(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
2 April 2009 (02.04.2009)

PCT

(10) International Publication Number
WO 2009/040166 A1

(51) International Patent Classification:
C02F 11/14 (2006.01) *C02F 1/48* (2006.01) *C02F 1/56* (2006.01)

(21) International Application Number: PCT/EP2008/059925

(22) International Filing Date: 29 July 2008 (29.07.2008)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
0718696.8 26 September 2007 (26.09.2007) GB

(71) Applicant *(for all designated States except US)*: **CIBA HOLDING INC.** [GB/GB]; Patents Department, PO Box 38, Bradford, West Yorkshire BD12 OJZ (GB).

(72) Inventors; and
(75) Inventors/Applicants *(for US only)*: **ADKINS, Stephen** [GB/GB]; 6 Bronte Close, Gomersal, Cleckheaton West Yorkshire BD19 4PU (GB). **OLDFIELD, Simon** [GB/GB]; 34 Coronation Street, Elland, Halifax West Yorkshire HX5 0DF (GB). **TIMMONS, Alban** [GB/GB]; 7 Cartwright Lane, Beverley Humberside HU17 8NB (GB). **THOMPSON, John** [GB/GB]; 12 Lakeside, Funtley, Fareham Hampshire PO17 5EP (GB).

(81) Designated States *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States *(unless otherwise indicated, for every kind of regional protection available)*: ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MT, NL, NO, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:
— *with international search report*

WO 2009/040166 A1

(54) **Title:** TREATMENT OF AN AQUEOUS SUSPENSION OF SOLID PARTICLES

(57) **Abstract:** The invention relates to a process of concentrating or dewatering an aqueous suspension of solid particles comprising the steps of, providing an aqueous polymeric flocculant composition or an aqueous polymeric coagulant composition by combining polymeric flocculant material or polymeric coagulant material with water, adding an effective flocculating or coagulating amount of the aqueous polymeric flocculant or coagulant composition into the aqueous suspension of solid particles thereby bringing about flocculation or coagulation of the solid particles, subjecting the so formed flocculated or coagulated suspension of solid particles to a concentrating or dewatering stage, wherein the aqueous polymeric flocculant or coagulant composition and/or the water which is to be combined with the polymeric flocculant or coagulant material is/are treated by a magnetic field and/or electric field with non-sinusoidalwave pulses at a sweep offrequencies. The invention also relates to a process of preparing an aqueous polymeric flocculant or coagulant composition.

Treatment of an Aqueous Suspension of Solid Particles

The present invention relates to an improved flocculation process for the concentration or dewatering of aqueous suspensions of solids. The process provides improved flocculant and coagulant performance.

It is known to concentrate suspensions of solids in aqueous liquids by use of flocculants resulting in flocculation of the solids which facilitates the separation of the solids from the liquid. In many processes the flocculated solids settle to form a bed by sedimentation. In other processes separation can be facilitated by mechanical dewatering, for instance in pressure filtration, centrifugation, by belt thickeners and belt presses.

It is well known to apply polymeric flocculants to aqueous suspensions in order to separate solids from the suspension. For instance it is common practice to flocculate and then dewater suspensions containing either suspended solid organic material or mineral solids. For instance it is common practice to flocculate sludges such as sewage sludge, waste waters, textile industry effluents, red mud from the Bayer Alumina process and suspensions of coal tailings etc. Flocculants are also commonly used in paper-making processes by addition of polymeric flocculants to the cellulosic suspension. Flocculation is usually achieved by mixing into the suspension polymeric flocculant, allowing the suspended particles to flocculate and then dewatering the flocculated suspension. In papermaking this removal of water from the cellulosic suspension is often referred to as draining.

High molecular weight polymeric flocculants are commonly used for this purpose.

High molecular weight flocculants may be cationic, anionic, nonionic or amphoteric in nature. The choice of polymeric flocculant will largely depend upon the substrate, which is being treated. For instance it is common practice to use high molecular weight cationic flocculants to treat aqueous suspensions comprising suspended organic material, for instance sewage sludge. In paper-making it is known to use any of cationic, nonionic, anionic or amphoteric flocculants. Flocculation of mineral suspensions is frequently effected by use of anionic flocculants.

