Influence of polymer charge, temperature, and surfactants on surface sizing of liner and greaseproof paper with hydrophobically modified starch

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ABSTRACT: The aim of this study was to investigate the properties of hydrophobically modified (HM) quaternary ammonium starch ethers for paper sizing. These starches possess temperature-responsive properties; that is, gelation or phase separation occurs at a certain temperature upon cooling. This insolubility of the HM starches in water at room temperature improved their performance as sizing agents. The contact angles for water on sized liner were substantially larger than on unsized liner. When the application temperature was well above the critical phase-separation temperature, larger contact angles were obtained for liner independently of pH compared with those at the lower application temperature. $Cobb_{60}$ values for liner decreased upon surface sizing, with a low pH and high application temperature giving lower water penetration. Contact angles on greaseproof paper decreased upon surface sizing as compared to unsized greaseproof paper, independently of pH and temperature. Greaseproof paper showed no great difference between unsized substrates and substrates sized with HM starch at different pH. This is probably due to the already hydrophobic nature of greaseproof paper. However, the $Cobb_{60}$ values increased at low pH and low application temperature. Surfactants were added to investigate how they affect the sized surface. Addition of surfactant reduces the contact angles, in spite of indications of complex formation.

Application: Surface sizing using HM starches and their interaction with surfactants give information on the nature of the interaction in coating formulas using latices stabilized by surfactants. Temperature-responsive starches can be adjusted to industrial applications because fiber coverage is much better when the starch is applied above the critical phase-separation temperature.

Film formation and prevention of retrogradation are important criteria when using starch in papermaking and surface treatment [1]. During the last decade, cationic starches have been used instead of oxidized starches in the size press because of the greater environmental benefits they offer, and also because they improve paper properties such as ink-jet printability, sizing degree, and sheet porosity [2]. The cationic starches will bond with the anionic fiber in the manner of a wet-end starch, which means they will form a stronger, more surface-oriented film. This results in an elevated starch concentration at the surface and a pronounced effect in improving opacity, brightness, print gloss, and ink density of paper compared with values achieved with oxidized starch, because of the better hold-out properties of cationic starches [3].

Hydrophobically modified starches (HM starches) have been of great interest for surface applications in the paper industry. Mešić et al. [4] showed in bench-coating trials of temperature-responsive HM starches that hydrophobic character and print quality increased when sizing was performed at a higher temperature (70°C). These temperature-responsive HM starches were also investigated on a molecular level in previous work at the authors' laboratory [5], where it was shown that the charge of the starch was of great importance for the starch properties in solution. When the starch had a zero net charge, pronounced phase separation was observed with particular precipitation as a result.

In this work, we have investigated surface sizing with temperature-responsive HM starches, taking into account the pH of the solution at application because the charge density of the starches affects temperature-induced precipitation [5].

The aim of this work was to investigate whether polymer charge, temperature, and surfactants have any effect on surface properties after surface sizing of different substrates representing low and high Cobb values. If the starch solution is applied above the phase-separation temperature, spreading of polymer over the fiber surfaces is expected to be favored compared to when the solution is already phase-separated. It is interesting to study the effect of surfactants to determine whether the stabilizing effect that the surfactants showed with the starch solutions [6] also improves the final surface properties of the sized substrates.

EXPERIMENTAL

Materials

The substrates used were liner (120 g/m^2) with no internal or external sizing (SCA Packaging, Argovia, Switzerland) and greaseproof paper (45 g/m^2) with no internal sizing (Nordic

Paper, Säffle, Sweden). An HM starch provided by Lyckeby Stärkelsen Industrial Starches AB (Kristianstad, Sweden) was used with a mole fraction of carboxylic groups, X_{COOH}= 0.027, and a degree of substitution with respect to hydrophobic modification, DS_N= 0.027. The HM starch was produced by reaction of a pre-oxidized starch with a quaternary amine reagent (3-chloro-2-hydroxypropyldimethyl-dodecylammonium chloride). Synthesis mechanisms are described elsewhere [5]. An antifoaming agent (Glanopon DA 202) from Bussetti (Wien, Austria) was added during the hydrophobizing step. Alcohol residues might be present in the final product from reaction of the epoxide with water instead of starch. The final product was purified from byproducts by stripping off residual amounts of epoxide and alcohol. The residual epoxide content was less than 50 ppm and close to zero [7], according to the starch supplier. As a reference, a cationic quaternary ammonium starch ether without hydrophobic modification (C*size 05962) from Cerestar (Mechelen, Belgium) was used. Cationic surfactant, dodecyltrimethylammonium chloride (DoTAC, 98% titr.), from Acros (New Jersey, USA) was used.

