

PROCESS FOR MAKING PAPER

This application claims the benefit under 35 U.S.C. §119(e) of prior U.S. Provisional Patent Application No. 60/538,622 filed January 23, 2004, which is incorporated in its entirety by reference herein.

FIELD OF THE INVENTION

[0001] The present invention relates to papermaking pulps, papermaking processes employing the pulps, papermaking apparatus and paper and paperboard products made from the pulps. More particularly, the present invention relates to treating papermaking pulp with at least one microparticle-containing retention aid system.

BACKGROUND OF THE INVENTION

[0002] Retention aid systems containing microparticles and other particulate materials have been added to papermaking pulps as process aids to improve retention and other properties such as formation and drainage. For example, U.S. Patent No. 5,194,120 to Peats et al., incorporated herein by reference in its entirety, describes a retention aid system comprising a cationic polymer and an amorphous metal silicate material. One type of silicate material mentioned by Peats et al. system is Laponite®, a synthetic layered silicate. According to Peats et al., the use of a retention aid system comprising an amorphous metal silicate material and a cationic polymer provides several advantages, including improved retention, drainage and formation while minimizing the amount of polymer and amorphous metal silicate added to the pulp.

[0003] The microparticle component of the retention aid system is typically added to the papermaking pulp in the form of a low viscosity aqueous colloidal dispersion, i.e. a sol. One problem with microparticle sols employed in papermaking pulps is instability. Because of the instability of sols used in connection with

papermaking pulps, the sols are often made on-site for immediate delivery to a papermaking process. A need exists for a stable microparticle sol retention aid for use in papermaking processes which can be formed off-site, exhibits a long shelf life, and can be shipped to a papermaking plant for immediate or future use in a papermaking process.

[0004] A need also exists for a papermaking pulp that exhibits even better drainage and retention of fines during a papermaking process.

SUMMARY

[0005] The disclosure relates to the use of a combination of synthetic layered silicate microparticles, a peptizer and at least one polymer as a retention aid system for a papermaking pulp or stock. The synthetic layered silicate is preferably a synthetic hydrous sodium lithium magnesium silicate and is preferably added to the papermaking pulp in the form of an aqueous colloidal dispersion which also contains the peptizer. The polymer can be cationic polymer, a nonionic polymer, or an amphoteric polymer used under cationic conditions. The polymer is preferably a synthetic nitrogen-containing cationic polymer, for example, a cationic polyacrylamide. If nonionic, the polymer can be, for example, a nonionic polyacrylamide or a polyethylene oxide.

[0006] The peptizer is present in the microparticle dispersion for the purpose of maintaining the dispersion in the form of a sol and to prevent the dispersion from setting to a gel for a predetermined period of time. This permits the formation of relatively concentrated microparticle sols which can be formed off-site, which exhibit a relatively long shelf life and can be shipped to the papermaking plant for immediate or future use.

[0007] The inventor has unexpectedly found that certain microparticle dispersions which include a peptizer also provide significant improvements over

microparticle dispersions which do not employ a peptizer. For example, the inventor has found that the use of a retention aid including a microparticle dispersion containing synthetic hydrous sodium lithium magnesium silicate and a peptizer significantly improves retention of fines, drainage and formation, thereby providing enhancements in the papermaking process and in the paper product.

[0008] In one aspect, there is provided a method of making paper or paperboard comprising: (a) forming a treated pulp by adding to a papermaking pulp a synthetic layered silicate, a peptizer and at least one polymer, the synthetic layered silicate comprising a synthetic hydrous sodium lithium magnesium silicate and the at least one polymer comprising one or more members of the group consisting of cationic polymers, nonionic polymers and amphoteric polymers under cationic conditions; and (b) forming the treated pulp into said paper or paperboard.

[0008a] In a second aspect, there is provided a method of making paper or paperboard comprising: (a) adding to a papermaking pulp, a synthetic layered silicate, a peptizer and at least one polymer, the synthetic layered silicate comprising a synthetic hydrous sodium lithium magnesium silicate, the peptizer comprising an inorganic polyphosphate peptizer, and the at least one polymer comprising one or more members of the group consisting of cationic polymers, nonionic polymer and amphoteric polymers under cationic conditions, wherein the synthetic layered silicate is added to the pulp in the form of a sol and wherein the sol contains said peptizer in an amount sufficient to maintain said sol in stable form for about 3 days to about 90 days; and (b) forming the pulp from (a) into said paper or paperboard wherein the pulp comprises more synthetic layered silicate than peptizer, and said, method further comprising shearing the pulp in one or more shear stages including a final high shear stage in which the pulp is passed through a screen prior to entering a headbox of a papermaking apparatus, and either (i) the polymer is added to the pulp before the final high shear stage and the synthetic layered silicate is added to the pulp after the final high shear stage, or (ii) the synthetic layered silicate is added to the pulp before the final high stage and the polymer is added to the pulp after the final high shear stage.

[0009] In one embodiment, there is provided a papermaking apparatus comprising a supply of synthetic layered silicate, a supply of a papermaking pulp, a device for feeding the synthetic layered silicate from the supply of synthetic layered silicate to the supply of papermaking pulp, a supply of a retention system polymer, a device for feeding the retention system polymer from the supply of retention system polymer to the papermaking pulp, and a device for forming the pulp into a paper or paperboard after treatment with the synthetic layered silicate and the retention system polymer, wherein said retention system polymer is a cationic polymer, a nonionic polymer, or an amphoteric polymer under cationic conditions, or combinations thereof and wherein the synthetic layered silicate comprises a synthetic hydrous sodium lithium magnesium silicate and is fed to the papermaking pulp in the form of an aqueous dispersion which also includes an inorganic polyphosphate peptizer.

[0010] In another embodiment, there is provided a paper or paperboard made from a drained paperweb, the paperweb comprising a treated pulp, the treated pulp comprising cellulosic fibers, synthetic hydrous sodium lithium

magnesium silicate, at least one retention system polymer and an inorganic polyphosphate peptizer, said retention system polymer comprising a cationic polymer, a nonionic polymer, or an amphoteric polymer under cationic conditions, or combinations thereof.