The types of flocculant added to the suspension will often depend upon the substrate. Generally suspensions tend to be flocculated by high molecular weight polymers. Examples of this are described in WO-A-9314852 and US3975496 regarding the flocculation of mineral suspensions such as red mud. Other disclosures of high molecular weight polymeric flocculants include US 6447687, WO-A-0216495 and WO-A-02083258 dealing with the flocculation of sewage sludge. It is known to sometimes add other chemical additives to condition the suspension. For instance suspensions may be first coagulated by a high charge density polymeric coagulant such as polyDADMAC or inorganic coagulants including ferric chloride.

It is known to apply a magnetic field to aqueous suspensions in order to enhance precipitation of impurities. For instance, GB 2335656 describes a method for flocculating and precipitating impurities from a liquid by treating with a flocculating agent in the form of a metal salt and then subjecting the so treated liquid to a magnetic field with flat wave pulses at a sweep of frequencies. Square waves and triangular waves are proposed.

Japanese patent application 2002 307071 describes a process in which waste water is fed into a raw water tank followed by addition of a first inorganic flocculating agent which is passed to a tank in which a high molecular weight flocculating agent is added followed by passing into a coagulation sedimentation tank. A magnetic apparatus, preferably a permanent magnet, is positioned at any of the aforementioned stages.

WO-A-9703925 describes a method for treating wastewater by first addition of an anionic polymer and then passing the treated waste water through a magnetic field and wherein at least some of the contaminants flocculate. The flocculated contaminants are separated from the waste water.

US 4089779 and US 4110208 describe a classification method in which the precipitation of metal hydroxide is improved by adding ferromagnetic additive e.g. ferromagnetic lignosulfonate and then using a magnetic field to aid settling.

GB 1310321 describes dehydration of sludge by addition of flocculation aids. The sludge is treated with high frequency electrical or magnetic field before addition of the flocculation aids.

Soviet Union patent 1694479 describes a method of thickening red mud by subjecting a farinaceous flocculant mixture with alkali aluminate solution to a continuous magnetic field. This is followed by adding the mixture to the pulp and subjecting the resulting mixture to a magnetic field.

Soviet Union patent 1286533 describes the purification of wastewater by addition of aluminium containing coagulant followed by a dilute solution of polyacrylamide. The flocculant is prepared using water that has been subjected to a magnetic field and then to electrolysis where it is saturated with anodically dissolved iron and again subjecting to a magnetic field.

An objective of the present invention is to improve the flocculation and/or coagulation efficiency in solid liquid separation processes.

According to the present invention we provide a process of concentrating or dewatering an aqueous suspension of solid particles comprising the steps of,

providing an aqueous polymeric flocculant composition or an aqueous polymeric coagulant composition by combining polymeric flocculant material or polymeric coagulant material with water,
adding an effective flocculating or coagulating amount of the aqueous polymeric flocculant or coagulant composition into the aqueous suspension of solid particles thereby bringing about flocculation or coagulation of the solid particles,
subjecting the so formed flocculated or coagulated suspension of solid particles to a concentrating or dewatering stage,
wherein the aqueous polymeric flocculant or coagulant composition and/or the water which is to be combined with the polymeric flocculant or coagulant material is/are treated by a magnetic field and/or electric field with non-sinusoidal wave pulses at a sweep of frequencies.

The present invention also provides a novel process of preparing an aqueous polymeric flocculant composition or an aqueous polymeric coagulant composition comprising combining polymeric flocculant material or polymeric coagulant material with water,
wherein the aqueous polymeric flocculant or coagulant composition and/or the water which is to be combined with the polymeric flocculant or coagulant material is/are treated by a magnetic field and/or electric field with non-sinusoidal wave pulses at a sweep of frequencies.

In accordance with the present invention the non-sinusoidal wave forms may include any of the wave forms in this category, for instance square waves, rectangular waves, ramp waves, triangle waves, spiked waves and sawtooth waves. Preferably the non-sinusoidal waves are square waves.

