HIGH STIFFNESS SURFACE COATING OPTIMIZATION THROUGH STARCH ENCAPSULATION OF PLATY KAOLIN

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Modified fillers consisting of kaolin particles encapsulated by starch have recently been demonstrated in mill trials to achieve significant filler loading levels without accompanying strength losses. In this work, laboratory experiments were conducted to explore the potential advantages of using starch-treated pigment for strength increases by application of surface coating. It is found that a platy clay coating will produce a higher increase in strength per unit weight of application compared to a fine clay, and more-so if the clay is encapsulated in starch. Starch encapsulation of clay produces a greater increase in strength than an equivalent weight proportion addition of starch to a kaolin formulation blend. The observations and measurements of changes in various physical properties of the coated samples are explained by a proportionate loss of void volume in the coating from the encapsulation process and the increase of stress transfer through introduction of higher platelet aspect ratio.

INTRODUCTION

A never-ending quest in the paper industry is to produce a product with optimized physical properties at minimal cost. A principal requirement for many applications is for paper sheets to have a high bending stiffness to weight ratio. Replacement of fiber by other materials is a common approach. Notably, many pigments are less costly per unit weight than fiber. Increasing the bending stiffness of paper with application of modified platy clay coatings is the objective of the current research project.

This paper explores the concept of using high aspect ratio kaolin as a surface coating for strength increase, which is a different application of clay from the traditional objective of attaining high brightness and opacity for printing quality. Platy clays when present in surface coatings have shown increased stiffness (Husband et al. 2006, 2009; Popil and Joyce 2008) through the mechanism of stress transfer achieved by overlapping platelets. If such platelets are fused with binder in a coating structure such that the air void volume is minimized, then increases in bending stiffness should be optimal. This can be achieved by a coating consisting of a cationic starch encapsulated clay and binder matrix. The targeted scenario is air knife or rod coated linerboard or alternately surface sizing of linerboard with an encapsulated platy clay formulation.

Application of a platy clay coating is an inexpensive alternative to achieving high bending stiffness through either higher basis weights and/or paper densification. Stiffness

is usually achieved through increase in paper basis weight or densification of a sheet of some prescribed basis weight through wet pressing. Both means involve high cost, the first in fiber, the second in capital equipment, usually involving an extended nip press at the wet end of the paper machine. Although densification of the sheet adds in-plane stiffness and compression strength, this causes a larger proportionate loss of bending stiffness from the corresponding loss of caliper required to achieve this result. This is expected from a beam mechanics point of view, since the in-plane stiffness of a sheet is dependent on its modulus E and caliper t as Et, and the bending stiffness is $Et^3/12$. For example, if caliper is decreased by 10% through wet pressing, the stiffness may increase by 10% as well from the higher E resulting from increased fiber bonding area. However, the bending stiffness will decrease by 20%. The data of Whitsitt 1988 shows moderate increases in compression strength accompanied by larger corresponding decreases in bending strength for a series of linerboards prepared with increasing densities obtained by progressive wet pressing times and pressures.

Alternatively to wet pressing, the replacement of fiber by high-density coatings lowers material and energy (pulping and drying) costs. The application of a high stiffness coating will provide the advantage of increased bending stiffness and increased compression strength using conventional coating equipment or a size press. Increased popularity of white top linerboard for product boxes comes at an increased cost of a secondary headbox and weaker short fiber bleached kraft pulp. Replacement of this layer with a strength-adding pigment layer allows reduced fiber usage, decreased chemical and energy consumption for pulping, and lower capital costs for a secondary bleached pulp line and headbox. Additionally, it is expected that such high-density coatings should have a moderate degree of water vapor and water resistance, thus eliminating the requirement for costly polymer overcoats. Coated linerboard is a growing market opportunity to supplant white top for the shipment and marketing of produce.

Therefore, the proposed development of a coating which produces a high bending stiffness is desirable for the paper industry to be able to reduce its fiber and energy usage to produce products that meet the requirements for high stiffness papers.

Principles of the Concept

Conventional kaolin coatings have considerable void volume between the platelets interspersed with specks or blobs of binder when considered on a microscopic scale. This microscopic level of porosity allows a controlled amount of ink penetration that is desirable for certain printing processes. The idea behind increasing stiffness with coating is to apply a coating such that the void volume is minimized. Furthermore, a platy clay coating with platelets aligned as a deck of playing cards should allow a stress transfer mechanism that would manifest itself as a increase in elastic modulus and hence stiffness.

