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Insights into Bridging Flocculation

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Summary: The phenomenon of bridging flocculation is very widely encountered in many industrial applications, but is rarely explained with any clarity. In this paper, a cohesive mechanistic model is used to explain the extraordinary effectiveness of bridging flocculation and to guide a body of work on synthesis and application of cationic polymers for the cane sugar industry.

Keywords: flocculation; hydrophilic polymers; solid/liquid separation; sucrose

Introduction

A number of different understandings of the word ‘flocculation’ appear frequently in the chemical and chemical engineering literature. These include:

- The use of flocculation in a completely interchangeably way with coagulation; any disruption of a sol to that the disperse phase is collected into macroscopic rather than microscopic lumps. This is the usage sanctioned by IUPAC[1,2].
- Referring to any anthropogenic coagulation process as flocculation, where something is *done to* a dispersion in order to aggregate the disperse phase.
- Using the term flocculation to refer specifically to the addition of a *polymeric* agent in order to bring about aggregation[3].
- With reference to electrical double layer theory[4], a coagulated dispersion may be said to consist of particles that have aggregated in the primary minimum, while a flocculated dispersion consists of particles that have approached no closer than the secondary minimum.
- Using flocculation to refer to the appearance of the separated material: a coagulated material is dense, like cheese (derived from the Latin *coagulare*, to curdle) while flocculated material is fluffy and wooly (derived from the Latin *floccus*, a tuft of wool)[2,3]. While this is a qualitative definition, it is also empirical - anyone may look at the separated material and determine whether it looks more like curds or wool – and makes no unverifiable mechanistic assumptions.

This paper will use the word ‘flocculation’ in the sense of the third definition, as aggregation facilitated by the addition of a polymeric additive- a flocculating agent, or *flocculant*.

Most sols of industrial importance are primarily stabilised by electrostatic charges on the surfaces of the disperse phase particles, rather than by steric stabilisation, and can be made to aggregate by addition of a counter-ion. This mechanism of coagulation of a sol in the presence of a counter-ion is well described by DLVO (Derjaguin-Landau-Verwey-Overbeek) theory[4]. Charged particles in solution are not stabilised by direct repulsion between each other: rather, each is surrounded by a diffuse atmosphere in which counter-ions are more numerous than ions of the same charge as the surface, and the interaction between these clouds of counter-charge will provide electrostatic stabilisation. In qualitative terms, the size of these atmospheres and their effectiveness in stabilising the particles will be reduced by any change that forces/allows a higher density of charge. Increasing the concentration of the electro-

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lyte is one option, as can be seen by the destabilisation of colloidal clays when rivers flow into the sea. Increasing the charge z on the individual counter-ions is another, known from the empirical Hardy-Schulze rule (rate of destabilisation $\propto z^3$). Both these approaches have been quantified in DLVO theory. In conventional coagulation the formation of strong localised bonds will lead to brittle, freely-draining dendritic aggregates, as each incoming particle will adhere irreversibly at the position where it first encounters the aggregate. Under shear, these dendritic aggregates will break and recombine to give aggregates that are small and dense on the macroscopic scale, showing little or no 'woolly' morphology.

Small-angle neutron scattering (SANS) measurements made on such systems suggest that they show little or no short-range order, consistent with a random and irreversible process of aggregation giving rise to a highly fractal morphology[5], as shown in Figure 1(a).

There are three primary mechanisms by which addition of a polymeric additive can encourage aggregation of the dispersed phase of a sol. These are:

- Depletion flocculation
- Charge annihilation/charge patch flocculation
- Bridging flocculation.

Depletion flocculation requires that the continuous phase be filled with polymer coils, generating an osmotic force between two particles whenever they approach closely enough that polymer is excluded from the volume between them. This clearly requires relatively large amount of flocculant, on the order of 1000 ppm or greater. The particles will be coagulated, rather than flocculated, by the DLVO definition, in that they will reach the primary minimum and be held together by Van der Waals forces. Under depletion flocculation relatively dense aggregates are typically formed, as in conventional coagulation, owing to the inflexible nature of the particle-particle bonds generated.

