

## Stabilization of Potentially Sticky Substances in Pulp Mill Process Water with Polysaccharides

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Characterization of the dissolved and colloidal substances (DCS) in the process water of a bleached chemithermomechanical pulp (BCTMP) production line showed that the major lipophilic substances (wood resins) were dehydroabietic acid, linoleic acid, oleic acid, and palmitic acid. Model compound experiments indicated that polysaccharides contained in DCS had significant effects on the stability of the system. Effects of neutral and acidic polysaccharides on the stability of wood resins (model resin acid R and fatty acid O) in the presence of metal ions were then investigated. No calcium-induced aggregation occurred when the concentration ratio of added neutral polysaccharide to R and O exceeded 0.2 and 1.0, respectively. Acidic polysaccharide could further degrade the stability of resin dispersions in the presence of calcium ions and yet have a positive effect in the presence of only sodium ions.

*Keywords:* Stabilization; Process water; Polysaccharide; Metal ion

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### INTRODUCTION

During the production and further treatment of mechanical wood pulps, approximately 1% to 5% of the components of the wood will be released from its tissue into the process waters. The released substances are mostly composed of hemicelluloses, pectin, dispersed wood resin, and lignin-like compounds, along with small amounts of lignin, acetic acid, formic acid, and inorganic salts (Ekman and Holmbom 1989; Sjöström 1990a,b; Thornton *et al.* 1994). These dissolved and colloidal substances (DCS) are potentially sticky, and they may accumulate to high concentrations in integrated pulp and paper mills, affecting the papermaking processes and product quality in various ways (Wearing *et al.* 1985; Francis and Ouchi 2001; Richardson *et al.* 2012).

Lipophilic wood resin is often blamed for the formation of tacky deposits on papermaking equipment. Uncontrolled aggregation of wood resin is a major factor affecting its deposition. Surface charges (in a double electric layer) are formed once the carboxylic groups of resin acids and fatty acids begin to dissociate (Back and Allen 2000; Lee 2012); thus, colloidal resins can stabilize themselves *via* an electrostatic mechanism, even though there are no steric stabilizing polymers present in the system (Sundberg *et al.* 1996a; Lee *et al.* 2010). However, inorganic electrolytes can compress the double electric layers to some extent, leading to the de-stabilization of colloids (Hiemenz and Rajagopalan 1997; Stack *et al.* 2014). In addition to wood resins, anionic pectin has been shown to interact with cationic additives like retention and drainage aids, rendering them less effective (Gao *et al.* 2011; Petzold *et al.* 2012).

The present study is focused on the stability of wood resins that remain in the pulp and how they are affected by other pulp components such as non-ionic and anionic polysaccharides in the presence of typical metal ions.

## EXPERIMENTAL

### Materials

The main unit operations and equipment involved in the production of bleached chemithermomechanical pulp (BCTMP) from wood chips include pre-steam treatment, screw press extruder, chemical treatment with sodium sulfite and sodium hydroxide, 1<sup>st</sup> refiner, dilute chest, 2<sup>nd</sup> refiner, latency chest, multi-disc filter, screw compressor, and bleaching tower. The pulps were taken from the second refiner and the bleaching tower of a Masson pine chemithermomechanical pulp (CTMP) production line. The consistency was approximately 35%, and the pulp was stored in a freezer until use.

Model wood resins R (resin acid) and O (fatty acid), ethanol, calcium chloride, sodium chloride, and non-ionic polysaccharide (guar gum, GM) were all of AR (analytical reagent) grade. Acidic polysaccharide (pectic acid, PA) in sodium salt was of chromatographic grade. All standard reagents were of chromatographic grade.

### Methods

#### *Sampling of DCS from mechanical pulp*

The pulps were suspended in distilled water (1.0% consistency) and gently agitated for 3 h at 60 °C to ensure release/sorption equilibrium between the fibers and the water phase. To remove fibers and other non-colloidal substances, the suspensions were centrifuged at 500 x g for 30 min, and the resulting upper supernatant was pipetted off and collected as a DCS sample.

