

Nanocellulose–cationic starch–colloidal silica systems for papermaking: Effects on process and paper properties

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ABSTRACT: Laboratory tests were conducted to better understand effects on the papermaking process and handsheets when recycled copy paper furnish was treated with combinations of nanofibrillated cellulose (NFC), cationic starch, colloidal silica, and cationic retention aid (cPAM; cationic polyacrylamide). Dosage-response experiments helped to define conditions leading to favorable processing outcomes, including dewatering rates and the efficiency of fine-particle retention during papermaking. Effects were found to depend on the addition amounts of cationic starch and colloidal silica added to the system. It was shown that the presence of a polymer additive such as cationic starch was essential in order to achieve large strength gains with simultaneous usage of NFC.

Application: Papermakers can regard NFC as an option for increasing the bonding strength of the resulting paper beyond what generally can be achieved with conventional wet-end additives. However, as shown in the present work, other additives, such as cationic starch and colloidal silica, may be required at suitable levels of addition to achieve the potential benefits of nanocellulose.

The dry strength of paper products is critically important in many applications, including printing and packaging. Strength is often the limiting factor that determines whether it is possible to reduce the basis weight or the amount of premium virgin fibers within a paper product. It is well known that paper's strength critically depends on the interfiber bonding [1]. Papermakers employ mechanical refining as a primary tool to increase the bonding between kraft pulp fibers [2]. However, there are practical limits to refining. With increased refining, the material becomes increasingly broken down with the release of cellulosic fines. Especially when they are not attached to the fiber surfaces by the use of retention aids, these fines can adversely affect the rates of dewatering [3]. Excessive refining reduces the inherent strength of a fiber, which eventually damages paper strength [1]. In addition, kraft pulp fibers that have been recovered from used paper tend to have a reduced ability to bond to each other [4].

Very highly fibrillated cellulose material, which in this research paper will be called nanofibrillated cellulose (NFC), offers a means to greatly increase interfiber bonding within paper. This is an attractive option, since NFC can be made from widely available, relatively inexpensive material, i.e., kraft pulp fibers [5,6]. Some recent studies have shown that NFC offers a way to achieve high increases in the bonding strength of paper [5,7]. However, due to its small size, NFC particles can be difficult to retain efficiently during paper's formation [8,9]. In addition, the NFC can choke up drainage channels within the wet web, leading to slow dewatering [7].

Though the overall effects of NFC on paper strength and the dewatering during paper's formation have been well known [5], it has been less common for such studies to also report effects due to other papermaking additives. Three exceptions to that rule are worth noting. Petroudy and co-authors showed large benefits when a cationic acrylamide copolymer (retention aid) was used in combination with microfibrillated cellulose prepared from bagasse pulp [10]. The tensile strength of the resulting paper was increased by 28% with the addition of 1% of the MFC to the fiber suspension. The dewatering rate was maintained when using the retention aid, whereas it became slower when the MFC was used alone. Merayo et al. achieved an 11% increase in paper's tensile strength with just 0.5% of NFC added to the furnish, and again a cationic retention aid was used to maintain high fines retention and to avoid a decrease in drainage [11]. Followup work achieved a higher tensile strength increase at a 1.5% addition level of NFC, with use of a cationic retention aid to maintain retention of the fine materials [12]. Cationic starch had been utilized in a study by Manninen et al. [13], but drainage properties had not been monitored. Thus, there has been a need to consider paper strength, dewatering, and other outcomes in systems that include such additives as NFC, cationic starch, cationic retention aid, and colloidal silica.

Recent work by Rice et al. [14] demonstrated a treatment strategy whereby NFC was pretreated first by cationic starch and then by colloidal silica. Not only did this combination contribute to a large increase in the paper's dry strength, but it also helped to overcome the negative effect

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of NFC on the drainage rate. The focus of the cited work was on the preparation of paper products having low apparent density. In particular, the described treatment system made it possible to achieve high gains in paper stiffness with greatly reduced refining of the main kraft pulp component. The objective of the present work is to better understand the results obtained in the earlier study and to widen the focus beyond the high-bulk category of paper products. Thus, the attention here will include relatively dense paper products, such as writing paper, linerboard, and other packaging products. A further goal is to study the factors contributing to dewatering, first-pass retention, the uniformity of formation, and physical properties of the resulting paper.

