

Influence of mechanical pulp fines on clay retention

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ABSTRACT: We investigated deposition of clay filler on fibrillar and flake-like fines and long fibers of mechanical pulp to study the effects of the fines on filler retention. The role of different retention mechanisms was illustrated by comparing clay retention in deposition and filtration experiments. The amount of clay deposited varied significantly between the pulp fractions in the presence of polymeric flocculants. Cationic polyacrylamide (CPAM) promoted filler deposition on fines, especially flake-like fines, more than on long fibers. Presumably, this was due to the high surface area and surface charge density of fines, which increase their bonding strength with polymer chains. Polyethylene oxide (PEO) slightly increased the deposition of clay on fines but started to disperse the clay, and especially the fines, as the polymer dose increased. PEO releases small particles from the fines that exist as bundles or entanglements in the suspension. When the deposition-based retention mechanism and the filtration-based mechanism were compared, mechanical attachment of clay in the filter cake had a significant effect on its retention. Also, long fibers are better able to form a filter layer on the screen than fines, which in turn adsorb more filler through chemical interactions.

Application: In mechanical pulps, kaolin clay filler retention might be influenced by fibrillar and flake-like fines fractions.

INTRODUCTION

In papermaking, the retention of filler particles is significant in filtration-based separation operations. Both the sheet forming process in the paper machine and separation processes such as solids recovery from the circulation water involve the attachment of small particles, including mineral fillers, to the fiber network during filtration. Filler retention by mechanical entrapment is poor, so the retention is closely associated with the ability of different pulp fractions to adsorb fillers. The influence of organic pulp fines fractions on filler retention is of interest because the fines contribute significantly to particle adsorption in the stock due to their high external surface area. In turn, they are retained better than individual filler particles in the fiber network.

The fines in mechanical pulps contain various types of particles with various physical and chemical properties. Fines are divided morphologically into flake-like fines, containing pieces from the middle lamella, primary walls and ray cells; and fibrillar fines, containing thread-like particles from the secondary walls. Flakes originating from the outer part of the cell wall contain more lignin and less cellulose than fibrils that originate mainly from the secondary walls [1]. This difference in the surface composition of fines fractions affects their ability to adsorb other components. For example, flakes tend to adsorb more colloidal resin in the presence of PEO than do fibrils [2], whereas more carbohydrates are adsorbed onto fibrils than onto flakes [3].

The interaction of fines and other pulp fractions with the polymeric flocculant is crucial for efficient filler retention because flocculants are needed to promote the attraction be-

tween anionic fillers and negatively charged pulp fractions. High molecular weight cationic polyelectrolytes and neutral polymers, such as cationic polyacrylamide (CPAM) or polyethyleneoxide (PEO), are common flocculants used to create links between particles by mechanisms based on bridging. The polymers' ability to attach filler particles to pulp depends on the physical and chemical properties of the pulp components, such as accessible surface area, porosity, chemical composition of the surface, and its charge density [4]. Earlier investigations studied the deposition of fillers on only one pulp fraction, the long fiber fraction [5-9]. The properties of fines and of long fibers differ significantly so the effect of the various fines fractions on filler retention is still unclear.

Our aim was to clarify the influence of various types of mechanical pulp fines on kaolin clay filler retention. We measured the amount of clay deposited on the fibrillar and flake-like fines and long fibers of refiner mechanical pulp (RMP) in the absence and presence of polymeric flocculants (CPAM and PEO). We also clarified the roles of the different retention mechanisms in filler retention by comparing filler retention in deposition experiments with that in the filtration process.