#### *Preparation of model wood resin dispersion*

The model resins R and O were first dissolved in anhydrous ethanol at a concentration of 50 g/L; then, the resultants were injected into distilled water under agitation at approximately 300 rpm. After 30 min of agitation, the dispersion was dialyzed to remove the solvent using distilled water. All the concentrations of model dispersions were the same as 500 mg/L.

#### *Stability experiments*

Resin dispersions in the presence/absence of polysaccharides were heated to 60 °C. During constant agitation, calcium or sodium chloride solution preheated to 60 °C was added to the above dispersions at varying concentrations. After agitation at 60 °C for 30 min, the samples were centrifuged at 500 x g for 30 min. Finally, the supernatants were pipetted off for turbidity analysis.

#### *Analyses*

Lipophilic extractives (resin) were extracted from DCS samples and model dispersions with methyl *tert*-butyl ether (MTBE) according to Örså and Holmbom (Orsa and Holmbom 1994) and were then silylated with 25 µL of pyridine, 25 µL of trimethylchlorosilane (TMCS), and 100 µL of N,O-bis(trimethylsilyl)trifluoroacetamide (BSTFA); then, the TMS (Trimethylsilyl) derivatives were analyzed using an Agilent

6890 GC (Gas Chromatograph) (USA) with a long column (HP-5ms, 30m x 0.25 mm) coupled to an Agilent 5973N mass spectrometer. The column temperature was initially set to 150 °C; 30 s after injection, the temperature was ramped up to 230 °C at a rate of 7 °C/min, then to 290 °C at a rate of 10 °C/min where it was held for 10 min. Compounds were identified by comparing the mass spectra obtained with those of NIST computer libraries and by mass fragmentography.

The dissolved polysaccharides were determined using methanolysis to obtain monomeric methyl glycosides of the polysaccharides and the methyl glycoside methyl esters of uronic acids. After being silylated, the monomer concentrations were quantified by gas chromatography (Bertaud *et al.* 2002). Turbidity was measured with a HACH 2100N turbidimeter (USA). The instrument expresses turbidity in nephelometric turbidity units (NTU). Particle size distribution was determined with a Malvern Nano ZS instrument (UK). The results are given as the averages of the intensity functions.

## RESULTS AND DISCUSSION

### Characterization of Lipophilic Extractives (Resin)

The DCS samples and dispersions were prepared according to the above mentioned procedures and had particle sizes of approximately 0.2 to 0.6 µm, which is in the colloidal range. The chemical compositions of DCS samples before and after bleaching were identified using gas chromatography-mass spectroscopy (GC-MS) techniques and are listed in Table 1.

**Table 1.** Compositions of DCS from Process Water (Expressed in Area Percentage of Total Ion Chromatogram)

	Unbleached	Bleached
	(%)	(%)
<b>Fatty acids</b>		
1 Tetradecanoic acid	6.92	2.50
2 Pentadecanoic acid	4.22	1.87
3 Methyl palmitate	0.00	1.77
4 Palmitoleic acid	7.00	2.49
5 Palmitic acid	32.90	12.88
6 Methyl linolein	5.03	3.38
7 Linoleic acid	6.36	7.63
8 Oleic acid	7.08	3.91
<b>Resin acids</b>		
9 Pimaric acid	3.29	11.76
10 Sandarackpimaric	ND <sup>a</sup>	2.06
11 Isopimaric acid	ND <sup>a</sup>	1.99
12 Levopimaric	3.75	1.83
13 Dehydroabietic	19.04	40.84
14 Abietic	0.00	3.26
15 Dibutyl phthalate	4.42	1.89

