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CONFORMATION OF ADSORBED POLYMERS AND FLOCCULATION OF MICROCRYSTALLINE CELLULOSE AND PULP SUSPENSIONS

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ABSTRACT

This paper consists of three parts. In the first part, the reconformation of polyelectrolytes adsorbed on fiber surfaces has been studied by measuring simultaneously the amount of adsorbed polymer and the number of released counterions. It was found that the time for reconformation from an initial, extended conformation to a more flat conformation was of the order of 60 seconds for a high molecular mass polyacrylamide. In the second and third parts, the flocculation of suspensions of microcrystalline cellulose and pulp fibers has been investigated. The results of these flocculation experiments can be related to the conformation and adsorbed amount of the polyelectrolyte.

INTRODUCTION

In this paper, an attempt is made to relate the flocculating efficiency of adsorbed polyelectrolytes to their conformation on the particle surface. The paper consists of three parts. In the first part, results from experiments designed to measure the conformation of adsorbed polymers as a function of time after adsorption are discussed. In the second and third parts, flocculation by polyelectrolytes of suspensions of microcrystalline cellulose (MCC) and cellulosic fibers respectively are reported. The conformation of the adsorbed polyelectrolytes is the basis for the discussion of these results.

CONFORMATION OF ADSORBED POLYELECTROLYTES

General remarks

Polymers, mainly cationic, have been used for many years as retention aids in paper manufacture to promote the retention in the paper of fine cellulosic material and added fillers. To be effective, these polymers have to be adsorbed on fibers, fines and filler. In order to optimize and understand the adsorption process, extensive research has been undertaken. In these studies both cellulosic fibers (<u>1-11</u>) and microcrystalline celluose, MCC (<u>12-14</u>) have been used as adsorbent.

The adsorption of cationic polymers on cellulosic fibers is in most cases achieved by an electrostatic attraction between anionic charges on the fibers and cationic charges on the polyelectrolyte. This dependence on charge interaction raises the question of whether or not the adsorption of cationic polymers may be considered to be an ion-exchange process. Results presented by Bates ($\underline{7}$) and Wågberg et.al. ($\underline{15,16}$) where the adsorption of both highly charged, low molecular mass polymers and low charged, high molecular mass polymers were studied, show that the adsorption may indeed be considered to be an ion-exchange reaction.

Despite the fact that the wet end of a paper machine is a very dynamic system, only a limited amount of work has been devoted to an increased understanding of the adsorption process during short contact times ($\langle 30 \ s \rangle$) (17-20). Together with

other studies on kinetics of adsorption $(\underline{1},\underline{10},\underline{13})$ these studies, with the exception of $(\underline{17})$, show that the initial adsorption is a fairly rapid process whereas a time of the order of hours is needed to reach adsorption equilibrium. It is hence also very important to clarify the kinectics of adsorption in order to understand the flocculating action of cationic polyelectrolytes. Due to kinetic effects, polymers may be effective by different flocculation mechanisms at different stages after polymer addition.

Experimental approach

In order to investigate the kinetics of adsorption, a high molecular mass, cationic, medium charged, polyacrylamide (MAPTAC) and a low molecular mass, highly charged 3.6.-ionene, were studied with a series of carboxymethylated, bleached pulps as substrates. Chemical structures of the polymers are given in Fig. 1. The cationic polyacrylamide was a copolymer between acrylamide and (N,N,N)-trimethylaminopropylmethacryl-amide with a charge density of $2.6 \cdot 10^{-3}$ eq/g and a relative molecular mass of $4 \cdot 10^{6}$. The charge density of the 3.6.-ionene was calculated from the chemical structure to be $5.35 \cdot 10^{-3}$ eq/g and the molecular mass of this polymer was $5.9 \cdot 10^{3}$.

The degree of polymer adsorption was determined by polyelectrolyte titration ($\underline{21}$). From the extrapolated plateau levels in the adsorption isotherms in Fig. 2, and from similar isotherms for the 3.6-ionene, a suitable level of polymer addition for the kinetic experiments was chosen. To evaluate the average adsorption conformation of the adsorbed polymers, the charge stoichiometry was determined. This entity is defined as the ratio (in mole %) of the number of counterions (to the polymer) released upon adsorption to the total number of adsorbed polymer charges. A high value indicates a rather flat conformation on the surface whereas a low value indicates an extended conformation with a large number of loops and tails protruding out from the surface. For full experimental details the reader is referred to ($\underline{19}$).



