

# *Effects of orders of addition in nanocellulose–cationic starch–colloidal silica systems for papermaking*

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**ABSTRACT:** Two orders of addition were compared when preparing paper handsheets from recycled copy paper furnish in combination with nanofibrillated cellulose (NFC), cationic starch, colloidal silica, and cationic retention aid (cPAM; cationic polyacrylamide). Faster dewatering and higher fine-particle retention were obtained at equal optimized dosages of additives when the colloidal silica was added last, after addition of the cPAM. The same order of addition also provided a higher gain in the paper's tensile strength. However, higher paper stiffness was achieved when the colloidal silica was instead added to the NFC, after its pretreatment with cationic starch. Results were consistent with the principle that papermaking additives added shortly before sheetforming tend to have the largest effects on drainage and retention. The results also demonstrated a sensitivity to the relative dosages of positively and negatively charged additives.

**Application:** Changing the order of addition of various chemical additives to the papermaking process often can be achieved with near-zero capital investment. The results in this paper provide an example where the order of addition can be expected to have important consequences.

Two articles published in this journal showed that favorable combinations of paper properties and rates of drainage of the wet web of paper can be achieved when using certain combinations of nanofibrillated cellulose (NFC), cationic starch, and colloidal silica [1,2]. It was reported recently that the efficiency of fine-particle retention depended on the order in which the ingredients were mixed together [2]. Accordingly, the present article considers two specific options regarding when to add colloidal silica. In one set of tests ("Option 1"), the colloidal silica was used as part of the pretreatment of NFC, subsequent to the addition of cationic starch. This was the procedure employed in work reported by Rice et al., which focused on the achievement of strong, stiff paper sheets at low values of apparent density [1]. In another set ("Option 2"), the colloidal silica was instead used as the final additive to the system, after a cationic polyacrylamide (cPAM) retention aid. Such conditions are consistent with modern papermaking processes in systems that do not contain NFC. The goal of this work was not only to shed light on potentially favorable papermaking practices, but also to explore a wider picture of effects related to retention, flocculation, and properties of the resulting paper when NFC is used with other papermaking additives.

The topic of order of addition of papermaking additives, though well known to practicing papermakers, has received only minor attention in published articles. For instance, it has been shown that the order of addition can be key to the performance of certain two-component retention

aid systems [3]. Such cases are explained by the fact that a high-charge cationic additive needs to be applied first, so that there will be positively charged sites on fiber surfaces upon which the other additive, a negatively charged retention aid, is able to bind.

Some of the earliest published reports describing the usage of combinations of high-mass cationic polymers and either colloidal silica [4] or bentonite [5,6] indicated that the cationic polymer solution would be added first and the tiny mineral products would be added last to the mixture during the papermaking process. In that way, it has been proposed that the nano- or microparticles would be able to complete the system of bridging between solid surfaces. In particular, it was proposed that some idle loops and tails of cationic polymer extending from adjacent solids surfaces might both become bound to the same negatively charged bentonite particle [7].

By selecting the points of addition, papermakers can purposely subject the treated furnish to the relatively high hydrodynamic shear stress of a pressure screen [8]. The tendency of the screen system to disperse fiber flocs after treatment with a retention aid appears to explain why cPAM has been recommended to be added before the screen, whereas the bentonite was recommended to be added post-screen [5,9]. It has been established that the hydrodynamic conditions needed to shear apart fibers from each other are much less severe than the conditions needed to shear small particles, such as mineral fines, from fiber surfaces, if one assumes that the same retention aid systems are binding

all such solids together [10,11]. In the case of colloidal silica, however, sometimes the opposite order of addition has been practiced [12,13]. It has been proposed that the very small size of the colloidal silica particles allows them to pass into the interior of coils of cationic polymers [14]. Then, as the cationic polymer wraps itself onto the negatively charged particles, the bridges are shortened. The resulting contraction of the system is credited with a wringing effect that squeezes water out of the wet web of paper, thus promoting the release of water during the paper forming process.