a. ND: not detected

As can be seen in Table 1, for unbleached pulps, the major lipophilic substances observed in DCS samples included palmitic acid (32%), dehydroabietic acid (19%), oleic acid (7%), hexadecenoic acid (7%), tetradecanoic acid (7%), and linoleic acid (6%). It is of note that this result was different from that determined by solvent (acetone, hexane) extraction of unbleached pulps. Only the dissolved and dispersed resin and fatty acids were identified and determined, since they better represent the actual DCS in process waters. The ratio of fatty acids to resin acids was approximately 3:1. After bleaching, the ratio became 2:3, with the amount of dehydroabietic acid having markedly increased. The alkaline conditions during bleaching facilitated the swelling of fibers and improved the solubility of resin acids.

### Stability of DCS in Process Waters

The DCS that dissolved or dispersed in process waters formed a very complex system. The concentration of lipophilic extractives correlates well with the turbidity of a disperse sample (Sundberg *et al.* 1993). The stability of colloids can be described in terms of their aggregation behavior in the presence of simple electrolytes (Shaw 1992). The dispersion becomes more stable against aggregation with increasing residual turbidity. Based on these theories, the stability of DCS samples from CTMP and BCTMP pulps against two typical metal ions ( $\text{Ca}^{2+}$ ,  $\text{Na}^{+}$ ) was investigated in the present study.

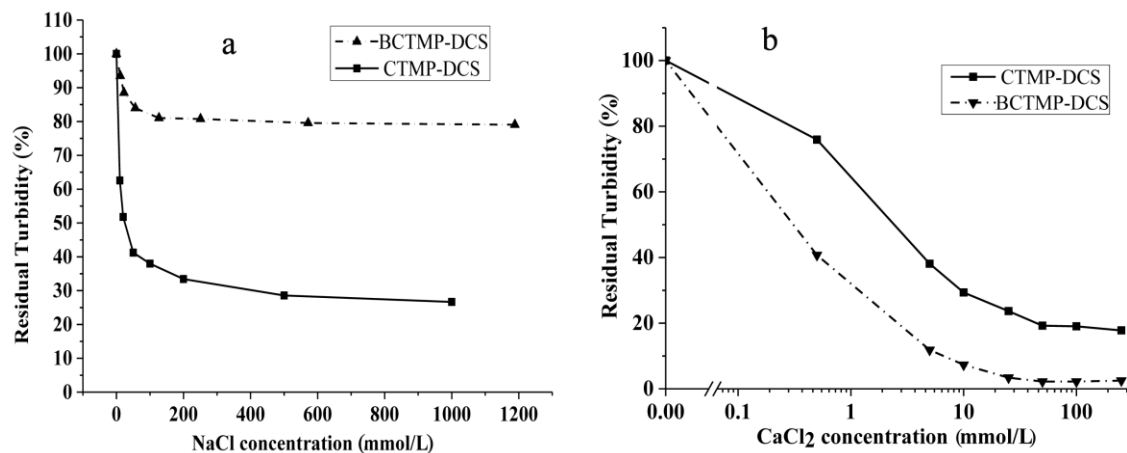
**Table 2.** Concentration of Sugar Units in DCS of Masson Pine Pulps

mg/L	CTMP	BCTMP
Ara	5.66	18.95
Rha	20.99	39.34
Xyl	9.34	40.32
Man	25.40	77.01
Gal	58.50	66.81
GalA	10.60	31.89
Glc	20.82	39.98
GlcA	18.47	39.27
Total	169.78	353.56

Note: Ara, Rha, Xyl, Man, Gal, GalA, Glc, and GlcA stand for arabinose, rhamnose, xylose, mannose, galactose, galacturonic acid, glucose, and glucuronic acid, respectively.

