Preferred citation: H. Xiao, R. Pelton and A. Hamielec. Novel non-ionic polymeric flocculants for mechanical pulps: an overview. In **The Fundametals of Papermaking Materials**, *Trans. of the XIth Fund. Res. Symp. Cambridge, 1997*, (C.F. Baker, ed.), pp 1121–1134, FRC, Manchester, 2018.
DOI: 10.15376/frc.1997.2.1121.

Novel Non-ionic Polymeric Flocculants for Mechanical Pulps: An Overview

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ABSTRACT

Novel comb copolymer with long polyacrylamide backbones bearing very short polyethylene glycol (PEG) pendant chains were prepared by the free-radical copolymerisation of acrylamide (AM) and PEG acrylate macromonomers. The copolymers are effective retention aids for mechanical pulps in retaining fines and precipitated calcium carbonate (PCC). The optimum copolymer stricture had a molecular weight greater than 3 million and contained 0.5 - 1.0 % of PEG pendant chains with 9 to 23 polyether repeat units. The retention of fines induced by copolymer/phenolic resin (PFR) dual-polymer system follows the mechanism called "complex bridging flocculation". According to this mechanism, the copolymer molecules aggregate in the presence of PFR to form a colloidal dispersed polymer complex which hetero-flocculates with fine particles. The interpolymer complex formation were experimentally observed through precipitate isotherms measurement, dynamic viscosity measurement and fluorescent microscopy.

Key words: copolymer; retention aids; flocculation mechanism; PEO; complex formation.

INTRODUCTION

Polyethylene oxide (PEO) has been used to improve fine retention of newsprint for over decade.[1],[2]. The advantage of using PEO is that the flocculation performance is less affected by the presence of wood based dissolved and colloidal substance (DCS) which bear anionic surface charges. By contrast, DCS causes the excess consumption of conventional cationic flocculants. The molecular weight (MW) of PEO is important. Only PEO with MW higher than 4 million gave significantly improved flocculation. However, very high molecular weight PEO is expensive and susceptible to degradation which lowers the molecular weight and thus the flocculation efficiency. PEO is particularly susceptible to degradation when in aqueous solution. The degradation can be caused by either shear forces or oxidation. Thus, in commercial applications, PEO solution must be freshly prepared and carefully handled.

In order to overcome the fragile properties of PEO, a series of the novel non-ionic comb copolymers with long polyacrylamide (PAM) backbones and short poly(ethylene glycol) (PEG) pendant chains have been developed in our work. The comb copolymers were prepared by free radical copolymerisation of acrylamide (AM) and poly(ethylene glycol) (PEG) macromonomers either in aqueous solution using potassium persulfate as an initiator, or in inverse emulsion polymerisations. PEG macromonomers are methoxy PEG esters of acrylic and methacrylic acid. The copolymerisation potentially provides full control over the pendant chain length, the number of pendant chains per macromolecule and molecular weight of copolymers.

The objectives of this work included 1) to prepare a series of novel acrylamide-PEG macromonomer comb copolymers with well-defined structures; 2) to correlate the flocculation performance to the structures of the copolymers; 3) to reveal retention mechanism in a non-ionic dual-polymer retention system; 4) to investigate the interpolymer complex formation.

EXPERIMENTAL

1. Materials

Acrylamide monomer(Aldrich Co.) and methoxy poly(ethylene glycol) monomethacrylate macromonomers (Polysciences, Inc.) were used as received. The macromonomer structures have been verified by ^1H and ^{13}C NMR measurements. More details on characterisation of macromonomers are described elsewhere.[3,4] Potassium persulfate (KPS) (BDH Chemicals) was used as an initiator. Milli-Q treated distilled water was used to prepare all aqueous solution.

Polyethylene oxide (PEO) (Union Carbide) and phenol formaldehyde resin (PFR) Cascophen C-271 (40 % in aqueous solution) (Borden Chemical) were used as supplied. Weight average molecular weight (Mw) of PEO-309 was 8 million, as indicated by the supplier. The Mw of the C-271 was approximately 13,000, measured by GPC in this work.

The newsprint pulp (Abitibi Price Co; Iroquois Falls) was based on 65 % stone groundwood and 35 % high yield sulfite pulps (70 % yield, acid sulfite). The pulps were made from a mixture of mostly black spruce plus a few percent jackpine and white spruce. Fines content of the pulp was 58 % (wt).

Precipitated calcium carbonate (PCC) used in this work was supplied by Speciality Minerals Inc.