Potential increases in interfiber bonding can motivate papermakers to consider adding highly fibrillated cellulose products to the papermaking furnish, but there have been concerns about slower rates of dewatering. The present work made use of differently prepared NFC, which was provided in dry form by the University of Maine (Orono, ME, USA). It is well known that NFC can be prepared from, for instance bleached softwood kraft pulp, by providing sufficiently high levels and durations of hydrodynamic shear or micro-grinding. When such materials are dried, for instance in preparation for storage or shipping, it has been shown that a partly irreversible loss of surface area can occur [15]. The loss can be attributed to the action of capillary forces that bring the cellulosic surfaces into molecular contact as the water evaporates [16]. Dense hydrogen bonding that forms between the adjacent cellulosic surfaces may be only partly reversible if and when the material is placed back into water [17]. These problems can be at least partly overcome by subjecting the bleached cellulose pulp to TEMPO-mediated oxidation before preparation of the NFC [18]. Not only does such treatment decrease the amount of energy needed to prepare the NFC, but it also facilitates the full redispersion of the dried NFC when it is mixed with water.

Following up on results shown by Garland et al. [2], the present work focusses on systems in which slurry from a master batch of repulped 100% recycled copy paper was treated with set amounts of NFC that had been pretreated with cationic starch. Colloidal silica was added either to the cationic starch-treated NFC or as the final additive to the process. The slurry of treated NFC was combined with the furnish, then treated with retention aid (cPAM), and finally with optional colloidal silica, if it had not been added earlier. The test runs with earlier addition of colloidal silica correspond to the procedures followed by Rice et al. [1]. In that work, the focus was on how best to prepare the NFC, and the option of adding the colloidal silica later was not tried due to lack of time. The addition of colloidal silica as the final additive corresponds to conventional papermaking practices that do not involve NFC [4,12]. Focusing on the role of the colloidal silica, addition very shortly before the headbox can be justified by anticipated benefits from fresh interactions between the cationic polymer and the colloidal silica. The transient nature of such effects has

been demonstrated with respect to retention [19,20]. Thus, late addition of colloidal silica, just after cPAM, might be expected to achieve a boost in retention and dewatering.

## EXPERIMENTAL

### *Chemicals and materials*

The chemicals and materials employed in this work were all the same as those specified in a previous article [2]. Briefly stated, the main fiber furnish was prepared by repulping of a 100% recycled copy paper product (trade-marked Boise ASPEN 100 Multi-use Recycled Copy Paper, Packaging Corporation of America, Boise Paper Division). The NFC consisted of the spray-dried, freeze-dried, and TEMPO-oxidized products supplied by the University of Maine. The cationic starch was Chargemaster L340 from Grain Processing Corp. (GPC; Muscatine, IA, USA). The colloidal silica used in the reported tests was Fennosil 2180 from Kemira Oyj (Helsinki). The cationic retention aid was Accurac 90, a very-high-mass cationic copolymer of acrylamide (American Cyanamid). Dilution and other preparation steps were the same as reported earlier [2].

As was noted at the beginning of this paper, the TEMPO-oxidized NFC product is of interest because the chemical treatment facilitates the fibrillation process and allows the material to be more fully redispersed after drying. The freeze-drying procedure provides an intermediate option whereby drying can take place with much less development of inter-particle hydrogen bonding and greater ability to be redispersed upon immersion in water.

### *Procedures*

The basic procedures used for the reported work were the same as reported earlier [2].

Because the focus of this part of the work was on the order of addition, some details are shown in **Table I**. A key aspect is that the addition levels of different substances generally referred to the total dry mass of the ingredients used to form a sheet. Such a manner of presentation was intended to allow easier focus on effects of changing the order of addition.

Matched levels of chemical treatments were combined with the same repulped 100% xerographic copy paper furnish in the two patterns shown in Table I. A figure depicting the options for order of addition of the NSF, cationic starch, cellulosic fiber furnish, cationic retention aid, and colloidal silica, relative to a hypothetical paper mill process, was presented in an earlier paper in this journal [1].

