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(54) Title: DEWATERING OF AQUEOUS SUSPENSIONS

(57) Abstract

Separation of solids from an aqueous suspension by sedimentation is improved by mixing a cationic polymeric flocculant into the suspension, aggregating the flocculated material by mixing an anionic colloidal material into the flocculated suspension, and then separating the aggregated material by sedimentation. The suspension is preferably a sewage suspension and the process is preferably a primary sedimentation stage. The cationic polymeric flocculant is preferably a low molecular weight coagulant polymer, especially a diallyl dimethyl ammonium chloride polymer. A high molecular weight, generally anionic, flocculant can be added after the anionic colloidal material and before the sedimentation.

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Dewatering of Aqueous Suspensions

This invention relates to a process for separating suspended solids from an aqueous suspension and which includes a solids-liquids separation stage that is conducted by sedimentation, and is of particular value in the sedimentation of sewage.

It is well known to add a flocculant material to a suspension so as to cause the suspended material to flocculate before solids-liquids separation. Suitable flocculants for this purpose include alum and other polyvalent metal salts, bentonite, and various natural or synthetic polymers that can be non-ionic, anionic or cationic.

In this specification, for convenience, we use the term "flocculation" as a generic term to embrace processes that might more accurately be referred to as coagulation and processes that are conventionally referred to as flocculation and which probably involve bridging flocculation. In the coagulation processes, charge neutralisation is a major effect and if the coagulant (flocculant) is a polymeric material then the polymer is generally of relatively low molecular weight and high cationic charge density. In the conventional flocculation processes (referred to herein as "bridging flocculation") the polymeric flocculant probably acts primarily by a bridging mechanism and so is generally of relatively high molecular weight. The terms "flocculant" and "flocculate" are used in a similar generic sense.

Although it is well known, as a generality, to use a polymeric flocculant to flocculate a suspension before subjecting it to a solids-liquids separation process there is still a very large amount of skill required in selecting optimum polymeric materials and use conditions for particular processes. For instance processes known for improving the retention of fibres during the manufacture of paper would not be thought to be relevant to the improvement of sewage sedimentation processes.

When sedimenting sewage or similar suspensions it is desirable that the sedimentation should occur at a reasonable speed to produce a reasonably dense sediment and a reasonably clear liquor. It is also desirable that a process should be reasonably versatile in the sense that it should produce reasonable results even if the sewage quality or conditions varies, for instance due to a change in temperature or a temporary change in the quality of the sewage. It is desirable that the biological oxygen demand and the chemical oxygen demand of the effluent liquor should be as low as possible. Various coagulation and bridging flocculation treatments, and combinations, have been proposed in an attempt at improving results. However there is a continuing need for further improvement in one or more of these respects.

It is known to promote sedimentation in the primary sedimentation stage by the addition of bentonite or by the addition of inorganic coagulants such as aluminium sulphate, optionally with a polymeric bridging flocculant. Also, it is known (for instance EP 235893) to improve paper production (for instance as regards retention and drying) by including a cationic polymer in the suspension, shearing the suspension and then adding bentonite prior to drainage filtration of the suspension through a screen to produce paper. However this is of no relevance to sewage sedimentation.

Existing methods of promoting sewage sedimentation are not entirely satisfactory in that they tend either to be rather slow (and therefore need large apparatus) or to be rather inefficient in that the clarity of the resultant liquor is rather poor.

A process according to the invention for separating suspended solids from an aqueous suspension comprises flocculating the suspended material by mixing a cationic polymeric flocculant into the suspension, aggregating the flocculated material by mixing an anionic colloidal material into the flocculated suspension, and separating the aggregated material from the suspension by sedimentation.

The invention can be applied to a wide variety of sedimentation processes where the flocculated particles are allowed to sediment under gravity either in a static tank or while flowing through a tank provided with a stack of plates or other surfaces to promote sedimentation. A preferred plate settler apparatus is described in EP-A-354744. Although the process can be used for, for instance, treating industrial effluents such as paper mill effluent or pigment effluent, the invention is preferably used for suspensions of organic material and is of particular value for sewage sedimentation stages in which raw sewage or a sewage sludge or biologically treated sewage is allowed to undergo sedimentation to separate solids from a clarified liquor.