Methods

We prepared all starch solutions in the same way: approximately 13 g of starch was dispersed in 120 ml water, and the starch suspension was cooked in a water bath at 95°C for 30 minutes under vigorous stirring. The solutions were diluted with warm water to obtain concentrations of 8 wt%, and pH was adjusted to between 3 and 10.

We measured the surface tension using a TD1 Lauda tensiometer (Dr. R. Lauda, Wobser GmbH & Co. KG, Lauda-Königshofen, Germany). The surfactant (DoTAC) was added as a micellar solution (0.67 M) to the starch solution in consecutive additions. The total surfactant concentration in the sample was calculated and corrected for the increase in sample volume. The experiment was performed at room temperature. Water was used as a reference medium.

Surface sizing was performed using a bench coater (K-Coater M101, RK Print Coat Instruments Ltd., Royston, UK) with HM starch on liner and greaseproof paper. A closely wound bar with a wire diameter of 0.15 mm (12-µm nominal wet-film deposit) was used at speed 2. Starch solutions at different pH values with and without surfactant were applied at room temperature (23°C) and at 70°C. For sizing at 70°C, the rod was preheated in an oven at 80°C and mounted on the bench coater. The hot starch solutions were applied on a polyester film mounted above the paper substrate at the application point to prevent the solution from absorbing into the paper substrate before sizing could begin. All sheets were allowed to dry at room temperature and then stored in a controlled environment (23°C and 50% RH) for one week until further analysis. Starch pickup was 5.1 ± 0.4 g/m² for liner and 2.2 ± 0.3 g/m² for greaseproof paper. The reference starch was used for comparison only on liner.

The $Cobb_{60}$ values were measured according to ISO 535. We took an average of six measurements for the liner and three measurements for the greaseproof paper.

We measured contact angles on an FTA 200 Dynamic Contact Angle Analyzer (First Ten Ångstroms, Portsmouth, USA) using a drop of deionized water. The contact angles at 0.1 s and 2 s were monitored and the average taken of twelve measurements. The contact angles at 0.1 s on liner and the contact angles on greaseproof paper can be considered to be the real contact angles because no absorption effects were observed. On the other hand, the contact angles at 2 s are apparent contact angles because they were influenced by the absorption effects of the substrate.

Rheological properties of the HM starch solutions were measured using a controlled-shear-stress rheometer (Paar Physica, MCR 300, Graz, Austria). Oscillatory measurements were performed with concentric cylinder geometry in the linear viscoelastic region to determine the complex viscosity (η^*) as a function of temperature. The temperature varied between 80°C and 20°C (60 minutes for one cooling-reheating loop), while the amplitude (1) and the frequency (1 Hz) were held constant. The absolute value, $|\eta^*|$, was then plotted. The relation between complex viscosity and modulus is given by:

$$|\eta^*| = \frac{\left(G'^2 + G''^2\right)^{1/2}}{\omega} \quad [1]$$

where G' is the storage modulus, G'' is the loss modulus, and ω is the frequency of oscillation.

The transmittance was measured using a conventional UV spectrophotometer (Shimadzu, UV2101 PC, Japan) at 618 nm at 23°C. The turbidity (τ) was calculated from the transmittance (T) of a light beam passing through a cell of length (L), according to:

$$\hat{o} = -\frac{\ln T}{L} \quad . \quad [2]$$

The transmittance of the starch solution in the presence of the cationic surfactant DoTAC was measured in the same way as described above, with a surfactant:starch ratio of 0.2:1 (w/w).