2. Preparation of Copolymers

The copolymerisations were carried out in aqueous solution in a 1 L batch reactor equipped with a mechanical stirring paddle. The temperature of polymerisation was maintained at 25°C or 40°C. Between 2 and 8 hours were required for polymerisation with initiator KPS at concentration of 3.0×10^{-3} mol/L. Monomer concentrations were below 6 %(wt). The copolymer aqueous solution was further diluted before being used in retention or flocculation applications. Details of the copolymerisation and copolymer characterisation have been published elsewhere.[3] The copolymer can also be prepared in inverse emulsion copolymerisation.[5]

3. Retention Measurements

Evaluation of the copolymers as retention aids for newsprints was carried out in a Dynamic Drainage Jar (Paper Research Materials Inc.) which was fitted with a perforated stainless steel plate containing 0.6 mm holes. The first-pass retention (FPR) was measured at 50°C with the propeller speed ranging from 250 to 1000 rpm. Detailed operating procedures were the same as described by Pelton et al.[6]

In retention of precipitated calcium carbonate, PCC (20 wt % on o.d. pulp) was added to the pulp (final pulp consistency 0.5 wt %) and the pH of the furnish adjusted to 7.5 by the addition of H_2SO_4. The furnish was then dispersed in a DDJ at 1,000 rpm for 60 seconds. Known quantities of polymeric flocculants were added; the first-pass white water (100 mL) being collected 10 seconds after the addition of the final polymeric component. The first-pass retention was evaluated from the difference between the amount of PCC present in DDJ before polymer addition, and the amount of PCC present in the white water. PCC concentrations were obtained by dissolution of $CaCO_3$ in HCl (0.3 mL of 12 N HCl per 100 mL sample) after which the soluble Ca^{++} concentration was measured by an automated EDTA titration apparatus. The endpoint was detected with a Orion Model 93-20 calcium selective electrode.

4. Characterisation of Interpolymer Complex Formation

The precipitated isotherm of copolymer onto PFR was measured by an UV spectroscopy (Hewlett Packard). Between 0.02 and 0.2 mL of C-271 at the concentration of about 4000 mg/L was added to a test tube containing 15 mL of copolymer solution (70 to 240 mg/L). After shaking by hand the tubes were left for 20 minutes to allow the precipitated complex to settle. The C-271 concentration in the supernatant was measured by UV at wavelength of at 286 nm.

A Bohlin VOR Rheometer System (Bohlin, Sweden) was used to measure the rheological behaviour of mixtures of the phenolic resin and copolymer. Measurements were conducted in a concentric cylinder with a measuring geometry of C25. Continuous viscosity and shear stress were recorded as a function of time at a constant strain. Shear rate was 146/s. The total sample volume was about 13 mL.

Fluorescent microscopic observations on the complexes formed between the copolymer and phenolic resins were conducted using a Laborlux K Dage-Mti series Sit 66 microscopy system (Leitz) with a halogen laser source. Acridine Orange (3,6-bis[dimethylamino] acridine-HCl, from Sigma) was used as a probe molecule in fluorescent microscopic observation. A solution mixture of phenolic resins and the probe molecule was first put in a watch glass. The copolymer solution, at pH 5, was then dropped on the glass and mixed with phenolic and Acridine Orange solution. Images were videotaped.

RESULTS AND DISCUSSION:

1. Copolymers as Retention Aids

A series of copolymers with structures shown in Figure 1 have been prepared and characterised.[3] Most copolymers used in this work were synthesised from acrylamide and methoxy PEG methacrylate macromonomers. Therefore, the copolymers are denoted as AM/MPEGMA copolymers. Figure 2 shows the first-pass retention of newsprint fines as a function of the density of PEG pendant chains in copolymers. The density is expressed as a mole fraction of macromonomer in the copolymers. The data from two series of copolymers, labelled as "low MW" and "high MW", also indicated the influence of the molecular weights of copolymers on retention processes. Obviously, higher MW gave better retention improvement. However, with high MW, only 0.8 % (mole) of macromonomer was required to increase the FPR from 37% to 75 %. No significant improvement was observed when the PEG compositions

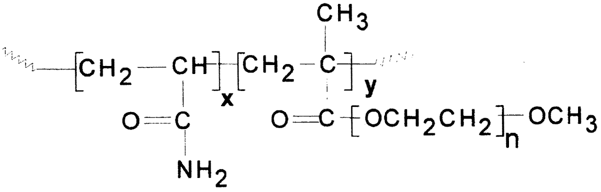

Figure 1. Structure of AM/MPEGMA copolymer

were further increased. It has also been found that the retention performance of the copolymer with high MW was not significantly influenced by the PEG pendant chain lengths ranging from 5 to 40 polyether repeat units.[7]