## RESULTS

### *Dewatering tests*

As was shown in Table I, Option 1 involves optional colloidal silica being added to the suspension of NFC after treatment with cationic starch. The NFC is added to the fiber furnish only after these pretreatments. The cationic retention aid (cPAM) is the final additive to the mixture of

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Item (in order of addition)	Level, % of dry mass of sheet	Role in Papermaking
Nanofibrillated cellulose (NFC)	5%	Potential bonding agent
Cationic starch	Various	Retention adjunct for NFC; also as a bonding agent
Colloidal silica (Option 1)	Either 0.5X or 1.0X the dry mass of cationic starch	Dewatering aid when used in sequence with a cationic polymer
100% recycled copy paper	To make 100%	The fiber furnish
Cationic retention aid	0.05%	Fine-particle retention
Colloidal silica (Option 2)	Either 0.5X or 1.0X the dry mass of cationic starch	Dewatering aid when used in sequence with a cationic polymer

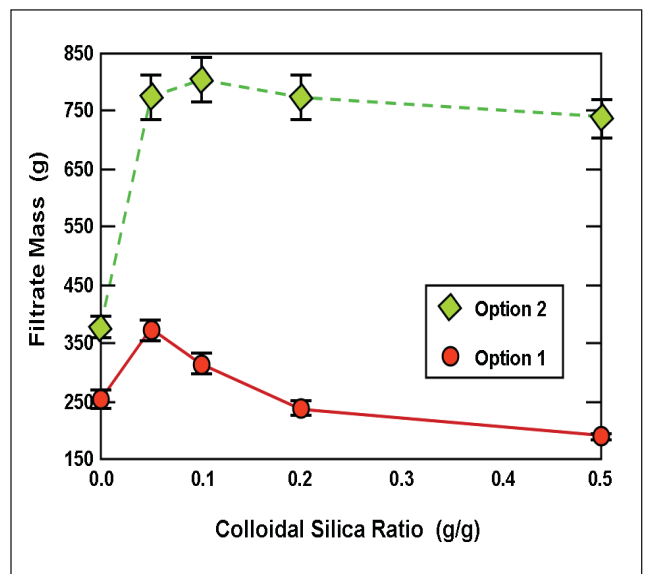
## 1. Orders of addition of the additives in the two main options considered in this study.

pretreated NFC and furnish. By contrast, in the case of Option 2, the NFC is pretreated only with cationic starch. After adding the cationic starch-treated NFC to the furnish, the final additives are cPAM and then the colloidal silica. As shown in the table, the net amounts of additives were the same in Options 1 and 2, but the order of addition was different. Option 1 matches the procedure used in earlier work reported by Rice et al. [1], whereas Option 2 is more typical of current industrial practices.

**Figure 1** shows that Option 2 resulted in much faster drainage than Option 1. The mass amounts shown correspond to readings taken 20 s after releasing the sealing cone. Here, the greatest drainage was achieved at the 0.1 silica-to-solids ratio. There did not seem to be much significant difference when the amount of silicon dioxide ( $\text{SiO}_2$ ) was increased above this ratio, with the mass of filtrate remaining at a plateau level. Option 1 resulted in overall slower rates of dewatering, but there was a more prominent peak at the optimum colloidal silica level of 0.05%.

When silica levels were equal to that of starch, higher drainage rates tended to be achieved, as shown in **Fig. 2**. This ratio of colloidal silica to cationic starch was more significant the higher the starch level became. Regardless of the order of addition that was used, less silica used than starch resulted in less drainage than with equal dry masses of silica and starch, and this effect was most notable at higher cationic starch levels.

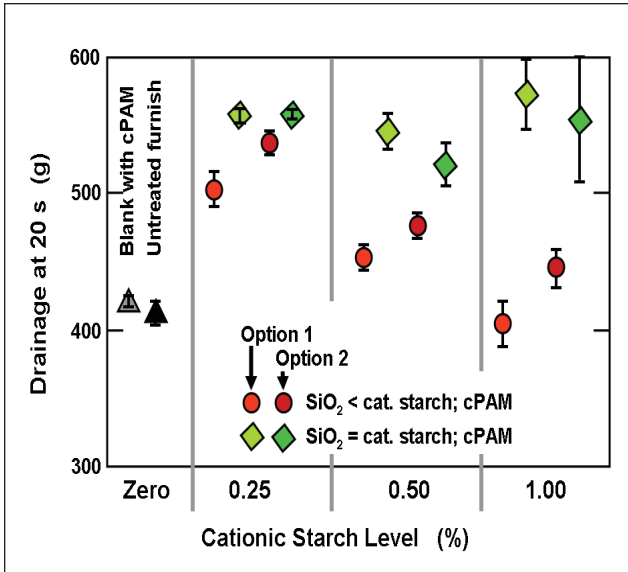
An unanswered question remaining after the work reported by Rice et al. [1] was whether strength benefits of NFC addition might continue to rise with increasing addition of cationic starch, up to very high levels. Though such a practice would be costly in terms of the required amounts of additives, sometimes papermakers have a need to boost strength gains in order to meet product specifications. Earlier work by Heermann et al. revealed continual strength increases to very high levels when forming in-situ polyelectrolyte complexes [21]. A concern, however, is that the higher chemical addition levels might have potential to hurt paper machine operating variables, such as the dewatering rates.