The physics behind the concept is straightforward and can be discussed succinctly as follows: from elastic beam theory, the bending stiffness for a symmetric three-ply board consisting of 2 surface ply facings designated by the suffix 1 and a middle core designated by 2 is (e.g., Navaee-ardeh and Nazhad 2008):

$$S = \frac{E_2 t_2^3}{12} + \frac{E_1(t^3 - t_2^3)}{12} \tag{1}$$

The total thickness of the paperboard is simply

$$t = 2t_1 + t_2. \tag{2}$$

So that upon substitution, the bending stiffness (1) becomes:

$$S = \frac{E_2 t_2^3}{12} + E_1 t_1 \left[t_1 t_2 + \frac{t_2^2}{2} + \frac{2t_1^2}{3} \right] \tag{3}$$

In the ideal case, when the facing modulus $E_1 \gg E_2$ and $t_2 \gg t_1$, the dominant term prevails, and the familiar sandwich approximation for bending stiffness is obtained:

$$S \approx \frac{E_1 t_1 t_2^2}{2} \tag{4}$$

Thus the bending stiffness of a sandwich plate structure is principally governed by the substrate separation thickness squared, separating thin coating layers of high modulus. To consider how the modulus can be engineered in a composite coating, the well known result for the effective modulus E_{eff} of composite materials is considered,

$$E_{eff} = \sum_i E_i \alpha_i \tag{5}$$

where the volume fraction and modulus of each ith component is represented by α_i and E_i respectively. For the ideal case of a coating layer consisting of no air void volume, a starch binder of high modulus E_s, a pigment of modulus E_p and critical pigment volume concentration (CPVC) being taken as the pigment volume fraction α_p:

$$E_c \cong E_p \alpha_p + E_s (1 - \alpha_p) \tag{6}$$

Therefore, based on the model suggested by the mechanics equations 1 through 3 above, the concept consists of developing: a high modulus pigment to be bonded with a high modulus binder containing no or minimal air void volume. Thereby the aim is to maximize the bending stiffness by creating a sandwich plate panel structure with the bulk of the sheet comprised of coarse non-collapsible fibers. This discussion excludes consideration of the coating-fiber interface as a contributing layer, since at high coating solids impregnation of fibers by coating is minimal as may be gleaned from inspection of SEM cross section micrographs of coated papers or linerboards (Popil 2005).

Preparation and advantages of starch-encapsulated clay as a filler have been documented for some time (Yan et al. 2005; Yoon et al. 2006; Zhao et al. 2005) and recently summarized (Deng et al. 2010). It has been shown that a starch-to-clay proportion of 2.5% by dry weight is sufficient to encapsulate clay platelets such that increased retention of clay is realized without appreciable loss of strength properties when introduced into the wet-end of the paper machine. In the case considered in this

paper, once dispersed onto a surface coating, the swollen starch jackets of the starch-treated clay will serve to bind the platelets or agglomerations of platelets to each other in addition to the usual latex. Figure 1 serves to illustrate the concept.
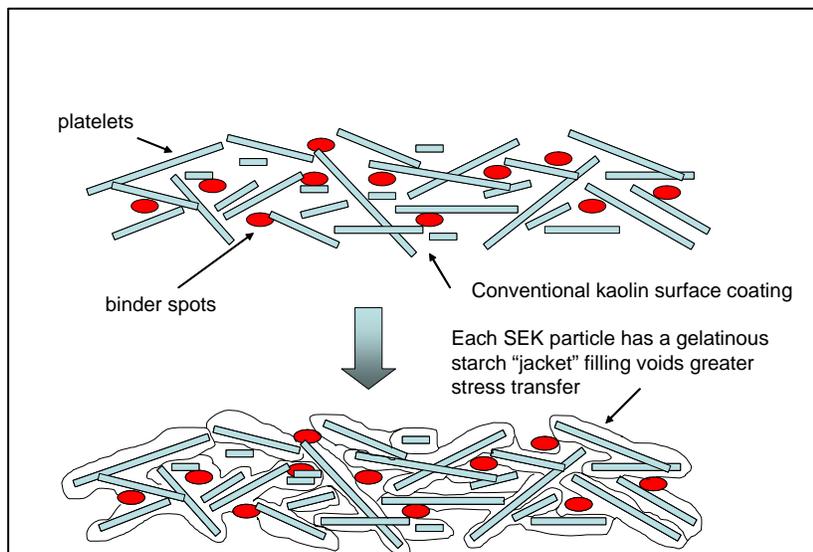

