütek SZP 04) to determine the zeta potential. In addition, a particle charge detector, Mütek PCD 03 was used in conjunction with an automatic titrator (Titraline alpha RS-232-C) to determine the anionic charge of the pulp filtrate. The standard solution for the titration was a 0.001 eqL⁻¹ cationic poly-DADMAC solution.

In the study, the cationic polymer retention aid dose was maintained at 300 ppm for all cases. Varying doses of NS were then added with combinations of P3NS2, P3NS4, P3NS6, P3NS8 and P3NS10. In the determination of first pass retention rates, a dynamic drainage jar (DDJ, Electrocraft & QMC, USA) was used to obtain pulp filtrate. A pulp sample of 500 mL at consistency C_i was put in DDJ and stirred with a impeller at 1000 rpm for 20 s, the distance between impeller and wire was 3.175 mm. Then the spigot was turn on to drain the suspension. Two hundred mL of filtrate was collected and oven-dried to obtain its solids content and calculate filtrate consistency C_0 . Then FPR % was defined as $(C_i - C_0)/C_i \times 100$.

In addition, in order to understand the effects of various additive, we've also examined the cases of pure pulp only; pulp with other additives and no microparticle (either GS or NS), in which to 0.3% consistency pulp, 300 ppm of cationic polymer, sizing agent (AKD TD15, 1 %), cationic starch 1%, and filler 25% were sequentially added; and pulp with addition of all other chemical without the dual chemical addition were carried out for comparisons.

We've also conducted a set of 2² factorial experiments. In which the 2 dual-chemical retention systems were compared. Based on the optimal commercial doses in which P3GS = 300: 430 ppm; and the optimal NS substitution amount, at the same zeta potential, of P3NS6 = 300: 600 ppm. These doses were set

as the middle values of the 2² factorial. In the design, the P and NS doses respectively varied by increment of 100 ppm and 400 ppm. Thus, in the 2² factorial design, in addition to the middle point condition of P3NS6, there were P2NS2, P2NS10, P4NS2, and P4NS10.

Handsheet preparation

Handsheet formation was based on the method of TAPPI T205 sp-95. The actual procedure was as follows: 1) pulp was disintegrated at 0.3 % consistency with a standard disintegrator; 2) sizing agent (AKD, 1 %), cationic starch 1%, filler 25% was added in sequence separately and maintained stirring for 30 min; 3) polyacrylamide was added and stirred for 60 s; 4) microparticle was added and stirred for 60 s; 5) handsheets of 60 g/m² was formed using a standard sheet mold, air-dry overnight; 6) handsheets conditioned in the standard atmosphere for > 24 h; and 7) handsheet physical and optical properties were measured.

Handsheet standards

The physical and optical properties of handsheets were tested according to various ISO standards: i.e., ash content, ISO 2144: 2000; brightness, ISO 2470-1: 2008; grammage, ISO 536: 1995; smoothness, ISO 8791-2:2013; tear index, ISO 1974: 1990; tensile index, ISO 1924-2: 1994; air permeability, ISO 5636-5: 2003; opacity, ISO 8791-4: 1992, and stiffness, ISO 5628: 2012.

3. RESULTS AND DISCUSSION

Particle size distributions of the microparticles and their TEM observations

The particle size distribution of GS and NS and their TEM micrographs are shown in Figs 1 and 2.