The present article, which is based on laboratory research carried out by students at North Carolina State University (Raleigh, NC, USA), deals with some issues that have arisen in recent studies of the use of highly fibrillated cellulose products in papermaking. Laboratory tests can provide opportunities for multiple replications, as well as precise definition, of the compositions and procedures. The idea was to establish a baseline for additional needed work. To add to that baseline, results of further laboratory studies are embodied in two companion articles, which are also appearing in this journal. One of those companion articles focuses on previously unknown effects of the orders of addition of the chemical agents. The third article in this set focuses on the likely mechanisms. In addition, there will be an ongoing need for followup work at the pilot-scale and commercial levels of papermaking.

EXPERIMENTAL

Chemicals and materials

The fiber furnish used throughout this work was prepared by the repulping of 100% recycled copy paper (trademarked Boise ASPEN 100 Multi-use Recycled Copy Paper, Packaging Corporation of America, Boise Paper Division).

Nanofibrillated cellulose (NFC), in dry form, was obtained from the University of Maine (Orono, ME, USA). Three versions were employed: spray-dried, freeze-dried, and TEMPO-oxidized. In addition, a microfibrillated cellulose product was obtained in slurry form from Stora Enso Oyj (Helsinki). The default level of addition (except for the blank) was 5% on a furnish dry-mass basis. The way this was chosen is that (a) 5% is enough to have a very large effect in decreasing the rate of dewatering, and (b) the amount is not so high as to be outside the range of interest of papermakers (but on the high side).

The cationic starch was Chargemaster L340 from Grain Processing Corp. (GPC; Muscatine, IA, USA). This is a pregelatinized paste version having a relatively high cationic starch density. The paste was kept in a refrigerator. Batches of starch were prepared at 1% solids, using warm water.

The colloidal silica type used in most of the work was Fennosil 2180, a product of Kemira Oyj (Helsinki). This is

a high-structured (gel-type) colloidal silica. Some tests also were carried out with Fennosil 5000 (medium-structure gel-type) and Fennosil 615 PA EN (low structure). Each colloidal silica product was diluted to 1% actives before its use in the experiments.

Accurac 90, a very-high-mass cationic copolymer of acrylamide (American Cyanamid), served as the retention aid for this work. The dry powder was diluted with agitation for an hour to a solids content of 0.1% before its use in the tests.

Deionized water was used for dilution of all chemical additives. Tap water was used for preparation of hand-sheets. The sodium sulfate was reagent grade. A diagram depicting the options for order of addition of additives in this type of experiment already appeared in the article by Rice et al. in this journal [14].

Pulp preparation

For preparation of batches of 0.5% solids pulp suspension, the paper taken from reams was assumed to have a constant 6% moisture content. The kilograms of dry paper were determined by multiplying the total intended volume of pulp in liters (e.g., 60 L) by 0.005 over 0.94 (where 0.94 is the solids content of the paper). The paper was torn into pieces, each smaller than 1/8 of a sheet. The pieces were soaked overnight. Then, the damp paper pieces were subjected to no-load “brushing” treatment (no weight applied) for 15 min with a laboratory Hollander beater, with a pulp consistency of approximately 2%. The pulp was added to a roll-around plastic barrel and diluted to a consistency greater than 0.5% (slightly below the target volume). With a probe of electrical conductivity inserted into the pulp suspension, and with vigorous agitation, increasing amounts of ca. 5% sodium sulfate were added, aiming for a final conductivity of 1000 $\mu\text{S}/\text{cm}$. This was followed by final adjustment of the consistency to 0.5%. Pulp was kept in a cold room when being stored for up to two weeks, and it was allowed to warm up to room temperature overnight before testing.