Figure 1. Concept behind the likely mechanisms of using of starch encapsulated platy clay

EXPERIMENTAL

Materials and Methods:

KSC, Astraplate, and SEK kaolin clays were supplied as dry pigments by Imerys' Sandersville Georgia USA lab and were blended with Dow 6810A SB latex in the ratio of 100:16 by dry weight to formulate coating colors of 55% solids. No dispersing agents or additives were used. Kaolin "HX" was used in slurry from the supplier. KSC (A) is a fine clay, Astraplate (B) is a delaminated platy, HX (C) is super-platy, and SEK (D) is the acronym for starch-encapsulated kaolin produced by Imerys through a spray drying process. Information provided by the supplier is that the delaminated clay Sedigraph distribution reports 85% of the particles being < 2 μm, median size 0.4 μm, and the HX distribution 58% < 2 μm median size 1.6 μm. Another formulation, SE-HX (starch encapsulated HX, formulation E) was prepared by cooking carboxylated starch (Penford 180) for 90 minutes at 90 degrees C and mixing in dry HX in a weight proportion to pigment of 2.5:100. This blended slurry was dried at 105 °C, ground with a mortar and pestle, and subsequently passed through a #325 vibrating mesh screen. The accepts from the screen were then blended with the SB latex to 55% dry weight solids. Both supplied commercial SEK and prepared SE-HK may contain aggregates of clay platelets that have not been fully dispersed but are still small enough to pass through the screen. Starch-treated clays coatings were applied after heating to 70 °C to ensure that the starch surrounding each clay particle was swollen with absorbed water. Table 1 summarizes the pigments used in the coating formulations.

Table 1. Clays for the Various Coatings Used in the Experiments

Clay type	Sample ID	Trade name	Substrate
Fine	A	KCS	Liner and newsprint
Delaminated	B	Astraplate	Liner and newsprint
Super platy	C	HX	Liner and newsprint
Starch encapsulated	D	SEK	Liner and newsprint
Starch encapsulated platy	E	SE-HX	Newsprint

Coating Application Technique and Physical Testing

The formulations were continually mechanically stirred during application. Coatings were applied to the chosen substrate strips of dimensions 5.5 x 16.5 inches using an Autodraw II drawdown coater and a #15 wire rod all supplied by Industry Tech of Oldsmar Florida. The drawdown coater was modified to have the sample supporting table covered with a soft closed-cell polyurethane foam pad to ensure uniform coating coverage. The choice of applicator rod was to provide coatings of typical grammages found on LWC grades. The coated samples were dried by placing them immediately after coating on a slowly rotating cylinder illuminated by 4 250 W IR heating lamps. Coat weight measurements accurate to 0.5 g/m^2 were made by weighing each sample piece to at least 3 decimal places prior and after the coating process. This also required accurately trimming the sample area of 0.062 m^2 and basis weight measurements made on samples that were equilibrated to 50% RH moisture from a dry state to ensure the same moisture content.

Coatings were applied to one side of 205 g/m^2 unbleached softwood kraft linerboard in one series of experiments and on both sides of 50 g/m^2 newsprint in a following series of experiments. Physical properties were measured on coated samples after equilibration using standard TAPPI methods for caliper (T 411 and T551), basis weight (T 410), tensile properties (T 404), and bending stiffness (T 489). Measurements also included in-plane velocity squared (TSI) using the Lorentzen and Wettre tensile strength orientation (TSO) instrument. Values of TSI multiplied by the sample basis weight are equivalent to tensile stiffness. Correlation between mechanical and TSI measured stiffness has been determined (Popil 2010). In addition, out-of-plane stiffness C_{33}, which relates to the compressibility of the sheet, was measured using IPST-developed equipment (Habeger et al. 1986; Waterhouse 1994). Tensile strength (N/mm) was measured using an Instron universal testing machine Model 1122 with Series IX software, and 2 point bending resistance (mN-m) using an L&W tester.

RESULTS AND DISCUSSION

a) Applications on Linerboard

Measured physical properties regarding strength were not appreciably changed by application of the coatings onto 205 g/m^2 linerboard, as summarized in Table 2. This may be understood from the perspective of the modulus of the coatings relative to the modulus

of linerboard the latter being about 4.8 GPa in the MD and 2.1 in the CD (Haj-Ali et al., 2008). Measurements of platy and fine clay coatings in pigment/binder ratios of 1:2 by the author (Popil and Joyce 2008) have indicated 860 MPa for a platy clay coating being 15% higher compared to the fine clay coating. Husband et al. (2009) report measurements of platy clay coatings of convention 100:20 pigment/binder ratios to be in the range 2 - 2.7 GPa, the higher range obtained by increased starch level in the formulation.