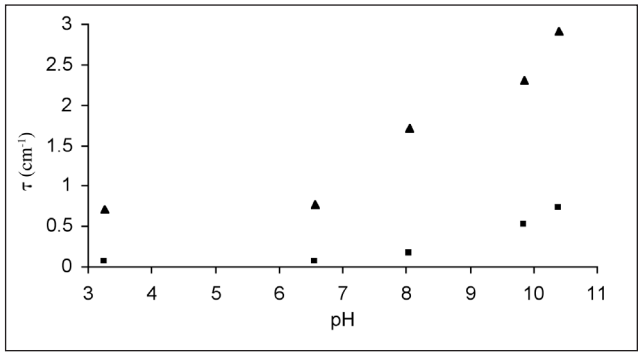
The yield was measured as the weight percentage precipitated based on the dry weight of starch. The pH of the cooked solutions was adjusted by addition of sodium hydroxide and hydrochloric acid, and the solutions were left to cool to room temperature. The samples were centrifuged at room temperature for two hours at 500 rpm, and the liquid phase was decanted. The precipitate was washed with water three times and stored in a desiccator until constant weight was obtained.

The charge density of the starches was measured using a Particle Charge Detector (Mütek, PCD 03, Herrsching Germany), and the average of six measurements was taken.

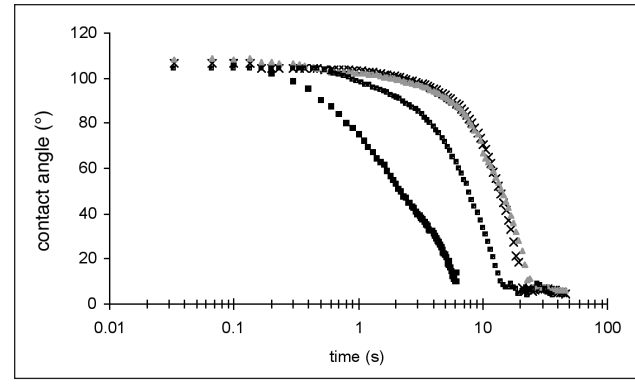
RESULTS

Effect of pH

The turbidity was measured at different pH at 23°C. We observed that the turbidity of HM starch increased with increas-



1. Turbidity as a function of pH (23°C). The symbol ▲ represents a starch solution (8 wt%) without surfactant, and ■ represents a starch solution with addition of 0.67 M DoTAC (starch-to-surfactant ratio is 1:0.2). Standard deviations were between 1% and 7% of the measured values.



2. Contact angles (23°C, 50% RH) as a function of time for untreated liner and for liner sized with HM starch at different pH values: unsized (■), pH 3.3 (x), pH 5.1 (▲), and pH 9.1 (●). The curves represent an average of six measurements.

ing pH (**Fig. 1**). When adding a surfactant to the starch solution, the turbidity was substantially lower compared with that of solutions without surfactant.

The pH also had a great impact on the precipitation yield. **Table I** presents the yields at different pH values; the highest precipitation yield was obtained around pH 10.

pH	Yield (%)
3.3	7.6 ± 0.2
6.5	8.2 ± 0.2
7.0	11.0 ± 0.1
7.2	11.2 ± 0.1
9.1	14.9 ± 0.3
9.9	15.8 ± 0.2

I. Precipitation yield at different pH values. Error limits indicate the standard deviation.

Contact angles were measured on unsized substrates, on substrates sized at different temperatures using the HM starch, and on liner sized with the reference starches. **Figure 2** shows the contact angles as a function of time for unsized liner and for liner sized with HM starch at different pH values. The contact angles decreased rapidly for unsized liner, but showed a plateau value at short times. The plateau was more extensive for liner sized with HM starch compared with the unsized liner. The same observation was made for the reference starch on liner (not shown), but the plateau was of shorter duration than for the HM-sized liner.