As shown in Fig. 1a, the turbidity of CTMP-DCS rapidly decreased with increasing concentration of sodium ions. The residual turbidity only accounted for approximately 27% of the original turbidity at sodium concentrations above 500 mM, meaning that 73% of the colloids were aggregated and removed during centrifugation. As for BCTMP-DCS, the residual turbidity remained at a level of 80% even at sodium concentrations above 100 mmol/L. This suggests that DCS samples from BCTMP pulps were hypothetically stabilized by polysaccharides (Stack *et al.* 2014), as larger amounts of polysaccharides were released from pulps during bleaching, as shown in Table 2. It is worth noting that pectic substances released during bleaching would not form insoluble flocs with sodium ions. Analysis of the carbohydrate composition of CTMP-DCS before

and after the addition of sodium ions indicated that sodium ions had a negligible effect on the composition and stability of carbohydrates.



**Fig. 1.** Effect of sodium and calcium ions on the stability and carbohydrate composition of DCS of Masson pine pulps

Different behavior was observed for calcium ions compared to sodium ions within the same system. As shown in Fig. 1b, the turbidities of both DCS samples dramatically decreased to a much lower level upon the addition of calcium ions. The residual turbidity of CTMP-DCS reached a plateau of about 20%, which was slightly lower than in the presence of sodium ions and further supported the hypothesis mentioned above. As for BCTMP-DCS, however, almost all of the colloids were aggregated and precipitated with the residual turbidity close to 0% at a calcium ion concentration of 50 mM. This might be explained by the composition of the DCS samples. Unlike CTMP-DCS, BCTMP-DCS contains more pectic substances released during the bleaching process. These pectic substances are prone to binding with calcium ions, forming insoluble flocs that act as flocculants for the colloids in the DCS samples (Garnier *et al.* 1993; Sundberg *et al.* 1996b). The reaction of pectic substances and calcium ions follows an egg-box model, which means that the affinity of some parts of the pectin chain for calcium gives rise to junction zones formation, thus promoting intermolecular associations (Durand *et al.* 1990). Analysis of the carbohydrate composition of BCTMP-DCS before and after the addition of calcium ions indicated that more than 2/3 of pectic substances, such as galacturonic acid (GalA), were removed with colloids.

Based on the results mentioned above, neutral polysaccharides might act as stabilizers for colloids against metal ion aggregation, while acidic polysaccharides are detrimental to the stability of colloids. Analysis of the composition of DCS samples seems to support these hypotheses. In contrast to sodium ions, calcium ions were more harmful to the stability of the process water. To further confirm these hypotheses, model colloid dispersions were used in the following experiments.

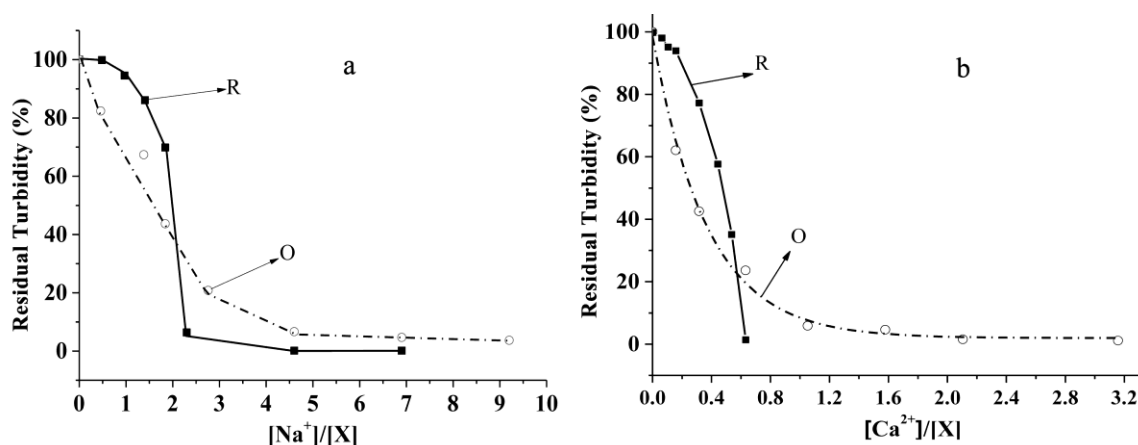
Based on the analysis of DCS samples, one fatty acid (O, oleic acid) and one resin acid (R, rosin acid) were chosen as the model wood resins to study their metal ion-induced aggregation in the absence and presence of polysaccharides.