MATERIALS

Separation and characterization of fines and fibers

Refiner mechanical pulp made from Norway spruce (*Picea abies*) was taken from a Finnish pulp mill after the first and second refining stages; consistency was approximately 40% and fractionated into the long fiber fraction and two fines fractions. The net specific energy consumption (SEC) for

Sample	Content of particles (% w/w)			Surface chemical composition (%)			Charge properties (μeq/g)	
	Flakes	Fibrils	Ray-cells	Lignin	Extractives	Carbohydrates	Total charge	Surface charge
Flake-like fines	73	24	3	53	23	24	164.9	47.1
Fibrillar fines	65	33	2	41	11	48	168.1	50.7
Long fibers	-	-	-	29	11	60	85.5	12.6

1. Properties of the pulp fractions used in the experiments.

these RMPs was 1.0 MWh/t in the first stage and 2.2 MWh/t in the second stage. The flake-like fines fraction was separated from the first-stage sample and the fibrillar fines fraction from the second-stage sample after washing and additional refining (575 kWh/t SEC). The long fiber fraction remained after the fibrillar fines were separated out. A vibro separator (Sweco 48 in.) equipped with a 75-μm opening (230 mesh) stainless steel wire mesh, corresponding to the standard 200 wire mesh on a Bauer McNett classifier, was used to wash and separate the fines. The fines suspensions were allowed to settle for 3 days. The supernatant was drained off and the fines were collected and centrifuged (Jouan GR422 centrifuge) for 5 min at 2000 G, after which the supernatant was again poured off. The resulting samples were frozen at -18°C. All the pulp fractions were treated with acid (0.01 M HCl) before the experiments in order to remove metal ions. They were then converted to sodium form by treatment with NaHCO₃ (0.001 M) at pH 9.5 (pH adjusted with 0.1 M NaOH solution). The samples were washed with deionized water until their conductivity was less than 5 μS/cm. Filter papers used in this washing were Schleicher & Schuell nr. 604 and Macherey Nagel nr. 617.

The particles in the fines fractions were analyzed with an image analyzer that classified them into fibrillar and non-fibrillar particles and ray cells and measured their apparent mass proportion [10]. We used Electron Spectroscopy for Chemical Analysis, ESCA (AXIS 165 spectrometer by Kratos Analytical, Chestnut Ridge, New York, USA) to measure the surface content of lignin, extractives, and carbohydrates in the pulp fractions [11]. The total and surface charges of the fractions were measured by polyelectrolyte titration [12] using poly(1,5-dimethyl-1,5-diazaundecamethylene) bromide (Polybrene, Sigma-Aldrich Chemie GmbH Buchs SB, Switzerland) for the total charge and poly(dimethyldiallylammonium) chloride (PolyDADMAC, Ciba Specialty Chemicals Ltd., Basel, Switzerland) for the surface charge. **Table I** displays the properties of the pulp fractions.

Clay filler

Kaolin clay from J. M. Huber Finland (Hamina, Finland) was used as mineral filler. The clay was supplied as a dry powder from which a water slurry with a solids content of 10% was prepared with deionized water without using dispersants. The particle size as obtained from a Sedigraph (D₅₀ value) and the surface area of the filler particles (BET) as given by the manufacturer, were 1.4 μm and 12 m²/g, respectively. The zeta potential and electrophoretic mobility of the clay, mea-

sured with a Coulter Delsa 440 Doppler Electrophoretic Light Scatter Analyzer, were -29.0 mV and -2.19 μm²/Vcm², respectively.

Polymers

PEO with a molecular weight of 10⁷ g/mol and CPAM with a molecular weight of 7×10⁶ g/mol and charge density of 1 meq/g were obtained from Kemira (Helsinki, Finland). PEO was used without any cofactor. The stock solutions of polymers, 0.5 g/dm³ for PEO and 5 g/dm³ for CPAM, were prepared by mixing polymer granules into deionized water and stirring at 200 rpm for 45 min for PEO and 90 min for CPAM. The stock solution of PEO was prepared daily because a long storage time would have altered its flocculation efficiency [13]. The stock solutions were diluted to 50 mg/dm³ before use.