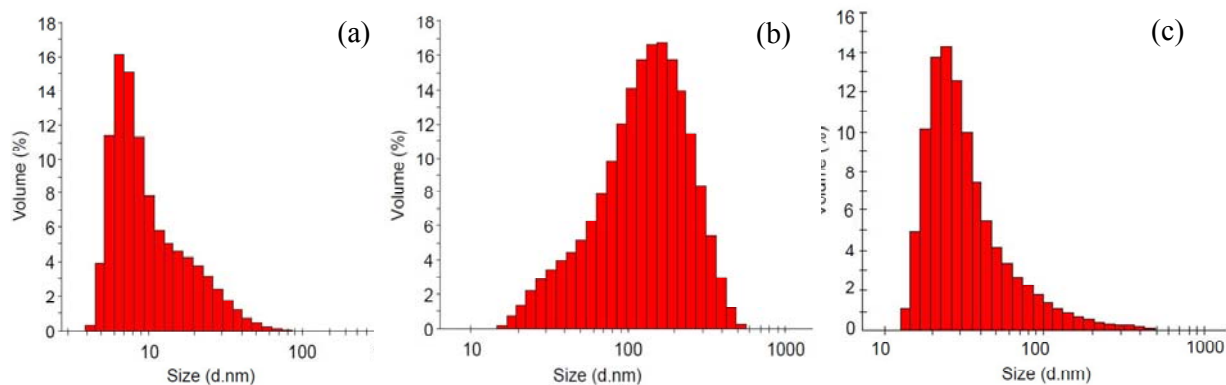


Fig. 1. The particle size distributions of (a) GS; (b) NS before coating, and (c) NS as aqueous solution after epoxy resin coating.

Figure 1(a) indicated that the commercial GS has sizes mostly around < 10 nm. The distribution was fairly uniform

Effects of adding nanoparticles on the pulp surface and effluent

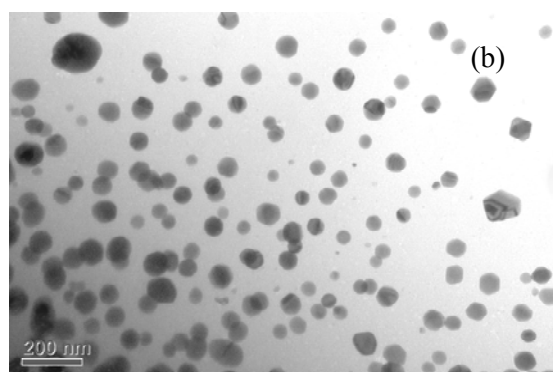
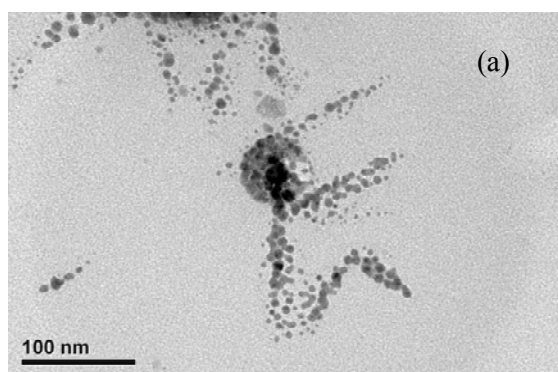


Fig. 2. The TEM micrographs of (a) GS, and (b) NS.

without large aggregate; Figs 1(b) and (c) suggested that before epoxy coating, NS particles were mostly about 200~300 nm diameter, whereas, after the treatment, the particles were less liable to self-aggregate and had dimensions mostly around 20~50 nm and average size of 120.7 nm.

In order to visualize the appearances of GS and NS, TEM micrograph observations are shown in Fig. 2.

Figure 2 shows that the GS was in a form of branched network of 25 silicate spheres each about 2.5 nm in diameter. The epoxy coated NS appeared spherical and uniformly suspended in medium. Each NS globule had diameter of ca. 50-100 nm. The GS and NS thus appeared to have significant differences both in appearance and state of dispersion in water.

charges

Changes in fiber surface charge and effluent charge upon addition of different doses of nanoparticles are shown in Figs. 3 and 4 below:

Figure 3 shows that at the GS dose of 600 ppm, the fiber surface charge reached a level most closely approximate that of the optimal condition at GS 430 ppm established by Solberg and Wågberg[8]. Figure 4 shows that at NS dose > 600 ppm, the anionic trash in filtrate increased markedly, indicating that excessive amount of NS was not retained on the fiber mat.