Dewatering tests

Two essentially equivalent devices were employed to obtain Schopper-Riegler filtrate mass as a function of drainage time. A majority of tests were carried out using the Mutek DFR-05 device from BTG Inc. (London), using the following settings: spin for 10 x at 700 rpm, then 10 s at 800 rpm, and then release.

Some supplementary tests were carried out with a portable modified Schopper-Riegler tester. The standard device of this type is described in the ISO 5267 Test Method “Pulps — Determination of drainability — Part 1: Schopper-Riegler method.” The modified device replaces the capillary opening and side spout with a simple large central opening for the filtrate to pass through. By this means, the filtrate mass can be measured as a function of time using an analytical balance having a measurement precision of 0.1 g.

Retention tests

The efficiency of retaining fine particles, including mineral fillers and cellulosic fines, was evaluated using a Britt dynamic drainage/retention jar [15,16]. A conventional 200-mesh stainless-steel screen was employed. In a typical test, there was a 30 s delay between addition of each successive additive to the mixture under a continuous agitation, usually at 600 rpm. An indication of the unretained fines was obtained by measurement of the turbidity of filtrate [17]. Reported mean values and 95% confidence intervals of the mean were based on ten replicate tests, which involved five samples taken from the device (with pairs of measurements obtained with a gentle overturning of the capped vial and retesting). Because the initial readings routinely exceeded the measurable range, it was standard procedure to dilute each specimen by a factor of ten and then multiply the reading by 10. Based on the Beer-Lambert Law [18], the turbidity in NTU units is expected to be approximately proportional to the contents of insoluble solids in the filtrate.

Flocculation tests

The degree of flocculation among fibers in the suspension, including change in flocculation due to various additives, was evaluated with use of a Photometric Dispersion Analyser (PDA, low-gain version) from Rank Brothers Ltd. (Bottisham, UK). The PDA unit was set up such that 0.5% consistency suspension was drawn into clear flexible tubing having an inner diameter of approximately 6 mm (Tygon S3 E-3603 NSI, OD ca. 8.5 mm). The tubing was passed through the sensing zone of the PDA, in which the change in transmission of white light was measured as a function of time. Both gain constants were set to 1.0, and the “Root-mean-squared” (RMS) and “Filter” buttons were depressed, thus providing the RMS value of deviations from the mean intensity of transmitted light through the tubing. This unitless value provides an indication of the degree of flocculation, though it is not specifically related to floc size or floc density by any known theory.

Streaming potential tests

Zeta potential information for the fiber surfaces was obtained by running streaming potential tests on pads of fibers drawn against a filter screen by a specified vacuum (700 mm Hg). The change in electrical potential was evaluated by placing welding wire probes on the two sides of the screen, with the use of fine Tygon tubing and plumber’s adhesive to fully insulate the submerged parts of the wires except for the intended measurement areas. For each individual measurement (with at least four replications), the initial value from a millivolt meter (with accuracy 0.1 mV) was obtained once a stable value had been reached, usually about 30 s after the start of vacuum application. At that point the vacuum was broken, using a quick-release fitting in the tubing, and a zero-vacuum value was obtained after waiting 2 s. The zeta potential was then calculated from Eq. 1:

$$\zeta = 4\pi \Delta E \eta \lambda / (\Delta P \epsilon) \quad (1)$$

where ΔE is the change in measured streaming potential (vacuum applied – without vacuum), η is the shear viscosity of the solution (assumed to equal that of pure water), λ is the electrical conductivity, ΔP is the difference between the applied vacuum level and zero, and ϵ is the dielectric constant. After applying corrections for the effects of temperature on electrical conductivity and viscosity, the following version of the equation was used in the calculations:

$$\zeta(\text{mV}) = 12.27 \Delta E(\text{mV}) * [\lambda(\mu\text{S}/\text{cm})/\Delta P(\text{mm Hg})] * [1+(25-\theta)/44.74]/ [1+(25-\theta)/223.7] \quad (2)$$

In this equation, θ stands for the temperature in °C.