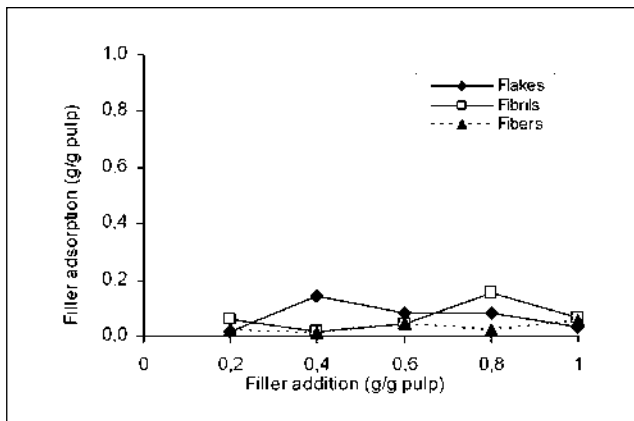
EXPERIMENTAL

In the first phase we measured the adsorption of clay particles onto fibrillar fines, flake-like fines, and long fibers to investigate the deposition-based retention process. In the second phase we examined the retention of clay in the filtration-based process.

Adsorption experiments

To investigate the adsorption of clay particles onto the pulp fraction, we analyzed the concentration of free filler particles in a stirred beaker (500 rpm) by measuring turbidity with a Hach Ratio/XR Turbidimeter. First we measured the deposition of clay on the pulp fractions as a function of clay dosage in the absence of flocculants, and then added a flocculant (CPAM or PEO) to the suspensions. The deposition of clay was obtained as a function of the flocculant dosage.

The experiments consisted of adding a suspension of 3–15 mg of clay to 15 mg of fibers or fines suspended in deionised water containing 10 mM of NaCl and 1 mM of NaHCO₃ as background electrolytes, followed by the addition of the polymer. The total volume of each suspension was 150 cm³. Free filler particles were separated from the suspension 120 seconds after the beginning of the experiment, as an equilibrium in filler adsorption was reached. A portion of the suspension was filtered while stirring through a screen that retained the wood material and adsorbed fillers, while the free fillers passed through. The smallest, colloidal size fraction of the fines, which might have passed through the screen, had been removed from the pulp during pre-treatment. In addition, the large clay aggregates were screened from the filler suspension. We used a calibration line for turbidity versus filler con-



1. Adsorption of clay on pulp fractions in the absence of flocculants (background electrolytes: 10 mM NaCl and 1 mM NaHCO₃).

centration (correlation constant $R^2 = 0.999$) to determine the amount of filler in the filtrate [14].

Filtration experiments

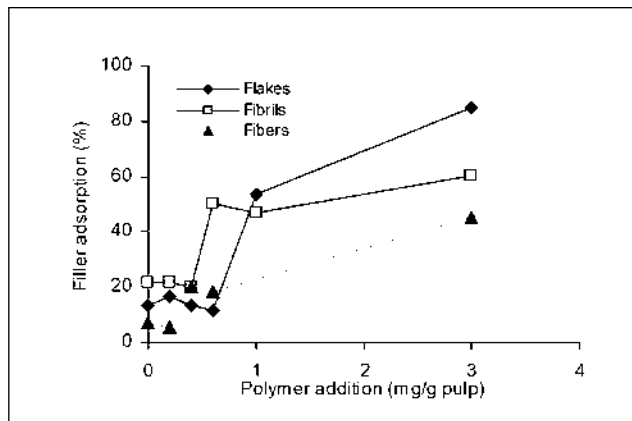
The filtration experiments were carried out by filtering the stirred (200 rpm) suspensions containing pulp fractions and clay particles through a wire mesh (50 μ m, 270 mesh) under a constant pressure difference (10 kPa) at room temperature. We chose a wire with a high mesh to promote filter cake formation from the fines. The experiments were conducted by adding 0.2–1.0 g of filler to 1.0 g of fibers or fines suspended in deionised water containing 10 mM of NaCl and 1 mM of NaHCO₃ as background electrolytes, followed by addition of the polymer and filtration of the suspension after 120 seconds of mixing. The total volume of suspension was 1000 cm³. The experimental setup for the filtration apparatus was similar to that described previously [15].

