Patented Apr. 13, 1954

2,675,312

UNITED STATES PATENT OFFICE

2,675,312

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No Drawing. Application September 29, 1950, Serial No. 187,665

1 Claim. (Cl. 92—21)

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My invention relates to a new and improved method of making cellulose pulp or paper products, which include paper. The invention also includes certain new and improved cellulose pulp or paper products. The invention includes certain cellulose pulp or paper products as the result of the improved method and certain other cellulose pulp or paper products irrespective of their method of manufacture.

Without limitation thereto, the new method is particularly useful in making paper products from reclaimed paper. Reclaimed paper is extensively used as a starting material in making various types of paper products including corrugated paper and uncorrugated paper, cartons, liners, and paper board.

The paper which is used as the starting material consists wholly or in part of cellulose chemical pulp, such as cellulose chemical pulp which has been made by the soda process or the sulfite process or the sulfate process.

In the known process of reclaiming paper, the paper starting material is beaten with water in a hydrator or beater or hydro-pulper, wherein the cellulose fiber is fibrillated and hydrated or plasticized, and the resultant fibrillated and plasticized cellulose fiber is uniformly intermixed with water. This pulp mixture is then treated in the old process in various well-known steps, such as forming a web by screening the mixture of cellulose pulp and water, dewatering the screened stock in order to thicken it by concentrating the cellulose pulp fibers, bleaching, removing ink from the starting material, conditioning, sheet fabrication, adding non-fiber or filler or loading ingredients such as size, starch, clay, talc, etc.

It has been well-known to use a mixture of kraft paper and newsprint paper as starting material in the old reclaiming process.

Kraft paper is a long fibered paper which is made originally by the sulfate process, in which chipped wood is cooked under pressure with an aqueous solution of sodium hydroxide and sodium sulfide.

Newsprint paper is usually made from a mixture of mechanical cellulose pulp and sulfite pulp, with about 75%–80% by weight of mechanical cellulose pulp. In the sulfite process, chipped wood is cooked under pressure in an acid aqueous solution of calcium bisulfite or other bisulfite and sulfurous acid.

In order to make paper board, cartons, wrapping material and various other paper products, it has been customary to use 80% of kraft paper

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and 20% of newsprint paper in the old reclaiming process, said ratio being by weight.

According to my invention, it is possible to use a mixture of 70% of kraft paper and 30% of newsprint paper as starting material in the new reclaiming process; and even as little as 60% by weight of kraft paper and 40% of newsprint paper, while providing the final paper product with the necessary hardness and resistance and sizing. This use of a small proportion of kraft paper is desirable because it lowers the cost of the starting material. Also, according to my invention, I can make reclaimed paper which is suitable for many uses, for which reclaimed paper has not been acceptable.

As one feature of my invention, I use sulfur of suitable small particle size as a hardening agent and also as a sizing agent.

Also, the sulfur protects the finished paper product from fungi, and spores, and other microorganisms.

As one feature of the invention, I beat and hydrate the paper or other cellulose starting material in an aqueous suspension or dispersion of sulfur, in which the suspensoidal or dispersed particles of sulfur have a suitable small particle size. The cellulose fibers are thus fibrillated and plasticized and hydrated in the beater or hydrator, in the presence of the suspensoidal or dispersed particles of sulfur. This step is performed at a temperature which is sufficiently low to prevent the sulfur from substantially reacting with the water in which said particles of sulfur are dispersed or suspended, or from substantially reacting with oxygen which is dissolved in said water or which is carried into the water as bubbles by the beating. I thus substantially prevent the formation of hydrogen sulfide and sulfur dioxide, which is an important advantage. The formation of these objectionable products is substantially prevented throughout the new process.

The suspensoidal or dispersed particles of sulfur are negatively charged in the water or aqueous phase of the suspension or dispersion, in the beater. The fibrils or tendrils of the cellulose fibers thus pick up and retain and adsorb the fine sulfur particles during the hydration. When the pulp is screened on a forming screen in order to form an initial web, the sulfur particles remain incorporated in the cellulose fibers and little or no sulfur particles remain in the water in the beater. At least 90% by weight of the sulfur particles can be thus removed from the aqueous phase of the aqueous suspension or col-

loidal dispersion of the sulfur particles. If the sulfur particles are too large in the water of the beater or hydrator, they are not picked up by the plasticized and fibrillated cellulose fibers during the beating. The web which is produced on the forming screen is heated under pressure, as by means of pairs of heated pressure rolls. The fibrillated cellulose fibers are thus compressed and retain the sulfur.

As an additional preferred and optional feature of the invention, the pH of the water in the beater is at least substantially seven, and said pH is preferably and optionally above seven. This aids in maintaining a stable suspension or dispersion of the suspensoidal particles of sulfur and other suspended particles.