### Stability of Model Dispersions in the Absence of Polysaccharides

The stability of model dispersions alone, without any polysaccharides, against metal ion-induced aggregation was investigated. The model dispersions were relatively

stable in the absence of calcium, as little aggregation occurred after centrifugation. This indicated that the colloidal resins could stabilize themselves *via* an electrostatic mechanism if no calcium ions were added. The electrostatic force originated from the surface charge of the dissociated fatty and resin acids.

Once the calcium or sodium ions were added, the colloidal resin dispersions aggregated rapidly. As can be seen in Fig. 2a, the resin dispersions completely aggregated when the concentration ratio of sodium ions to R and O reached 2.5 and 4.5, respectively. As for calcium ions, the corresponding concentration ratios, as shown in Fig. 2b, were approximately 0.6 and 1.6, respectively. The added calcium or sodium ions destroyed the double electric layers, resulting in the attraction forces being predominant. Aluminum chloride could also induce the aggregation of colloidal resin dispersions. By comparison of Fig. 2 with Fig. 1, it can be concluded that polysaccharides in DCS had noticeable effects on the stability of the DCS.



**Fig. 2.** Sodium and calcium-induced aggregation of model resin dispersions ( $[Na^+]/[X]$  and  $[Ca^{2+}]/[X]$  represent the weight ratio of sodium and calcium ions to model resins. X stands for R (resin acids) or O (fatty acids), respectively.

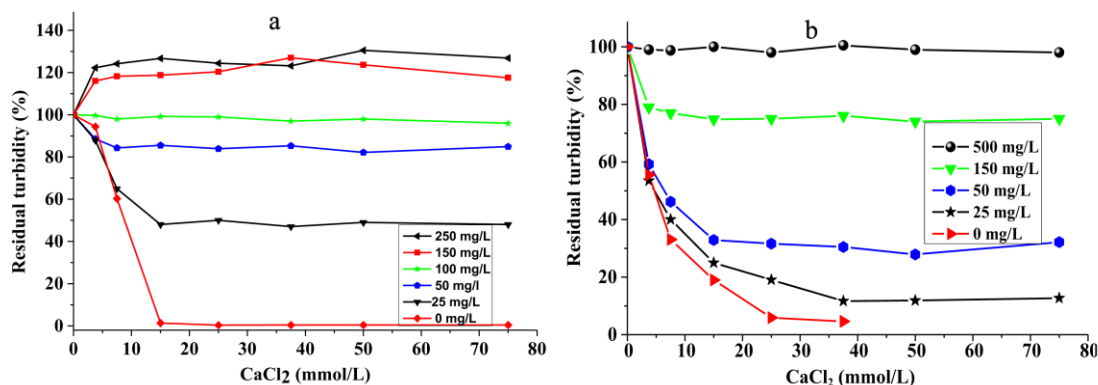
To investigate the effects of various polysaccharides on the stability of the model dispersions, model neutral polysaccharides (GM, guar gum) and acidic pectic polysaccharides (PA, pectic acid sodium) were used, either separately or jointly, in the following experiments.

### Effect of Neutral Polysaccharide on the Stability of Model Dispersions

As observed above, there was a considerable difference between the DCS from unbleached and bleached pulps regarding their stability against metal ion-induced aggregation. Regardless of the amount of calcium added, the DCS of unbleached pulps remained at approximately 40% of residual turbidity, which suggests that steric forces stabilize colloidal resin dispersions. The calcium-induced aggregation of DCS from peroxide-bleached pulps was much more pronounced than that of the unbleached one. Complete aggregation was seen upon the addition of sufficient  $CaCl_2$ .