We monitored the retention of clay during filtration by measuring the turbidity and ash content of the filtrates after obtaining a constant cumulative amount of filtrate (250 ml).

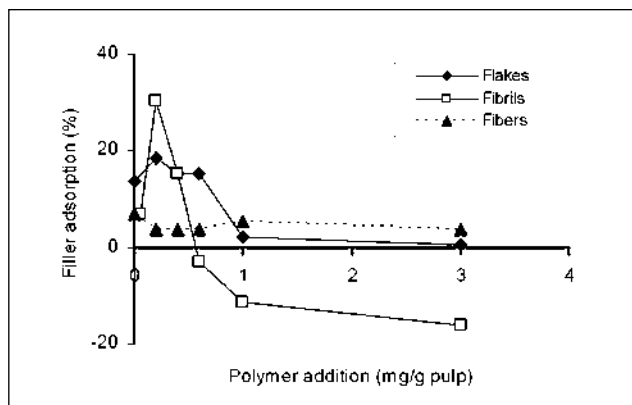
RESULTS

Properties of pulp fractions

Table I shows the particle properties measured for the long fibers and two fines fractions (fibrillar and flake-like fines). The fibril contents of the fines fractions, based on image analysis, were 33% for the fibrillar fraction and 24% for the flake-like fraction. The ESCA analysis of the surface chemical composition of the fractions showed that the flakes had a higher surface lignin and extractive content but lower surface carbohydrate content than did the fibrils, whereas the fines had lower surface carbohydrate content and higher surface lignin content than the long fiber fraction. Long fiber fraction had same surface extractive content as fibrillar fines. Both the total and surface charges per gram of pulp were higher for the fines than for the long fibers. Earlier results of Sundberg and Holmbom [3] support this finding, although there was a small difference in the charges of the two fines fractions.