Fig. 1 Chemical structures of (a) the cationic polyacrylamide and (b) the 3.6.-ionene.



Fig. 2 Adsorption in mg/g of the MAPTAC on different carboxymethylated pulps.(From ref.19 with permission of Academic Press)

Results and discussion

Results of experiments with MAPTAC-polymers are given in Figs. 3 and 4 where the adsorption and the adsorption stoichiometry are shown as functions of time after addition of polymer. The results in Fig. 3 show that the adsorption is very rapid and that only a small increase is detected with increasing time after addition. In contrast, the adsorption stoichiometry shows large changes, with a low initial stoichiometry and a fairly high value of 80 % after 30 min. These results indicate that the polymer is initially adsorbed in a conformation extending out from the surface with only a few segments attached to the surface and that with increasing time after addition the polymer adopts a more flat conformation. It should perhaps be emphasized that most of the reconformation is completed within 60 seconds.

The results in Fig. 4, where the level of addition has been doubled, show that an increased competition for the surface charges between the polymer molecules renders the molecules a more extended conformation with an initial adsorption stoichiometry of only 6 %. The stoichiometry after 30 min. has also decreased from 80 to 70 %. This decrease is most probably due to the increased interaction between the macromolecules on the surface, although the decrease in coil dimensions in solution at higher polymer concentration might also give the polymer a more coiled conformation on the surface as described by Lipatov et.al (22).

The slow increase in adsorption with increasing time after polymer addition may be ascribed to a reptation process $(\underline{23})$ where the polymer chain penetrates into the fiber wall despite a radius of gyration larger than the pores in the cellulosic fibers.

Results from experiments with the more highly charged, low molecular mass 3.6.-ionene are given in Fig. 5. Here, the value of the adsorption stoichiometry indicates that this polymer is initially adsorbed in a rather flat conformation on the surface. The comparatively large increase in adsorbed amount between 10 and 60 seconds after polymer addition is most likely due to a diffusion of these low molecular mass polymers into the porous fiber wall.



Fig. 3 Kinetics of adsorption and adsorption stoichiometry, Q %, for the MAPTAC-polymer on cellulosic fibers with D.S.=0.065. Added amount of polymer: 7.5 mg/g. (From ref. 19 with permission of Academic Press)



Fig. 4 Kinetics of adsorption and adsorption stoichiometry, Q %, for the MAPTAC-polymer on cellulosic fibers with D.S.=0.065. Added amount of polymer: 15 mg/g. (From ref. 19 with permission of Academic Press)



Fig. 5 Kinetics of adsorption and adsorption stoichiometry, Q %, for the 3.6.-ionene on cellulosic fibers with D.S.=0.065. Added amount of polymer: 80 mg/g. (From ref. 19 with permission of Academic Press)

FLOCCULATION OF SUSPENSIONS OF MICROCRYSTALLINE CELLULOSE (MCC)

General Remarks

Flocculation mechanisms are usually discussed in terms of bridging, patch or charge neutralisation flocculation. These mechanisms have been discussed in detail in overview articles (2,24,25) and these discussions will not be repeated here. It is, however, very important to remember, as has been carefully pointed out by e g Gregory (26), that flocculation is a complex process and must be viewed as a sequence of events. First the polymer adsorbs onto the particle and then the polymer starts to reconform to reach its equilibrium configuration on the particle surface which is different from the conformation of the polymer in solution. When two flocculating particles meet, one or both of the particles may carry polymer which may or may not have reached its equilibrium conformation. Obviously the relative rates of all these events are of great importance for the flocculating process.

Experimental approach

The experimental strategy in the flocculation experiments with MCC has been to add the polymers to the suspension and, by stirring the solution for a short time, obtain a very rapid adsorption of the polyelectrolytes onto the particles. The suspension is then allowed to stand for a certain time to permit reconformation of the adsorbed polymers before flocculation is initiated by stirring. The MCC suspension is so dilute and the particles are so large (9 µm), that no flocculation will occur by a pure diffusion process. The apparatus used for the flocculation experiments is a Britt Dynamic Drainage Jar (BDDJ) with a controlled speed of the propeller (27). The procedure used has been to add the polymer to the MCC-suspension and stir the suspension at 300 rpm for 10 seconds. This is the adsorption step. The suspension has then been left without stirring for t seconds. After t seconds the solution has been stirred for s seconds at 500 rpm to induce flocculation. The time t can be 0 seconds, which means that the speed of the propeller is in this case changed directly to 500 rpm to induce flocculation. The extent of flocculation was measured by taking samples from the BDDJ, diluting the samples and directly measuring the floc size in a particle size analyser Malvern 2600c. Full experimental details will be given in a separate report (28).