Preferably the magnetic field and/or electric field is produced by a frequency modulated non-sinusoidal wave generator. In general this non-sinusoidal wave generator will comprise at least one electrical coil. However, in some cases it may be desirable to use more than one electrical coil.

Generally the frequency modulated non-sinusoidal, preferably square, wave pulses have fundamental frequencies within the audio bandwidth. Nevertheless, due to the shape of the pulsed wave form energy will tend to be emitted in other bandwidths, including for instance within the magnetic, ultrasonic, radio and/or microwave bandwidths.

Unexpectedly, we have found that improvements can be achieved when the water used to make up the aqueous flocculant/coagulant composition is hard water. We have found that in such cases the treatment of the water or aqueous flocculant/coagulant composition according to the present invention brings about significant improvements in solid liquid separation by comparison to carry out the process in the absence of the aforementioned treatment. Thus the invention can be of particular value in circumstances where it is normal practice to use hard water in order to make the flocculant or coagulant solution.

The water used to make the aqueous flocculant/coagulant composition may contain small amount of dissolved compounds. Typically such compounds may be those normally associated with hard water, for instance calcium salts, such as calcium carbonate or calcium bicarbonate. Other compounds that may be present include other bicarbonates, carbonates, sulphates or chlorides etc. Such compounds may be alkali metal salts, alkaline earth metal salts, aluminium salts, transition metal salts etc. Generally, these compounds will be present only been trace amounts. Typically, the amount of dissolved compounds present in the water will be less than 1% by weight of water and normally, it will be below 1000 ppm. Usual levels of calcium carbonate or calcium bicarbonate in hard water will be up to 600 ppm and more usually no higher than 300 ppm.

Nevertheless, other circumstances it may be more desirable if the level of dissolved compounds, such as calcium carbonate, is below this, for instance below 100 ppm, especially below 50 ppm, particularly below 25 ppm or less.

The aqueous polymeric flocculant or coagulant composition is particularly suitable for providing improvements in the process of concentrating or dewatering suspensions of solids.

Typically the non-sinusoidal wave pulse will include a sweep of frequencies and can be frequency modulated to between 300 and 21,000 Hz. Preferably, the spectrum of frequencies is often between 500 and 10,000 Hz and especially within the range between 700 and 3000 Hz. Normally, the power output will be within the range of 5 milliamps and 5 amps. Preferably the power output will be between 25 milliamps and 2 amps. By varying the power the strength of the wave form (including magnetic and/or electric field) can be varied.

The magnetic field generated will tend to be relatively low and in the order of below 5 Gauss ($5X10^{-4}$ Tesla), and generally below 2 Gauss. Typically the magnetic field will be at least 0.1 Gauss often at least 0.5 and generally around 1 Gauss. The treatment will generally be for a period of between 100 milliseconds to 3 hours or more. In all cases the treatment may be applied by passing the liquid/solution to be treated through the energy or magnetic field generated.

Preferably, the treatment incorporates a complex field consisting of energy from the magnetic, audio, ultrasonic, radio and microwave bandwidths, generated by a Frequency Modulated Square-Wave Generator with pulses with extremely sharp rising edges, based on the apparatus covered by European patent EP0357102 and British patent GB 2335656.

Generally at least one coil is placed around the vessel holding the aqueous flocculant or coagulant composition or water used to make this (fluid to be treated). Preferably the energy is applied by at least one solenoid coil applied around a pipe or other conduit carrying the aforementioned fluid to be treated. In general the vessel (e.g. pipe or other conduit) desirably may have several coils and typically up to 40 or 50 coils. However, it is preferred that there are between 12 and 16 coils around the vessel. Desirably the aforementioned frequency modulated frequencies should be applied.

The aqueous polymeric flocculant or coagulant composition and/or the water which is to be combined with the polymeric flocculant or coagulant material may be passed along a pipe or other suitable flow line which is provided with a suitable means for treating the said composition or water by the frequency modulated energy field. Alternatively, the aqueous polymeric flocculant or coagulant composition or the water which is to be combined with the flocculant/coagulant material may be held in a suitable vessel, such as a tank, and then treated with the frequency modulated energy field.