Charge annihilation flocculation involves the addition of a counter-ion to the charges on the surface of the dispersed phase: e.g., the use of $FeCl_3$ and $Al(OH)_3$ in water treatment. To increase the counter-ion charge density and reduce the size of the electrical double layer, counter-ion charges can be provided using a charged polymer, where the charges are held together at a shorter distance than they would adopt if separate (Figure 3). This will increase the rate of particle destabilisation even at concentrations much less than those required to equalise, or 'annihilate', the surface charges of the particles, so in that sense the term 'charge annihilation' is a misnomer. The 'charge patch' mechanism is the suggestion that adsorption of a polymer on the surface of a particle, with its high density of counter-charge, can create an area of localised charge inversion that will be attractive to other particles. Both of

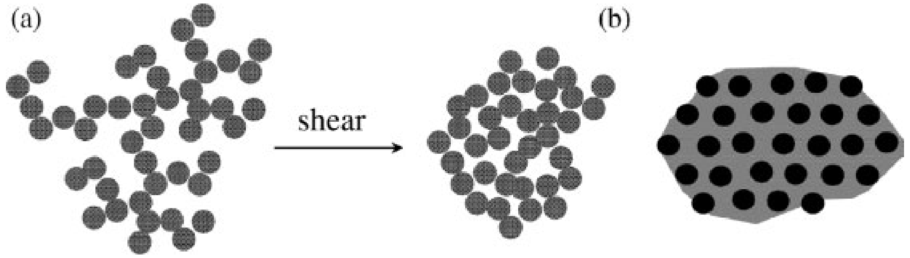

Figure 1.
Expected morphologies of aggregates of monodisperse particles formed by a diffusion-limited irreversible process such as coagulation of charge annihilation flocculation (a) and by a reaction-limited process with dynamic interparticle links, such as bridging flocculation (b).

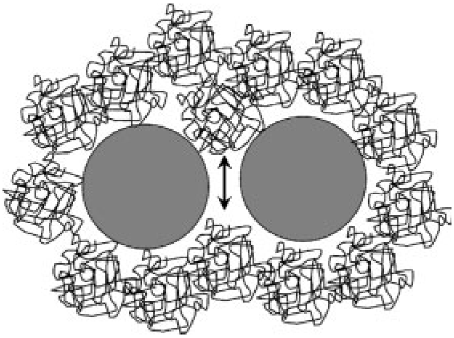

Figure 2.
Depletion Flocculation. When particles approach closely enough that no polymer coils can fit between them, an osmotic force will act to remove the solvent remaining between the particles and bring them together.

these mechanisms involving a polymer will tend to create coagulated rather than flocculated, aggregates by the DLVO definition of flocculation, where reduction of inter-particle repulsion allows particles to approach closely enough to be held together by van der Waals forces. Empirically, the lowest concentrations of polymer at which charge annihilation or charge patch flocculation mechanisms are effective are in the range 10-100 ppm.

If a high molecular weight charged polymer holds particles together such that they remain separated and cannot reach the primary minimum, it is probably more correct to describe the mechanism as bridging flocculation.

Bridging flocculation relies on the addition of high-molecular weight polymer, where a single polymer can adsorb to two or more particles, physically holding them together. It is normally extremely rapid, is effective at polymer concentrations as low as 1 ppm, and gives rise to aggregates that clearly show floc morphology. Typically, very high molecular weight polyacrylamides are used to flocculate aqueous dispersions, where a relatively small proportion of the residues in the polyacrylamide are derived either from anionic species (e.g., sodium acrylate) or cationic species (e.g., trimethylammoniumethyl acrylate chloride). In contrast to the other flocculation methods discussed, short-range order has been observed in SANS measurements of flocs prepared by bridging flocculation[6,7]. Bridging flocculation gives rise simultaneously to a diffuse, low-density floc on the macroscopic scale, and a fluid where particles of the disperse phase (if relatively disperse) show significant short-range order

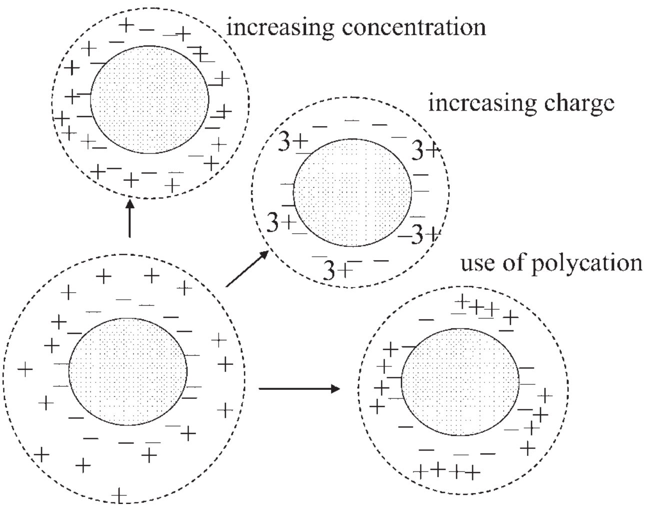

Figure 3.
Three strategies for enhancing destabilisation of a electrostatically stabilised colloid by addition of counterions.