BRIEF DESCRIPTION OF THE DRAWINGS

[00011] The invention will now be described, by way of example only, with reference to the accompanying drawings in which:

[00012] FIG. 1 is a flow chart showing a papermaking process according to an embodiment of the present invention;

[00013] FIG. 2 is a flow chart showing a papermaking process according to another embodiment of the present invention;

[00014] FIG. 3 is a flow chart showing a papermaking process according to yet another embodiment of the present invention;

[00015] FIG. 4 is a flow chart showing a papermaking process according to yet another embodiment of the present invention;

[00016] FIG. 5 is a flow chart showing a papermaking process according to yet another embodiment of the present invention;

[00017] FIG. 6 is a bar graph showing the time to achieve drainage of 200 ml of filtrate from paperwebs made of various exemplary and comparative paperstock formulations;

[00018] FIG. 7 is a bar graph comparing the turbidity of various exemplary and comparative paperstock formulations;

[00019] FIG. 8 is a bar graph showing the % total first pass retention (TFPR) of various exemplary and comparative paperstock formulations;

[00020] FIG. 9 is a plot of time vs. volume showing the drainage of various exemplary and comparative paperstock formulations;

[00021] Figure 10 is a bar graph showing the drainage in seconds of various exemplary and comparative paperstock formulations; and

[00022] Figure 11 is a bar graph showing the retention for various exemplary and comparative paperstock compositions.

DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

[00023] The present invention relates to the use of a retention aid system for a papermaking pulp, the system comprising a synthetic layered silicate, a peptizer and at least one polymer. More than one type of microparticle, more than one type of peptizer and more than one type of polymer can be used in the process of the invention. Paper and paperboard products made according to the method preferably exhibit excellent opaqueness and/or other desirable physical properties. Sheets of pulp from which the paper and paperboard products are made preferably exhibit excellent drainage and/or excellent retention of pulp fines.

[00024] The synthetic layered silicate preferably comprises a synthetic hydrous sodium lithium magnesium silicate which is manufactured and sold under the trademark Laponite® by Rockwood Additives Limited of Widnes, Cheshire, United Kingdom. These synthetic hydrous sodium lithium magnesium silicates are synthesized by combining salts of sodium, magnesium and lithium with sodium silicate at carefully controlled rates and temperatures. This produces an amorphous precipitate which is then partially crystallized under high temperature and pressure. The resulting product is filtered, washed, dried and milled to give a fine white powder.

[00025] For greater certainty, the terms "synthetic hydrous sodium lithium magnesium silicate" and "hydrous sodium lithium magnesium silicate" as used herein include silicates which are identified by CAS No. 533320-86-8 and have the following typical chemical analysis (wt%): SiO₂ 59.5; MgO 27.5; Li₂O 0.8; Na₂O 2.8; loss on ignition 8.2. Such silicates typically comprise a free flowing white powder having a bulk density of 1,000 kg/m³; surface area (BET) of 370 m²/g; pH (2% suspension) of 9.8; sieve analysis (<250µm) of 98%; and moisture content of 10%.

[00026] For greater certainty, the terms "synthetic hydrous sodium lithium magnesium silicate" and "hydrous sodium lithium magnesium silicate" as used herein do not include synthetic layered silicates identified by the TSCA name "hydrous sodium lithium magnesium fluorosilicate" and by CAS No. 64060-48-6 and which have the following typical chemical composition (wt% - dry basis): SiO₂ 51.0; MgO 25.0; Li₂O 1.3; Na₂O 6.0; P₂O₅ 3.3; F 5.0; loss on ignition 8.4.

[00027] The synthetic layered silicate microparticles can be added in any amount sufficient to improve the retention of fines or drainage or to reduce turbidity when the pulp or stock is formed into a wet sheet or web. Preferably, the microparticles are added in an amount of at least about 0.05 lb/ton (0.02 kg/tonne) of paperstock, based on the dried solids weight of both the microparticles and the paperstock. More preferably, the microparticles are added in an amount of from about 0.1 lb/ton (.05 kg/tonne) of paperstock to about 5.0 lb/ton (2.3 kg/tonne) of paperstock, for example, from about 0.2 lb/ton (0.09 kg/tonne) to about 1.0 lb/ton (0.5 kg/tonne), based on dried solids weight of the paperstock. For purposes of this patent application, the terms "furnish", "pulp", "stock", and "paperstock" are used interchangeably.

[00028] Preferably, the synthetic layered silicate is added to the pulp in the form of an aqueous, colloidal dispersion of relatively low viscosity. A colloidal dispersion having these characteristics is known as a "sol". In addition to the synthetic layered silicate, the dispersion preferably also contains a peptizer in an amount sufficient to maintain the dispersion in the form of a sol for a predetermined period of time. The

peptizer essentially stabilizes the sol to prevent it from setting to a gel for a period of time which depends at least partially on the concentration of the synthetic layered silicate. This permits the dispersion to be formed off-site in a reasonable concentration and then shipped to the paper making plant for immediate or future use.

[00029] The peptizer is preferably a water soluble salt which enhances dispersion of the synthetic layered silicate, more preferably a sodium salt selected from the group comprising sodium carbonate, sodium metaphosphates, sodium polyacrylates, sodium hydroxide, sodium chloride, sodium polyphosphates and sodium pyrophosphates. In particularly preferred embodiments of the present invention, the peptizer is an inorganic polyphosphate, more preferably tetrasodium pyrophosphate. The preferred peptizer, tetrasodium pyrophosphate, is present in certain grades of hydrous sodium lithium magnesium silicate available from Rockwood Additives Limited, including Laponite RDS, Laponite XLS and Laponite DS. One particularly preferred grade of Laponite is Laponite RDS which comprises synthetic hydrous sodium lithium magnesium silicate (CAS No. 53320-86-8) in combination with about 5 wt% tetrasodium pyrophosphate. Laponite RDS sols containing about 10wt% concentration of Laponite are stable for about 3 days. Preferably, the sol has a laponite concentration of up to about 6 wt% which is stable for at least about 90 days. Sols having a microparticle concentration in this range exhibit sufficiently long shelf to allow them to be formed off-site for shipment and subsequent use in a papermaking process.