A conventional process for treating raw sewage includes the following treatment stages:

(1) passing the sewage through screens and then settling to remove detritus;

(2) a first sedimentation stage to form a primary sludge, at which stage sewage solids which are mainly organic are removed as primary sludge and the biological oxygen demand (BOD) is reduced;

(3) a biological treatment stage to convert soluble and colloidal materials to separatable solids;

(4) a second sedimentation stage to remove said solids as secondary sludge; and

(5) effluent handling, for instance by being discharged without prior treatment or after treatment chemically and further separation by a tertiary treatment stage.

The invention is preferably applied to the first sedimentation stage, but can be applied to later stages in the process.

In preferred processes according to the invention we find it is possible to improve sewage treatment processes by adding a cationic polymer followed by the anionic colloidal material at a sedimentation stage. In particular, it is preferred to treat raw sewage by subjecting it to a primary sedimentation stage (generally after screening and detritus removal), and the cationic polymer and bentonite are added in this primary sedimentation stage.

The result of this process is the formation of a primary sludge and a clarified liquor that can then be subjected to biological treatment to convert soluble and colloidal materials in it to biomass which can then be separated from the liquor by a secondary sedimentation stage to form a secondary sludge and a secondary liquor that can either be discharged or subjected to a tertiary treatment. The treatment of the invention can, if desired, be applied to the second and/or third sedimentation stages as well as to the first.

The cationic polymer that is used in the invention can be a natural cationic polymer such as chitosan or a modified natural cationic polymer such as cationic starch. Preferably however the polymer is an organic synthetic polymer that is substantially water soluble and that is formed by polymerising one or more ethylenically unsaturated monomers, in generally acrylic monomers, that consist of or include cationic monomer. Suitable cationic monomers are dialkylaminoalkyl (meth) acrylates and dialkylaminoalkyl (meth) acrylamides, either as acid salts or preferably as quaternary ammonium salts. The alkyl groups may each contain 1 to 4 carbon atoms and the aminoalkyl group may contain 1 to 8 carbon atoms. Particularly preferred are dialkylaminoethyl (meth) acrylates, dialkylaminoethyl (meth) acrylamides and dialkylaminopropyl (meth) acrylamides. These cationic monomers are preferably copolymerised with a non-ionic monomer, preferably acrylamide. Cationic amphoteric polymers (including a minor amount of anionic groups) can also be used. Preferred polymers are particulate as in EP 202780.

Various other cationic polymers that may be used include polyethylene imines, dicyandiamide polymers, polyamine epichlorhydrin polymers and polymers of diallyl monomers such as diallyl methyl ammonium chloride (DADMAC), either as homopolymer or copolymer with acrylamide or other comonomer.

It is preferred that the cationic polymer that is added to the suspension before the anionic colloid should have a sufficiently low molecular weight that the flocculation process can more properly be described as coagulation. Preferably the molecular weight of the polymer is such that IV is not above 3dl/g, e.g., 0.2 to 2dl/g, or molecular weight 50,000 to 2 million. (IV is intrinsic viscosity measured at 25°C in IN sodium chloride solution.) Suitable low molecular weight polymers of this type are low molecular weight versions of polymers discussed above and in particular preferred polymers for use in the invention include polyamines, polyethylene imines, polymers of diallyl dimethyl ammonium chloride (for instance as homopolymer or copolymer containing a minor amount of acrylamide) and polymers of dialkylaminoethyl acrylate or methacrylate acid addition or quaternary salt, generally as homopolymers or copolymers with acrylamide.

The cationic polymer generally has a relatively high charge density, for instance above 0.2 and preferably 0.4 to 2.5 equivalents of catonic nitrogen per kilogram of polymer. When the cationic polymer has IV below about 3dl/g the cationic content is preferably relatively high. For instance the polymer may be a substantial homopolymer or formed from a monomer blend at least 50% and generally at least 80%, by weight cationic monomer, any remaining monomer generally being acrylamide or other non-ionic monomer.

The use of DADMAC polymers, especially homopolymers or, less preferably, copolymers with up to 20% or even 50% acrylamide, is especially preferred.

The low molecular weight polymers can be of the type that are available commercially as solutions, but it is particularly preferred that they should have IV above 1dl/g and that they should be of the type that is commercially available as beads since it seems better results are obtained using these bead polymers. They may be dissolved before addition to the sewage or they may be added as beads, for instance as published in WO92/00247.