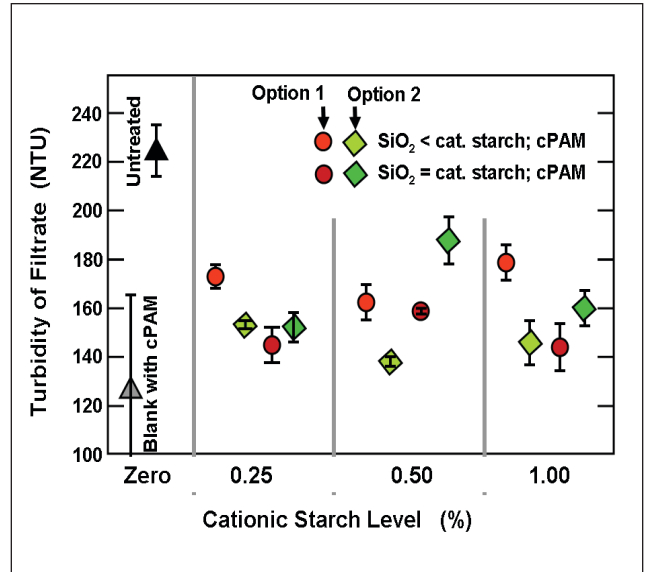
**1. Effect of the ratio of colloidal silica to nanofibrillated cellulose (NFC) on the mass of filtrate collected in 20 s after opening the sealing cone, with 0.1 parts of cationic starch added per 1 part of NFC before addition to the furnish.**

As shown in Fig. 2, at the lower addition ratio of colloidal silica, there appeared to be an optimum level of cationic starch, beyond which further increases produced no further gains in dewatering rates. However, as shown by the positions of the diamond symbols, it was possible to maintain rapid dewatering by employing a sufficient colloidal silica dosage, even at very high cationic starch levels.

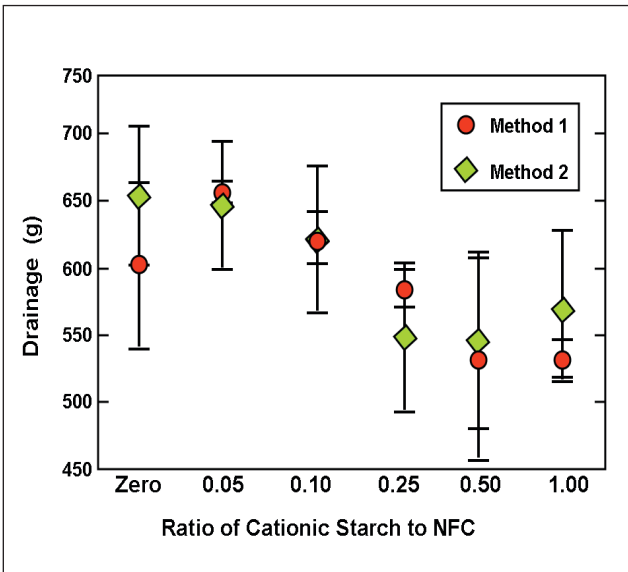
**Figure 3** considers a different set of experiments in which no colloidal silica and no cPAM retention aid were used. Two mixing procedures were compared. In Method 1, the cationic starch was mixed well with the NFC before addition to the fiber suspension. In Method 2, the NFC was added directly to the fiber suspension and then cationic starch was added to the mixture and agitated. As shown, regardless of the order of addition, dewatering became



2. Effect of cationic starch variable, orders of addition, and the ratio of colloidal silica (silicon dioxide; SiO<sub>2</sub>) to cationic starch on the mass of filtrate collected within 20 s of opening of the sealing can in systems with cationic polyacrylamide (cPAM) retention aid treatment.