In a preferred form of the invention the treatment comprises passing the aqueous polymeric flocculant or coagulant composition or water which is to be combined with the flocculant/coagulant material through an electrical coil which generates a field by passing an energising signal to the coil. Preferably, the energising signal is frequency modulated as described above. This may conveniently be achieved by passing the aqueous flocculant/coagulant composition or water along a pipe around which an electrical coil is placed. As described above the field strength required to effect the treatment will depend on the diameter of the coil or the pipe around which the electrical coil is placed, the number of coils around the pipe, the energising signal and electrical power transmitted through the coil. We have found that a greater electrical power is required where the diameter of the pipe or electrical coil is greater.

In another preferred form of the invention it may be desirable to treat the aqueous suspension of solid particles to be concentrated by the magnetic field and/or electric field with non-sinusoidal wave pulses at a sweep of frequencies as defined above in addition to treating the aqueous flocculant or coagulant composition or water used to make this. This additional treatment of the aqueous suspension may be carried out prior to, during or subsequent to the addition of the so treated flocculant or coagulant.

By polymeric flocculant material or polymeric coagulant material we mean flocculant or coagulant products that comprise flocculant or coagulant polymers respectively. This may for instance be solid particulate powders or beads of flocculant or coagulant polymer, concentrated solutions of flocculant or coagulant polymer or alternatively reverse phase emulsions or dispersions of flocculant or coagulant polymer.

The polymeric flocculant material may comprise synthetic or natural polymer. Suitable natural polymers include polysaccharides such as starches such as cationic, anionic, non-ionic or amphoteric starch, cellulose derivatives such as carboxy methyl cellulose and hydroxy ethyl cellulose, Guar gum, dextran, chitosan or chitin.

Preferably, the polymeric flocculant is a synthetic polymer. The polymeric flocculant may include high molecular weight polymers that are cationic, non-ionic, anionic or amphoteric. Usually, if the polymer is synthetic it should exhibit an intrinsic viscosity of at least 4 dl/g. More preferably though, the polymer will have significantly higher intrinsic viscosity. For instance the intrinsic viscosity may be as high as 25 or 30 dl/g or higher. Typically the intrinsic viscosity will be at least 7 and usually at least 10 or 12 dl/g and could be as high as 18 or 20 dl/g.

Intrinsic viscosity of polymers may be determined by preparing an aqueous solution of the polymer (0.5-1% w/w) based on the active content of the polymer. 2 g of this 0.5-1% polymer solution is diluted to 100 ml in a volumetric flask with 50 ml of 2M sodium chloride solution that is buffered to pH 7.0 (using 1.56 g sodium dihydrogen phosphate and 32.26 g disodium hydrogen phosphate per litre of deionised water) and the whole is diluted to the 100 ml mark with deionised water. The intrinsic viscosity of the polymers are measured using a Number 1 suspended level viscometer at 25°C in 1M buffered salt solution.

One desirable class of synthetic polymers includes polyethers such as polyalkylene oxides. Typically these are polymers with alkylene oxy repeating units in the polymer backbone. Particularly suitable polyalkylene oxides include polyethylene oxides and polypropylene oxides. Generally these polymers will have a molecular weight of at least 500,000 and often at least one million. The molecular weight of the polyethers may be as high as 15 million of 20 million or higher.

Another preferred class of synthetic polymers include polymers that are formed from an ethylenically unsaturated water-soluble monomer or blend of monomers.

The synthetic polymer may be cationic, non-ionic, amphoteric, or anionic. The polymers may be formed from any suitable water-soluble monomers. Typically the water soluble monomers have a solubility in water of at least 5g/100cc at 25°C. Particularly preferred anionic polymers are formed from monomers selected from ethylenically unsaturated carboxylic acid and sulphonic acid monomers, preferably selected from (meth) acrylic acid, allyl sulphonic acid and 2-acrylamido-2-methyl propane sulphonic acid, and their salts, optionally in combination with non-ionic co-monomers, preferably selected from (meth) acrylamide, hydroxy alkyl esters of (meth) acrylic acid and N-vinyl pyrrolidone.

Especially preferred polymers include the homopolymer of sodium acrylate, the homopolymer of acrylamide and the copolymer of sodium acrylate with acrylamide.