on the microscopic scale. A benefit of using a polymer is that an individual chain will have very numerous attractive interactions with a given particle, making complete dissociation entropically unfavourable; the chemical specifics of the polymer-particle interaction should be of comparatively little importance, as long as they are favourable to some degree. Bridging flocculation is therefore essentially a physical process, as has been demonstrated in studies showing that anionic, cationic, and non-ionic polyacrylamides all show a degree of effectiveness in flocculating primary cane sugar juice[8] (Figure 4).

The term 'bridging' flocculation implies that the dynamics of chain relaxation are important, as shown in Figure 5; for a polymer to act as an effective bridge between particles, it must remain extended significantly beyond the double layer thickness for a time that is greater than the average time between interparticle collisions[9]. Effective bridging flocculation would therefore be predicted for higher molecular weight chains, for chains that occupy a larger volume in solution at a given molecular weight, and for chains that relax relatively slowly (e.g., have substituents that show a relatively high degree of *intrachain* interactions between near neighbours that must be disrupted for chain collapse, or are very bulky). Under conditions of rapid chain relaxation, bridging flocculation will be expected to be relatively ineffective. However, under conditions such that relaxation is relatively slow, more effective bridging flocculation would be expected.

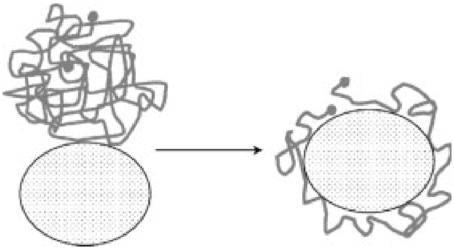

Figure 5.
Relaxation of a polymer onto a particle surface.

Additional characteristics of systems where bridging flocculants are used for clarification (municipal and industrial wastewater treatment, clarification of cane sugar juice, and other extractive industries) are the presence of a relatively high concentration of counter-ions and a corresponding relatively high rate of coagulation in the absence of a bridging flocculant. The role of the bridging flocculant is to *accelerate the rate* of aggregation and *increase the size* of the aggregates formed to encourage settling. Given these functions, we need to look at the effect of flocculant addition not only on primary particle aggregation, but on the behaviour of larger scale aggregates, i.e., microflocs.

While bridging flocculation is frequently illustrated in such a way as to suggest that the dimensions of the polymer random coils are small in comparison to the dimensions of the flocculated particles, e.g.,[10], this is not likely to be true for many practical

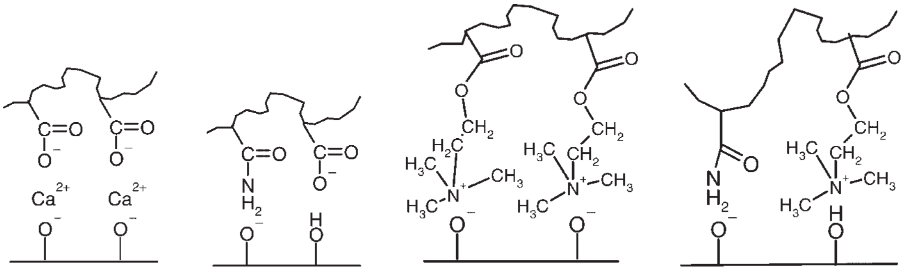

Figure 4.
Binding of anionic and cationic polyacrylamide-based flocculants to anionic surfaces (e.g., amorphous calcium phosphate, phenolics, flavonoids, silica) present in cane sugar juice.

systems where bridging flocculation is employed. For example, in primary cane sugar juice the suspended particles targetted by addition of an anionic polyacylamide flocculant are typically 500 to 5000 m in diameter[11]. This may seem large in comparison to the random coil diameter of typical polymers, but the flocculants used have both a very high molecular weight and a large value of the Mark-Houwink parameter a. Typical dimensions may be calculated as follows.