4. Effects of cationic starch level, order of addition of colloidal silica (SiO<sub>2</sub>), and ratio of colloidal silica to cationic starch on the turbidity of filtrate in Britt jar tests.



3. Effect of the relative amount of cationic starch, as well as its order of addition, on the amount of filtrate collected 20 s after opening of the sealing cone in systems not including either colloidal silica (SiO<sub>2</sub>) or cationic retention aid. (Method 1 = add cationic starch to NFC before its addition to the suspension; Method 2 = add cationic starch last.)

slower when the cationic starch level was above 0.1 (as a ratio relative to NFC). As can be seen from the overlapping of 95% confidence intervals of the mean values, there was no statistically significant difference in the results for the two different orders of addition of the cationic starch.

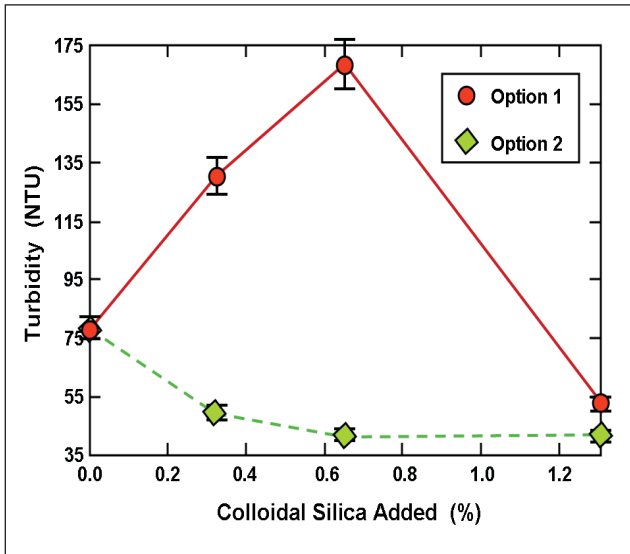
To understand why the order of addition did not appear as important in the absence of the colloidal silica and

cPAM, it is worth noting that those two additives have potential to contribute to very strong dewatering effects, which has been called the “microparticle effect” [4,12]. Most previously reported work involving those additives in papermaking systems have been limited to orders of addition in which colloidal silica and cPAM were the final additives, most often with the colloidal silica added last.

### Retention tests

In this work, the turbidity of filtrate from testing with a Britt drainage/retention jar [22,23,24] was used as an indication of the efficiency of fine-particle retention. As a general finding, both options involving NFC, cationic starch, colloidal silica, and cPAM resulted in effective retention of fine particles, contributing to lower turbidity values in comparison to the blank. As shown in **Fig. 4**, lower turbidity was most often achieved when using Option 2 in cases when the colloidal silica added was less than the starch. When Option 1 was applied for silica levels being less than that of the starch added, higher turbidity resulted. The overall finding was that all of the systems involving cationic starch, colloidal silica, and cPAM retention aid were shown to be effective in retaining fine materials within the ranges of addition considered.

Although changes in turbidity can be effectively used to estimate changes in retention efficiency in general, there will be a need in future work to quantify the retention efficiency of NFC itself. Due to the small diameters of NFC fibrils (e.g., 5 to 20 nm), their contribution to scattering visible light will be low. Thus, they are hard to detect by optical measurements. A practical approach may be to assume that the retention efficiency of NFC itself is likely to follow the same trends as the retention of fillers and cellulosic