Charge demand titrations

The cationic demand or the anionic demand values of various aqueous mixtures (usually sampled as 10 mL aliquots) were obtained using a CA Touch streaming current detector from emtec Electronic (Leipzig, Germany). The device features a polytetrafluoroethylene (PTFE) piston (diameter approximately 15 mm) that moves up and down at about 4 Hz within a loosely fitted PTFE boot (gap width less than 1 mm). By means of electrode probes near the base of the boot and above the annular region, the device essentially senses the presence and sign of charge of an electrical double layer at the PTFE surfaces. Because those surfaces will be coated by polyelectrolytes and colloidal material from an aqueous sample, it can be used for detection of the endpoint of a titration with known polyelectrolytes. Poly(diallyldimethylammonium chloride) (polyDADMAC) (0.001 molar solution) was used as the cationic titrant, and the potassium salt of poly(vinyl sulfate) (PVSK) was used as the anionic titrant (0.001 M).

RESULTS

Characteristics of the fiber suspension

As shown in **Table I**, the pH of the fiber suspension remained within the range of 8.3 to 8.9 for all of the experimental conditions considered. This pH range is consistent with the usage of 100% recycled fiber content xerographic copy paper as the fiber furnish for this work. Recycled office paper can be expected to contain about 10% to 25% of calcium carbonate (CaCO₃ filler), which will tend to buffer the pH when it is placed into water [19]. None of the listed treatment conditions included enough acid content to dissolve the CaCO₃ and overpower its pH-buffering effect.

Results for zeta potential (calculated from the streaming potential test results) showed clear effects related to the additives considered in this work. For example, adding cationic polyacrylamide (cPAM) retention aid at a level of just 0.05% (solids basis) to the suspension decreased the nega-

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Specimen	pH	Zeta Potential, mV	Cationic Demand, $\mu\text{eq/L}$
100% DIP (default)	8.6	-21.4	23.2
DIP + 0.05% cPAM	8.3	-14.9	17.8
DIP + 5% NFC	8.6	-32.4	31.6
DIP + NFC + cPAM	8.7	-25.6	20.2
1% cationic starch + NFC + DIP + cPAM	8.4	-19.5	7.8
2% cationic starch + NFC + DIP + cPAM	8.8	-8.8	-34.4
5% cationic starch + NFC + DIP + cPAM	8.6	14.9	-157
10% cationic starch + NFC + DIP + cPAM	8.7	19.1	-404
1% cationic starch + NFC + DIP	8.9	-15.6	9.6
2% cationic starch + NFC + DIP	8.8	8.0	-32
5% cationic starch + NFC + DIP	8.8	3.8	-161
10% cationic starch + NFC + DIP	8.7	4.2	-401

DIP = deinked pulp; NFC = nanofibrillated cellulose; cPAM = cationic polyacrylamide (retention aid).

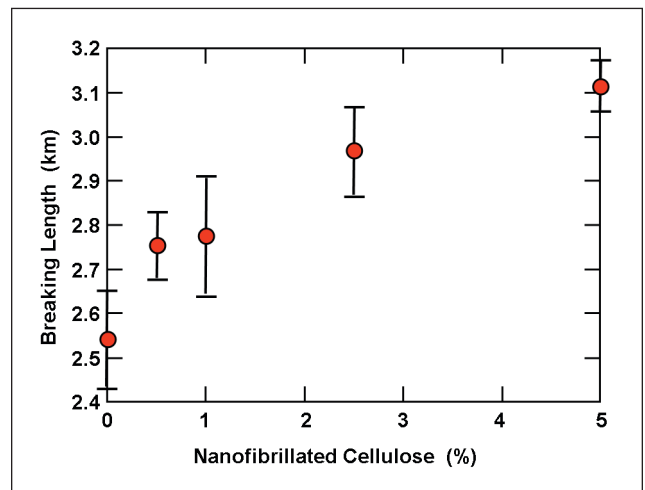
I. Properties of fiber suspensions.

tive zeta potential (absolute value) by about 30%. By contrast, the individual effect of adding nanofibrillated cellulose (freeze-dried type) to the deinked pulp (DIP) furnish was to make the zeta potential even more negative. This is consistent with the expected negative charge of freeze-dried NFC. The zeta potential was shown to become less negative or more positive with increasing addition levels of cationic starch. As shown in the table, between a 2% and a 5% level of cationic starch addition (based on solids) was sufficient to overcome the charge demand of the fibers and nanocellulose (whether or not cPAM was present), thereby giving a positive zeta potential.