In some instances it is desirable to include a relatively low molecular weight cationic polymer (for instance a relatively highly charged cationic polymer having molecular weight 50,000 to 2 million) as a coagulant followed by a higher molecular weight cationic polymer, for instance to act as a bridging flocculant, followed by the anionic colloidal material. In other instances it is suitable to use a bridging flocculant alone. The bridging flocculant is preferably a polymer made from ethylenically unsaturated monomers as described above and having an IV of at least 4dl/g. Often the amount of cationic monomer is 8 to 40, often around 10 to 20, mole percent with the balance being acrylamide.

Particularly preferred processes of the invention involve flocculation with a relatively low molecular weight cationic polymer (i.e., coagulation) followed by the addition of the anionic colloidal material, followed by the addition of further polymeric flocculant before the sedimentation. This further polymeric flocculant is preferably a bridging flocculant and so is preferably of high molecular weight, for instance IV above 4dl/g. It can be a cationic flocculant in which event it generally has been formed from 3 to 25, preferably 8 to 15, mole percent cationic monomer with the balance acrylamide. Preferably however it is a non-ionic flocculant or an anionic flocculant, usually formed from 3 to 25 mole percent anionic monomer, for instance sodium acrylate or other carboxylic or sulphonic ethylenically unsaturated monomer, copolymerised with acrylamide or other suitable non-ionic monomer.

Although the process can be conducted with relatively low degrees of mixing, with the result that there is little or no degradation of the initial flocs before the anionic colloidal material is added, it is often preferred to stir sufficiently to break down any large particles that may have aggregated before adding the anionic colloidal material. In some instances it may be desirable to achieve an overall reduction in floc size by applying sufficient stirring or other agitation to the flocculated suspension. Naturally the extent of agitation must not be so great that the initial solids are resuspended in the aqueous suspension but it is possible, particularly if relatively large amounts of the cationic polymer flocculant are used, to choose a degree of agitation that breaks the initial flocs down to microflocs that are stable in the system against further reduction in size, and which are then aggregated by the anionic colloidal material. An advantage of reducing the floc size in this manner is that it can facilitate separation to a higher solids content compared to the solids content than is conveniently available if the flocs do not undergo size reduction before adding the anionic colloidal material. Size reduction of the flocs by agitation is particularly desirable when the polymer has IV above 4dl/g.

The addition of cationic polymer is made at some position ahead of the clarifier or other sedimentation equipment and anionic colloidal material is added after the polymer addition but before the sedimentation stage, and preferably bridging flocculant is added after the anionic colloid but before sedimentation. There must be sufficient interval between the first two points of addition to allow flocculation to occur and, as indicated above, it can be desirable to apply sufficient mixing or other shear between the two points of addition so as to break down the initial flocs that are formed into microflocs.

The anionic colloidal material can be an emulsion of water-insoluble anionic organic polymer, or it can be an anionic inorganic polymer such as colloidal silicic acid, but preferably it is an anionic swelling clay. Such clays are known to swell to a very large extent when the dry clay is contacted with water. They are generally smectites and are frequently montmorillonites. Suitable materials are referred to as bentonites such as Wyoming bentonite, or Fullers Earth. The bentonite or other clay may have been chemically modified, e.g., by alkali treatment to convert calcium bentonite to alkali metal bentonite. The bentonite or other clay preferably swells by at least 10 or 20 times its own volume and the dry clay is contacted with water and the surface area of the anionic colloid before swelling is preferably at least $30m^2/g$ and the surface after swelling is preferably at least 400, e.g, up to $800m^2/g$.

The bentonite or other clay may be provided as a powder that is mixed with water to form a slurry for convenient addition to the aqueous suspension, or may initially be provided as a slurry, and this in turn may be provided as a concentrated slurry that contains low provided as a concentrated slurry that contains low molecular weight sodium polyacrylate or other dispersing agent or other material that will render the concentrate sufficiently fluid for ease of handling.

The amounts of cationic polymeric flocculant and anionic colloidal material used in the invention will depend upon the suspension that is being treated and the degree of agitation that is applied and the nature of the suspension and of the sedimentation process. The optimum can be determined by routine screening. If the flocculated suspension is to be agitated significantly prior to adding the anionic colloidal material, it is desirable to include that are present after the agitation all carry a relatively heavy cationic charge due to the flocculant polymer.