A non-ionic neutral polysaccharide (GM), whose structure and composition were much like those of the polysaccharides in DCS samples, was selected to investigate the possible steric effect. Figures 3a and 3b show that neutral polysaccharides undoubtedly had a positive effect on the stability of resin dispersions against calcium-induced aggregation. By comparison, the model resin acid R could easily be stabilized. When the

dosage of GM reached 100 mg/L, little aggregation occurred. It seemed that additional more GM led to the formation of larger particles, causing the residual turbidity to be greater than 100%. For model O, the dosage should be up to 500 mg/L. Similar results were seen for sodium-induced aggregation.



**Fig. 3.** Effect of GM on the stability of model R and O dispersions

The experiment results may be explained by the steric stabilization effect (Vercoe *et al.* 2005). The GM used was composed of sugars containing many hydroxyl groups. It was possible that there were enough hydrogen bonding sites for interactions between both the colloid and the surrounding water molecules. Greater amounts of hydrogen bonding sites between colloids and GM lead to a stronger interaction with the water than if the colloid was interacting with the water itself, *i.e.*, the GM-wrapped colloids had a greater affinity for water. The surfaces of the colloidal particles became non-ionic and had no affinity for calcium ions.

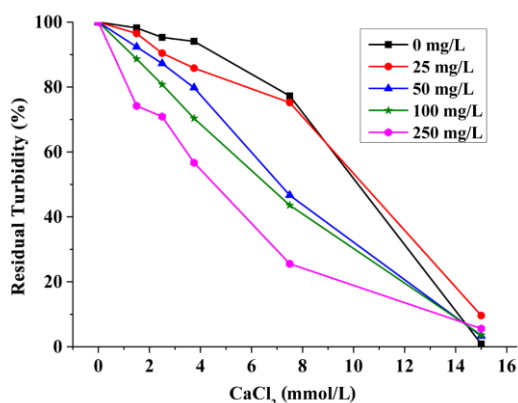
### Effect of Acidic Polysaccharide on the Stability of Model Dispersions

As shown in Fig. 1b, calcium chloride could completely aggregate the DCS from peroxide-bleached pulps. This phenomenon has been explained in light of interactions between calcium ions and polygalacturonic acids (Sundberg *et al.* 1994; Dai *et al.* 2011). To verify this, the effect of acidic polysaccharide (PA, in sodium salt form) alone on the stability of model resin R was investigated.

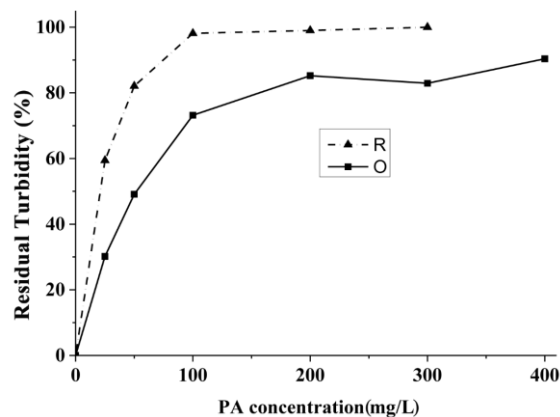
The procedure for studying the effect of acidic polysaccharide on the stability of model resin dispersion R was the same as for that of neutral polysaccharide GM, except PA was substituted for GM. As can be seen in Fig. 4, in the presence of calcium ions, an acidic polysaccharide could weaken the stability of colloidal model dispersions. On the one hand, the acidic polysaccharides could interact with the cationic polymers and might be considered potential “anionic trash.” On the other hand, these acidic polysaccharides could interact with calcium by a so-called “egg-box” model mechanism. Upon the addition of calcium chloride, polygalacturonic acid and colloidal resin dispersions co-aggregated. That was why the calcium-induced aggregation of DCS from peroxide-bleached pulps was much more pronounced than that of the unbleached pulps.