Analysis of handsheet physical properties

In this study, pure pulp, a base pulp furnish (pulp+, with

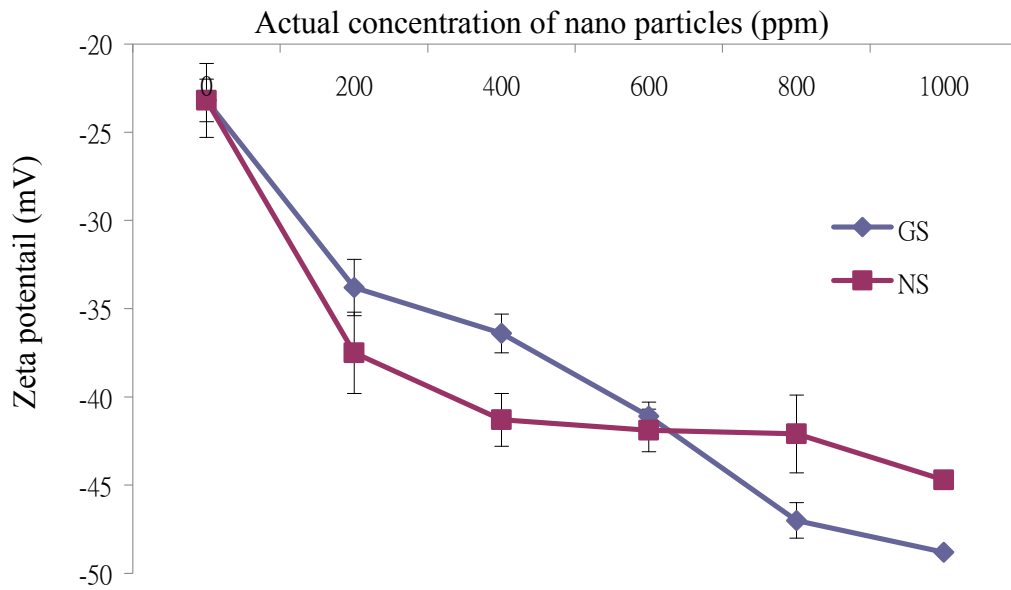


Fig. 3. Effects of different doses of nanoparticles on the fiber surface charges.

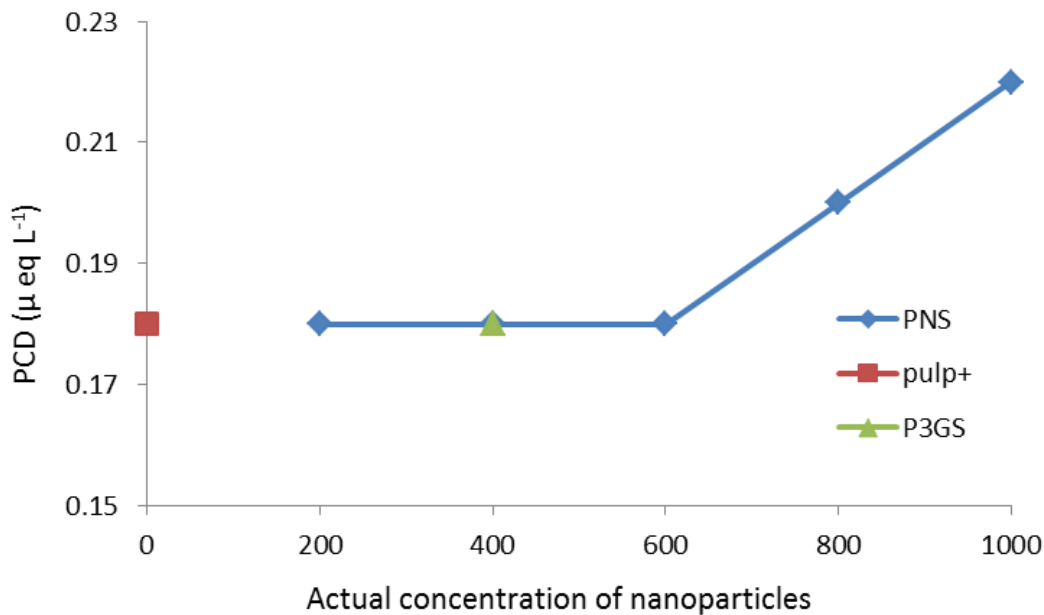


Fig. 4. Effects of nanoparticle doses on the charge titration results of the fiber filtrates.