Preferred non-ionic polymers are formed from ethylenically unsaturated monomers selected from (meth) acrylamide, hydroxy alkyl esters of (meth) acrylic acid and N-vinyl pyrrolidone.

Preferred cationic polymers are formed from ethylenically unsaturated monomers selected from dimethyl amino ethyl (meth) acrylate - methyl chloride, (DMAEA.MeCl) quat, diallyl dimethyl ammonium chloride (DADMAC), trimethyl amino propyl (meth) acrylamide chloride (ATPAC) optionally in combination with non-ionic co-monomers, preferably selected from (meth) acrylamide, hydroxy alkyl esters of (meth) acrylic acid and N-vinyl pyrrolidone.

It is particularly preferred that the polymeric flocculant material comprises a synthetic polymer that is a cationic, non-ionic or anionic polymer of acrylamide.

The synthetic polymer may be in any convenient physical form. Preferably however, it will be in the form of a particulate solid material, such as beads or powder or alternatively it may be in the form of a reverse phase emulsion or dispersion.

In the invention, the polymer may be formed by any suitable polymerisation process. The polymers may be prepared for instance as gel polymers by solution polymerisation, water-in-oil suspension polymerisation or by water-in-oil emulsion polymerisation. When preparing gel polymers by solution polymerisation the initiators are generally introduced into the monomer solution.

Optionally a thermal initiator system may be included. Typically a thermal initiator would include any suitable initiator compound that releases radicals at an elevated temperature, for instance azo compounds, such as azo-bis-isobutyronitrile. The temperature during polymerisation should rise to at least 70°C but preferably below 95°C. Alternatively polymerisation may be effected by irradiation (ultra violet light, microwave energy, heat etc.) optionally also using suitable radiation initiators. Once the polymerisation is complete and the polymer gel has been allowed to cool sufficiently the gel can be processed in a standard way by first comminuting the gel into smaller pieces, drying to the substantially dehydrated polymer followed by grinding to a powder.

Such polymer gels may be prepared by suitable polymerisation techniques as described above, for instance by irradiation. The gels may be chopped to an appropriate size as required and then on application mixed with the material as partially hydrated water soluble polymer particles.

The polymers may be produced as beads by suspension polymerisation or as a water-in-oil emulsion or dispersion by water-in-oil emulsion polymerisation, for example according to a process defined by EP-A-150933, EP-A-102760 or EP-A-126528.

Alternatively the synthetic polymer may be provided as a dispersion in an aqueous medium. This may for instance be a dispersion of polymer particles of at least 20 microns in an aqueous medium containing an equilibrating agent as given in EP-A-170394. This may for example also include aqueous dispersions of polymer particles prepared by the polymerisation of aqueous monomers in the presence of an aqueous medium containing dissolved low IV polymers such as poly diallyl dimethyl ammonium chloride and optionally other dissolved materials for instance electrolyte and/or multi-hydroxy compounds e. g. polyalkylene glycols, as given in WO-A-9831749 or WO-A-9831748.

Preferably the synthetic polymer is prepared as a water-soluble linear polymer. Such a polymer it generally prepared in the absence of branching, structuring or cross linking agent. In some cases it may be desirable to introduce some degree of structuring such that the polymer is nevertheless water-soluble. In another preferred instances the synthetic polymer may be prepared with small or moderate levels of branching or cross linking agent so as to provide a branched or cross linked polymer. Particularly suitable cross linked polymers will tend to have a volume average particles size diameter of below 10 microns and usually below 2 microns, usually has a reverse phase emulsion or dispersion, for instance as described in EP-A-202,780.

Preferably the polymeric flocculant material comprises a synthetic polymer in solid particulate form or as a reverse phase emulsion or dispersion, which synthetic polymer is formed from ethylenically unsaturated monomer or monomer blend.