[00030] As mentioned above, the inventor has unexpectedly discovered that certain microparticle dispersions which include a peptizer also provide significant improvements over microparticle dispersions which do not employ a peptizer. In particular, the inventor has found that the inclusion of a peptizer in the microparticle dispersion significantly improves retention of fines, drainage and formation, thereby providing enhancements in the papermaking process and in the paper product. For example, the inventor has found that Laponite RDS, which contains the peptizer

tetrasodium pyrophosphate (TSPP), provides significantly enhanced drainage and retention, with lower turbidity, than equivalent amounts of Laponite RD.

[00031] Without being bound by theory, it is believed that when the TSPP is blended and dissolved in a colloidal dispersion of Laponite, the pyrophosphate ions become associated with the positively charged edges of the Laponite crystals, making the whole particle negatively charged. This effectively increases the negative charge on the Laponite crystals and causes them to have a greater attraction to cationic particles present in the papermaking process.

[00032] The polymer is preferably added to the papermaking pulp before addition of the microparticles, though any order of addition can be used. Preferably, the polymer can be any polymer which does not adversely affect the formation of pulp or paper and may preferably comprise a coagulant and/or a flocculant. Preferably, the polymer is a medium to high molecular weight synthetic polymer, for example, a cationic nitrogen-containing polymer such as a cationic polyacrylamide or a copolymer thereof, or a cationic diallyldimethylammonium chloride or a copolymer thereof.

[00033] The polymer can be cationic, nonionic, or amphoteric. If amphoteric, the polymer is preferably used under cationic conditions. At least one other polymer of any kind can be used in addition to the polymers recited above so long as the at least one other polymer does not substantially adversely affect the retention properties of the present invention. The at least one other polymer can preferably be a polyamidoamineglycol (PAAG) polymer.

[00034] The polymer preferably has a molecular weight in the range of from about 100,000 to about 25,000,000, and more preferably from about 500,000 to about 18,000,000, though other molecular weights are possible to achieve the intended effect.

[00035] The polymer can preferably be a high molecular weight linear cationic polymer or a crosslinked polyethylene oxide. Exemplary high molecular weight linear

cationic polymers and shear stage processing suitable for use in the pulps and methods of the present invention are described in U.S. Patent Nos. 4,753,710 and 4,913,775 to Langley et al., both of which are incorporated herein in their entireties by reference.

[00036] The polymer is preferably added before at least one of the significant shear steps of the papermaking process and may be added in more than one step. The microparticles can be added before or after the various significant shear steps of the papermaking process. According to some embodiments of the present invention, the polymer can be added before the microparticles and before at least one significant shear step in the papermaking process. If the polymer is added before the microparticles, the microparticles can be added before or after a final shear step of the papermaking process. Although it is preferable to add the polymer to the papermaking pulp before the last shear point in the papermaking process, the polymer can be added after the last shear point.

[00037] The microparticles preferably form bridges or networks between various particles. The polymer is preferably partially attached (e.g., adsorbed) onto the surfaces of particles within the stock and can provide sites of attachment.

[00038] Aqueous cellulosic papermaking pulp or stock can be treated by first adding the polymer to the pulp or stock, followed by subjecting the paper stock to high shear conditions, followed by the addition of the microparticles prior to sheet formation. As discussed above, the polymer can be cationic, nonionic, or amphoteric under cationic conditions. Alternatively, the polymer can be added simultaneously with the synthetic layered silicate microparticles.

[00039] Preferred cationic polyacrylamides for use as the retention system polymer are described in more detail below. If a cationic polyacrylamide is used as the cationic polymer, the cationic polyacrylamide can have a molecular weight in excess of 100,000, and preferably has a molecular weight of from about 500,000 and 18,000,000. The combination of the polymer and the synthetic layered silicate

microparticles preferably provides a suitable balance between freeness, dewatering, fines retention, good paper formation, strength, and resistance to shear.

[00040] The polymer composition of the retention system is added in an amount effective to preferably improve the drainage or retention of the pulp compared to the same pulp but having no polymer present. The polymer is preferably added in an amount of at least about 0.01 pound of polymer per ton (0.005 kg/tonne) of paperstock, based on the weight of dried solids of both the polymer and the paperstock. More preferably, the polymer is added in an amount of from about 0.1 lb/ton (0.05 kg/tonne) of paperstock to about 5 lb/ton (2.3 kg/tonne) of paperstock, even more preferably from about 0.2 lb/ton (0.09 kg/tonne) to about 2 lb/ton (1 kg/tonne) based on the dried solids weight of the paperstock, though other amounts can be used.

[00041] If the polymer is cationic, any cationic polymer or mixture thereof can be used and preferably conventional cationic polymers commonly associated with papermaking can be used in the pulps or stocks of the present invention. Examples of cationic polymers include, but are not limited to, cationic starches and cationic polyacrylamide polymers, for example, copolymers of an acrylamide with a cationic monomer, wherein the cationic monomer may be in a neutralized or quaternized form. Nitrogen-containing cationic polymers are preferred. Exemplary cationic monomers which may be copolymerized with acrylamide to form preferred cationic polymers useful according to the present invention, include amino alkyl esters of acrylic or methacrylic acid, and diallylamines in either neutralized or quaternized form. Exemplary cationic monomers and cationic polyacrylamide polymers are described in U.S. Patent No. 4,894,119 to Baron, Jr., et al., which is incorporated herein in its entirety by reference.

[00042] The polymer may also be a polyacrylamide formed from comonomers that include, for example, 1-trimethylammonium-2-hydroxypropylmethacrylate methosulphate. Other examples of cationic polymers, include, but are not limited to,

homopolymers of diallylamine monomers, homopolymers of aminoalkylesters of acrylic acids, and polyamines, as described in U.S. Patent No. 4,894,119. Co-polymers, ter-polymers, or higher forms of polymers may also be used. Further, for purposes of the present invention, a mixture of two or more polymers may be used.

[00043] In embodiments wherein the polymer contains a cationic polyacrylamide, nonionic acrylamide units are preferably present in the copolymer, preferably in an amount of at least about 30 mol % and generally in an amount of no greater than 95 mol %. From about 5 mol % to about 70 mol % of the polymer is preferably formed from a cationic comonomer.