Remarkably, although coatings were all of the same solids and applied by a #15-wire rod, the SEK coat weight was the smallest weight (Fig. 2). The Mayer wire rod applies a coating thickness that is principally governed by the interstices resulting from the selected wire diameter contacting the surface to be coated. Therefore, the expectation is that all coatings from this application method should have the same wet film thickness of approximately 38 microns or 1.5 mils. If the encapsulated kaolin starch swells appreciably, as depicted conceptually in Fig. 1, then the density of the coating before drying would definitely be less than a corresponding clay coating without encapsulation, where the clay would be comparably more compacted. The observation that the dry weight of encapsulated coatings was smallest in comparison to the other coatings was consistent throughout all the experiments.
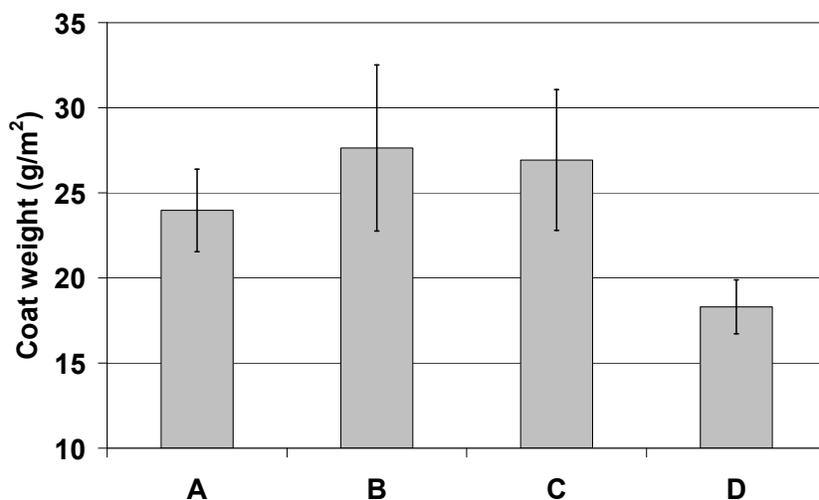

Fig. 2. Coat weight obtained on linerboard using various pigments. Error bars are standard deviations .

The effects of coatings on linerboard are more evident however, when the results are indexed i.e., normalized to the basis weight of the applied coating the effect of SEK stands out in the tensile strength as shown in Fig. 3. The expectation is that any relative strength increases are proportional to the dry coating grammage. Previous measurements (Popil 2006) have found that short span compression strength increases with increased clay coating grammage.

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Table 2. Summary of Coating Results on Linerboard.*

coating	Base bw	Coat wt	TSI MD	TSI CD	SCT	BS$_{MD}$	C$_{33}$	tensile st	TEA	stiffness	TSI MD st
	g/m^2	g/m^2	(km/s)^2	(km/s)^2	kN/m	mN-m	MPa	N/mm	J/m^2	N/mm	N/mm
A	205.1	21.5	10.84	6.15	7.26	10.25	108.6	17.9	256.1	1220.3	2456
A	207.5	25	10.53	6.13	7.44	10.675	106.3	17.2	225.2	1213	2448
A	207.5	25.4	10.59	6.24	7.63	9.25	102.7	17.4	247.7	1182	2466
B	203.3	22.8	10.69	5.89	7.60	10.95	107.7	17.3	221.8	1224.3	2417
B	205	31.1	10.24	6.29	7.89	10.3	113.2	17.1	239.4	1157	2418
B	207.6	29	10.56	6.22	7.72	10.1	116.6	16.8	226.5	1182.3	2498
C	207.4	25.3	10.03	5.78	7.77	9.135	111.2	16.9	240.6	1140	2334
C	205.7	28.5	9.77	5.71	7.51	10.135	106.1	18.3	266.8	1220	2288
C	202.2	26	10.38	5.79	7.79	10	109.7	16.6	224	1156.3	2369
C	205.4	27.9	10.27	5.8	7.47	9.05	117.5	17.5	255.4	1175.3	2395
D	206.8	19.3	10.61	5.73	7.60	10.95	104.5	18.2	249.4	1244.3	2399
D	206.4	16.7	10.13	5.73	7.54	10.75	100.9	17	220.4	1217.3	2260
D	207.3	18.9	10.53	5.78	7.42	10.15	102.5	18.3	269.3	1218	2382

Notes: "bw" stands for basis weight, "SCT" for short span compression strength, "BS" for 2 point bending stiffness, tensile strength is "tensile st", "TSI MD st" is the tensile stiffness obtained by multiplying "TSI MD" by basis weight and is about twice as large as mechanically measured stiffness.