The intrinsic viscosity $[\eta]$ of a solution of spherical particles is related to the volume of the individual spherical particles V_H by the relationship

$$V_H = \frac{[\eta]M}{2.5N_A}$$

where M is the molecular weight, N_A is Avogadro's constant, and the intrinsic viscosity is related to the molecular weight using the empirical Mark-Houwink parameters K and a.

$$[\eta] = K\mathrm{M}^a$$

The hydrodynamic radius of a polymer random coil in solution, R_H, is therefore given by:

$$R_H = \sqrt[3]{\frac{3KM^{a+1}}{10\pi N_A}}$$

The relationship between the radius of gyration of the polymer random coil, $\mathrm{R_G}$, and $\mathrm{R_H}$ is empirical. Below the theta temperature, R_G will be approximately 1.2 R_H for freely draining coils according to the frequently employed Flory constant[12]. The most probable distance of a polymer segment from the centre of mass of the random coil will be $2R_G$.

Given the Mark-Houwink parameters for poly(acrylamide) $K = 6.31 \times 10^{-5}$, $a = 0.80$ (0.10 M NaCl, 30 °C)[13], and alternative values $K = 5.31 \times 10^{-5}$, $a = 0.79$ (0.12 M NaCl, 30 °C)[14], the 'effective diameter' $4R_G$ of a random coil can be estimated to be 106–116 nm for a chain of molecular weight 4×10^5, and 920-1020 nm for a chain of molecular weight 1.5×10^7. Asselman and Garnier have estimated R_G values ranging from 50 nm ($M = 4 \times 10^5$) to 340 nm ($M = 1.5 \times 10^7$) for commerical cationic polyacrylamides[9]. Because of the similar dimensions of the polymer coils and the flocculated particles, it is reasonable to consider the process of bridging flocculation not so much as a process in which particle surfaces become covered with polymer as a ***heterocoagulation***, in which aggregation of polymer coils and particles is favoured over coil-coil or particle-particle aggregation, but where coils and suspended particles are of comparable size and occupy comparable total volumes.

In bridging flocculation, a relatively small amount of charge, if any, is added to the sol: hence, the flocculated particles will have the full degree of interparticle repulsion that they would show as independent entities. As they exist within a network of polymer chains that can readily respond to stresses, it can be expected that they will rearrange themselves to minimise the repulsive forces between them, and if relatively monodisperse would be expected to give an ordered array of particles (see Figure 1(b)). The higher the molecular weight of the bridging polymer, the greater capacity it has to rearrange to absorb stresses, and hence the more robust the aggregates formed. The dynamic response of the polymer chains to shear will prevent the breakup of aggregates as they are formed, giving a low-density homogeneous structure (Figure 1(b)) rather than the dendritic initial aggregates or final disordered dense particles formed by a strictly diffusion-limited aggregation process.

Kinetics of Flocculation: Orthokinetic and Perikinetic Mechanisms

Whether the repulsive force between particles in a sol is electrostatic or steric in origin, it will give rise to a potential barrier that must be overcome for aggregation to take place. For an electrostatic barrier, this repulsion will be supplied largely by a diffuse atmosphere of counter-ions (see above) and so would be expected to repel a charged bridging flocculant of opposite charge to the particles. Steric stabilisers such as the humic and fulvic acids commonly adsorbed onto the surface of clay particles in untreated freshwater sources, or the proteins and polysaccharides adsorbed on particles in cane sugar juice, will clearly repel polymer coils. Hence, it is unreasonable to expect that the polymer in bridging flocculation is simply thrown out across an electrical double layer like a rope thrown between two boats.

Thus, both polymer coils and multiparticle aggregates will still need to overcome some potential barrier. The force that serves to overcome this potential barrier between particles will not be the same for all particle sizes. For relatively small particles, random thermal energy (Brownian motion) will supply the energy to enable the barrier to be overcome: such a mechanism of aggregation is called perikinetic flocculation. The collision cross section, β, between two spherical particles of diameter d_1 and d_2 is proportional to $(1/d_1 + 1/d_2)\ (d_1 + d_2)$, which will be largest for particles where there is a large difference between d_1 and d_2[15].

For larger particles, the displacement due to Brownian motion becomes much less and the primary force bringing about aggregation is differential shear: a particle must move at a single velocity, no matter what its dimensions, and hence where there is a distribution of velocities in a fluid the surface of the particle will be moving at slightly different speeds relative to the bulk motion of the fluid. This aggregation of particles with different centre-of-mass velocities whose surfaces can come into contact is known as orthokinetic flocculation. The collision cross section β between two spherical particles of diameter d_1 and d_2 in this case is proportional to $(d_1 + d_2)^3$, and hence will be weakly selective for particles of equal size.