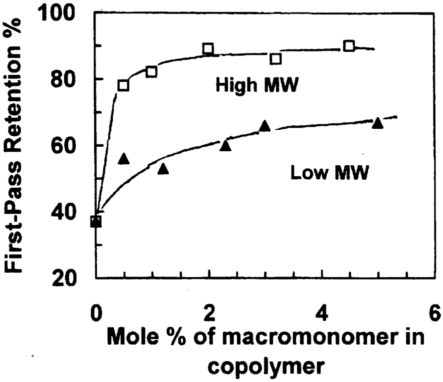

Figure 2. Influence of the density of PEG pendant chains in copolymers on first-pass retention of newsprint fines in DDJ measurements. Lower molecular weight (MW) copolymers were obtained at 40°C with 1.0 to 10 % (mole) of PEG macromonomer with 10 ether repeat units; higher MW copolymers were prepared at 25°C with 0.5 to 7 % (mole) of PEG macromonomer with 23 ether repeat units.

Figure 3 describes the influence of copolymer concentrations on FPR for newsprint pulps at constant phenolic resin concentration (= 2.0 mg/L). The copolymer contained approximately 0.7 % (mole) PEG pendant chains with average 9 polyether repeat units and a MW of 4.6 million. Fines retention increased with copolymer concentration to a maximum of 90 % at a concentration of 2.0 mg/L. Beyond 2.0 mg/L, there was little increase in retention. Therefore, the requirement on polymer dosage for the copolymer is similar to that for PEO homopolymer.

Precipitated calcium carbonate retention is shown in Figure 4 as a function of copolymer/PFR concentrations. The results from high-molecular-weight PEO are included for comparison. It has been shown previously that the high-molecular-weight

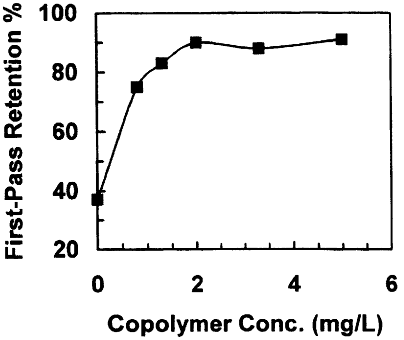

Figure 3. Effects of amounts of the comb copolymer addition on first-pass retention. Phenolic resin was fixed at 2 mg/L. The measurements were conducted at 50°C with propeller speed 250 rpm.

PFR/PEO system was an effective flocculant for pulp fines in newsprint pulp.[2] The PCC retention results obtained in the present work, however, suggest that this system is not a very effective retention aid for PCC (lowest curve in Figure 4). By contrast, the copolymer produced good PCC retention and the data indicate that there was significant improvement in retention over the PFR/ PEO system.

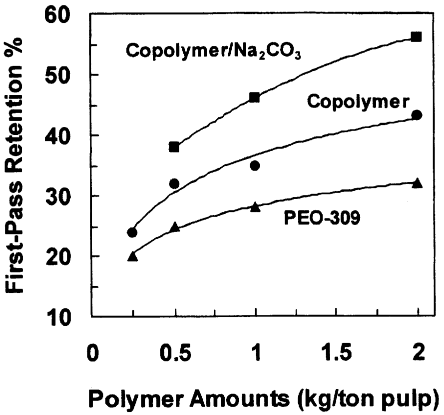

Figure 4. PCC retention induced by copolymer and PEO in the presence of cofactor PFR. Copolymer:PFR = 2:1 (wt). 0.01 M Na_2CO_3 was added as third component for copolymer/PFR retention system. First-pass retention was measured at 1000 rpm.

The PCC retention was increased further when Na_2CO_3 was used in conjunction with either PEO or the copolymer. Indeed, PFR/copolymer in the presence of 0.01 M Na_2CO_3 was the most effective combination of additives in this investigation. Addition of Na_2CO_3 lowered the free calcium ion concentration from 0.3 mM to 3 mM. Furthermore, retention was not dependent upon the order of addition of Na_2CO_3 and PCC to the pulp. The role of Na_2CO_3 has not been completely understood. We speculate that the calcium ions would interfere the complex formation between the copolymer and PFR. Thus, the reduction of Ca^{++} ions concentration induced by Na_2CO_3 would correspondingly increase the retention efficiency.