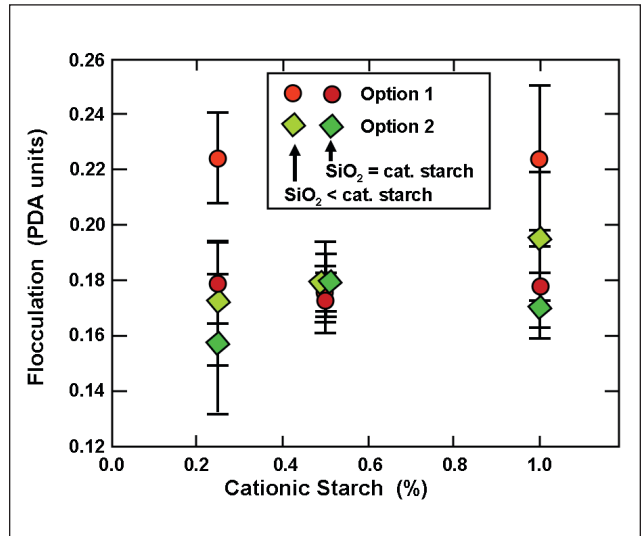


**5. Effect of colloidal silica addition level and order of addition on the turbidity of filtrate during Britt jar testing of systems in which NFC, cationic starch, colloidal silica, and cationic retention aid were added to a fiber suspension from 100% recycled copy paper.**

finer, both of which can be estimated by measurements of white water turbidity or filterable solids content. In principle, by maintaining a relatively high level of fines retention in a paper machine system, one might avoid a tendency for NFC to build up in the process water system.

**Figure 5** shows results for systems with constant levels of NFC (5%) and cationic starch (0.5%) relative to furnish solids. As indicated, Option 2 yielded far lower turbidity levels than Option 1. Additionally, Option 1 exhibited an increase in turbidity up to 0.65% silica added and then tended to decrease in turbidity to 1.3% silica added. It should be noted that it is unclear whether 0.65% silica addition was the turning point for this sudden decrease in turbidity for Option 1. Option 2 tended to decrease in turbidity but leveled off at around 0.65% silica addition.

The fact that the turbidity rose with increasing colloidal silica when using Option 1 (pretreatment of the NFC with cationic starch and colloidal silica) is consistent with an agglomeration mechanism. One can expect the cationic starch (positively charged) to coat the NFC surfaces. Then, the colloidal silica (negatively charged) will be expected to bridge those positively charged surfaces, resulting in agglomerates that are large enough to scatter light efficiently. But as long as the system has a net positive charge, due to the cationic starch, there will not be an excess of colloidal silica still available to interact with the cationic retention aid, which is added later in the case of Option 1. The strong decrease in turbidity at the highest level of colloidal silica (1.3% addition) is consistent with there being an excess of colloidal silica, after interacting with the cationic starch. That excess of colloidal silica then will be available to interact with the cationic retention aid, giving overall results



**6. Effect of cationic starch level, order of addition of colloidal silica ( $\text{SiO}_2$ ), and ratio of colloidal silica to cationic starch on the flocculation degree of fiber suspensions.**

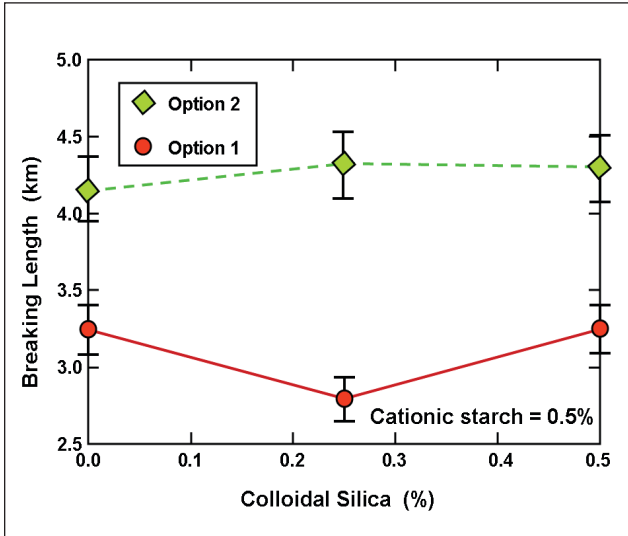
relatively close to those of Option 2, which represents a more typical order of addition in modern papermaking.