[00044] The papermaking pulp or stock can be any conventional type, and, for instance, can contain cellulose fibers in an aqueous medium at a concentration of preferably at least about 50% by weight of the total dried solids content in the pulp or stock. The retention system of the present invention can be added to many different types of papermaking pulp, stock, or combinations of pulps or stocks. For example, the pulp may comprise virgin and/or recycled pulp, such as virgin sulfite pulp, broke pulp, a hardwood kraft pulp, a softwood kraft pulp, mixtures of such pulps, and the like.

[00045] The retention aid system can be added to the pulp or stock in advance of depositing the pulp or stock onto a papermaking wire. The pulp or stock containing the retention aid system has been found to exhibit good dewatering during formation of the paperweb on the wire. The pulp or stock also exhibits a desirable high retention of fiber fines and fillers in the paperweb products under conditions of high shear stress imposed upon the pulp or stock.

[00046] In addition to the retention aid system used in accordance with the present invention, the papermaking pulp or stock according to the present invention may further contain other types of microparticles. One or more different types of secondary microparticle additives, different from the synthetic layered silicate microparticles, may be added to the pulp at any time during the process. The secondary microparticle additive can be a natural or synthetic hectorite, bentonite,

zeolite, non-acidic alumina sol, or any conventional particulate additives as are known to those skilled in the art. Exemplary synthetic microparticles are described in U.S. Patent Nos. 5,571,379 and 5,015,334, which are incorporated herein in their entireties by reference.

[00047] In addition to the synthetic layered silicate microparticles retention aid system used in accordance with the present invention, the papermaking pulps or stocks according to the present invention may further contain a coagulant/flocculant retention system having a different composition than the retention system of the present invention.

[00048] The papermaking pulps of the present invention may also contain a conventional papermaking pulp-treating enzyme that has cellulytic activity. Preferably, the enzyme composition also exhibits hemicellulytic activity. Suitable enzymes and enzyme-containing compositions include those described in U.S. Patent Nos. 5,356,800 and 6,342,381 to Jaquess, and International Publication No. WO 99/43780, all incorporated herein in their entireties by reference. Other exemplary papermaking pulp-treating enzymes are BUZYME™ 2523 and BUZYME™ 2524, both available from Buckman Laboratories International, Inc., Memphis, Tenn. A preferred cellulytic enzyme composition preferably contains from about 5% by weight to about 20% by weight enzyme. The preferred enzyme composition can further contain polyethylene glycol, hexylene glycol, polyvinylpyrrolidone, tetrahydrofuryl alcohol, glycerine, water, and other conventional enzyme composition additives, as for example, described in U.S. Patent No. 5,356,800. The enzyme may be added to the pulp in any conventional amount, such as in an amount of from about 0.001% by weight to about 0.100% by weight enzyme based on the dry weight of the pulp, for example, from about 0.005% by weight to about 0.05% by weight.

[00049] In one preferred embodiment of the present invention, an enzyme composition is included in the pulp or stock and contains at least one polyamide oligomer and at least one enzyme. The polyamide is present in an effective amount to

stabilize the enzyme. Exemplary enzyme compositions containing polyamide oligomers and enzymes are described in International Published Application No. WO 99/43780, which is incorporated herein in its entirety by reference.

[00050] If an enzyme composition is included, it can include a combination of two or more different enzymes. The enzyme composition can include, for example, a combination of a lipase and a cellulase, and optionally can include a stabilizing agent. The stabilizing agent may be a polyamide oligomer as described herein.

[00051] One particular additive for use according to the methods of the present invention is a cationic starch. Cationic starch may be added to the pulp or stock of the present invention to form a starch treated pulp. Starch may be added at one or more points along the flow of papermaking pulp through the papermaking apparatus or system of the present invention. For instance, cationic starch can be added to a pulp at about the same time that the acidic aqueous alumina sol is added to the pulp. Preferably, if a cationic starch is employed, it is added to the pulp or combined with the pulp prior to introducing the synthetic layered silicate microparticles to the pulp. The cationic starch can alternatively or additionally be added to the pulp after the pulp is first treated with an enzyme, a coagulant, or both. Preferred cationic starches include, but are not limited to, potato starches, corn starches, and other wet-end starches, or combinations thereof.

[00052] Conventional amounts of starch can be added to the pulp. An exemplary amount of starch that can be used according to the present invention is from about 5 to about 25 pounds per ton based on the dried solids weight of the pulp.

[00053] A biocide may be added to the pulp in accordance with conventional uses of biocides in papermaking processes. For example, a biocide may be added to the treated pulp in a blend chest after the pulp has been treated with the optional enzyme and polymer. Biocides useful in the papermaking pulps according to the present invention include biocides well known to those skilled in the art, for example,

biocides available from Buckman Laboratories International, Inc., Memphis, Tenn., such as BUSAN™ biocides.

[00054] The pulps or stocks of the present invention may additionally be treated with one or more other components, including polymers such as anionic and non-ionic polymers, clays, other fillers, dyes, pigments, defoamers, pH adjusting agents such as alum, microbiocides, and other conventional papermaking or processing additives. These additives can be added before, during, or after introduction of the synthetic layered silicate microparticles. Preferably, the synthetic layered silicate microparticles are added after most, if not all, other additives and components are added to the pulp. Thus, the synthetic layered silicate microparticles can be added to the papermaking pulp after the addition of enzymes, coagulants, flocculants, fillers, and other conventional and non-conventional papermaking additives.

[00055] The addition of the retention system in accordance with the present invention can be practiced on most, if not all, conventional papermaking machines.

[00056] A flow chart of a papermaking system for carrying out one of the methods of the present invention is set forth in FIG. 1. It is to be understood that the system shown is exemplary of the present invention and is in no way intended to restrict the scope of the invention. In the system of FIG. 1, an optional supply of enzyme composition at a desired concentration is combined with a flowing stream of papermaking pulp to form a treated pulp. The supply of pulp shown represents a flow of pulp, as for example, supplied from a pulp holding tank or silo. The supply of pulp shown in FIG. 1 can be a conduit, holding tank, or mixing tank, or other container, passageway, or mixing zone for the flow of pulp. The supply of enzyme composition can be, for example, a holding tank having an outlet in communication with an inlet of a treated pulp tank.