One of the important features of the comb copolymer is the ageing resistance. Compared with PEO homopolymer, copolymer appeared to be more stable in aqueous solution. For example, PEO homopolymer showed severe degradation at diluted concentration (e.g. 0.03 % wt) in less than 3 days with almost complete loss of flocculation ability whereas the copolymer maintains high flocculation efficiency after having been stored at the same conditions for up to 20 days.

Overall, the AM/MPEGMA copolymers provide a novel series of non-ionic polymer retention aids in conjunction with phenolic resin or other effective cofactors. The copolymer displayed improved retention performance on PCC and newsprint fines. Moreover the copolymer apparently is less sensitive than PEO to degradation. As an effective retention aid, the copolymer molecular weight must be greater than 3×10^6, however, the PEG content in copolymer can be as low as 0.5 % (mole) with chain lengths ranging between 9 to 23 repeat units.

2. Retention Mechanism

The retention of colloidally dispersed fine particles with a mixture of PEO and PFR as well as cellulose fibres is complicated because many interactions are possible. AM/MPEGMA copolymers have polyether groups as does PEO homopolymer. The interaction between PEO and phenolic resin is presumed to occur between PEG pendant chains in the copolymer and phenolic resin. Furthermore, all evidence suggests that the same retention mechanism applies to for both PEO/PFR and copolymer/PFR systems.

We have proposed a "complex bridging retention" mechanism in which the fines particles are flocculated due to adsorption of PEO or copolymer/phenolic resin complex possibly in colloidal form on their surfaces. Essentially the same mechanism was proposed by Van de Ven and Alince.[8] The flocculation process is complicated and includes the following processes:

Copolymer molecules first form a soluble polymer complex with one or more PFR molecules. Copolymer/PFR complex then aggregates to form a time dependent distribution of colloidal sized species called colloidal complex. Complexes, both colloidal and soluble, adsorb onto fines particles. The bridging of fines particles by adsorbed complexes. Continuing heterocoagulation of fines particles and colloidal complexes, this constitute a mechanism herein called "complex bridging".[9] Finally, the deposition of large fines flocs onto fibre surfaces.

The PFR cofactor used in this work formed a precipitate when mixed with aqueous PEO; other cofactors do not display thus behaviour.[7,10] The bridging of fines induced by colloidal polymer complex must occur before the collapse of aggregation of colloidal complex to give a dense precipitate which is inactive in flocculation. Perhaps, the rate of complex collapse and precipitation is molecular weight dependent, thus explaining the molecular weight sensitivity in PEO/PFR or copolymer/PFR retention system. That is, we speculate that the interpolymer complexes based on PEO or copolymer with low molecular weight (e.g. 10^4 to 10^5) collapse too quickly for flocculation to occur. More work is required.

3. Interpolymer Complex Formation

The key point in the complex-bridging retention mechanism is the complex formation between copolymer and cofactor PFR. It has been noticed that the precipitates formed when the copolymer aqueous solution was mixed with PFR at a relatively high concentration (e.g. about 0.4 % wt). Figure 5 shows the precipitation isotherms, i.e., the mass of precipitate as a function of the amount of added PFR. The experimental conditions are summarised in the figure caption and the mass of PFR in the precipitate was divided by the total mass of polyether (i.e. copolymer) to give a dimensionless quantity. The mass of precipitated PFR increased with the amount of added C-271, and nearly saturated at 0.6 mg C-271/mg copolymer. Similar precipitation was observed for PEO/PFR system.[11,12]

Another evidence for the interpolymer association is the rheological behaviours of the copolymer/PFR mixtures. The experimental result in Figure 6 shows that a spike in the viscosity was observed after the PFR was added into the copolymer solution. After approximately 80 seconds, the system viscosity was reduced to close to the initial

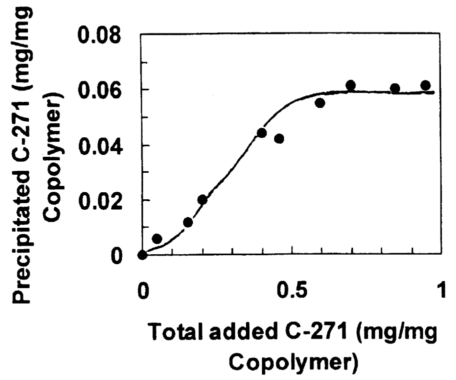

Figure 5. Precipitated complex as functions off the amount of added PFR C-271. The dimensionless PFR concentrations were based on the ratios o PFR to the copolymer concentration which was 249 mg/L. The initial pH was 5.