Results and discussion

The results of the flocculation of a MCC-suspension (100 mg/l) with a high molecular mass cationic poyacrylamide (Allied Colloids, Bradford, UK) are shown in Fig. 6. According to the manufacturer, the polymer has 10 mole-% cationic groups. The level of addition of the polymer is 1.75 mg/g, which corresponds to a negative zeta-potential as can be seen in Fig. 7. For a polymer with such a low charge density added at this level, flocculation of the bridging type is obviously expected.



Fig. 6 Floc size of MCC as a function of waiting time. 1.75 mg/g high molecular weight cationic polyacrylamide (10 mole -%) was added. Flocculation time was 30 s.

The results shown in Fig. 6 give a detailed picture of the flocculation process. If the waiting time t is 0, the flocculation is very weak. The polymers have just been adsorbed when the flocculation phase starts. The polymers have obviously not been anchored sufficiently firmly to the surface to give flocs that are resistent to the shear level used.



Fig. 7 Zeta-potential for MCC-particles as a function of added amount of high molecular weight cationic poly-acrylamide (10 mole -%).

At this point it is pertinent to point out that we have checked that all polymer is adsorbed after the initial 10 seconds. This has been done by quickly filtering off the particles and measuring the polymer concentration using polyelectrolyte titration (21). The particles will of course also collide during the adsorption phase, and it is indeed easy to show (26) that for the case of orthokinetic flocculation the collision between the particles is a faster process than the adsorption of polymer. However, as already discussed, the polymer bridges are obviously too weak at this stage.

Fig. 6 also shows that there is a clear maximum in the flocculating efficiency if the polymers are given a waiting time of approximately 30 seconds. At this point the polymers have obviously had time to become firmly attached to the surface while still having a conformation that make them effective for bridging flocculation. If the waiting time is more than 60 seconds, the polymer has lost much of its flocculating ability. It has now adopted a flat conformation on the particle surface and is no longer effective for bridging flocculation. It should be kept in mind that the level of polymer addition is such that the particles are negatively charged and the charge density of the adsorbed polymer is so low that patch flocculation is not very likely. It is of course satisfying to notice that the time during which bridging flocculation seems to be effective for this polymer is definitely of the same order as the reconformation time reported for high molecular weight polymers in the first part of this paper. The charge density is lower for the present polymer, but in experiments in progress in our laboratory no great variation in the reconformation time with changes in polymer charge density has been found.



Fig. 8 Floc size of MCC as a function of waiting time. 3 mg/g high molecular weight cationic polyacrylamide (10 mole -%) was added. Flocculation time was 60 s.

The flocculation behaviour when the level of addition of polymer is increased to 3 mg/g is shown in Fig. 8. The charge on the particles is now close to zero. The flocculation mechanism will thus be mainly charge neutralization and no strong dependence of the flocculation efficiency on polymer conformation will be expected. The weak decrease in flocculation that occurs especially during the first 60 seconds of waiting time can probably be attributed to a loss of flocculation by bridging.

These experiments have been repeated with a medium charge density, high molecular mass cationic polyelectrolyte, a copolymer of acrylamide and (N,N,N)-trimethylaminopropylmethacrylamide, MAPTABr. The same polymer in chloride form was used in the work described in the first part of this paper (see Fig. 1). The charge density of this polymer is $2.6 \cdot 10^{-3}$ eq/g



Fig. 9 Floc size of MCC as a function of waiting time. 0.5 mg/g MAPTABr was added. Flocculation time was 30 s.

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which corresponds to approximately 40 mole-% charged monomers. The results of measurements when 0.5 mg/g of MAPTABr is added are given in Fig. 9. This level of addition gives a zeta-potential of -12 mV. Zero zeta-potential is reached at addition of 0.8 mg/g. At zero waiting time, weak flocculation is observed which again is probably due to bad anchoring of the adsorbed polymers. After a certain waiting time, the flocculation has increased dramatically. In this early phase, flocculation by bridging should be the dominating mechanism. In contrast to the case for the polyelectrolyte with low charge density, the flocculation decreases only moderately at long waiting times. The reason is most likely that for this high charge density polymer a patch type flocculation is also possible.