[00057] The pulp treated with the enzyme composition is passed from the treated pulp tank through a refiner and then through a blend chest where optional additives, for example, a biocide, may be combined with the treated pulp. The refiner has an inlet

in communication with an outlet of the treated pulp tank, and an outlet in communication with an inlet of the blend chest.

[00058] According to the embodiment of FIG. 1, the pulp treated in the blend chest is passed from an outlet of the blend chest through a communication to an inlet of a machine chest where optional additives may be combined with the treated pulp. The blend chest and machine chest can be of any conventional type known to those skilled in the art. The machine chest ensures a level head, that is, a constant pressure on the treated pulp or stock throughout the downstream portion of the system, particularly at the head box.

[00059] From the machine chest, the pulp is passed to a white water silo and then to a fan pump. The retention system polymer of the present invention is preferably introduced into the flow of pulp between the silo and the fan pump. The supply of retention system polymer composition can be, for example, a holding tank having an outlet in communication with a line between the white water silo and the fan pump. As pulp passes from the fan pump to a screen, the synthetic layered silicate microparticles are preferably added. Conventional valving and pumps used in connection with introducing conventional additives can be used. The screened pulp passes to a head box where a wet papersheet is made on a wire and drained. In the system of FIG. 1, drained pulp resulting from papermaking in the headbox is recirculated to the white water silo.

[00060] In the embodiment shown in FIG. 2, the synthetic layered silicate microparticles are added first to the refined treated pulp between the white water silo and the fan pump. The retention system polymer is added after the fan pump and before the screen.

[00061] Another embodiment of the present invention is shown in FIG. 3. A pulp optionally treated with a cationic starch is refined, passed to a blend chest, passed to a machine chest, and then passed to a white water silo. Between the white water silo and the fan pump the retention system polymer is preferably added to the pulp. The

synthetic layered silicate microparticles are preferably added after the pulp passes through the screen and just prior to sheet formation in the head box.

[00062] The apparatus of the present invention can also include metering devices for providing a suitable concentration of the synthetic layered silicate microparticles or other additives to the flow of pulp.

[00063] A cleaner, for example, a centrifugal force cleaning device, can be disposed between, for instance, the fan pump and the screen, according to any of the embodiments of FIGS. 1-3 above.

[00064] Figures 4 and 5 are flow charts illustrating the polymer and microparticle addition steps in two particularly preferred embodiments of the present invention. It will be appreciated that Figures 4 and 5 illustrate only those components (i.e. fan pump, screen and headbox) and addition steps which are necessary to describe the polymer and microparticle addition steps in these preferred processes, and that the processes and apparatus illustrated in Figures 4 and 5 may preferably include some or all of the optional additives, apparatus components and/or process steps shown in Figures 1 to 3 and described above.

[00065] The pulp passes through the apparatus of Figures 4 and 5 in the direction indicated by the arrows, passing through the fan pump and the screen on its way to the headbox. The pulp is sheared by both the fan pump and the screen, however the shear applied to the pulp by the screen is greater than that applied by the fan pump, so that the screen is the final high shear stage in the papermaking process prior to entry of the pulp into the headbox of the papermaking apparatus.

[00066] In both Figures 4 and 5, a coagulant polymer is preferably added before the fan pump. The coagulant preferably comprises a relatively low molecular weight, cationic, high charge density polymer to scavenge and collect colloidal particles, primarily anionic fibers and fillers. The colloidal particles are coagulated to form macro-colloids which are larger in size and more easily retained in the sheet upon

drainage. The coagulant is preferably a polyamine or diallyldimethylammonium chloride (DADMAC) polymer, or copolymers thereof. A particularly preferred coagulant for use in the processes illustrated in Figures 4 and 5 is BUFLOC™ 5376, available from Buckman Laboratories International, Inc., which is a cationic DADMAC having a 95% charge density and a molecular weight of about 500,000. The coagulant polymer is preferably added to the pulp in an amount of from about 0.05 to about 1.0 kg/tonne of pulp on a dry basis, more preferably from about 0.1 to about 0.5 kg/tonne and even more preferably about 0.3 kg/tonne.

[00067] The processes of Figures 4 and 5 both include the addition of a microparticle-containing retention aid system comprising a retention system polymer and a microparticle. The microparticle is a synthetic layered silicate, more preferably a synthetic hydrous sodium lithium magnesium silicate, and even more preferably a synthetic hydrous sodium lithium magnesium silicate in combination with a peptizer, the most preferred peptizer being tetrasodium pyrophosphate. The microparticle preferably comprises one or more of Laponite RDS, XLS and DS, and more preferably comprises Laponite RDS. The microparticle is preferably added in an amount of from about 0.1 to about 1.0 kg/tonne of pulp on a dry basis, more preferably from about 0.2 to about 0.6 kg/tonne and even more preferably about 0.4 kg/tonne.

[00068] The retention system polymer is preferably a flocculant and may preferably comprise any of the synthetic nitrogen-containing cationic polymers described above. A particularly preferred retention system polymer is BUFLOC™ 5511, available from Buckman Laboratories International, Inc., which is a cationic polyacrylamide having a molecular weight of about 10,000,000. The retention system polymer is preferably added to the pulp in an amount of about 0.05 to 1.0 kg/tonne of pulp on a dry basis, more preferably from about 0.05 to about 0.5 kg/tonne and even more preferably from about 0.1 to about 0.2 kg/tonne.

[00069] The flow charts of Figures 4 and 5 differ from one another in the order of addition of the retention system polymer and the microparticle. In Figure 4, the

microparticle is added before the screen, more preferably between the fan pump and the screen, while the retention system polymer is added after the screen, more preferably between the screen and the head box. In Figure 5, the order of addition is reversed, with the polymer being added before the screen, more preferably between the fan pump and the screen, and the microparticle being added after the screen, more preferably between the screen and the head box. The order of addition in Figure 5 is preferred.

[00070] The invention is further described in the following examples.

EXAMPLES

[00071] In the examples below, various components used in the examples are abbreviated. In the examples, the components identified as "RD", "RDS" and "JS" are Laponite RD, Laponite RDS and Laponite JS respectively, available from Rockwood Additives Limited. Laponite RD is a hydrous sodium lithium magnesium silicate; Laponite RDS is a hydrous sodium lithium magnesium silicate with tetrasodium pyrophosphate; and Laponite JS is a hydrous sodium lithium magnesium fluorosilicate with tetrasodium pyrophosphate. When followed by a numerical value, for example, "RDS 0.5", the numerical value represents the amount of pounds on a dry basis of the Laponite microparticles per ton of paperstock based on the dried solids weight of the paperstock.