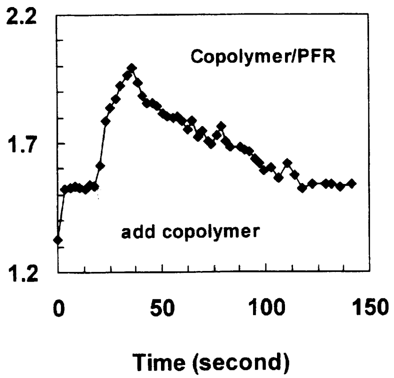

Figure 6. The viscosity behaviour during interpolymer associations between copolymer and phenolic resin C-271. Experimental conditions: copolymer conc. = 60 mg/L; [C-271] = 60 mg/L; pH = 5.0; temperature = 23 ± 0.1°C; shear rate = 146 sec^{-1}; and, constant shear strain. Adding copolymer at time = 15 seconds.

copolymer aqueous solution value. The curve indicate the transit properties of the interpolymer association between the copolymer and PFR. The similar curve was found for PEO/PFR system,[13,14] however, the collapse of PEO/PFR complex apparently faster. It only took about 30 seconds to reduce the peak viscosity to original viscosity for the PEO/PFR system.[12] This phenomenon suggests that the copolymer/PFR complex might last longer than PEO/PFR complex does before collapse.

Fluorescent microscopy was also used to observe the complex formation. Acridine orange, a cationic water soluble dye, was used as a probe. When acridine orange was mixed with phenolic resin, the uniformly dispersed white spots were observed. It was assumed that the cationic dye formed electrostatic complexes with the negatively charged PFR and generated the fluorescent emission. However, when the copolymer was added to the mixture of the dye and phenolic resin, highly fluorescent aggregates with sizes greater than 15 μm were immediately observed. Figure 7 shows such an example. It is assumed that the large white fluorescent spot indicates the concentrated PFR complexed with the copolymer. It should be noted that the PFR concentrations used in these measurements (275 mg/L) were much higher than those used in flocculation applications (2 mg/L). If polymer concentrations were reduced to 25 mg/L, no luminescent aggregates were observed under the present instrumental conditions.[12]

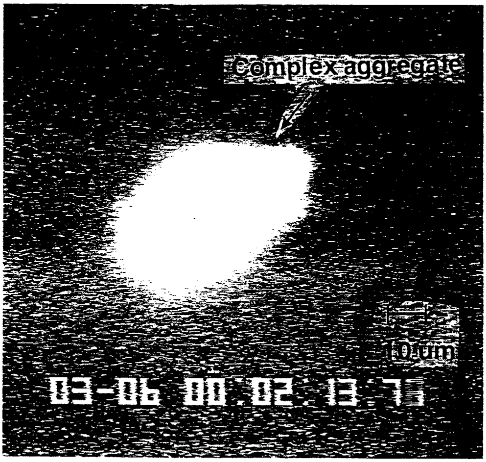

Figure 7. Fluorescent microscopic observation on the mixture of Acridine Orange and PFR C-271 with copolymer; [Acridine Orange] = 30 mg/L; [PFR] = 275 mg/L; [copolymer] = 490 mg/L. Luminescent aggregates were formed in this case. Copolymer 56-107 containing 1.7 % (mole) of PEG macromonomers with average PEG chain length of 23 repeat units.

It has been found that the interpolymer complex formation occurred to the copolymer with PEG pendant chain lengths as short as 9 repeat units (i.e., MW of PEG about 400). This was attributed to the simultaneous interaction of more than one PEG chain with a phenolic resin molecule. Figure 8 shows the possible topologies of the copolymer/PFR complexes. A PEG chains can be simultaneously bonded to adjacent PEG chains on the same PAM backbone (Figure 8(a)). Since the copolymer normally contained less than 1.0 mole % of macromonomers, this case might seldom occur due to a large average distance between neighbouring PEG pendant chains. Figure 8(b) shows the possibility of complex formation resulting in intramolecular crosslinking whereas Figure 8 (c) indicates a PEG molecule bridging tow different copolymer molecules. Case (b) should reduce the viscosity of the mixture solution whereas case (c) should increase the viscosity. The rheological result in Figure 5 is evidence for the presence of case c structures.