The dynamic contact angles at 0.1 s and 2 s were monitored and are summarized in **Tables II**, **III**, and **IV**. The contact angles of untreated liner were 99 ± 4 at 0.1 s and 45 ± 5 at 2 s. For untreated greaseproof paper, the contact angles were 106 ± 5 at both 0.1 s and 2 s. Table II presents the contact angles for liner sized with the reference starch.

Table III presents the contact angles at 0.1 s for substrates sized with the HM starch. We observed that there was no large difference in contact angle for any of the substrates when sized with solutions of different pH values; they all fall within the same range, taking the standard deviations into consideration. Furthermore, the contact angles of water for sizing at different application temperatures did not differ significantly.

Temperature (°C) Liner	Contact angle (°) 0.1 s	2 s
20	75 ± 2	62 ± 3
70	78 ± 3	67 ± 3

II. Contact angles (23°C, 50% RH) for liner sized with conventional cationic starch, using 8 wt% starch solution at pH 8 and at two different allocation temperatures (20°C and 70°C). Error limits indicate the standard deviation.

pH	Charge (μeq/g)	Contact angle (°) Liner 20°C	Greaseproof 20°C	Liner 70°C	Greaseproof 70°C
3.3	115 ± 1	108 ± 5	69 ± 2	107 ± 4	73 ± 2
5.1	100 ± 5	108 ± 5	72 ± 4	110 ± 4	74 ± 3
6.3	83 ± 7	101 ± 5	71 ± 3	109 ± 4	77 ± 3
9.1	57 ± 5	104 ± 5	N/M	110 ± 4	N/M

III. Contact angles (23°C, 50% RH) measured at 0.1 s for substrates sized with HM starch at 20°C and 70 °C. Error limits indicate the standard deviation.

pH	Charge (μeq/g)	Contact angle (°) Liner 20°C	Greaseproof 20°C	Liner 70°C	Greaseproof 70°C
3.3	115 ± 1	104 ± 5	68 ± 2	105 ± 4	74 ± 2
5.1	100 ± 5	97 ± 4	72 ± 4	105 ± 5	74 ± 3
6.3	83 ± 7	93 ± 5	71 ± 3	106 ± 5	76 ± 3
9.1	57 ± 5	94 ± 4	N/M	107 ± 4	N/M

IV. Contact angles (23°C, 50% RH) measured at 2 s for substrates sized with HM starch at 20°C and 70°C. Error limits indicate the standard deviation.

However, there was a weak trend for contact angle to decrease with increasing pH for liner sized at 20°C. Liner sized with HM starch showed an increase in contact angle over both the reference starch and the unsized substrate. Greaseproof paper sized with HM starch showed decreased contact angles compared with those of the unsized substrate.

Temperature (°C)	pH	q (μeq/g)	$Cobb_{60}$ (g/m²)	
			Liner	Greaseproof
20°C	3.3	115 ± 1	141 ± 3	30.3 ± 2.1
	5.1	100 ± 5	139 ± 3	24.0 ± 1.9
	6.3	83 ± 7	145 ± 3	25.9 ± 2.0
70°C	3.3	115 ± 1	128 ± 3	25.3 ± 2.1
	5.1	100 ± 5	139 ± 3	25.6 ± 2.0
	6.3	83 ± 7	139 ± 3	25.5 ± 2.2

V. $Cobb_{60}$ values on substrates (23°C, 50% RH) sized with HM starch solution (8 wt%) and at different pH values at 20°C. $Cobb_{60}$ values on liner with the reference starch (pH 8) were 140 ± 2 g/m² and 143 ± 3 g/m² at 20°C and 70°C, respectively. Error limits indicate the standard deviation.

When the contact time was increased to 2 s, the contact angles at the higher application temperature did not depend on pH (Table IV). However, for liner sized at room temperature, there was a decrease in contact angle as pH was increased. Both the short- and long-time contact angles for sized liner indicated that the temperature at application affected the contact angle at high pH but not at low pH.