addition of cationic starch, AKD, calcium carbonate), a polymer pulp furnish (pulp+, with addition of cationic polymer, P 300 ppm, or P3), a dual-chemical pulp furnish (pulp+, with P#GS, or P300 ppm and GS 430 ppm), and a dual-chemical pulp furnish (pulp+, with P3NS6) are shown in Table 1 to demonstrate the effects of adding cationic polymer retention aid and various microparticles.

Table 1 shows that addition chemical such as AKD, cationic starch and calcium carbonate helped with ash retention of the handsheet. However, comparisons of the first pass retention values of P3, P3GS, and P3NS6, indicated that the main salient objective was to retain fines and other minute particles. Of the treatments, cationic polymer alone allowed increased retention of calcium carbonate; whereas

Table 1. Effects of adding different chemicals to pulp furnishes on the forming and handsheet properties.

	Ash content (% of paper)	First -pass retention (%)	PCD ($\mu\text{eq L}^{-1}$)	Zeta potential (mV)	Tensile index (N·m/g)	Tearing index (mN · m ² /g)
pure pulp	0.16±0.22	77.39±0.34	0.24±0.00	77.5±0.01	46.39±3.83	6.03±0.40
pulp+	15.86±0.42	80.34±0.17	0.18±0.00	45±0.01	36.55±1.97	5.25±0.23
pulp+/P (P3)	17.59±0.28	78.15±0.47	0.16±0.00	20±0.02	35.30±1.98	4.94±0.13
P3GS	16.90±0.38	84.10±0.43	0.18±0.00	41.1±0.00	35.80±1.02	5.12±0.42
P3NS6	16.67±0.02	84.01±0.16	0.18±0.00	41.3±0.00	41.13±1.26	5.12±0.44
	Burst Index (kgf · m ² /g)	Elongation (%)	Smoothness (s/100 mL)	Air permeab. (sec/100cc)	Folding endurance (times)	Brightness (% ISO)
pure pulp	2.99±0.12	3.14±0.52	25.96±1.81	20±4.86	17±1.41	81.79±0.10
pulp+	2.36±0.08	3.27±0.39	25.95±1.82	13.81±1.10	9.38±0.92	82.81±0.14
pulp+/P (P3)	2.16±0.06	3.28±0.32	27.66±0.76	10.14±0.59	6.75±0.71	83.06±0.94
P3GS	2.2±0.08	3.33±0.18	26.49±2.71	11.21±2.14	8.13±0.35	82.93±0.07
P3NS6	2.43±0.04	4.57±0.19	29.73±1.35	14.49±0.88	12.63±1.60	82.67±0.05