As described earlier, the cationic demand results shown in Table I were based on filtrates obtained from the corresponding mixtures after stirring. In most cases, the sign of cationic demand was opposite to that of the measured zeta potential. This is because slurry systems having a negative zeta potential are likely to have an excess of negatively charged dissolved and colloidal titratable substances in the aqueous phase. An exception to the rule was found in the system with 2% cationic starch with NFC, DIP furnish, and cPAM. In that case, the zeta potential was still negative, though the charge titration showed an excess of cationic substances in the filtrate. Related observations were reported by Strazdins [20], who attributed such findings to progressive permeation of cationic polymers into the mesopores of kraft fibers.

Effects of nanofibrillated cellulose addition

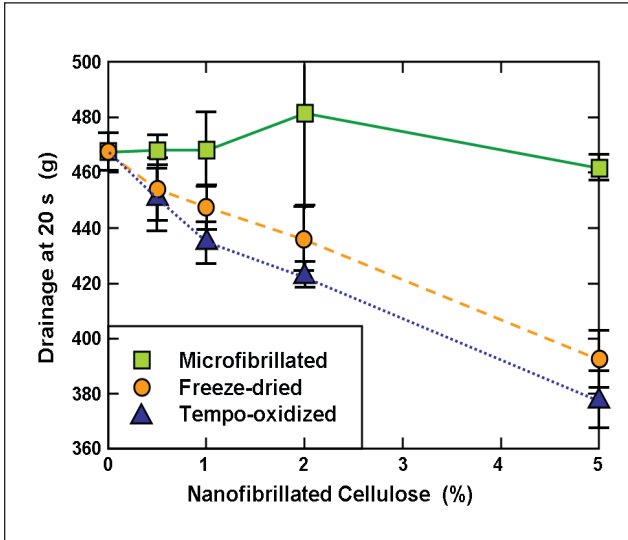
In principle, nanofibrillated cellulose can improve paper strength due to increased interfiber bonding. The high flex-



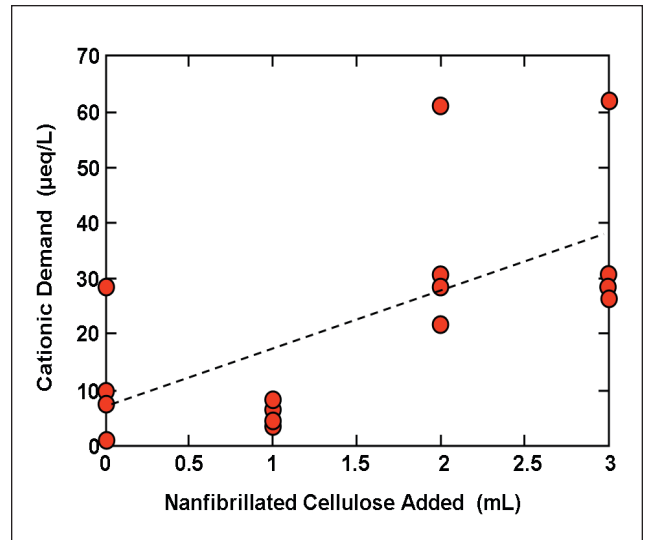
1. Effect of nanofibrillated cellulose addition level on the tensile strength of handsheets.

ibility of NFC in the wet state allows it to efficiently fill spaces between adjacent rough fiber surfaces. It is assumed that progressively more solid-to-solid hydrogen bonds will be established both within the NFC and also with the fiber surfaces as water becomes evaporated during drying of the paper. The strength testing data (Fig. 1) showed that increasing the nanofibrillated cellulose percentage increased the breaking length of the paper. As suggested by the 95% confidence interval bars, a significant increase in strength was already apparent at the 0.5% level of addition (based on solids). Thereafter, the breaking length continued to increase, though less sharply.