Finally, when particles are sufficiently large and dense to move under the influence of gravity, aggregation by differential shear will be surpassed by aggregation by differential sedimentation. Both differential shear and differential sedimentation are subject to a hydrodynamic interaction ('bow wave' effect) that increases their tendency to select for equal-sized particles: the fluid in front of a moving particle will be pushed out of the way, taking small particles that might otherwise have collided with the large particle with it. For shear, the reduction in the cross section is given by the empirical expression[16]

$$\beta_{\mathrm{new}}/\beta_{\mathrm{old}} = 8/(\lambda+1)^3 \times \mathrm{e}^{(4.5+3.5\log\gamma+\lambda(20.7+11.5\log\gamma)}$$

Where

$$\lambda = d_2/d_1$$

and

$$\gamma = A/18\pi\eta G(d_1)^2$$

Here A is the Hamaker constant, η is the viscosity of the suspension, and G is the shear applied.

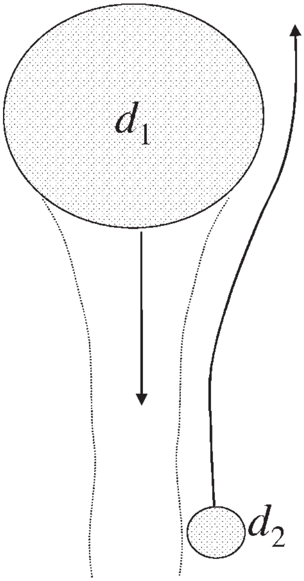

Figure 6.
Hydrodynamic interaction: the 'bow-wave' effect in particle aggregation.

Mode of Action of Polymer

What is the expected effect of adding a large population of low density particles (polymer coils) to a sol, *independent* of any specific interaction between particles and polymer coils that may increase the aggregation rate?

(1) There will be an increase in collisions due simply to the increase in the number concentration of particles, N_C.
(2) Because adsorbed polymers will occupy a reasonable volume, and because the continuing electrostatic repulsion between particles bridged by polymers will maintain a separation between them, aggregates generated by bridging flocculation will move more rapidly from the perikinetic to the orthokinetic domain than aggregates generated by other mechanisms.
(3) Because the aggregates formed will consist largely of fluid with a small amount of polymer, they will be of low density, and hence will resist moving from the orthokinetic domain to the domain of differential sedimentation.
(4) To the extent that aggregates formed are free-draining, the bow-wave effect would be expected to be reduced, increasing the collision cross section for orthokinetic flocculation.
(5) While in the orthokinetic domain, aggregation will be selective for aggregates of similar size. This can be expected to have two effects:

All the polymer may well be incorporated into macroscopic flocs before the smaller particulate material can be flocculated (a very common phenomenon in practical bridging flocculation).

Selection for aggregation between particles of similar size will lead to a degree of self-similarity, generating dendritic structures. This morphology will persist only on the macroscopic scale, where polymer relaxation is too slow to bring about homogeneity, generating the floccules typical of bridging flocculation. While this model is consistent with the observed features of bridging flocculation, it is not readily amenable to direct experimental testing.

Hogg has reached similar conclusions by considering flocculation as a process governed by irreversible polymer-particle collisions and modelling this absorption process[17]. Polymer will be adsorbed rapidly, primarily to larger particles, making high molecular weight polymer unsuitable for effectively flocculating populations of small, stable particles. These small particles can normally be separated effectively only if pre-assembled by high concentrations of another flocculant, commonly of lower molecular weight and of opposite charge, which will henceforth be referred to a coagulant. Use of two populations of polyelectrolytes (frequently of opposite charge), one a coagulant and one a flocculant has previously been employed in a range of industries with good results[18–21]; this is the process of dual flocculation. The primary role of the coagulant is to assemble small particles together into aggregates that have a higher affinity for the second flocculant, enabling more efficient adsorption and bridging[22]. Alternatively, the coagulant may conceivably target a second population of particles that do not interact strongly with the flocculant, either on the basis of size or surface characteristics. Within a model where adsorption kinetics alone determine flocculation behaviour, there would appear to be little scope for adjusting such a system in order to reduce the amount of coagulant required, or to improve the ability of the large flocs to capture unaggregated small particles. The physical model suggested above, however, suggests that flocculation of small particles that are missed by formation of the initial aggregates may be improved in two ways: (1) using polymers that are more likely to give free-draining aggregates, which will have a reduced bow-wave effect: this could be achieved by using no more charge than is necessary, by hindering chain motion with bulky substituents to retard polymer chain collapse, or (2) generating loose, rather than dense, aggregates with the coagulant, which will be large and low-density enough to participate in orthokinetic flocculation. This could be achieved by again using a relatively high molecular weight, slowly collapsing coagulant. The use of inorganic cationic microparticles rather than a polymer coagulant[23] may also generate a more open framework, due to the relative incompressibility of the particles and the more heterogeneous charge distribution unavoidably obtained in such a system. Such relatively large and porous primary aggregates also have practical advantages in the dewatering of flocculated material, e.g., in the papermaking industry[24].