Table 3. Data for Coatings Applied to Newsprint

pigment	coating wt.	TSI MD	TSI CD	BS$_{MD}$	caliper	MIT folds	C$_{33}$	Tensiles	TEA	Ultrasonic
	g/m^2	(km/s)^2	(km/s)^2	mN-m	um		MPa	stre N/mm	J/m^2	stiff N/mm
A	47.2	7.3	3.51	0.28	136	23	156	3.2	31.7	311.3
A	51.5	7.3	3.55	0.31	139	22	173.2	3.041	25.9	330.6
A	45.5	7.36	3.45	0.28	140	14.7	148	3.063	32.3	315.3
B	56	8.58	4.66	0.34	133	10	166.4	3.09	25.18	336.1
B	55.4	8.86	4.76	0.45	137	11.3	174.3	3.129	26.27	290.8
B	51.1	8.77	4.36	0.33	130.8	15.3	146.8	3.18	25.05	344
C	44.3	9.23	4.83	0.32	143	7.3	153	3.45	33.75	343.3
C	41.4	9.1	4.65	0.36	137.6	23.7	130.1	3.05	20.71	364.4
C	48.1	9.11	4.5	0.37	138.1	18	140.2	2.895	20.8	295
D	31.4	8.19	3.86	0.29	134.3	33.3	139.7	2.911	21.26	321.7
D	39.3	8.28	3.89	0.35	134.6	23.3	153.6	2.888	21	309.8
D	36.5	8.25	3.91	0.37	138.9	29.3	138.1	3.52	33.94	352.6

Increases in compression strength should in principle be accompanied by similar increases in tensile strength, since the stress-strain curve for paper is identical in compression as it is in tension (Calvin and Fellers 1975). The vertical axis is the tensile strength N/mm divided by the dry coating weight in g/m^2. The platy clay coatings generally show larger increases in strength properties compared to the fine clay A (KCS).
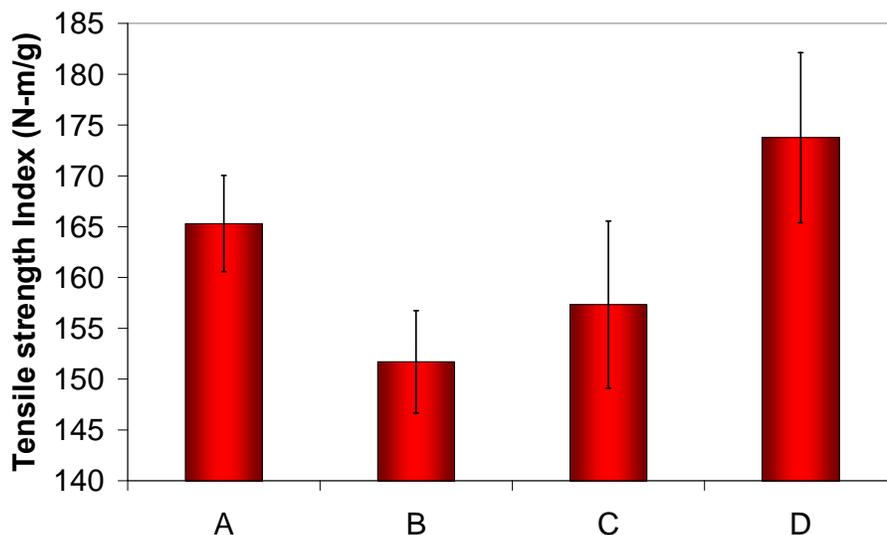

Fig. 3. Coated linerboard with starch-treated clay (D) shows an increase in tensile strength (vertical axis) compared to other coatings when normalized to the basis weight of the dry weight of the applied coating.

b) Application on Newsprint

Results of the application of coating on linerboard suggested that the modulus of the coatings was not substantially above that of the substrate. Therefore, attention was turned to the application of coatings to a lightweight newsprint to accentuate the differences imparted by different coatings to the measured physical properties. In this case, coatings were applied similarly as for the linerboard but on both sides of the sheets. The data for the sample set are summarized in Table 3.