### Flocculation tests

At two of the conditions considered, Option 2 resulted in a lower level of flocculation than Option 1. As shown in **Fig. 6**, this was not the case for a starch level of 0.5%. In that case, the flocculation was essentially the same for the two optional orders of addition. When the amount of silica added was equivalent to that of the added starch, as compared to less silica than added starch, flocculation levels were typically less. The least amount of flocculation achieved was associated with 0.25% starch added when silica was equivalent to the amount of starch added for Option 2. This small amount of flocculation was also achieved for the same conditions as just mentioned, when starch addition was at 1%. In general, though some of the experimental conditions showed significant increases in fiber flocculation, most of the results did not show important differences in flocculation, even when the colloidal silica was added last (Option 2). This is notable in light of the earlier statement that agents added very late to a papermaking wet-end system often have the largest effects on retention aid dewatering. The generally low levels of flocculation found in this work suggest that relatively uniform, unflocculated fiber sheets can be prepared using the described program of papermaking additives.

### Handsheet properties

During handsheet testing, ratios of colloidal silica to cationic starch were 0%:0%, 0.25%:0.5%, and 0.5%:0.5%, respectively. As shown in **Fig. 7**, Option 2 (adding colloidal silica last) consistently yielded greater breaking length than Option 1 (with the colloidal silica added as part of the pre-



**7. Effect of colloidal silica level and order of addition on the tensile strength of handsheets containing NFC, cationic starch, colloidal silica, and cationic retention aid: Option 1 is to add colloidal silica to an NFC-cationic starch mixture before its addition to the fiber suspension. Option 2 is to add the colloidal silica at the very end, after cationic retention aid.**

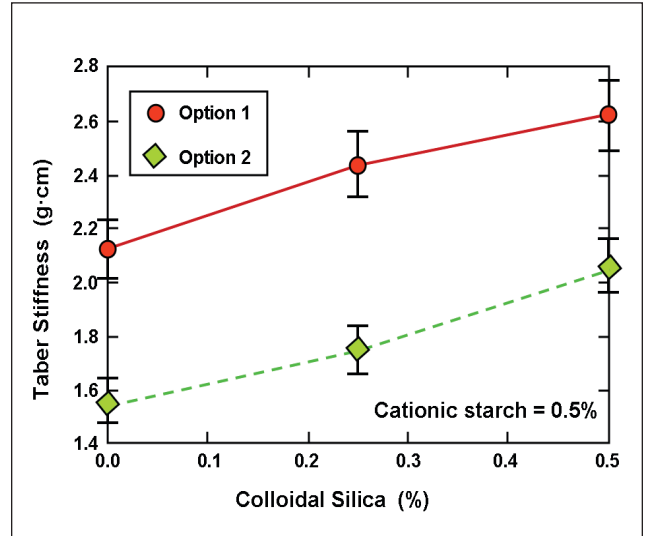
treatment of NFC, together with cationic starch). Option 1 gave a noticeably lower breaking length. However, the surprise in Fig. 7 is the fact that the level of cationic starch (in the range of zero to 1% of total solids) did not have a statistically significant effect when considering the results as a whole. Note that in the case of Option 2 (upper curve), the 95% confidence bars overlap each other for all of the conditions. In the case of Option 1 (lower curve), there was not a consistent trend. It is worth bearing in mind that handsheet tests often underestimate paper strength due to the low hydrodynamic shear conditions employed, which can lead to higher flocculation of the fibers. When handsheets are being prepared with the addition of dry strength additives and flocculating chemicals, the strength results may be unreliable [25].

From **Fig. 8**, as the silica additive percentage was increased, Taber stiffness increased, regardless of the order of addition that was used. However, Option 2 had noticeably lower Taber stiffness than Option 1 throughout all trials.

It is worth noting that stiffness results tend to be less affected by nonuniformity of the paper, due to the presence of fiber flocs. The fact that the results agree with the earlier findings of Rice et al. [1] support the concept that adding at least a part of the colloidal silica earlier in the process may provide an important option for papermakers.