2. Adsorption of clay on pulp fractions as a function of CPAM dosage (filler dosage 0.7 g/g pulp).

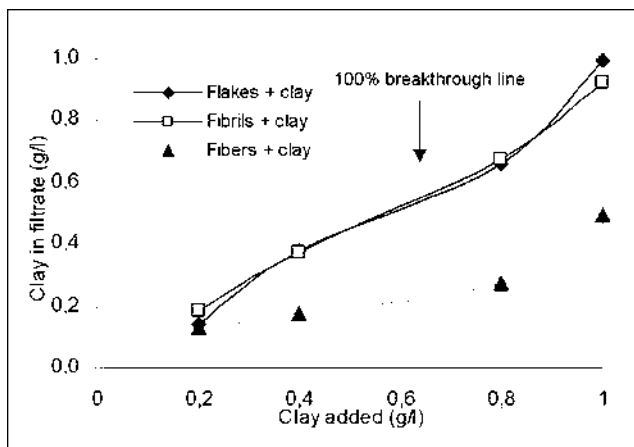


3. Adsorption of clay on pulp fractions as a function of PEO dosage (filler dosage 0.7 g/g pulp).

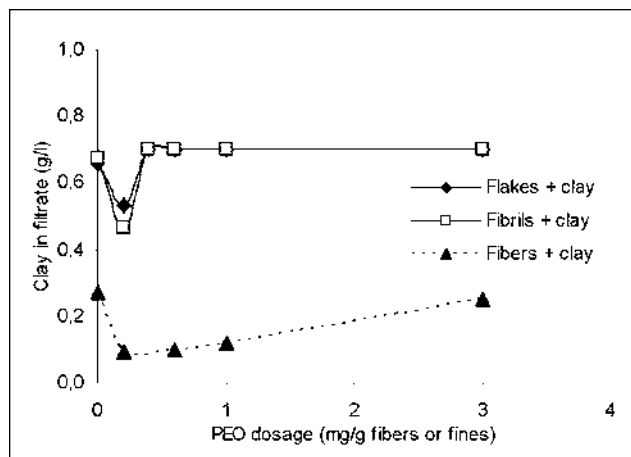
Adsorption experiments

Figure 1 shows the adsorption of clay onto the RMP pulp fractions in the absence of flocculants. As expected the clay, which is mainly negatively charged, showed only slight adsorption onto the anionic pulp fractions in the absence of flocculants, despite the background electrolytes that partly screened the electrostatic repulsion between like charges. The amount of adsorbed filler as a function of filler dosage was almost constant for all the fractions. Also, the average amount of filler adsorbed was 30 mg/g pulp for the long fibers and 70 mg/g pulp for the fines fractions.

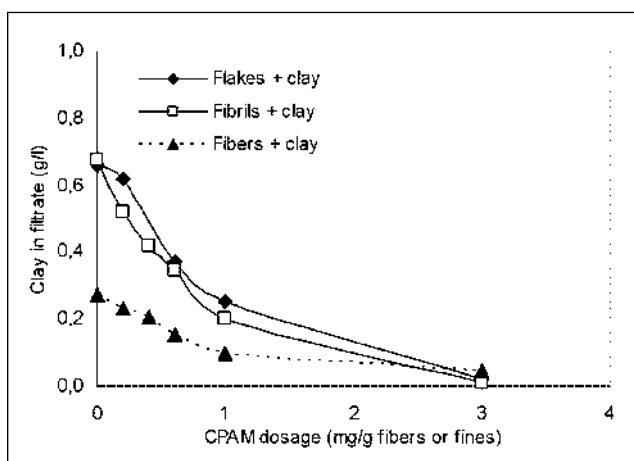
The adsorption of clay (dosage 0.7 g/g pulp) onto the pulp fractions in the presence of flocculants (CPAM and PEO) as a function of flocculant dosage is shown in **Fig. 2** and **Fig. 3**. These flocculation agents promote the deposition of clay particles on the pulp fractions and enhance flocculation among the filler particles. Both of these mechanisms increase filler retention and both could contribute to the results we obtained. Assuming that flocculation among the clay particles is the preferential retention mechanism, retention would be independent of the pulp properties. However, the results for the suspensions containing CPAM demonstrated that both fines fractions retained more filler than did the long fibers at



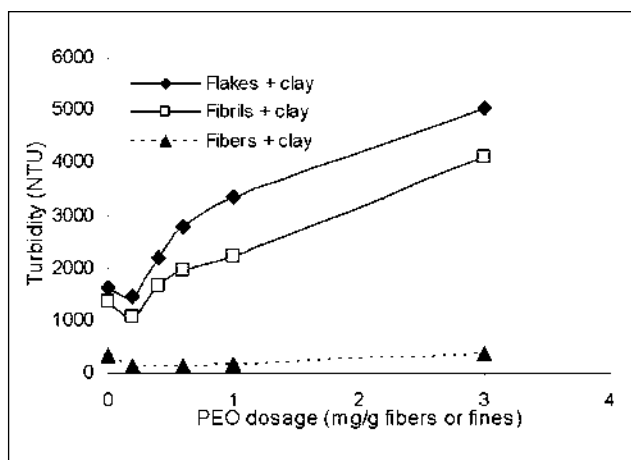
4. Retention of clay in filtration in the absence of flocculants.



6. Retention of clay in filtration as a function of PEO dosage.



5. Retention of clay in filtration as a function of CPAM dosage.



7. Turbidity of filtrates as a function of PEO dosage.

polymer doses higher than 1.0 mg/g pulp. The amount of filler adsorbed onto flakes at high CPAM dosages was nearly twice that observed in the case of the long fibers.