In this case, differences between the different coatings become more apparent than in the case of linerboard, since the coating and substrate basis weights are about equal. Folding endurance (Tappi method T 511) was measured using the MIT tester to assess the propensity for fold cracking. The effects of various pigments becomes more evident once the results are normalized i.e., "indexed" to the applied coating weight, and such results are graphically summarized in Fig. 4.

In the case of newsprint, there were clear differences in the increases in physical properties with platy clay formulation C, and more so with starch-treated D. The per-weight increase in bending stiffness was most significant (58%) for the application of formulation D, the starch-treated platy clay. An interesting result is that the out-of-plane stiffness was least for the case of applied platy and starch-treated coatings C and D, which could have an advantage for contact printability, where surface compressibility is required. Starch-treated clay coating D showed a greater fold resistance compared to delaminated clay B or platy C.
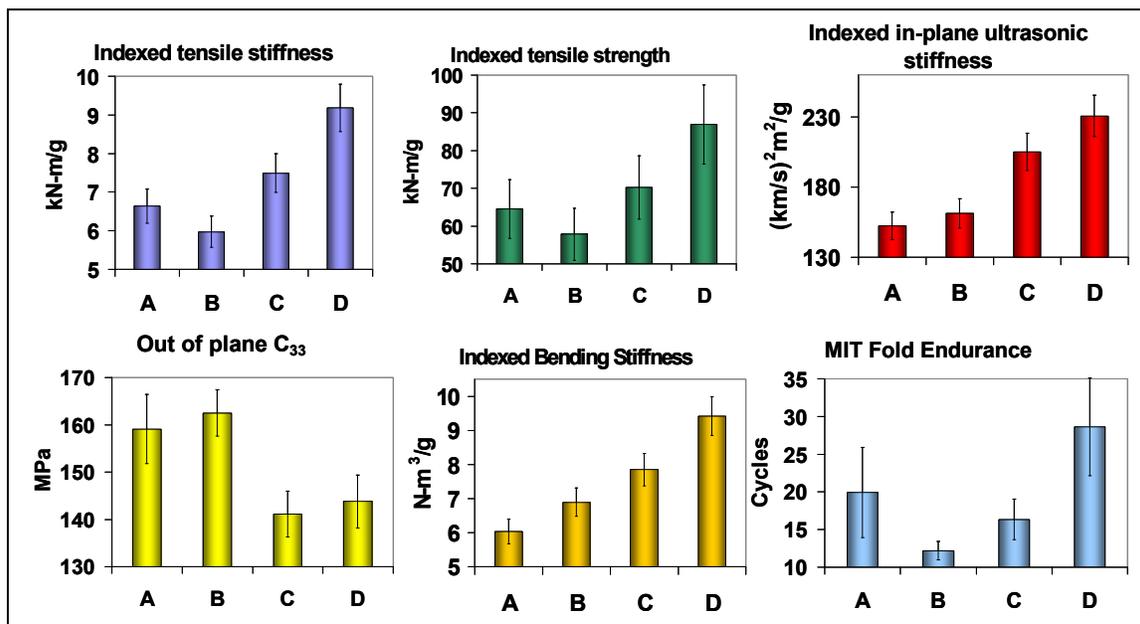
Fig. 4. Summary of results of coatings applied to newsprint

c) Encapsulation of Platy Clay versus Clay Addition

Another consideration is to compare the effect of starch added separately to the formulation blend in the same relative amount as contained in a starch-treated clay formulation. The question is whether encapsulation of clay by starch is an advantageous intermediate step compared to simply including the starch separately in the same starch-to-clay dry weight proportion. In this case, cooked starch carboxylated Penford 180, 2.5% in proportion to pigment by dry weight was added directly to the coating blend. The aspect ratio of commercially supplied SEK was unknown; however, since it is intended as a filler clay, it is likely calcined blocky clay. Since there may be an advantage towards attaining larger strength properties if platy clay were to become encapsulated, the experiment of starch addition versus starch encapsulation was conducted with pigment C.