**DISCUSSION**

Two principles are known to be important when seeking an optimum order of addition of papermaking additives. First is a general expectation that additives injected into the furnish very shortly before formation of the sheet are most



**8. Effect of colloidal silica addition level and order of addition on the Taber stiffness of handsheets prepared from 100% recycled copy paper with NFC, colloidal silica, and cationic retention aid.**

likely to have big effects on retention and drainage attributes. Evidence to support this concept comes from the fact that very high-mass copolymers of acrylamide, i.e., “retention aids,” are almost always added to papermaking furnish in commercial papermaking operations [26]. The point of addition for cPAM is usually either after or immediately before a screen system in the approach to a paper machine forming section. This first rule was evidenced most clearly in the present results for Britt jar tests (Fig. 5). It is clear that the combination of cationic starch with subsequent colloidal silica as the final additive was very effective. When the colloidal silica had instead been utilized as part of a pre-treatment system for the NFC, it apparently was no longer available to interact with the cPAM retention aid (as the final additive), which explains the much higher filtrate turbidities when employing Option 1 (adding the colloidal silica to the NFC and cationic starch mixture).

The second rule is that any negatively charged additives ought to be injected at a point in the system where the surfaces have already been treated with enough of a positively charged additive to create suitable anchoring points on fiber surfaces.

A third lesson that the present experimental results are providing is that a specific chemical or mechanism treatment (or order of addition) can often have complex effects relative to the goals of papermaking. Thus, an order of addition providing high stiffness of the paper (Option 1) was not conducive to the fastest dewatering or efficient fine-particle retention. Thus, once some interesting findings are obtained in laboratory work, it may be necessary to consider more than one arrangement of addition points on a commercial paper machine when attempting to find the overall most suitable additive program.

As has been shown at various points in the previous dis-

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cussion, the ratio of colloidal silica to cationic starch appears to have had significant effects. In principle, that might be due to the interactions among oppositely charged additives to the system. It can be hypothesized that peak performance of some systems might be achieved when there is a balance between positive and negative charge amounts. Such issues will be considered in a future article in this series.

## CONCLUSIONS

The order of addition of colloidal silica showed important effects in papermaking wet-end systems including nanofibrillated cellulose (NFC), cationic starch, colloidal silica, and cationic retention aid in the presence of fibers from 100% recycled copy paper. Adding the colloidal silica last, after the cationic retention aid, yielded the highest drainage, first-pass retention, and breaking length of the resulting handsheets. However, the highest stiffness of the paper was achieved when the colloidal silica was added to a mixture of cationic starch and NFC before their addition to the fiber suspension. By increasing the amount of colloidal silica relative to cationic starch in either order of addition, it was possible to maintain a favorable dewatering rate, even at a high addition level of cationic starch. **TJ**

## ABOUT THIS PAPER

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## ABOUT THE AUTHORS

Papermakers can switch addition points for chemical additives at almost zero cost. As shown in this paper, there sometimes can be benefits to the papermaker. This is the second paper in a set. The first, which appears on p. 563 of this issue, describes the experimental approach and gives evidence that the order of addition could be important. This article follows up in detail.

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 from the Engineering Enhancement Fee during the summer of 2021. 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

During this project, it was a challenge to study a large number of variables. The project required the work of four primary researchers (the authors) and a team of additional helpers described in the Acknowledgements.

The authors found that tests involving retention



Leib



Garland



Barrios



Hubbe

efficiency, drainage rates, fiber flocculation, and handsheet properties need to be viewed together in order to better understand chemical additives for papermaking. An interesting finding was that while the state-of-the-art recommendation for order of addition of some chemical additives often gave the most promising results, this was not so in every case.

Depending on production constraints or product goals, this study shows that mills can choose between two promising orders of addition for chemical additives. The third and final paper in this series by Barrios et al. will consider mechanistic aspects.

*Garland and Leib are undergraduate students, Barrios is a graduate student, and Martin A. Hubbe is a professor in the Department of Forest Biomaterials at North Carolina State University, Raleigh, NC, USA. Email Hubbe at: [hubbe@ncsu.edu](mailto:hubbe@ncsu.edu).*

their work in carrying out some of the experimental procedures, processing data, and contributing to the interpretation of data: Benen Tarpley, Blaine Mercer, Parker Janson, Wallace Layman, James Lewis, Erik Dahlgren, Peter Gough, Samuel Webb, Sophia Mills, Abby Saul, Jared Brew-Thompson, Autumn Satterfield, John Bedard, Katie Krull, Allie Temple, Jack Powell, Eduardo Sanchez, Austin Schott, and Cade Wanner.

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