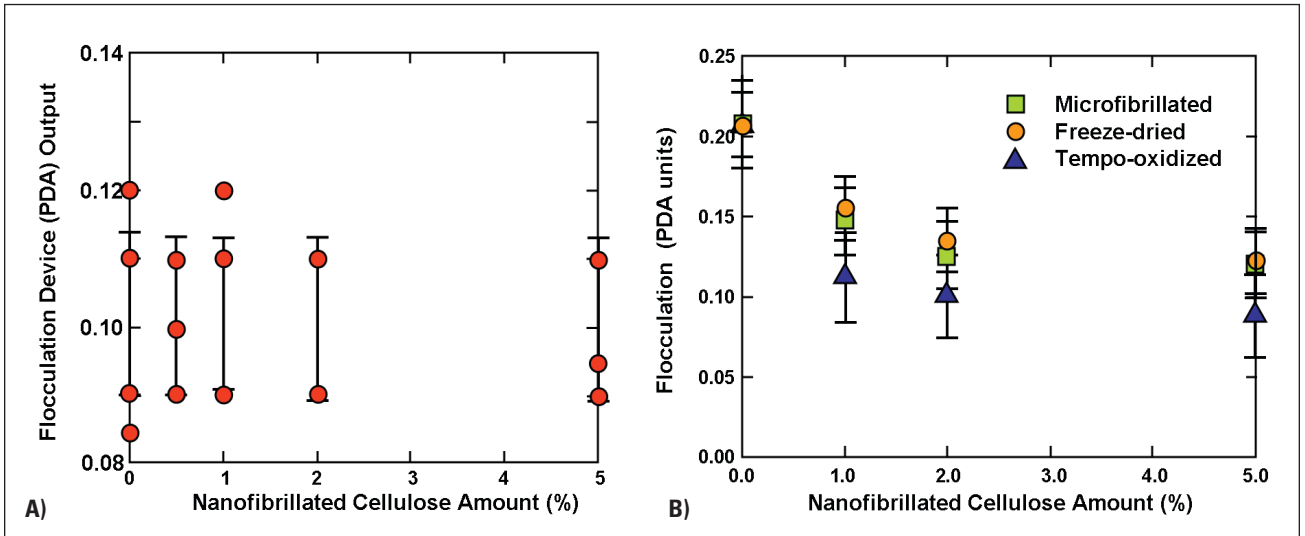
Beyond the issues related to statistical significance, it is important to also stress that papermakers have a range of



2. Effect of nanofibrillated cellulose addition level on the dewatering rate of 100% recycled copy paper furnish.



3. Cationic demand as a function of the addition level of nanofibrillated cellulose to default furnish.



4. Effect of nanofibrillated cellulose addition on the flocculation level of the default furnish: A) with no other additives; and B) with subsequent treatment by cationic polyacrylamide (cPAM) retention aid at the 0.05% level based on solids.

options to increase paper strength, and some of them may be cheaper or easier to implement than the addition of NFC. For example, papermakers usually will have the option of increasing the level of refining of the main fiber furnish. Production teams continually weigh such issues as refining levels of the main furnish, as well as the usage of cationic starch. The addition of NFC may or may not provide an additional approach that can be considered, depending on whether there are constraints on refining levels or the use of strength additives.

However, in addition to increasing bonding strength, the usage of NFC can also decrease drainage rates (Fig. 2). For drainage testing, three types of nanofibrillated cellulose were tested. Those types were microfibrillated, freeze-dried, and TEMPO-oxidized. The data showed that drain-

age measured 20 s after opening of the sealing cone decreased as more nanofibrillated cellulose was added. It can be challenging to retain nanofibrillated cellulose particles on the fiber. This can result in flow channels within the wet web of paper being blocked, resulting in a decrease in drainage [21,22,23]. Based on the results shown in Fig. 2, the three types of NFC did not all have the same effects on dewatering. The two NFC products from the University of Maine gave relatively large decreases in drainage rates that were almost linear with respect to added amounts.