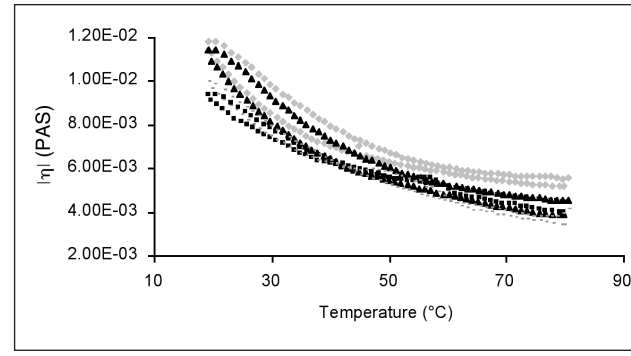
Table V shows the $Cobb_{60}$ values on liner and greaseproof paper sized with HM starch. The lower the $Cobb_{60}$ value, the less water penetrated into the base paper. The $Cobb_{60}$ value for the unsized liner was measured as 185 ± 3 g/m² and, for unsized greaseproof paper, 26.5 ± 2.0 g/m². The unsized greaseproof paper had a very low $Cobb_{60}$ value because of intensive beating of the pulp during manufacture. $Cobb_{60}$ values for liner did not change with pH at low application temperatures, but a lower $Cobb_{60}$ was observed at low pH and high application temperature. Greaseproof paper showed higher $Cobb_{60}$ values for starch solutions with low pH and low application temperature, while $Cobb_{60}$ was not influenced by either temperature or pH for the other measurement points.

The rheological temperature ranges of HM starch solutions at different pH values are summarized in **Fig. 3**. It was observed that all the starch solutions behave fairly similarly, with the solution at pH 3 being somewhat more viscous compared with the solutions at higher pH values.

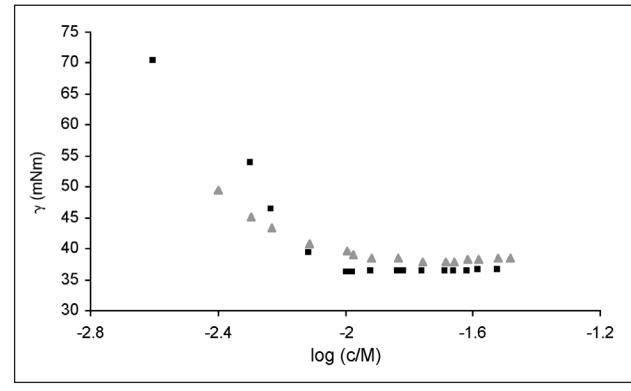
Effect of surfactants

The surface tension of the starch solution after addition of the cationic surfactant, DoTAC, was determined (**Fig. 4**). As a reference, the surface tension of pure water with addition of DoTAC solution is shown in the same figure. The surface tension is a measure of the amount of surfactant monomer in a solution. The more monomer (DoTAC) is added to the solution, the lower the surface tension will be. The critical micelle concentration (cmc) was determined as the breakpoint in the graphs and calculated as 10 mM for the surfactant in water and 7–10 mM for the surfactant in the starch solution of 8 wt%.

Contact angles on liner sized with HM starch solutions of 8 wt% containing different concentrations of the cationic surfactant DoTAC were investigated (**Table VI**). Addition of



3. Complex viscosity as a function of temperature for solutions of HM starches (8 wt%): pH=3.27 (♦), pH=5.09 (■),pH=6.32(▲), and pH=9.07 (—).



4. Surface tension as a function of increasing surfactant (DoTAC) concentration in an oxidized starch solution of 8 wt% and pH 8 at 23°C (▲) and in pure water at pH 6.8 (■).

DoTAC (8 mM to 80 mM) prevented precipitation of the starch. For both application temperatures, there was a decrease in contact angle as the surfactant concentration increased.

DISCUSSION

Effect of pH

It is of interest to investigate whether the pH of the starch solution could have a significant effect on surface properties, as it has on the net charge of the starch backbone. The turbidity (Fig. 1) and the precipitation yield (Table I) of an HM starch solution increased as pH increased, showing that the amount of precipitate depends on the pH of the solution. Therefore, starch solutions at lower pH, i.e., a higher net charge, give more stable solutions. In previous work [5], when the net charge of the starches was adjusted by DS_N, it was concluded that low charge gave more precipitation, i.e., less stable solutions. This shows that the stabilizing effect is more complex than stated in [5]. Addition of surfactant showed partly inhibiting effects on turbidity.