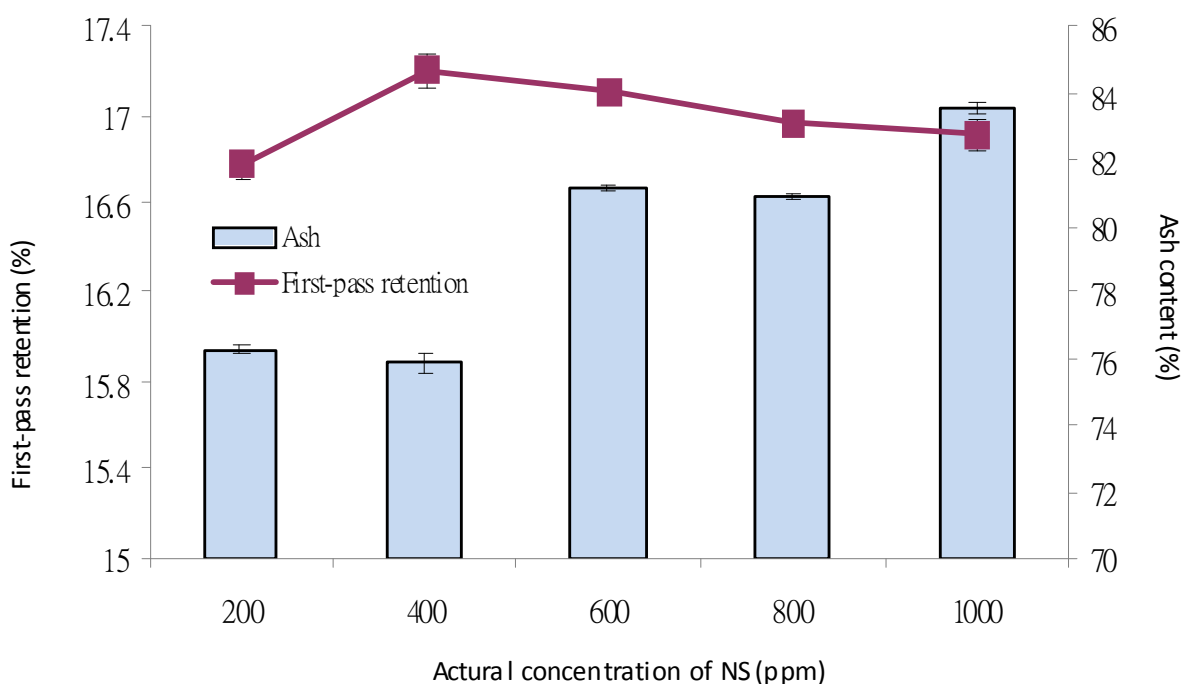


Fig. 5. Effects of NS doses on the first pass retention and ash content of the paper.

microparticles seemed to help more with the retention of fines.

Comparing the various handsheet physical properties of P3GS and P3NS6, one can note that at the optimal conditions, substituting NS for GS helped to increase tensile strength, rate of elongation, smoothness, air permeability and folding

endurance of the handsheets.

In order to probe this in more detail, we've included a series of P3NS at NS doses of 200, 400, 600, 800, and 1000 ppm to understand the effects of NS concentrations on paper properties. The results are shown in Figs. 5 to 7.

From Fig. 5, with NS dose increases, first pass retention tended to increase and then decrease slightly. Ash content of the

retention reached maximum. However, the ash retention was at minimum, suggesting that the fibrous content of comparable