The microfibrillated cellulose, by contrast, did not have a significant effect on dewatering (at least not in the absence of other additives). As shown by the top line in Fig. 2, even at an addition level of 5%, the microfibrillated cellulose did not adversely affect dewatering under the condi-

NANOCELLULOSE

tions of testing. These results help to reinforce the point made earlier that papermakers will have choices. By employing a cellulose product having an intermediate level of fibrillation, there may be opportunities to achieve benefits of enhanced bonding with less or no penalty in terms of decreased rates of water removal.

Cationic demand gives information on how much of a highly charged polymer is needed to neutralize the surface of a sample when added to papermaking furnish. The data in **Fig. 3** show that the cationic demand increased as the amount of nanofibrillated cellulose dosage was increased. This is to be expected, since nanofibrillated cellulose has a negative charge.

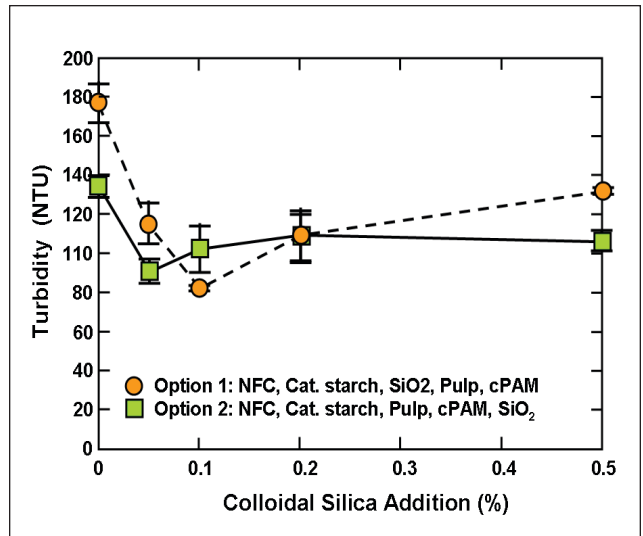
Part A of **Fig. 4** shows that addition of NFC (type = freeze-dried) had no significant effect on fiber flocculation in the absence of other additives. By contrast, part B of the figure shows that in cases where the furnish had also been treated with cPAM copolymer retention aid, subsequent addition of any of the three types of NFC decreased the level of flocculation. The latter findings can be attributed to the NFC adsorbing the cPAM from the aqueous phase, thereby leaving less of the cPAM available to participate in bridging flocculation among the fibers [24].

Effects of cationic starch addition

As was shown earlier in Table I, increasing cationic starch dosage led to an increase in cationic character of the furnish. These effects were shown by the less negative or more positive values of zeta potential of the fiber, based on the streaming potential tests. In addition, the filtrate had a decreased cationic demand with increasing cationic starch addition levels. These results are to be expected due the cationic starch's positively charged nature. Furthermore, as shown in Fig. 1, cationic starch addition led to increased strength in paper.

Effects of nanoparticle addition

Addition of colloidal silica or sodium montmorillonite (bentonite) to untreated papermaking furnish is known to not have significant effects on drainage, flocculation, or other aspects of the papermaking process. This is because, like the fibers and nanocellulose, the colloidal silica or bentonite have a negative charge, and hence they don't interact with each other. By contrast, **Fig. 5** shows clear effects of colloidal silica addition to systems with nanofibrillated cellulose that also involved treatments with cationic starch. The "Option 1" mentioned in the figure involves pretreatment of the NFC with cationic starch, followed by colloidal silica, final addition of the treated NFC to the fiber suspension, and final addition of the retention aid. "Option 2" is similar, except that the addition of colloidal silica is moved to the very end of the preparation, after the addition of cPAM to the combined furnish. As shown, in both cases the addition of even 0.05% SiO₂ (on a solids basis to the combined furnish) yielded a major increase in retention ef-



5. Effects of colloidal silica addition level and order of addition on the efficiency of fine particle retention in systems where nanofibrillated cellulose (NFC) at the 5% level had been treated with cationic starch (cat. starch), then added to the pulp, followed by treatment by cationic polyacrylamide (cPAM) retention aid. The addition orders were as shown in the legend (SiO₂ = silicon dioxide).

iciency, as indicated by the turbidity of the filtrate in a Britt jar test. Increased amounts of SiO₂ above 0.1% did not further increase retention efficiency.