[00072] In the examples below, the abbreviations "B 594" and "594" represent BUFLOC™ 594, available from Buckman Laboratories International, Inc., which is a high molecular weight cationic polyacrylamide having an average molecular weight of from about 5,000,000 to about 7,000,000 units and a 21% charge density. When followed by a numerical value, for example, "594 0.5", the numerical value represents the amount of pounds on a dry basis of the Bufloc 594 polymer per ton of paperstock based on the dried solids weight of the paperstock.

[00073] The abbreviations "B 5511" and "5511" represent BUFLOC™ 5511, available from Buckman Laboratories International, Inc., which is a cationic

polyacrylamide having a molecular weight of about 10,000,000. When followed by a numerical value, for example, "5511 0.5", the numerical value represents the amount of pounds on a dry basis of the Bufloc 5511 polymer per ton of paperstock based on the dried solids weight of the paperstock.

[00074] In samples where both a polymer and a microparticle component are added, the order of addition is specified. For example, the abbreviation "B 5511 0.5/RDS 0.5" indicates that the polymer component Bufloc 5511 is added to the furnish before the microparticle component Laponite RDS. This simulates a papermaking process in which the polymer is added prior to the final high shear stage (typically before the screen) and the microparticle is added after the final high shear stage (typically between the screen and the headbox). Similarly, the abbreviation "RDS 0.5/B 5511 0.5" indicates that the polymer component Bufloc 5511 is added to the furnish after the microparticle component Laponite RDS. This simulates a papermaking process in which the microparticle is added prior to the final high shear stage (typically before the screen) and the polymer is added after the final high shear stage (typically between the screen and the headbox).

Example I – Drainage and Turbidity Tests

[00075] Tests were conducted at a paper mill. Drainage was performed using a small screen through which 500 ml samples were drained using a modified Schopper Riegler method. Mixing was carried out in a food blender.

[00076] Equipment used for the modified Schopper Riegler drainage test included the following: a Modified Schopper Riegler (MSR); a 1000 ml graduated cylinder; a stopwatch; a 5-gallon (18.9 l) plastic bucket; wires for MSR; a vacuum flask and funnel (for retention); Whatman ashless filter papers (for ash retention); a turbidity meter; a hemocytometer; and a microscope.

[00077] Samples to be tested were taken from the headbox of the papermaking apparatus. For each test, 1000 ml was required. Because temperature has an impact on drainage, each test was run immediately after the sample was taken. For lab

studies with the retention aids, the furnish was kept at the same temperature as the headbox temperature.

[00078] If the MSR was cold and the sample was hot, the MSR was warmed up by running hot water over the outside and inside of the MSR. If no hot water was available, cold water was used. All tests were conducted in the same way. It was imperative that the MSR wire was devoid of any fibers or fines. The wire was backflushed with water before the test was run. Uniform fiber, fines, and filler distribution in the sample was ensured by agitating the fiber slurry in the bucket. 1000 ml of the slurry was measured in a graduated cylinder and poured into the MSR while holding the plunger down. The graduated cylinder was placed under the MSR. The plunger was then released and the stop watch started at the same time. The time required for drainage of the sample in incremental units of 100 ml was measured and recorded. The incremental units of 100 ml chosen were purely empirical. For example, very slow draining stock samples were instead measured at 100, 150, and 200 ml drainage times. Sometimes several tests were needed to determine the starting volume tests. The different levels of polymers in the various samples were compared, and for this purpose, furnish samples were obtained from the machine before addition of the retention/drainage aid. Drainage and retention values were compared against blank furnishes to determine improvement. To measure retention performance, the MSR filtrate was filtered through a pre-weighed filter paper, dried in an oven at from 105°C. to 120°C and weighed again. The weight difference was recorded in mg/ml.

[00079] Drainage times were compared based on different levels of additives (i.e. polymer and/or microparticle) in the furnish. Drainage times were recorded in seconds for each volume level. The total suspended solids were estimated with a turbidity meter. The filtrate could also have been filtered to determine suspended solids. Solids contents of MSR filtrate could be reported in mg/ml and used to indicate the retention capabilities of different systems, with lower numbers indicating better retention.

[00080] For repeated tests, the sample was taken from the same place along the papermaking system. It was ensured that the furnish composition was the same for the repeated test. Repeated tests that did not agree within reason with a corresponding original test were suspect.

[00081] The MSR was kept clean and constantly rinsed with water to keep residual fibers from building up on the sides. The screen was periodically cleaned to remove resin build-up, and brushed clean with a mild detergent. The wires were checked to make sure bent or damaged wires were not used. All tests were conducted in the same manner and at the same consistency.

[00082] The paper mill employed a newsprint furnish comprising 70 wt % thermomechanical pulp (TMP) and 30% de-inked pulp (DIP). The pulp had a headbox conductivity of 1,000 microsiemens, a cationic demand of 0.15 ml/l of 0.001N solution and a consistency of 0.65. The headbox pH of the paperstock was 4.83. Additives combined with the paperstock included calcined clay as a filler in an amount of 2 wt% based on the dried solids weight of the paperstock. The calcined clay was present in the DIP component.

[00083] Polymer was added to the paperstock in varying amounts up to 0.75 lb/ton (0.35 kg/tonne) of paperstock, based on the dried solids weight of both the polymer and the paperstock.

[00084] The microparticle was added to the paperstock in varying amounts up to 1.0 lb/ton (0.46 kg/tonne). All microparticle dosages were calculated on dry basis.