Starch (Penford 180) in dry weight proportion of 2.5% was added to 55% solids platy clay slurry C and was heated for 30 minutes at 90 °C. Platy clay slurry supplied as a commercial product contained 0.4% polyacrylate dispersant and was presumed to consist of delaminated platelets. The starch/clay blend was dried in an oven at 105 ºC and ground using a mortar and pestle. The resulting powder was sent through a # 325-mesh vibrating screen. The screen accepts were blended with latex binder to make a 55% solids coating color to be consistent with the previous applications of coatings. This coating color with platy clay C and blended starch, no encapsulation, is dubbed "C+" in the results presented in Table 4 and Fig. 5. The coating colors with starch enclosed platy clay are labeled E1 and E2. Another new set of samples and measurements were made for coatings A, B, C, and D to complete this comparison.
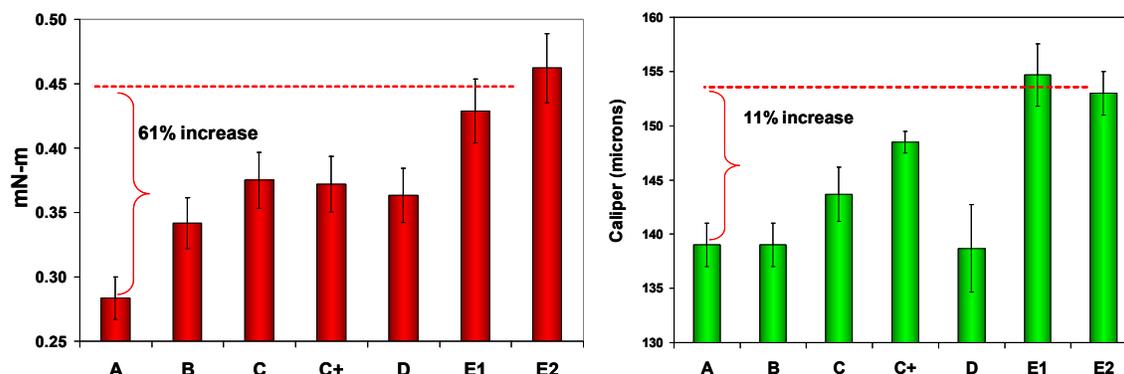
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Fig. 5. Increase in bending stiffness using coatings on newsprint. Right graph: calipers of the corresponding samples

Table 4. Summary of Results (averages) for Another Set of Samples Including Platy Clay with Added Starch "C+", Starch-Treated Platy Clays "E1", and Repeat "E2"

sample ID	pigment	coating bw	TSI MD	TSI CD	BS_{MD}	caliper	C_{33}	Strength	Stiffness	TEA	Strain	Modulus	Folds
		g/m^2	(km/s)^2	(km/s)^2	mN-m	µm	MPa	N/mm	N/mm	J/m^2	%	N/m^2	
A	KCS	51.3	7.22	3.54	0.28	139.0	155	3.00	310.9	24.3	1.5	2129	20.5
B	ASTRA	52.1	7.86	3.93	0.34	139.0	162	2.99	304.8	23.3	1.5	2087	16.1
C	HX	47.9	9.13	4.69	0.38	143.7	151	3.18	307.6	26.0	1.6	2106	19
C+	HX+ starch	56.5	9.07	4.86	0.37	148.5	159	3.54	324.6	33.6	2.2	2223	15.3
D	SEK	38.6	8.07	3.88	0.36	138.7	162	3.28	330.3	26.0	1.6	2261	19.9
E1	SE-HX (a)	55.3	8.66	4.53	0.43	154.7	167	3.37	336.9	29.2	1.7	2306	22.1
E2	SE-HX (b)	56.2	8.99	4.84	0.46	153.0	171	3.47	323.3	30.6	1.7	2220	16.4

Figure 5 shows that the encapsulated platy clay in two separate cases "E1" and "E2" increased bending stiffness over the value obtained by the coating A by 61%. This increase cannot be explained by the 11% increase in caliper shown in the right-hand graph of Fig. 5, since as discussed previously, bending stiffness is expected to be proportional to the change in caliper squared or cubed, depending on whether the appropriate bending stiffness model is taken to be a beam or a sandwich structure. Caliper change alone can only account for at most a 33% change in bending stiffness. Therefore, encapsulation of kaolin by starch has a mechanical effect beyond what is attainable through caliper increase alone. This also demonstrated by the fact that the sample "C+" 100:2.5 clay starch ratio but no encapsulation, did not show as large an increase in bending stiffness as the starch encapsulated platy clay formulations "E1" and "E2".