The polymeric coagulant material will comprise a suitable coagulant polymer. In general the coagulant polymer will be of a molecular weight below that of a flocculant polymer. Typically a coagulant polymer will exhibit an intrinsic viscosity of below 4 dl/g and usually below 3 dl/g, for instance below 2 dl/g. Frequently, the coagulant polymer may exhibit an intrinsic viscosity of at least 0.1 dl/g and usually at least 0.2 or 0.5 dl/g. Such polymers will tend to have molecular weights in the range of between 50,000 and 2 million, for instance 150,000 to 1.5 million. The coagulant polymer will normally be ionic and usually will exhibit a high charges density, for instance at least 2 or 3 milliequivalents/g and in some cases as high as 4 or 5 milliequivalents/g or higher. The coagulant polymer may be anionic and for instance be formed from any of the aforementioned monomer or monomer blends for producing anionic polymers, suitably an anionic monomer or blend of anionic monomer(s) optionally with non-ionic monomer. Preferably the coagulant polymer is cationic.

Such a cationic water soluble polymer may be a relatively low molecular weight polymer of relatively high cationicity. For instance the polymer may be a homopolymer of any suitable ethylenically unsaturated cationic monomer polymerised to provide a polymer with an intrinsic viscosity as mentioned above in regard to coagulant polymers. The cationic coagulant may be prepared from any of the aforementioned monomer are all monomer blends for producing cationic polymers, typically employing at least one cationic monomer optionally with a non-ionic monomer. Homopolymers of diallyl dimethyl ammonium chloride are preferred. The low molecular weight high cationicity polymer may be an addition polymer formed by condensation of amines with other suitable di- or tri- functional species. For instance the polymer may be formed by reacting one or more amines selected from dimethyl amine, trimethyl amine and ethylene diamine etc and epihalohydrin, epichlorohydrin being preferred.

The polymeric flocculant or coagulant material is combined with water to form an aqueous composition. In accordance with the present invention either the water or the aqueous composition is/are treated using the aforementioned energy and/or magnetic field. When the polymeric flocculant or coagulant material comprises water-soluble polymers, the polymer will dissolve in the water to form an aqueous solution as the aqueous polymer flocculant or coagulant composition. In the case where the polymeric flocculant or coagulant material is an aqueous solution concentrate the combination with water will form a more dilute solution which will be the aqueous polymeric flocculant or aqueous composition. Where the polymeric flocculant or coagulant material is cross linked and therefore insoluble, the combination with water will form an aqueous dispersion, which may be referred to as an aqueous dilution. Typically this will be in the case of reverse phase emulsion or dispersions of cross linked flocculant polymers as described in EP-A-202,780. The aqueous dispersion (or aqueous dilution) would then be the aqueous flocculant or coagulant composition.

Generally, solid particulate polymer, for instance in the form of powder or beads, is dispersed in water and allowed to dissolve with agitation. This may be achieved using conventional make up equipment. Desirably, the polymer solution can be prepared using the Auto Jet Wet (trademark) supplied by Ciba Specialty Chemicals. Where the flocculant or coagulant material is supplied in the form of a reverse phase emulsion or dispersion this can then be inverted into water to form an aqueous solution or aqueous dispersion as the aqueous flocculant or coagulant composition.

Desirably the aqueous polymeric flocculant or coagulant composition has a polymer concentration of between 0.05% and 1% by weight. This may for instance be between 0.1 and 0.5% by weight.

The aqueous flocculant or coagulant composition treated with the energy or magnetic field, either directly or by treating the water forming it, can then be fed to the aqueous suspension of solid particles. The process according to the present invention is applicable generally to solids liquid separation processes. Thus the aqueous suspension of solids may comprise organic material including for instance sewage sludge or cellular material from fermentation processes. The suspension may also be a suspension of cellulosic material, for instance sludges from papermaking processes.

Alternatively, the suspension is an aqueous suspension comprising mineral particles. Thus the process may involve the treatment of aqueous suspensions resulting from mined mineral processing and other mining wastes, for instance from carbon based industries such as coal and tar sands, comprising suspensions of mineral particles, especially clays. In this case the aqueous suspension of solids is derived from mineral or energy processing operations and/or tailings substrates. By energy processing operations we mean preferably processes in which the substrate involves the separation of materials useful as fuels. The process may also involve suspensions selected from mining and refining operations the group consisting of bauxite, base metals, precious metals, iron, nickel, coal, mineral sands, oil sands, china clay, diamonds and uranium.