However, the addition of the acidic polysaccharide had a positive effect on the stability of colloidal model dispersions when sodium was the only metal ion present in the system, as shown in Fig. 5. The acidic polysaccharide did not form insoluble flocs with sodium ions and seemed to act as a dispersant. This is why the residual turbidity of

BCTMP-DCS remained at 80% at sodium concentrations of above 100 mmol/L, as shown in Fig. 1a.

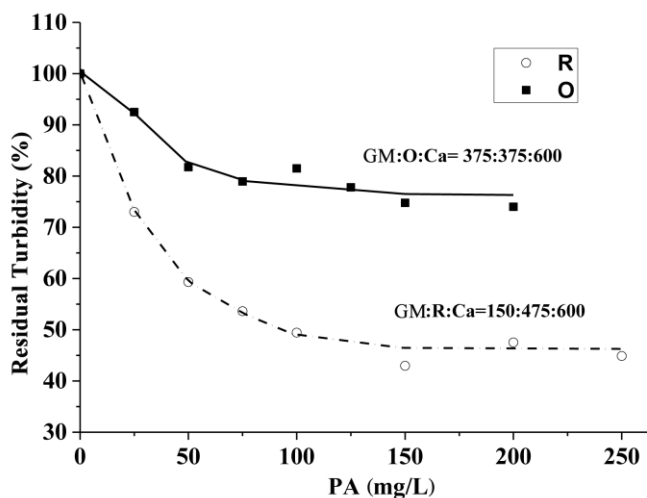


**Fig. 4.** Effect of PA on the stability of model R dispersion against calcium-induced aggregation



**Fig. 5.** Effect of PA on the stability of model dispersions against sodium-induced aggregation

To study the effect of both GM and PA on the stability of model dispersions, the concentration of GM and dispersions were kept constant, with the ratio of GM to R/O being large enough to stabilize the colloidal resins. Figure 6 shows that when the neutral and acidic polysaccharides coexisted, the sterically stabilized resin acid R and fatty acid O retained a certain level of stability, which indicated the limited adverse effect of acidic polysaccharides. This might give us a clue to stabilizing the colloidal resins by improving the proportion of neutral polysaccharides in DCS and greatly reducing the content of acidic ones.



**Fig. 6.** Effect of PA and GM on the stability of model dispersions

## CONCLUSIONS

1. The major lipophilic substances (wood resins) of DCS samples were palmitic acid (32%), dehydroabietic acid (19%), oleic acid (7%), hexadecenoic acid (7%),



tetradecanoic acid (7%), and linoleic acid (6%). The ratio of fatty acids to resin acids was approximately 3:1 before bleaching. After bleaching, the ratio became 2:3, with the amount of dehydroabietic acid having increased markedly.

2. A considerable difference between DCS from unbleached and bleached pulps regarding their stability against metal ion-induced aggregation was observed. It appeared that neutral polysaccharides might act as stabilizers for colloids against metal ion aggregation, while acidic polysaccharides were detrimental to the stability of the colloids.
3. Model compound experiments showed that model resin dispersions could stabilize themselves *via* an electrostatic mechanism without any aggregation. However, once metal ions were added, regardless of the presence or absence of sodium or calcium ions, the dispersions aggregated rapidly. Polysaccharides that were contained in the DCS had noticeable effects on the stability of the system.
4. No calcium-induced aggregation occurred if the concentration ratio of added neutral polysaccharide to R and O dispersions exceeded 0.2 (w/w) and 1.0 (w/w), respectively. Acidic polysaccharides could further worsen the stability of resin dispersions in the presence of calcium ions yet have a positive effect in the presence of only sodium ions. Adjusting the proportion of neutral polysaccharides in DCS and greatly reducing the content of acidic ones could be a promising way to stabilize the colloidal resins.

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