The amount of polymer that is included in the suspension is generally at least 1ppm or 2ppm (based on the volume of suspension) and is generally at least 5 or 10ppm. It can be up to, for instance, 500ppm but the amount is generally below 100ppm and is frequently below 50ppm. Values of 2 to 50, often around 2 to 30, mg/l are often preferred. These are all based on the total weight of suspension. Based on the solids content of the suspension, the amounts typically are at least 0.1% and often at least 0.5%, but generally below 5% and often below 2%, by weight of the solids content of the dispersion. If too much polymer is used, the disadvantages of overdosing will occur.

The amount needed for optimum results may be reduced if the suspension already contains cationic polymer from a previous stage, for instance coagulant polymer may already be in the suspension in which event the amount of low molecular weight polymer that should be added can be reduced.

The amount of anionic colloidal material is generally at least 10ppm and usually at least 30ppm and preferably at least 50ppm, based on the volume of the suspension that is to be sedimented. The amount is generally below 500ppm and frequently it is below 250ppm. Amounts around 20 to 200mg/l with best results often in the range 30 to 100mg/l are often preferred. Based on the solids content of the suspension, the amounts typically are above 0.1% and generally above 0.3% and often above 1%, but the amount is generally below 10%, and preferably below 3% by weight.

The solids content of the liquor that is treated is normally typical for primary sedimentation liquors, for instance 100 to 400ppm, but if the liquor is a sludge then the solids content will be higher, tyically 2,000 to 8,000ppm.

By the invention it is possible to obtain rapid separation of relatively firm solids leaving a liquor of improved clarity and reduced biological oxygen demand.

The following test procedure was used in each of the examples:

1. addition of coagulant to sewage;
2. 40 seconds flash mix;
3. 90 seconds coagulation;
4. addition of bentonite;
5. 30 seconds flash mix;
6. 270 seconds flocculation;
7. addition of flocculant;
8. 30 seconds flash mix;
9. 150 second flocculation;
10. 300 seconds settlement.

The test as a whole typically takes around 10 minutes.

The products used in the following examples were:

As coagulants: (low molecular weight flocculant)

A: bead polymer of diallyl dimethyl ammonium chloride having an IV of 1-2.5, in the form of a 1% solution or added as beads direct to the sewage.

B: polyamine in the form of a 1% solution.

C: solution polydadmac having an IV of around 1.4, in the form of a 1% solution.

0.5% slurry of bentonite was used.

As bridging flocculant: D: low anionic high molecular weight polyacrylamide, in the form of a 0.05% solution.

In each of the examples, each run is given a relative order of effect of magnitude 1-4 as compared to the other runs in that example, 1 being the best and 4 being the worst. The order of effect in each example is independent of that in every other example.

Example 1

This example investigates the combined effect of coagulant and bentonite as compared to their separate effects. The results are shown in Table 1.

Table 1

Run	A mg/l	Bentonite in mg/ l	D mg/ l	Order of Effect
1	20		0.5	3
2	40		0.5	4
3		40	0.5	2
4	20	40	0.5	1

Run 2, having an order of effect of 4, was the worst run in this example and illustrates that a dosage of 40ppm A overdosed the system. The presence of D (the flocculant) had minimal effect when subsequently added to the A floc but in contrast worked relatively well on the A + bentonite flocc.

The run in which A and bentonite were used in combination produced a very heavy floc with a very low turbidity supernatant.

Examples 2 and 3

In these two examples the amount of coagulant used in the test procedure was varied while maintaining the amount of organosorb and D at a constant level. The results are shown in Tables 2 and 3 respectively.

Table 2

Run	A mg/l	Bentonite in mg/ l	D mg/ l	Order of Effect
1	5	40	0.5	4
2	10	40	0.5	3
3	15	40	0.5	2
4	20	40	0.5	1

Table 3

Run	A mg/1	Bentonite in mg/ 1	D mg/ 1	Order of Effect
1	1	40	0.5	3
2	3	40	0.5	2
3	5	40	0.5	1
4	0	40	0.5	4

As can be seen from Table 2, results improved as the amount of A was increased. Although Run 1 had a rating of 4, this is only relative to the other runs in that example and the test illustrated that a concentration of A of 5mg/l in the invention was still effective.

From Table 3 it can be seen that the concentration of A of 3mg/l in the invention is still effective, whereas concentrations of around 1 mg/l and lower are of little use in the invention.