Contrary to CPAM, PEO slightly increased filler deposition on the fines at low dosage levels but had no effect on filler retention in the suspensions containing long fibers. In addition, PEO started to disperse the suspensions containing fines and clay at higher dosages, which caused a decrease in the adsorption of clay. Since the analysis of the amount of filler adsorbed was based on the separation of free filler from the pulp suspension and measurement of the turbidity of the filler solution, the negative amount of adsorption noted with fibrils implies that the amount of particles in the size range of the filler particles (i.e., able to pass through the screen in the separation process) was actually higher after the addition of PEO than at the beginning of the experiment.

Filtration experiments

The vacuum filtration experiments illustrated the retention of clay in filtration. Unlike in the deposition experiments, filler retention was based on two phenomena: (1) the chemical interactions of the fillers with the particles in suspension

and (2) mechanical attachment of the filler to the filter cake, the formation of which was promoted by the use of low stirring speed and the high screen mesh number. **Figure 4** shows the amount of clay in the filtrates in the absence of flocculants. Despite the conditions, there was poor retention of fines on the screen, which led to the formation of a thin filter cake and 90%–95% permeability of the clay. The long fibers formed a thicker filter cake, however, which retained filler particles efficiently. Compared with the results of the deposition experiments (Fig. 1), mechanical attachment in filtration significantly increased filler retention in the suspensions containing long fibers, leading to 35%–50% retention.

Figure 5 shows the amount of clay in the filtrates in the presence of CPAM, which increased filler retention in all the pulp suspensions. Retention was higher in the suspension containing long fibers than in the fines suspensions at low CPAM dosages. Clay retention in filtration was directly related to pulp retention and the structure of the filter cake, so the results also show how CPAM affected flocculation of the pulp fractions.

PEO had a similar effect on clay retention in the fines sus-

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pensions as in the deposition experiments. Clay retention increased at low dosages but started to disperse suspensions at higher dosages (**Fig. 6**). PEO also increased clay retention in the suspensions containing long fibers. **Figure 7** shows the turbidities of the filtrates in the PEO experiments. Turbidity correlates with the total solid content of the filtrate, i.e., it is affected by both filler content and pulp content. As the permeability of the filler in fines suspension was nearly 100% in the absence of a flocculant, the increase in turbidity from that level was associated with an increased fines content in the filtrate; thus PEO effectively dispersed the pulp fines.

DISCUSSION

The results showed that clay retention in the presence of polymeric flocculants was significantly affected by the properties of the pulp fractions. This finding supports the earlier results published by Cadotte et al. [16] that interaction between the filler and pulp material (filler deposition) contributes more to filler retention than does flocculation among the filler particles. This may depend on both the physical and chemical properties of the particles. The larger particle size and concentration of the pulp material than that of the fillers promote collisions of clay with the pulp, as stated in collision theory, whereas the higher relative difference in size between the pulp material and fillers than between two filler particles reduces the probability of filler deposition due to hydrodynamic restrictions. It has also been suggested [17] that the lower negative surface charge on the fibers, σ_f , than on the clay, σ_c , may favor clay deposition, since electrostatic repulsion is lower between pulp and clay than that between clay particles, i.e., $\sigma_f \sigma_c < \sigma_c^2$.

Deposition of clay filler on fines with no flocculants

Asselman and Garnier [18] and Middleton and Scallan [19] reported that the retention of anionic fillers such as clay is low in the absence of flocculants. The measured amount of clay adsorption in fines suspensions corresponded to only about 2% of the maximum surface coverage of clay [20]. Since the measured critical coagulation concentration of clay (CCC) is 1.8 mM of CaCl_2 [21], and the critical deposition concentration is presumably only slightly below this value, electrostatic repulsion is expected to be the main reason for the low filler retention under the electrolyte concentrations used here (10 mM of NaCl and 1 mM of NaHCO_3). According to the Schulze-Hardy rule, the corresponding CCC for the monovalent ions is over 100 mM, so that any attractive interactions that might exist (e.g., hydrogen bonding between silanol or aluminol groups in the clay and hydroxyl groups in the cellulose) would be too weak to promote flocculation among the clay particles or attach clay to the pulp under the present shear conditions.