DISCUSSION

Results shown in this article have considered mainly individual effects of different components in a system of highly fibrillated cellulose (including different types), cationic starch (including different levels), and colloidal silica (at different levels) on some process-related aspects and product quality aspects of papermaking. The present work is related to earlier reported results that focused on the manufacture of bulky paper products [14]. It was shown in the cited work that the combination of NFC, cationic starch, and colloidal silica could be optimized, along with major decreases in refining levels, to achieve high increases in strength, while maintaining high bulkiness and satisfactory dewatering rates. The present article supports those results, but it also shows potential for improving the strength of more typical paper grades, such as copy paper.

The final figure shown in this article provides a segue to the main topic of the next article in this series. As shown in Fig. 5, different results were obtained depending on the detailed order of addition of additives. The earlier-reported work by Rice et al. [14] showed promising results when colloidal silica had been added to a suspension of NFC that had been pretreated with cationic starch. It is more conventional in the paper industry to add colloidal silica as the final additive, after a very high-mass cationic retention aid, i.e. cPAM [25,26]. Such issues, and more, will be considered in the next article [27].

CONCLUSIONS

In terms of dewatering, fine-particle retention, and the level of flocculation among fibers, promising results were obtained in a 100% recycled copy paper furnish when adding combinations of nanofibrillated cellulose (NFC), cationic starch, and colloidal silica. Dosage-response curves were developed for individual additives to the furnish. The addition of NFC, at increasing levels with no other additives, decreased dewatering in an approximately linear manner. The cationic demand of the furnish increased with increasing addition of NFC. Adding NFC to the furnish affected the level of fiber flocculation only when the furnish was subsequently treated by cationic polyacrylamide (cPAM) retention aid. This was because the cPAM interacted with colloidal silica rather than just with fibers and fines. Fine-particle retention was optimized at a certain level of colloidal silica, with the results depending on the order of addition of the additives. **TJ**

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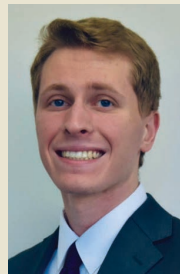
Nanocellulose represents an important opportunity for papermakers to increase the bonding strength of their products, but it tends to interfere with drainage. The authors of this paper are at North Carolina State University in the Department of Forest Biomaterials. The work was supported in part by Research Experience for Undergraduates funds during the summer of 2021.

Important findings were reported in the past by Rice, Pal, Gonzalez, and Hubbe in *TAPPI J.* 17(7): 395(2018). This work, by contrast, deals with dense paper products. The authors were motivated to carry out this work due to a need to follow up on earlier promising research results, with the help of some newly acquired equipment for automated evaluation of dewatering rates.

The challenge in this research was to characterize the effects of different combinations of nanofibrillated cellulose with cationic starch, colloidal silica, and cationic acrylamide. It was discovered that there appear to be optimum treatments levels. The most interesting finding was that the combined system did not have any adverse effect on paper uniformity.



Garland



Leib



Barrios



Hubbe

This research shows that mills finding themselves in a situation where they need additional bonding strength have an additional option, for which the factors are now better known.

This is the first in a set of three articles on the topic in *TAPPI Journal*. The second one, which appears in this issue as *TAPPI J.* 21(9): 572(2022), deals with issues of orders of addition. The third one (Nelson et al.) will deal with mechanistic aspects.

Garland and Leib are undergraduate students, Barrios is a graduate student in the Ph.D. program, and Hubbe is a professor in the Department of Forest Biomaterials at North Carolina State University, Raleigh, NC, USA. Email Hubbe at hubbe@ncsu.edu.

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