Optimising Flocculation in The Sugar Industry

Cane sugar juice is a heterogeneous material containing a range of particles varying in size from tens of nm to the macroscopic scale. These include silica particles, clay, polysaccharides, and proteins, with anionic surfaces predominating. Cane sugar juice clarification based on simple defecation uses a process of sweep flocculation by amorphous calcium phosphate, generated by adding lime/lime saccharate to the process stream at concentrations sufficient to adjust the pH to between 7.8 and 8.0. Anionic polyacrylamides are customarily added in order to accelerate the rate of settling and increase the strength of the flocs formed, at a concentration of 3 ppm or less.

We have attempted to use the paradigm outlined in the section above to guide our synthetic work in the preparation of flocculants for application in the cane sugar industry[25]. Rather than vary the anionic flocculants currently used, our experiments on optimising this system have employed dual flocculation (i.e., both cationic and anionic polymers) in laboratory-scale trials[25].

A preliminary empirical study of a range of commercially available cationic polymers as coagulants[26] identified low molecular weight polymers of triemethylammonium ethyl methacrylate (TMAEMAC), presumably operating by a charge annihilation mechanism, as effective in significantly improving cane sugar juice clarity at concentrations of 40 ppm and greater, but also gave positive results for high molecular weight polyacrylamides containing a perecentage of TMAEMAC or trimethylammonium ethyl acrylate (TMAEAC). Further experiments with a series of TMAEAC homopolymers gave improved clarification of cane sugar juice[27]. Again, better clarification was obtained for lower molecular weight polymers, but all were ineffective at concentrations below 20 ppm, suggesting that use of this cationic homopolymer gave 'classical' dual flocculation.

Of greater interest were the cationic copolymers which were more effective at higher molecular weights, suggesting they enhanced overall flocculation by a bridging mechanism. Further experiments were carried out using TMAEAC and TMAEMAC-containing poly(acrylamides) of varying molecular weights and cationicities[28] and the following trends identified: (1) Much better clarification was obtained at 40 ppm than 10 ppm, with meaningful but unimpressive improvements in clarity at 10 ppm; (2) Improvements in clarity were seen if the polymers were added before or after the addition of calcium saccharate, although improvements in clarity were generally better when the polymer was added after calcium saccharate. This suggests that adsorption of the cationic polyacrylamide to particles is not an absolute limiting factor in its flocculation effectiveness; (3) Better clarity, and reduced settling rate, were seen with increasing cationicity and with lower intrinsic viscosity (Figure 7); (4) while the difference between polymers containing methacrylate and acrylate based cationic monomers was small, TMAEAC-containing polymers were generally more effective in improving clarity than TMAEMAC-containing polymers of equivalent intrinsic viscosity, particularly at high concentrations after liming (Figure 8).