Deposition of clay filler on fines with CPAM

The fact that significantly more clay was adsorbed onto the

fines fractions in the presence of CPAM than onto the long fibers is not surprising, since the external surface area of fines is significantly higher. The external surface area of TMP long fibers has been reported to range from 1.38–2.63 m^2/g depending on the freeness level [22], whereas the average surface area of TMP fines is 12 m^2/g [23]. Among the fines fractions, however, the flakes adsorbed more clay than did the fibrils, despite having a smaller surface area. The difference in surface charges might explain this, because the driving force for adsorption and flocculation by CPAM is charge interaction (electrosorption) between polymer segments and the surface charges. Even though the charges per gram of pulp were almost equal for both fines fractions, the surface charge relative to the surface area of the pulp was higher for the flakes. Based on the specific surface areas proposed by Mosbye et al. [24], 7.2 m^2/g for flakes and 13.5 m^2/g for fibrils, the surface charge densities were calculated to be 6.5 $\mu\text{eq}/\text{m}^2$ and 3.8 $\mu\text{eq}/\text{m}^2$, respectively. Due to the high charge density, clay particles were strongly attached to the flakes because there were more anchoring sites available for the polymer segments than on the surfaces of the fibrils. The high surface charge might also alter the polymer to a flatter conformation, although this did not reduce the clay retention, since the thickness of polymer layer, δ , was greater than the double layer thickness of the charged surfaces, κ^{-1} [21].

Deposition of clay filler on fines with PEO

PEO promoted the adsorption of clay onto the fines fractions slightly but had no effect on filler retention in the suspensions containing long fibers. The reason for this might be its low affinity for fibers or the highly abrasive shear forces caused by the fibers [16]. The low deposition efficiency of PEO in the absence of any cofactor or adjuvant supports the earlier findings published by Alince and van de Ven [25] and Wu et al. [26], although the reason for the significantly lower deposition efficiency of PEO than of CPAM is not completely clear.

The main factor affecting the adsorption energy of polyelectrolyte (CPAM), and thus its ability to flocculate particles, is the entropy gain, ΔS_{ads} , arising from the displacement of counter-ions from the particle surfaces [27]. For neutral polymers such as PEO, on the other hand, the entropy gain due to release of the solvent molecules from the solvation sphere of the polymer, ΔS_{ads} , is presumably small. Consequently, the strength of non-electrostatic interactions (enthalpy of adsorption, ΔH_{ads}) between the polymer segments and the surface should be relatively high, since the Gibbs energy of adsorption, ΔG_{ads} , entails contributions from both the enthalpy of adsorption and the entropy of adsorption, $\Delta G_{\text{ads}} = \Delta H_{\text{ads}} - T\Delta S_{\text{ads}}$. Since PEO contains ether oxygens, which could in principle interact with OH groups on the pulp surface, an interaction is assumed to exist between PEO and pulp. The affinity of PEO for components of the pulp surface is known to be low, however, except in the case of phenolic groups, which are found mainly in the lignin [28]. Therefore, in order

to achieve good flocculation by PEO, a cofactor that adapts the PEO into a good absorbing polymer is needed (i.e., ΔH_{ads} is more negative such a cofactor is used). This cofactor might also affect the entropy of adsorption, ΔS_{ads} , by modifying the stiffness of the PEO [29].