The suspensions will often contain at least 5% by weight suspended solids particles and may contain as much as 30% or higher. Preferably suspensions will contain at least 0.25% more preferably at least 0.5% Usually the suspensions will contain between 1% and 20% by weight suspended solids.

It is preferred that the aqueous suspension to be treated in the process comprises at least some organic material. This organic material may for instance be sewage sludges or other waste water derived substrates. It may also be cellular material, for instance from a fermentation process. Alternatively it may include cellulosic material, for instance as in paper or paperboard manufacturing or treatment of cellulosic wastes e.g. cellulosic sludges. Preferably the suspended solids will comprise at least 10% by weight of organic material based on total weight of suspended solids. More preferably this will be at least 30% by weight, frequently at least 50% by weight, and especially at least 70% by weight and most preferably entirely organic derived material or at least at least 90% by weight.

Suitable doses of organic polymeric flocculant range from 5 grams to 10,000 grams per tonne of material solids. Generally the appropriate dose can vary according to the particular material and material solids content. Preferred doses are in the range 10 to 3,000 grams per tonne, especially between 10 and 1000 grams per tonne, while more preferred doses are in the range of from 60 to 200 or 400 grams per tonne.

The invention is illustrated by the following examples which in no way limit the scope of invention.

Examples

Example 1

Polymer A = A liquid dispersion polymer (50% active in a mineral oil) of a 80:20 Methyl Chloride quaternised Dimethyl ammonium ethyl acrylate: acrylamide copolymer of IV 12.

A 0.125% active aqueous solution of Polymer A was prepared in deionised water and allowed to age for 1 hour. The sample was split into 2. The first solution was evaluated with no further treatment, the second solution was treated by passing through a tube (approx 7mm diameter) around which was wound the wire from a frequency modulation generator (model MOD -2 (D13519) supplied by Environmental Treatment Concepts Ltd, such that in formed 40 coils around the tube to create a solenoid. The power output applied was 12 volts peak-to-peak and the frequency range scanned was 700 to 3,000 Hz.

Each solution was then evaluated as a flocculant for treating sewage sludge whereby 250 cm^3 of the sludge was taken in a 400 cm^3 beaker. The appropriate volume of flocculant solution was added and the sludge was mechanically stirred for 15 seconds at a speed of 2000 rpm. The flocculated sludge was then poured onto a sieve of 8cm diameter and the volume of filtrate produced after allowing 5 seconds drainage was measured.

Table 1

Dose (mg/l active)	5 second filtrate Volume (cm^3)	
	Polymer A untreated	Polymer A Treated
40	73	103
45	118	129

50	121	140
60	137	154
70	129	145
80	119	129

These results show that the use of treated polymer A results in the release in a greater volume of water at a lower dose that the untreated polymer A.

Example 2

A 0.125% active solution of polymer A was prepared in deionised water in a 600 cm^3 beaker (approx 10 cm diameter) and allowed to age for one hour by stirring at 800 rpm using a flat bladed mechanical stirrer. A second 0.125% active solution of polymer A was prepared in a 600 cm^3 beaker wound round which were 15 coils of wire from the same generator as used in Example 1. The power applied and frequencies scanned were also the same as in Example 1. These were applied continuously for set periods of time. The solutions produced were tested on a sample of sewage sludge as described in Example 1 with the exception that the stirring speed applied was 4000 rpm.

Table 2

	5 second filtrate volume (cm^3)			
Active Dose (mg/l)	Untreated	Treated for 30 minutes	Treated for 120 minutes	Treated for 180 minutes
65	47	73	90	91
75	76	90	104	99
90	71	70	83	80

These results show that treating the solution by stirring in a container surrounded by the coil has a similar effect to passing through the coil.

Example 3

Polymer B = 50:50 sodium acrylate : acrylamide copolymer of IV approximately 24 made as a solid grade.

A 0.5% active aqueous solution of polymer B was prepared in deionised water and allowed to age for 2 hours. The solution was diluted to 0.05% active and split into two solutions. One solution was left untreated whilst the second solution was treated for 60 minutes as outlined in example 2.