Contact angles decreased rapidly with time for unsized liner (Fig. 2). The contact angle measured on unsized liner was the apparent value affected by absorption, not the true contact angle as described in the methodological section. For liner treated with HM starch, the contact angles reached a

DoTAC (mM)	Contact angle (°)			
	20°C		70°C	
	0.1 s	2 s	0.1 s	2 s
0	101 ± 5	93 ± 5	109 ± 4	106 ± 5
8	90 ± 4	87 ± 4	98 ± 4	96 ± 3
11	87 ± 4	86 ± 3	88 ± 4	85 ± 5
80	63 ± 3	62 ± 3	75 ± 3	72 ± 3
Ref. starch	75 ± 2	62 ± 2	78 ± 3	67 ± 2

VI. Contact angles (23°C, 50% RH) at 0.1 s and 2 s for liner sized with HM starch solution at 20°C and 70°C (pH 8) containing the cationic surfactant DoTAC. Error limits indicate the standard deviation.

plateau value, and the true contact angles were measured. Table III presents the contact angles at 0.1 s for substrates sized with HM starch. It was observed that the pH of the starch solution had no effect at either application temperature; all the values fell within the same range for each substrate. Still, liner sized with HM starch solution showed greater contact angles than either the reference starch or the unsized liner. A trend toward decreased contact angles with increasing pH was observed for substrates sized at 20°C and was significant at 2 s (Table IV). Because the contact angles were greater at 70°C and did not differ over the time scale of the measurements (0.1 s–2 s), we can conclude that the liner surface had a more hydrophobic character when the application temperature was well above the critical phase-separation temperature.

Greaseproof paper sized with HM starch showed similar values for contact angles independently of application temperature and contact time. However, the contact angles decreased after sizing compared with those on unsized greaseproof paper.

The temperature-responsive polymers showed different solubility at different temperatures. The results in Table I and Fig. 1 show that a higher pH promoted precipitation and that the contact angles decreased with increasing pH (Tables III and IV) when starch was applied at 20°C. This indicates less coverage of the fiber substrate. For sizing performed at 70°C, there was less precipitation, indicating improved fiber coverage, and pH was expected to have little effect on the surface properties. The contact angles at 70°C on liner were very similar over the pH range, while the contact angles decreased with increasing pH at 20°C. From Fig. 2 and Tables II, III, and IV, we can conclude that the charge of the starch did affect the final surface properties.

By applying the starch solution at 70°C (well above the phase-separation temperature), the phase separation occurred while the starch solution cooled and dried on the paper surface. A more even fiber coverage was obtained because a completely dissolved starch is assumed to give better coverage than a partly particulate fluid. This showed that the hydrophobic character of the substrate could be improved if the HM starches were applied at temperatures above the critical phase-separation temperature [5].

Liner sized with HM starch (Table V) gave almost the same $Cobb_{60}$ value as the reference starch (140 g/m²), but a lower value than that for unsized liner (185 g/m²). The unsized liner had very poor water resistance and was totally penetrated by water in the $Cobb_{60}$ experiments before the sorption sequence was completed. However, when sizing was performed at 70°C and low pH, the $Cobb_{60}$ value was lower than for the reference starch, probably because of improved film formation due to reduced particulate precipitation. The HM-sized greaseproof paper showed no essential difference in $Cobb_{60}$ compared with unsized greaseproof paper (26.5 ± 2 g/m²), except for a slightly higher $Cobb_{60}$ value for the application at 20°C and pH 3.3 (Table V). A similar trend was observed for contact angle (i.e., low contact angle at pH 3.3 and 20°C), indicating spreading and absorption of water into the surface.

The complex viscosity as a function of temperature (Fig. 3) showed similar behavior to that reported earlier [5]. In the earlier work, it was concluded from phase-angle measurements that formation of a gel network in combination with normal Arrhenius behavior occurs for these kinds of starches. When the charge of the starch was low, gelation was promoted, while particulate precipitation was predominant when the charge was high. A trend in complex viscosity, indicating an increase in gelation propensity with decreased particular precipitation at low pH, was observed in this work (Fig. 3 and Table I).