[00085] The results of the tests are shown in Tables 1 and 2 below. (Table 1 will contain the drainage/turbidity data and Table 2 will contain the retention data). In Table 1, the column headings "100", "150", "200" and "250" represent the number of milliliters of filtrate collected that drained through the wire. The corresponding numbers underneath the column headings represent the number of seconds needed for the respective number of milliliters (ml) of filtrate to drain through the wire and be

collected. For example, in the first entry of Table 1, the paperstock identified as "Blank", (having no microparticle retention system) required 27 seconds for 100 ml of filtrate to be drained through the forming wire and collected, required 58 seconds for 150 ml of filtrate to be collected, and required 90 seconds for 200 ml of filtrate to be collected. In Table 1 the turbidity, measured in units of nephelometric turbidity units (NTU), is listed in the last column. For each of the various samples tested and reported in Table 1 which include both a microparticle additive and a polymer, the order of addition is specified in the table. For example, in samples 5 to 7 and 11 to 13 of Table 1, the polymer was added first. In samples 8 to 10 the microparticle was added first.

TABLE I – Drainage and Turbidity

Sample No.	Sample Composition*	100 ml.	150 ml.	200 ml.	250 ml.	Turbidity
1	Blank	27	58	90	141	249
2	B 5511 0.25	25	55	87	129	193
3	B 5511 0.5	22	46	76	112	211
4	B 5511 0.75	24	49	78	112	210
5	B 5511 0.5/RDS 0.5	17	38	64	93	114
6	B 5511 0.5/RDS 0.75	15	31	52	75	70
7	B 5511 0.5/RDS 1	13	29	48	72	91
8	RDS 0.5/B 5511 0.5	14	30	50	70	97
9	RDS 0.75/B 5511 0.6	15	30	50	70	94
10	RDS 1/B 5511 0.7	13	28	46	64	101
11	B 5511 0.5/RD 0.5	15	45	74	102	150
12	B 5511 0.5/RD 0.75	15	39	64	94	129
13	B 5511 0.5/RD 1	14	34	55	83	91
14	Blank 2	29	58	93	139	241

* All sample compositions in lb/ton

The data shown in Table 1 is graphically represented in Figures 6 and 7.

Example 2 – Retention Tests

[00086] Britt Jar tests were performed at 1,000 rpm to evaluate performance of the retention aid system according to the invention based on increased first pass retention and increased first pass ash retention. In the Britt Jar, the furnish is under continuous movement, so no fiber mat is formed and the water can drain continuously through the wire. This simulates the stock and water dewatering occurring on the paper machine.

[00087] The furnish used in the Britt Jar tests was identical to the furnish used in Example 1.

[00088] In the Britt Jar tests, the chemicals are applied in the correct sequence and the furnish is mixed to a degree to simulate the treatment of the furnish by paper machine equipment such as fan pumps and centri-screens. As it is sheared, the stock spends a second (at the most) actually in the fan pump or the screens. Another way to simulate the shear and to represent the actual short contact time is to carry out all chemical mixing outside the Britt Jar Retention tester. Chemical additive mixing is carried out using a common household food blender.

[00089] The mixing process is the same for the retention as well as drainage work.

[00090] All high shear mixing is carried out outside the Britt Jar using a food blender since this is more representative of the high shear points in the papermaking process. This also avoids plugging the Britt Jar wire. Each chemical addition is followed by a short two-second burst in the food blender. This simulates stock (including coagulant, starch, high molecular weight polymer) going through the fan pump.

[00091] To simulate pre-screen addition of polymer or microparticle component a short one-second burst in the food blender is used. To simulate post-screen addition of polymer or microparticle the treated stock sample is poured into a cylinder, followed by addition of the polymer or microparticle component and inversion of the Britt Jar four times.

[00092] The following equipment was used in the Britt Jar tests: Britt Jar tester; wires— 80P for newsprint furnishes; pre-conditioned ashless filter papers – Whatman # 41 or 43; diluted polymer samples; stock sample (at least 20 liters); balance to 0.001g; buchner funnel; drying oven; furnace for ash determination; plastic containers with lids; syringes – 1, 5, and 10 mL; and a blender—standard household food blender with pulse feature.

[00093] Each of the stock and polymer samples were prepared as follows: prepare stock and polymer samples; make sure the wire for the Britt Jar is wet; set the Britt Jar speed at the required set point and turn it on; mix chemicals into stock; pour treated stock into Britt Jar; wait 5 seconds; open clamp and start collecting filtrate; collect first 100 mL of filtrate; filter the filtrate through the pre-conditioned filter papers and dry in the oven at 110°C; and calculate the %TFPR. If required ash the dried filter papers to determine the %FPAR.

[00094] If required ash the dried filter papers to determine the %FPAR.

Calculations

1. Consistency = % solids in sample, and is represented by [] symbol
$$= 100 \times (\text{wt suspended solids}) / (\text{wt or vol. of the original sample})$$

2. FPR, First Past Retention = % of HB solids retained in sheet
$$= ([\text{HB}] - [\text{TW}]) / [\text{HB}] \times 100$$

Example: [HB] = 0.75%, [TW] = 0.18%

$$\text{FPR} = 100 (0.75 - 0.18) / 0.75 = 76\%$$

3. $\text{FPAR, First Pass Ash Retention} = ([\text{HB ash}] - [\text{TW ash}]) / [\text{HB ash}] \times 100$

[00095] [HB ash] is determined by ashing the HB, then multiplying the % ash value by the [HB]. [TW ash] is determined by ashing the TW, then multiplying the % ash value by the [TW].

[00096] The results of the retention tests are shown below in Table 2.

TABLE 2 – Retention

SAMPLE NO.	COMPOSITION	TFPR/percent
1	Blank	24.22
2	594 0.5	48.99
3	594 0.5/RD 0.5	59.05
4	594 0.5/RD 1	63.26
5	594 0.5/RDS 0.5	65.66
6	594 0.5/RDS 1	71.71

The data shown in Table 2 is graphically illustrated in Figure 8.

Example 3 – Drainage Tests Comparing Laponite RD, RDS and JS

[00097] Drainage tests were conducted on an alkaline furnish following the procedure described in Example 1.

[00098] All the samples containing a microparticle also contained a polymer, Bufloc 5511. In most of these tests, Bufloc 5511 was added to the furnish before the microparticle. For Laponite RDS, tests were also conducted with the reverse order of addition.

[00099] The results of the drainage tests are illustrated in Figures 9 and 10. As shown in these figures, the furnish containing Laponite RDS had better drainage

properties than the furnish containing either Laponite JS or Laponite RD. In addition, drainage results using RDS were better when the polymer (Bufloc 5511) was added before the Laponite RDS than when Laponite RDS was added last.