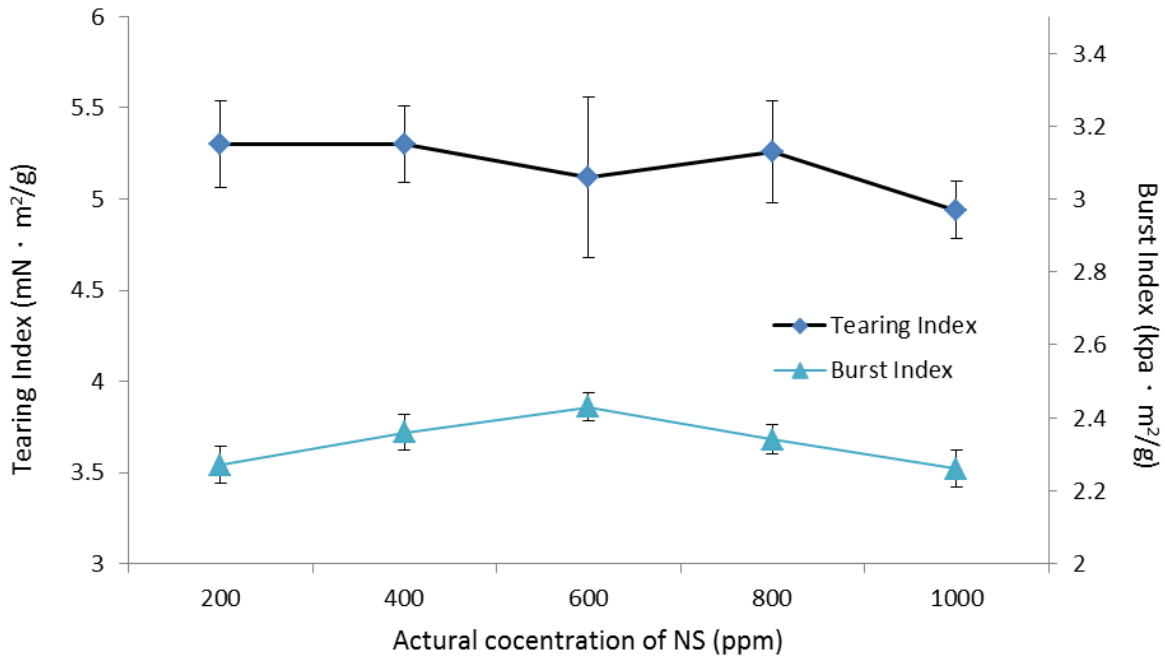


Fig. 6. Effects of NS dose on the tear and burst indices of the paper.

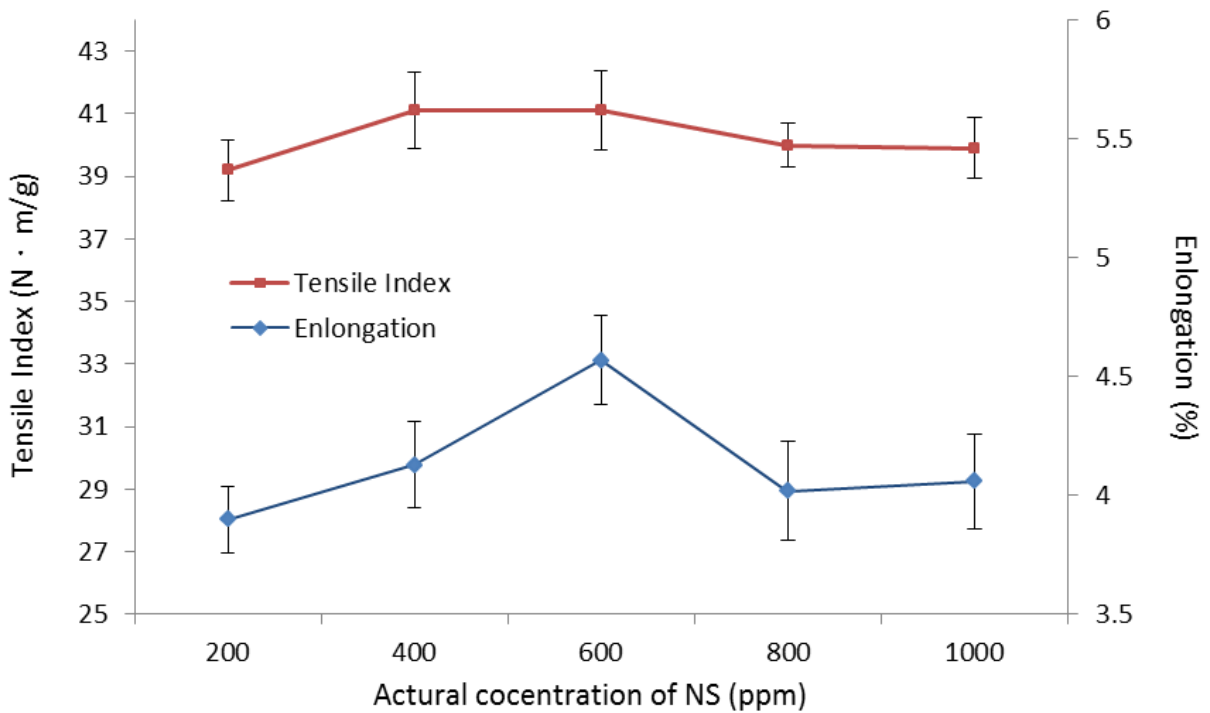


Fig. 7. Effects of NS dose on the tensile index and elongation rate of the paper.

paper also initially increased and then decreased with further increase of NS doses. At NS dose of 400 ppm, the first pass

grammage paper was at maximum then.