The results showed that PEO started to disperse not only the clay but more especially the fines at very low dosages (<1.0 mg/g of fines) that corresponded to a surface coverage of about 10% of the external surface area of the fines [26], too low an amount for dispersing the individual fines by steric stabilization. Wu et al. [26] also observed that the maximum polymer adsorption of a PEO/cofactor complex onto fines is very low, suggested that the fines exist as entanglements or bundles in the suspension, and that the accessible surface area is thereby lower than the external surface area of the fines because the polymer can be adsorbed only to the surfaces of these entanglements. Our findings support this assumption, and we propose that the small particles that are caught in the entanglements are released by the dispersing activity of PEO. In particular, the long and flexible fibrils that have been reported to flocculate in the absence of flocculants [30] must have released small particles from the entanglements.

Role of filtration in clay retention

The results presented by Solberg and Wågberg [8] showed that filtration or sieving has only a minor effect on the retention of ground calcium carbonate (GCC) filler, and they proposed that filler deposition is the predominant mechanism for GCC retention. By contrast, Davison [31] concluded that filtration is the primary mechanism of fine particle retention, because the filter cake can entrap flocs that are formed from fine particles. Our results also indicate that clay retention is significantly affected by filtration. These contradictory findings suggest that the mechanism of filler retention is highly dependent on the properties of the suspension components and their interactions and on details of the experimental conditions such as stirring intensity. In our experiments the plate-like shape of the clay and the low stirring rate may have promoted filler retention during filtration.

Even though the deposition experiments showed that the fines adsorbed more filler than the long fibers in the presence of CPAM, the retention of clay was lower in the fines suspensions because the fines were poorly attached to the screen. Wu et al. [26] suggested the size of fines flocs seemed to increase as a function of polymer dosage, leading to higher fines retention and increased the filler retention, because clay was adsorbed onto the fines.

The results indicated that long fibers and fines fractions affect filler retention in different ways. Long fibers are better able to form a filter layer on the screen than fines, which in turn adsorb more filler through chemical interactions. Fines also have an important effect on filtration because they reduce the pore size of the filter cake.

CONCLUSIONS

Fines adsorb more clay than long fibers in the presence CPAM due to the higher external surface area, while the flake-like fines retains more clay than the fibrillar fines, due to the higher surface charge density of the flakes, which increases the bonding strength between the clay and the pulp surface. PEO increases clay deposition on fines at low dosages but starts to disperse the clay, and more especially the fines, at higher dosages. The results suggest that fines exist in the suspension as entanglements from which smaller particles are released by the dispersive activity of PEO. The results also suggest different filler adsorption mechanisms for fines and long fibers. The latter have a better ability to form a filter layer on the screen than do fines, whereas fines can adsorb more filler through chemical interactions. **TJ**

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INSIGHTS FROM THE AUTHORS

The retention of filler particles is closely associated with the ability of different pulp fractions to adsorb fillers, because filler retention by mechanical entrapment is poor. Earlier investigations were mainly restricted to studying the deposition of fillers on only one pulp fraction, the long fiber fraction. Since the properties of fines and long fibers differ significantly, the effect of the various fines fractions on filler retention is still unclear.

Our aim was to clarify the influence of mechanical pulp fines of various types on kaolin clay filler retention. We also illustrated the role of different retention mechanisms by comparing clay retention in deposition and filtration experiments. The amount of clay deposited varied significantly between the pulp fractions in the presence of polymeric flocculants. Cationic polyacrylamide (CPAM) promoted filler deposition on fines, especially flake-like fines, more than on long fibers, presumably due to the high surface area and surface charge density of fines, which increases their bonding strength with polymer chains. Polyethylene oxide (PEO) slightly increased the deposition of clay on fines but started to disperse the clay, and especially the fines, as the polymer dose increased.



Niinimäki



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Haapala

Our results suggest that fines exist in the suspension as entanglements from which smaller particles are released by the dispersive activity of PEO. When the deposition-based retention mechanism and the filtration-based mechanism were compared, mechanical attachment of clay in the filter cake had a significant effect on its retention. Long fibers are better able to form a filter layer on the screen than fines, which in turn adsorb more filler through chemical interactions.

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