We have also conducted some experiments in which the polymer has merely been mixed into the suspension and the adsorption of the polymer left to a diffusional process. If one analyses the situation in detail (26), this is a case where the adsorption can take place first and the flocculation then initiated by stirring. However, to reach an adsorption time that is short compared to the reconformation time, highly concentrated suspensions must be used. Fig. 10 shows the flocculation results for a dilute latex suspension (100 mg/l) into which 0.5 mg/g DADMAC (dimethyldiallylammoniumchloride, Allied Colloids, Bradford, UK) had been mixed. This is a highly charged rather low molecular mass polymer $(6.19 \cdot 10^{-3} \text{ eq/g}, \text{ M.W.}=40.000)$. This level of addition gives at equilibrium a negative zeta-potential of -20 mV. If the suspension is gently stirred directly after polymer addition only weak flocculation is obtained since almost no polymer has had time to adsorb. After five minutes there is much more extensive flocculation since the polymer has now had time to adsorb and at least part of the absorbed polymers are in a conformation that is favourable for flocculation. At longer times, 15 and 30 minutes, most of the polymers have been adsorbed and have also had time to adopt a flat equilibrium conformation on the surface. Consequently there is only a weak flocculation at these longer times.



Fig. 10 Floc size of polystyrene latex as a function of waiting time. 0.5 mg/g DADMAC was added. Flocculation time (gentle stirring) was 60 s.

FLOCCULATION OF PULP SUSPENSIONS

Experimental approach

To evaluate the kinetics of flocculation of cellulosic fibers, a special equipment was constructed (29). The system is built around a circulation system, Fig.11, in which flocculation is monitored with a laser-optical device (8 in Fig.11). The back-scattered laser light from the fiber suspension is transformed into an electrical signal by the phototransistor (9 in Fig.11), and is taken as a measure of the local fiber concentration. This signal is fed to a frequency analyser and the frequency power spectrum (P(n)) is recorded. This spectrum is then transformed to a wavelength power spectrum (E(λ)) and by comparing the wavelength power spectra with and, without polymer addition, it is possible to evaluate both the degree of polymer-induced flocculation and the average size of the flocs formed. Only the degree of flocculation is however considered in the present paper.

From the wavelength power spectrum a coefficient of variation of the signal, which is a measure of the degree of flocculation according to Wahren (<u>30</u>), can be calculated according to Equation (1).

$$V = \frac{1}{c} \int_{a}^{b} E(\lambda) d\lambda$$
 (1)

a and b = integration limits determined by the experimental set up

c = average fiber concentration

To characterize the degree of polymer-induced fiber floccualtion with a single quantity, the coefficients of variation V_A of the signal with polymer addition and V_B without addition are used to calculate the flocculation index F according to the equation:

$$F = \frac{\sqrt{\frac{V_{A}^{2} - V_{B}^{2}}{V_{B}}}}{V_{B}}$$
(2)



Fig. 11 Diagram of the device used for detection of polymerinduced fiber flocculation. (From ref. 29)

1. Stirrer. 2. 250 l tank (for fibers and deionized water). 3. Pump, all parts in contact with the suspension being of plastic material. 4. Magnetic valve. 5. Plunger for polymer dosage. 6. Stepmotor, driven and controlled by the microcomputer unit 10. 7. Polymer solution. 8. He-Ne laser (632.8 nm). 9. Photo-transistor with amplifier. 10. Micro-computer unit (ABC-80). 11. Frequency analyser. 12. Plotter for presenting wavelength power spectra. 13. Magnetic valve. 14. Glass pipe. 15. Point of polymer addition.

If this measurement is repeated at different positions downstream from the polymer addition (15 in Fig. 11), it is possible to evaluate the kinetics of polymer-induced flocculation.

In these experiments, the same types of cationic polyacrylamide were used as in the work reported in the previous parts of this paper, (see Fig. 1). D.S.-levels in mole-% cationic groups and molecular mass data for these polymers are given in Table 1.

D.S.	[ŋ]	M _η (visc) ¹	Mw 2
%	(dl/g)	•10 ⁻⁶	• 10 ^{- 5}
1.8	8.5	3.5	4.7
5.0	10.8	5.4	3.0
20	10.55	5.7	1.9
40	8.65	5.8	3.5

1 From intrinsic viscosity

2 From HPLC measurements

Table 1 Molecular mass characteristics of MAPTAC-polymers.

Only decrilled bleached softwood fibers were investigated in the experiments presented here. The average velocity in the pipe was 1.12 m/s which corresponds to a shear field of 483 s⁻¹. Only deionised water was used. For full experimental details the reader is referred to (31).