The solutions were evaluated using 4% china clay slurry (containing 4g/l NaOH) as test substrate. 500 cm3 samples of the test substrate were taken in 500 cm3 cylinders. The appropriate volume of the solutions of polymer B were added and mixing was achieved using 3 plunger strokes. The resultant settlement rate was measured between two fixed points and the supernatant turbidity was measured after allowing 10 minutes settlement time.

Table 3

Sample	Polymer Dose (mg/l)	Settlement Rate (cm/min	Turbidity (NTU)
0.05% Polymer B untreated	4	4.8	1884
	5	27.6	1034
	6	40.1	1075
	8	107.1	424
Polymer B treated	4	9.5	1072
	5	37.4	751
	6	42.5	584
	8	87.5	406

These results show that the treated solution was more effective as it produced faster settlement rates at lower dose levels together with improved overflow turbidities.

Claims

1. A process of concentrating or dewatering an aqueous suspension of solid particles comprising the steps of,
providing an aqueous polymeric flocculant composition or an aqueous polymeric coagulant composition by combining polymeric flocculant material or polymeric coagulant material with water,
adding an effective flocculating or coagulating amount of the aqueous polymeric flocculant or coagulant composition into the aqueous suspension of solid particles thereby bringing about flocculation or coagulation of the solid particles,
subjecting the so formed flocculated or coagulated suspension of solid particles to a concentrating or dewatering stage,
wherein the aqueous polymeric flocculant or coagulant composition and/or the water which is to be combined with the polymeric flocculant or coagulant material is/are treated by a magnetic field and/or electric field with non-sinusoidal wave pulses at a sweep of frequencies.

2. A process according to claim 1 in which the magnetic field and/or electric field is produced by a frequency modulated non-sinusoidal wave generator comprising at least one electrical coil.

3. A process according to claim 1 or claim 2 in which the sweep of frequencies is frequency modulated to between 300 and 21,000 Hz.

4. A process according to any preceding claim in which the aqueous polymeric flocculant or coagulant composition and/or the water which is to be combined with the flocculant or coagulant material is/are passed through at least one electrical coil which generates the magnetic field and/or electric field.

5. A process according to any preceding claim in which polymeric flocculant material is a synthetic polymer in solid particulate form or as a reverse phase emulsion or dispersion, which synthetic polymer is formed from ethylenically unsaturated monomer or monomer blend.

6. A process according to any preceding claim in which the polymeric flocculant material comprises a synthetic polymer that has an intrinsic viscosity of at least 4 dl/g.

7. A process according to any preceding claim in which the polymeric flocculant material comprises a synthetic polymer that is a cationic, non-ionic or anionic polymer of acrylamide.

8. A process according to any preceding claim in which the aqueous polymeric flocculant or coagulant composition has a polymer concentration of between 0.05% and 2% by weight.

9. A process of preparing an aqueous polymeric flocculant composition or an aqueous polymeric coagulant composition comprising combining polymeric flocculant material or polymeric coagulant material with water,
wherein the aqueous polymeric flocculant or coagulant composition and/or the water which is to be combined with the polymeric flocculant or coagulant material is/are treated by a magnetic field and/or electric field with non-sinusoidal wave pulses at a sweep of frequencies.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2008/059925

A. CLASSIFICATION OF SUBJECT MATTER
INV. C02F11/14
ADD. C02F1/56 C02F1/48

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C02F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, COMPENDEX

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	CHAUHAN S P: "FEASIBILITY OF BIOSLUDGE DEWATERING USING PULSED ELECTRIC FIELDS" BATTELLE FINAL REPORT, X, XX, 17 September 1998 (1998-09-17), pages 1-42, XP002942843 table 1 table 2 page 7 - page 20 page 25 - page 26 -----	1-9
X	US 6 030 538 A (HELD JEFFERY S [US] ET AL) 29 February 2000 (2000-02-29) table 1 -----	1-9

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"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.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
29 October 2008	24-11-2008

Name and mailing address of the ISA/	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Janssens, Christophe

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/EP2008/059925

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
US 6030538	A	29-02-2000	NONE	

Form PCT/ISA/210 (patent family annex) (April 2005)