Example 4 – Retention Tests Comparing Laponite RD, RDS and JS

[000100] Britt Jar tests as described in Example 2 were performed using various combinations of polymer (Bufloc 5511) and microparticles. The tests were performed with an alkaline fine paper furnish comprised of 60% hardwood and 40% softwood, having a pH of 7.9, conductivity of 670 microsiemens and ash content of 20% precipitated calcium carbonate (PCC) added at the machine chest in the paper process. The retention data, including both total first pass retention (TFPR) and first pass ash retention (FPAR) is shown below in Table 3 and is also illustrated in Figure 11.

TABLE 3

SAMPLE NO.	COMPOSITION	TFPR/percent	FPAR/percent
1	Blank	50.97	18.14
2	5511 0.5	78.72	69.16
3	5511 0.5/JS 0.5	78.93	71.49
4	5511 0.5/JS 0.75	82.80	73.05
5	5511 0.5/RD 0.5	81.44	73.15
6	5511 0.5/RD 0.75	84.99	76.46
7	RDS 0.5/5511 0.5	84.22	79.59
8	RDS 0.75/5511 0.5	86.63	80.58
9	5511 0.5/RDS 0.5	87.21	81.02
10	5511 0.5/RDS 0.75	89.99	82.04

[000101] As shown in Table 3 and Figure 11, the retention results for Laponite RDS were better than the results obtained for comparable concentrations of Laponite

JS and Laponite RD. Also, the results show that addition of Bufloc 5511 followed by addition of Laponite RDS provided the best results.

[000102] It will be apparent to those skilled in the art that various modifications and variations can be made to the embodiments of the present invention without departing from the spirit or scope of the present invention. Thus, it is intended that the present invention covers other modifications and variations of this invention within the scope of the appended claims and their equivalents.

What is claimed is:

1. A method of making paper or paperboard comprising:
 - (a) forming a treated pulp by adding to a papermaking pulp a synthetic layered silicate, a peptizer and at least one polymer, the synthetic layered silicate comprising a synthetic hydrous sodium lithium magnesium silicate and the at least one polymer comprising one or more members of the group consisting of cationic polymers, nonionic polymers and amphoteric polymers under cationic conditions; and
 - (b) forming the treated pulp into said paper or paperboard.
2. The method of claim 1, wherein the synthetic layered silicate comprises laponite.
3. The method of claim 1, wherein the synthetic layered silicate is added to the pulp in an amount of at least about 0.05 pound on a dry basis, per ton of pulp based on the dried solids weight of the pulp.
4. The method of claim 1, wherein the synthetic layered silicate is added to the pulp in an amount of from about 0.1 pounds to about 5.0 pounds on a dry basis, per ton of pulp based on the dried solids weight of the pulp.
5. The method of claim 1, wherein the synthetic layered silicate is added to the pulp in an amount of from about 0.2 pounds to about 1.0 pounds on a dry basis, per ton of pulp based on the dried solids weight of the pulp.
6. The method of claim 1, wherein the peptizer comprises an inorganic polyphosphate peptizer.
7. The method of claim 1, wherein the peptizer comprises tetrasodium pyrophosphate.

8. The method of claim 1, wherein the synthetic layered silicate is added to the pulp in the form of an aqueous dispersion and wherein the dispersion contains said inorganic polyphosphate peptizer in an amount sufficient to maintain said dispersion in the form of a sol for a predetermined period of time.
9. The method of claim 5, wherein the aqueous dispersion contains said synthetic layered silicate in an amount of up to about 10 wt%.
10. The method of claim 1, wherein the cationic polymer is present and comprises a synthetic nitrogen-containing cationic polymer.
11. The method of claim 1, wherein the cationic polymer is present and comprises one or more members of the group consisting of cationic polyacrylamides and copolymers thereof; and cationic diallyldimethylammonium chloride and copolymers thereof.
12. The method of claim 1, wherein the cationic polymer is present and is added to the pulp in an amount of at least about 0.01 pound on a dry basis, per ton of pulp based on the dried solids weight of the pulp.
13. The method of claim 1, wherein the cationic polymer is present and is added to the pulp in an amount of from about 0.1 pound to about 5 pounds on a dry basis, per ton of pulp based on the dried solids weight of the pulp.
14. The method of claim 1, wherein the cationic polymer is present and is added to the pulp in an amount of from about 0.2 to about 2 pounds on a dry basis, per ton of pulp based on the dried solids weight of the pulp.
15. The method of claim 1, further comprising shearing the pulp in one or more shear stages including a final high shear stage in which the pulp is passed through a screen prior to entering a headbox of a papermaking apparatus.

16. The method of claim 15, wherein the polymer is added to the pulp before the final high shear stage and wherein the synthetic layered silicate is added to the pulp after the final high shear stage.

17. The method of claim 15, wherein the synthetic layered silicate is added to the pulp before the final high shear stage and wherein the polymer is added to the pulp after the final high shear stage.

18. A method of making paper or paperboard comprising:

(a) adding to a papermaking pulp, a synthetic layered silicate, a peptizer and at least one polymer, the synthetic layered silicate comprising a synthetic hydrous sodium lithium magnesium silicate, the peptizer comprising an inorganic polyphosphate peptizer, and the at least one polymer comprising one or more members of the group consisting of cationic polymers, nonionic polymer and amphoteric polymers under cationic conditions,

wherein the synthetic layered silicate is added to the pulp in the form of a sol and wherein the sol contains said peptizer in an amount sufficient to maintain said sol in stable form for about 3 days to about 90 days; and

(b) forming the pulp from (a) into said paper or paperboard

wherein the pulp comprises more synthetic layered silicate than peptizer,

and said, method further comprising shearing the pulp in one or more shear stages including a final high shear stage in which the pulp is passed through a screen prior to entering a headbox of a papermaking apparatus, and either (i) the polymer is added to the pulp before the final high shear stage and the synthetic layered silicate is added to the pulp after the final high shear stage, or (ii) the synthetic layered silicate is added to the pulp before the final high stage and the polymer is added to the pulp after the final high shear stage.

19. The method of claim 1, wherein the synthetic layered silicate comprises 59.5% SiO₂; 27.5% MgO; 0.8% Li₂O; and 2.8% Na₂O.