Considering both the results of examples 2 and 3 together, and from visual observation of the results obtained in each run, it was concluded that a concentration of A of between 3-5 mg/l was optimum.

Example 4

In this example the type of coagulant was varied and the results are shown in Table 4.

Table 4

Run	Coagulant in mg/l	Bentonite in mg/ 1	D mg/ 1	Order of Effect
1	3 A	40	0.5	1
2	3 B	40	0.5	4
3	3 C	40	0.5	3
4	5 C	40	0.5	2

Each of the coagulants worked in the invention, but the results showed that A was the optimum coagulant for the process, and even a 5 mg/l dosage of C did not produce as good as results as a 3mg/l dosage of A.

Example 5

This example investigated the effect of varying the concentration of bentonite used in the process of the invention.

Table 5

Run	A mg/l	Bentonite in mg/ l	D mg/ l	Order of Effect
1	3	10	0.5	4
2	3	30	0.5	3
3	3	60	0.5	2
4	3	90	0.5	1

The results indicated that in this example a bentonite dosage of greater than 30mg/l was necessary to attain the required level of flocculation, and decrease in turbidity, with 3mg/l dosage of A. Greater order of effects were obtained for bentonite doses of 60 and 90mg/l, however the effect of both these doses are only slightly better than that obtained when using a 40mg/l dose.

CLAIMS

1. A process for separating solids from an aqueous suspension by mixing a cationic polymeric flocculant into the suspension and then separating the flocculated solids from the suspension by sedimentation, characterised in that the flocculated solids are aggregated by mixing an anionic colloidal material into the flocculated suspension and the suspension containing the aggregated solids is subjected to the sedimentation.

2. A process according to claim 1 in which the aqueous suspension is a sewage suspension.

3. A process according to claim 2 and which is a primary sedimentation stage to form a primary sludge.

4. A process according to any preceding claim in which the cationic polymeric flocculant comprises polymer that has IV 0.2 to 3dl/g and is formed from monomer of which at least 50% by weight is cationic.

5. A process according to claim 4 in which the polymer is selected from polyamines, polyethylene imines, polymers of diallyl dimethyl ammonium chloride, and polymers of dialkylaminoethyl acrylate or methacrylate acid addition or quaternary salt.

6. A process according to claim 4 in which the polymer is a homopolymer of diallyl dimethyl ammonium chloride or a copolymer with up to 50 mole percent acrylamide.

7. A process according to claim 6 in which the polymer has IV at least 1dl/g and is supplied as a bead polymer.

8. A process according to any of claims 4 to 7 in which a polymeric flocculant polymer having IV at least 4dl/g is mixed into the suspension after the anionic colloidal material and before the sedimentation.

9. A process according to claim 8 in which the polymeric flocculant having IV above 4dl/g is selected from non-ionic and anionic flocculants.

10. A process according to any preceding claim in which the anionic colloidal material is an anionic swelling clay.

INTERNATIONAL SEARCH REPORT

International Application No ’CT/GB 92/01431

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)[6]

According to International Patent Classification (IPC) or to both National Classification and IPC

Int.Cl. 5 C02F1/52

II. FIELDS SEARCHED

Minimum Documentation Searched[7]

Classification System	Classification Symbols
Int.Cl. 5	C02F

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched[8]

III. DOCUMENTS CONSIDERED TO BE RELEVANT[9]

Category °	Citation of Document, [11] with indication, where appropriate, of the relevant passages [12]	Relevant to Claim No.[13]
X	GB,A,1 188 394 (FOSECO INTERNATIONAL LIMITED) 15 April 1970 see the whole document ---	1-5
A,P	US,A,5 071 587 (PERMAN,C.A.) 10 December 1991 see claims 1-4,21 -----	1,10

° Special categories of cited documents : [10]

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"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

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IV. CERTIFICATION

Date of the Actual Completion of the International Search 13 NOVEMBER 1992	Date of Mailing of this International Search Report 07.12.92
International Searching Authority EUROPEAN PATENT OFFICE	Signature of Authorized Officer GONZALEZ ARIAS, M.L.

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO. GB 9201431 SA 62920

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.
The members are as contained in the European Patent Office EDP file on 13/11/92
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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
GB-A-1188394	15-04-70	None	
US-A-5071587	10-12-91	WO-A- 9118838	12-12-91

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82