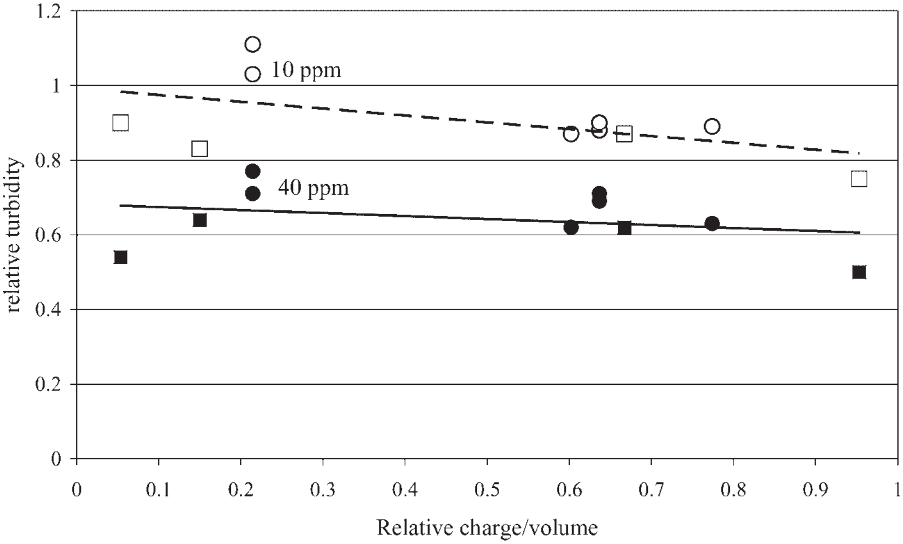

Figure 7.
Relative turbidity (absorption at 900 nm of dual-flocculated sample, divided by the absorption at 900 nm of a control flocculated using anionic polyacrylamide only) for 10 ppm (open symbols) and 40 ppm (filled symbols) cationic polyacrylamide added after liming, containing either TMAEAC (■,□) or TMAEMAC (●,○). Data are plotted as a function of relative charge per unit volume of polymer coil, assuming $V_H \propto [\eta]^{1.8}$ (i.e., $a = 0.8$).

These trends are consistent with the cationic polyacrylamide acting by a bridging flocculant, more or less independently of the anionic flocculant. The tradeoff between higher settling rates and lower clarity as molecular weight increases is very common for bridging flocculants, and is adequately explained by the models above; this trend would be expected to be more marked for TMAEMAC-containing copolymers, which will be expected to relax more slowly on adsorbing to particles and

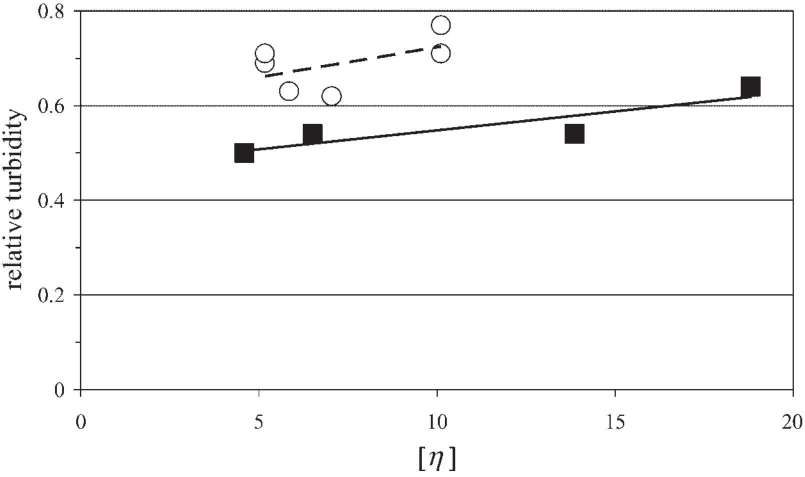

Figure 8.
Relative turbidity (absorption at 900 nm of dual-flocculated sample, divided by the absorption at 900 nm of a control flocculated using anionic polyacrylamide only) for 40 ppm cationic polyacrylamide added after liming, containing 15–30% either TMAEAC (■) or TMAEMAC (○).

hence maintain a larger molecular coil size over time than the TMAEAC-containing copolymers.

Under the conditions employed, the cationic flocculants clearly form primary aggregates which can readily be assembled by the anionic flocculant, whether they are added before or after the formation of amorphous calcium phosphate in the system. While the trends in behaviour with variations in cationic polyacrylamide suggest that the primary aggregates are relatively large and low density, the aggregates expected to be less dense on the basis of polymer size are not significantly more effective in adsorbing additional small particles once formed (Figure 8). A weak trend is seen for greater effectiveness in reducing turbidity for polymers that have a high ratio of charge to random coil volume, which might be expected to relax to weakly-charged surfaces most slowly (Figure 7). These data are insufficient as a basis for any confident mechanistic conclusions, but they are not inconsistent with the mode of bridging polymer action outlined here. Clearly, there is a need for additional work on well-defined model systems less complex and heterogeneous than primary cane sugar juice.

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