Results and discussion

First of all, the experiments show that the flocculation induced by the MAPTAC-polymers is a very rapid process with almost all the flocculation completed in less than one second after polymer addition. This among other things is shown in Fig. 12 where the flocculation index is plotted as a function of time after polymer addition.

A constant addition of 0.3 mg polymer/g fiber was used in these experiments (this level was chosen since it was not sufficient to recharge the cellulosic fibers).



Time after polymer addition (s)

Fig. 12 Flocculation index as a function of time after polymer addition for differently charged MAPTAC polymers. Addition level of MAPTAC was 0.3 mg/g and fiber concentration was 1 g/l. (From ref. 31)

The figure shows that the D.S. of the polymer is very important for the flocculation efficiency. This D.S.-dependence may be explained by several factors. A different expansion of the polymers out from the fiber surface, in accordance with the results presented in the previous two sections, may affect the efficiency. When the charge density is increased, the polymer chain is extended by the repulsion between the charges in the chain. This extension increases the ability of the polymers to form bridges between the cellulosic fiber surfaces in the fiber-fiber contacts. However, if the polymer charge is increased above a critical level, the forces associated with polymer-surface interactions tend to give the polymer a flatter conformation on the surface and hence a decreased bridge-forming ability. This was shown in the first section of this paper.



Fig. 13 Flocculation index as a function of time after polymer addition at different pH-values. Addition level of MAPTAC (D.S.=0.2) was 0.3 mg/g and fiber concentration was 1 g/l. (From ref. 31)

Another factor that affects the flocculation is the number of adsorbed polymer molecules. When the charge density is increased there is also a decrease in adsorption and hence a smaller number of polymer-bridges between the fibers.

Since one of the phenomena discussed (increased polymer expansion) tends to increase and the others to decrease the bridge-forming ability of the polymer, a maximum at some D.S.value as observed is expected.

This hypothesis is also supported by the experiments with the polymer of D.S. = 0.2 at different pH-values shown in Fig. 13. When the pH is increased from 6 to 8 and hence the surface charge of the fibers is increased, there is a large decrease in flocculation index, most probably due to a flatter conformation of the polymer on the surface. The decrease in flocculation index with decreasing pH is attributed to a lower number of polymer bridges due to a lower adsorption at lower pH and to an increase in electrolyte concentration which also reduces the extension of the polymer.

Finally it should also be added that the phenomena discussed in this last section are caused by the initial adsorption phenomena described in the first section of this paper. The differences in adsorption stoichiometry at different levels of addition of MAPTAC-polymers were, for example, much larger at short contact times, which makes the large differencies shown in Fig. 12 and 13 much easier to understand.

CONCLUDING REMARKS

It is evident that the conformation and reconformation processes of adsorbed polymers are very important factors in flocculation. In the practical use of polyelectrolytes for flocculation the point of polymer addition can obviously be of crucial importance.

There are, however, many aspects of polymer conformation and flocculation which remain for future investigation. A systematic study of the effects of molecular mass and charge density of the polymers has not been made and the influence of added electrolyte has not been covered in this investigation. The strength of the flocs formed is also an important aspect of flocculation most certainly connected to the conformation of the adsorbed polymers.

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Transcription of Discussion

CONFORMATION OF ADSORBED POLYMERS AND FLOCCULATION OF MICROCRYSTALLINE CELLULOSE AND PULP SUSPENSIONS

L. Wagberg, L. Odberg and G. Glad-Nordmark

Dr. T. Herington, Reading University, UK

Thank you Lars for a very interesting and stimulating paper. You have given us lots of ideas - just one point - you talked about pores, I wonder what you actually mean by a pore and what physical evidence you have for their existence?

Dr. L. Wagberg

What do you mean by physical evidence for a pore? I rely on the papers we discussed yesterday and that we hence are talking about different kinds of pores. First of all we are talking about fissures in the fibre wall of the order of a micrometre - and secondly we discuss the pores that are measured by the solute exclusion technique. These latter are of the order of 50 Angstroms in diameter. This is what I mean by pores. I assume that your question is concerned with how this kind of polymer can reach all the smallest pores? The fibres I have discussed are in most cases carboxymethylated pulp and so they are highly swollen. Which means that the pores I am referring to are really the micropores, if I may call them that. We have seen that when we used a noncarboxymethylated pulp we could soon saturate the outer surface of the fibres with polymers and if we left the fibres for 5 days it was possible to increase both the absorption stoichiometry and the absorbed amount. This can only be achieved if the polymers are removed from the outer surface so that new polymer can come in. That is a naturally indirect measure and it is in short how we interpreted the data.