Figure 6 shows that the coat weight of SEK was substantially lower than the other coatings, which is consistent with previous experiments. Indexed tensile stiffness and correspondingly tensile strength were still highest for SEK in comparison to the other coatings. Addition of starch by blending in C+ was not effective in increasing tensile strength or specific modulus as by encapsulation shown by E1 and E2.
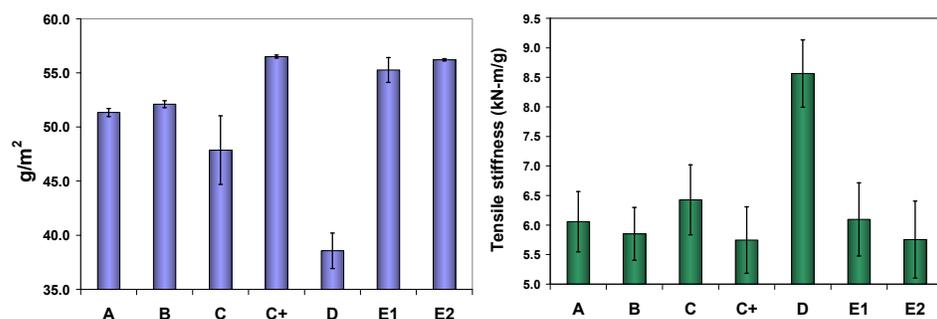
Fig. 6. Coating weights (dry) and indexed tensile stiffness for the coated newsprint set
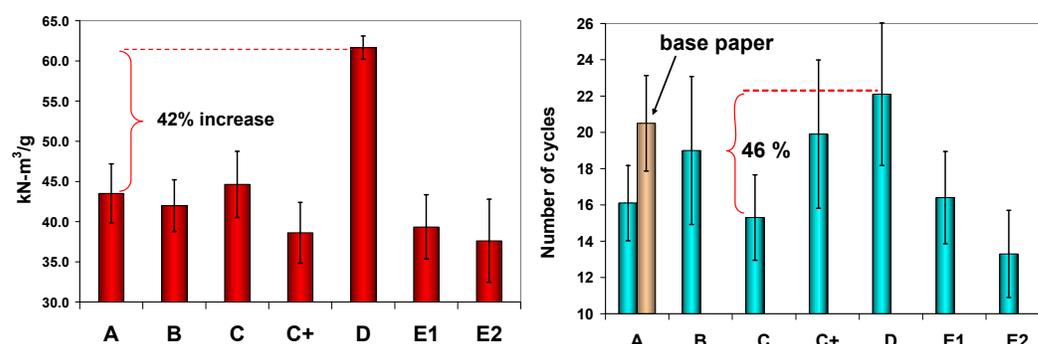

Fig. 7. Indexed in-plane modulus and MIT fold endurance, coatings applied to newsprint

The indexed modulus is shown in Fig. 7 to have a maximum for the case of SEK formulation D. The values for the in-plane moduli were obtained from the tensile strength tests and take into account the caliper of each specimen. The right-hand side graph of Fig. 7 shows the results for MIT fold cycles, which indicate for SEK on newsprint that the fold endurance was comparable to uncoated paper.

Inclusion of pigments and starch will increase stiffness, as seen here, but can also lead to loss of fold endurance from the abrasive action of the pigment combined with inelastic brittle starch adhered to the surface. This leads to possible surface fissures once coated samples are subjected to shearing stresses from folding. The introduction of a platy clay "HX" in formulations C, E1, and E2 appears to limit fold endurance. Overall, the surface application of SEK appears to have the largest comparative benefits per unit dry weight of application.

CONCLUSIONS

1. Substitution of existing pigments by starch-treated kaolin SEK or starch encapsulated platy clay is straightforward and requires no extra expenditures in an existing surface coating process equipment; however this study has not addressed high-speed runnability of the experimental formulations.

2. Application of SEK in a surface coating will increase tensile and bending, as shown in the lab results. Starch encapsulation definitely has mechanical effect that cannot be duplicated by starch addition to the coating color. The caveat is that surface properties may not be optimal in terms of brightness, gloss, or printability since these properties were not measured in these experiments.
3. Indications are that improved printability may be expected on the basis that a platy kaolin or an SEK coating will have a lower out-of-plane stiffness and hence greater surface compressibility compared to other kaolin coatings investigated in this paper. However, further trial work should be performed to optimize a coating formulation to suit a particular purpose or target.
4. The conclusion drawn from the presented work is that SEK overall has the best increase in physical properties per unit basis weight of application compared to the other clays investigated: fine, delaminated, super-platy, and super-platy starch-treated clay.

ACKNOWLEDGMENTS

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