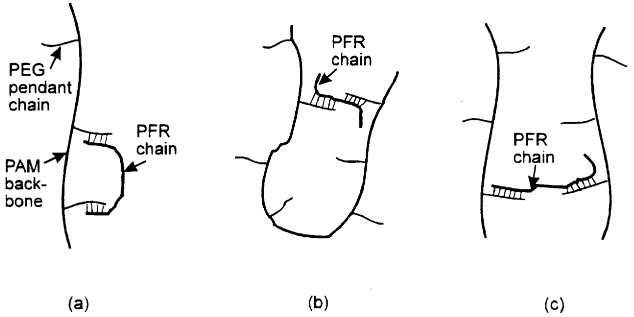

Figure 8. A schematic of the complex formed between PAM-co-PEG copolymer and PFR. Cases (a) and (b) involve isolated polyether chains whereas Case (c) would lead to macroscopic interpolymer associated structures.

It is emphasised that the structure in Figure 8 are speculative. The copolymer/PFR interaction have not been completely illustrated. It is possible that there are some interactions between the PAM backbone and PFR. In addition, hydrophobic interaction may also contribute to complex formation. To further understand the complex formation between copolymer/PFR, the phenolic resins or model compounds with well-defined structures are also required.[15,10]

CONCLUSIONS:

1. Copolymers with long polyacrylamide backbones and short poly(ethylene glycol) pendant chains appear to be effective retention aids. The copolymers can be prepared both in solution polymerisation and inverse microemulsion polymerisation.

2. To obtain the optimum retention performance, MW of the copolymers must fall between 3 to 5 million. With these molecular weights, it is only necessary for the copolymer to contain 0.5 to 1.0 % (mole) PEG pendant chains with chain length in the range 9 to 23.

3. The retention mechanism of copolymer/phenolic resins system follows the "complex bridging flocculation". Copolymer/PFR complex must be adsorbed onto fines particles for retention to occur.

4. The complex formation between copolymer and phenolic resin was demonstrated by observations on the precipitation of complexes, rheological behaviour of polymer mixtures and fluorescent microscopy.

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Transcription of Discussion

Novel non-ionic polymeric flocculants for mechanical pulps: an overview

Dr Huining Xiao, Lecturer, UMIST, UK

Professor Tom Lindström, Royal Institute of Technology (KTH), Sweden

Your complex bridging mechanism is a new invention. There are simpler explanations and you probably know that from our previous correspondence and discussions. Apart from this, I would like to ask you about the interesting effect of sodium carbonate on PCC retention. If you have some polyacrylamide molecules which are hydrolysed then you could possibly explain this effect. If you are adding sodium carbonate to PCC it becomes not negatively charged. If you have a few co-polymer groups which are hydrolysed the PAM will absorb onto the anionic PCC. This will lead to more efficient network mechanism because if you can avoid interactions between the polymer and the surface then you would get more polymer in solution available to network formation. So this is a way to explain your data from our original proposal of the network flocculation mechanism.

Huining Xiao

I guess your point is if you reduce the adsorption of polymer onto PCC then in fact we are left with the network flocculation mechanism. I wish we could prove that. We did try to synthesise two types of terpolymers - one combines acrylamide, cationic and PEO macromonomer, another consists of acrylamide, acrylic acid and PEO macromonomer. However, experimental results indicated that the copolymers of acrylamide and PEO macromonomers are better than the terpolymers in PCC retention.

Dr Theo van de Ven, Director, Paprican/McGill, Canada

I have a question about your mechanism. Obviously it can only be the bridge route because you started off with particles which are already coated with PEO. PEO does not absorb on most broke fibres and cellulosic fines so you haven't explained in your mechanism any of that first step. Also you had three cases of complex formation and you said only one or two work. We have at least three cases where PEO molecules which remain individual molecules which work as co-factors.

Huining Xiao

We do have evidence to support a PEO adsorbed on the polystyrene latex which has been done by NMR. However, for bleached kraft pulps which were used in our model experiments, there is no absorption of PFR on the fibres based on UV measurements. What I propose here is no direct link (chemical bonding) between fibres and fillers. You have two polymers form a filler floc. These flocs might be only mechanically entrapped by fibre networks. There is no hydrogen bonding between filler and fibres in our model systems.

Theo van de Ven

I have another question about hydrogen bonding. We have one co-factor, if it is pure it will contain phenolic groups, that's polynapthylene sulphonate and that is an excellent co-factor which brings into question whether we really need hydrogen bonding.

Huining Xiao

In our co-factor (PFR)-PEO system, hydrogen bonding is important. Retention efficiency is pH dependent, which is the evidence to support this. However, we cannot distinguish the contributions from hydrogen bondings and hydrophobic associations. Future work is required.