Comparing Figs. 6 and 7, although at NS dose of 400 ppm

the resulting handsheet had maximum tear index and rate of elongation, however, due to its lower ash retention, the production cost for the paper was higher. At NS dose of 600 ppm, on the other hand, the first pass retention was slightly lower than that at 400 ppm, but ash retention increased 0.79% while other paper physical properties were not significantly different from those of 400 ppm ones. Therefore, at the same grammage, paper made with 600 ppm of NS would have a slight cost advantage.

The figures show that handsheet rate of elongation and tensile index tended to initially increase with increasing NS concentrations and then decreased with further increases. The probable reason for the phenomenon was that at NS dose < 600 ppm, the fines retention increased which helped tensile strength and elongation. However, when NS dose increased further, the retained calcium carbonate in paper also increased which interfered with interfiber bonding and reduced individual fiber pliability leading to decreased tensile and elongation. Prior research indicated that cationic polymer retention aids have the functions of flocculating fiber flocs and retaining calcium carbonate particles. The functions of microparticle, on the other hand, tend to break up the large flocs, causing interstices of fibers to increase and helping with the paper formation and further increased retention of calcium carbonate[14-16]

Based on the experimental results, although NS appeared to differ from GS in morphology, its performance in a dual-chemical system was nevertheless every bit as effective. In addition, NS helped to create more fiber interstices for calcium carbonate particle to fill-in. Changing NS concentrations did not appear to affect smoothness, folding endurance, air permeability and brightness of the paper much. At NS dose > 600 ppm, however, gain in ash retention leveled off and first pass retention and paper physical properties tended to decrease gradually. Titration of the filtrates in Fig. 2 indicated that at NS dose > 600 ppm created an overly anionic condition and retention of NS in fiber flocs was questionable, as indicated by the titrant amount for the filtrates. In other words, at higher NS doses, no further functional performance can be expected. Henceforth, in this phase of the study, we've selected

P3NS6 as the optimal condition of addition for the dual-chemical retention system.

ANOVA Statistics

A one-way ANOVA analyses of the 2² factorial experimental design based on P and NS as variables and with respect to first pass retention, ash retention, tensile, burst, stiffness, smoothness, air permeability, rate of elongation, and brightness were carried out to delineate the main and interactive effects. At a confidence interval of 95% with 12 sets of experiments and 4 degree of freedom, the ANOVA results indicated that for the P3NS system, changing NS concentrations caused significant effects to the aforementioned paper physical properties; whereas the cationic polymer concentration exerted significant effects on the following properties: First pass retention (F-factor: 27.871, significant to $p < 0.000$); burst index (F-factor: 5.325, significant to $p < 0.009$); folding endurance (F-factor: 8.935, significant to $p < 0.001$); air permeability (F-factor: 4.538, significant to $p < 0.017$); smoothness (F-factor: 9.282, significant to $p < 0.001$); elongation rate (F-factor: 8.935, significant to $p < 0.001$). Thus, substituting NS for GS exerted significant effects on first pass retention, and both main effects of NS and P significantly affected first pass retention rate. There was, however, no significant interaction between them. In the P3GS system, Both P and GS main effects and their interaction had significant influences on the ash retention of paper; however, substituting NS for GS exerted no significant effect on paper ash retention, and only P concentrations significantly affected ash content. In addition, substituting NS for GS showed no significant effect on paper brightness either.

4. CONCLUSIONS

Based on the experimental results, we've pegged optimal dose of cationic polymer in the dual-chemical system at 300 ppm. At the condition, optimal dose of NS was 600 ppm. At the identical P dose, the first pass retention, and ash retention attained by either NS or GS microparticles were similar. However, NS substituted system produced paper with gains in tensile index, burst index, elongation, smoothness, air

permeability, and folding endurance respectively by 14.9%, 10.5%, 37.2%, 12.2%, 29.3%, and 55.4%, and clearly demonstrated its superior performance. The ANOVA results indicated that in the P3NS system, changing NS dose exerted significant effects on various paper physical properties. Substituting NS for GS also significantly affected first pass retention while there were no significant effects on ash content and paper brightness.

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