Effect of surfactants

One interesting feature of these HM starches is their behavior in contact with surfactants. Upon addition of surfactant to the starch solution, the turbidity decreased dramatically compared with that of the starch solution without surfactant (Fig. 1), indicating that the surfactant stabilized the starch and prevented precipitation. Figure 4 shows the surface tension as a function of amount of surfactant added. The cmc of the surfactant in pure water was calculated as 10 mM, which was lower than the value of 16 mM reported in the literature [8], probably due to impurities in the surfactant.

The starch solution had lower surface tension than the corresponding starch-free solution at the same surfactant concentration in the concentration range up to the cmc (Fig. 4). The lower surface tension could be due to the surface-active nature of the starch, which might create an enriched concentration of polymer at the air/liquid interface, or it could result from carryover products from the synthesis step. Normally any complex formation between the polymer and the surfactant should have resulted in a higher cmc value in the presence of polymer. This was not observed, even if the exact value of the cmc in the starch-DoTAC system was difficult to detect due to the indistinct breakpoint. Still, the possibility of aggregation between the starch and the surfactant could not be excluded, because the observed cmc value for the starch-DoTAC system might have been affected by impurities such as antifoaming agent. Aggregate formation between HM starch at low net charge and DoTAC has been reported in previous work [6].

Cationic surfactant was added to the starch solution before sizing. At both application temperatures, the contact angles on liner sized with HM starch solutions containing different concentrations of DoTAC decreased as the surfactant concentration increased (Tables III and VI). This indicates that the surfactants are located at the surface of the paper and that the possible formation of complexes between HM starch and surfactant did not prevent the increase in the hydrophilic character of the surfaces resulting from the presence of surfactant molecules.

The surfactants prevented starch from precipitating (Fig. 1) and thus promoted better film formation by the starch. The contact angles did not change significantly between 0.1 s and 2 s, which indicated little absorption of water into the paper.

CONCLUSIONS

The final surface properties of paper surface-sized with HM starch are dependent on the application temperature, the surfactant concentration, and the charge of the polymer. The contact angles indicated that the hydrophobic character of the surface was enhanced when the application temperature was above the phase-separation temperature. By adjusting the pH of the starch solution to a high value and performing surface sizing above the critical phase-separation temperature, we were able to enhance the hydrophobic character of the surface-sized liner. Upon addition of surfactant, there was a substantial deterioration in hydrophobic character due to the presence of surfactants on the surface. $Cobb_{60}$ values were reduced upon sizing with HM starch compared with those for unsized liner, while greaseproof paper was not affected by sizing except at a low application temperature and a pH of 3.3. **TJ**

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INSIGHTS FROM THE AUTHORS

Because starches are renewable materials, their use in packaging can minimize the requirements for other, nonrenewable additives. This study has investigated the properties of hydrophobically modified (HM) quaternary ammonium starch ethers for paper sizing.

The authors previously showed that hydrophobically modified (HM) starch forms inclusion complexes with surfactants. It was therefore interesting to explore further the effect of surfactants to determine whether the stabilizing effect of surfactants on starch solutions also improves the final surface properties of the sized substrates.

Addition of surfactant was found to reduce contact angles, although the effect was less than might have been expected. The amount of surfactant required was not previously known and had to be investigated here, because many latices are stabilized with surfactants, but in unknown quantities.

Hydrophobically modified starches were clearly demonstrated to have a positive effect on short-time interactions between water and paper and liner substrates, on the condition that film forming and spreading of the size solution are controlled to a sufficient degree. The discoveries reported here may be of particular interest for inkjet grades. Surfactants may have both negative and positive effects on a paper surface-sized with hydrophobically modified starches.

Starch application at temperatures above the phase-separation temperature of the HM starch was observed to promote better coverage of the fiber substrate.

This research could be extended by investigating other coating and sizing formulations with components such as pigments that offer hydrophobic properties.

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