As another additional preferred and optional feature of the invention, said suspension in the beater or hydrator includes finely divided suspensoidal or colloidally dispersed particles of a non-sulfur filler. These particles of the filler are negatively charged in the aqueous phase of the suspension or dispersion in the beater or hydrator. Such non-sulfur fillers are exemplified by clay. The suspensoidal sulfur particles agglomerate very readily to form aggregates in the aqueous dispersion or suspension. The negatively charged suspensoidal non-sulfur filler particles prevent this undesirable agglomeration of the sulfur particles.

As another additional preferred and optional feature of the invention, said dispersion or suspension in the beater includes a wetting agent or surface-active agent. This agent is exemplified by di-octyl sulfosuccinate, which is sold commercially as "Vatsol OT." This is a powdered wetting agent or surface-active agent which is used to increase the spreading of insecticidal dusts and sprays. It is described at page 457 of "Handbook of Material Trade Names," by Zimmerman and Lavine, published in 1946 by Industrial Research Service. This wetting agent is insoluble in water, but I can use other wetting agents which are soluble in water. This wetting or surface-active agent aids in causing the cellulose fiber to pick up the sulfur particles.

As another additional preferred and optional feature of my invention, I use a solution of a base or alkali or salt as the aqueous phase of the suspension or dispersion in the beater, so that the aqueous phase has a preferred pH of 7.4 to 7.8. As one example of this ingredient, which also acts as a peptizing or dispersing agent when dissolved in the water, I use ammonium carbonate. This also has the advantage of preventing the sliming of the water. Also, a mixture of dry, undissolved ammonium carbonate particles with dry sulfur particles prevents the dry particles of sulfur from agglomerating. The pH of the solution of ammonium carbonate or other base or alkali or salt, maintains a stable dispersion of the water-insoluble ingredients in the water. By maintaining said pH, instead of acidifying the aqueous dispersion before the cellulose fiber and the picked-up sulfur particles, I maintain the dispersed particles in the undeflocculated state.

In order to use these four ingredients, it is preferred to mix and grind relatively coarse particles of the sulfur, clay, di-octyl sulfo-succinate, and ammonium carbonate, in order to form an initial coarse and intimate and solid mixture. This initial coarse mixture is then reduced to final small particle size under agitation, preferably by passing it through a micronizer mill. The micronizer and the micronizing process are described in "The Rubber Age, N. Y.," issue of January 1944 and in numerous other publications. In the micronizing process, the material is circulated at high speed through a circular micronizer or grinding chamber by jets of air, steam, etc., so that the material is rubbed against the wall of the grinding chamber, and said material is milled to small particle size.

It is advantageous to form an initial dry mixture of the di-octyl and sulfo-succinate and clay and sulfur and ammonium carbonate, and then to reduce the ingredients of this initial dry mixture simultaneously to final particle size. For best results, the average and maximum particle size of the micronized mixture is 3.5 microns. The invention is not limited to this size. The average or maximum particle size may be 4–5 microns, or even more. The average or maximum particle size may be less than 3.5 microns. The dry sulfur agglomerates readily in the nozzles of the micronizer, thus clogging said nozzles. The ammonium carbonate prevents this agglomeration of the fine, dry sulfur particles, by preventing the formation of large charges of static electricity on the fine, dry sulfur particles during the micronizing process.

The invention is further explained in the following example, without being limited to its ingredients or proportions or other details.

In this example, the starting material consists of 70% by weight of kraft paper and 30% of newsprint paper.

A dry micronized mixture is made as above described. This dry mixture consists of 2.5% of di-octyl sulfo-succinate; 2.45% of clay; from 0.5% to 1.0% of ammonium carbonate, and 94.3% of sulfur. These proportions are by weight. The weight of the clay particles in this milled mixture is substantially 2.6% of the weight of the sulfur, and the weight of the ammonium carbonate is substantially 0.53% to 1.16% of the weight of the sulfur.

As above noted, the particles of the four ingredients of this dry mixture preferably have a particle size of 3.5 microns or substantially 3.5 microns.

As one illustration, 1,200 pounds of the paper starting material are heated and hydrated and plasticized in the hydrator or beater with 60,000 pounds of water in which 20–25 pounds of the dry mixture are incorporated. Hence one part by weight of the paper starting material is beaten with fifty parts of water, and the weight of the dry mixture is substantially 1.7% to 2.2% of the weight of the starting material, at least substantially 1.3%. Since the weight of the sulfur is substantially 94% of the weight of the mixture, the weight of the sulfur is substantially 1.5% to 2% of the weight of the starting material to which the sulfur is added. As is well-known, a beater is a machine which operates on a batch or charge of cellulosic material mixed with water, to defiber said cellulosic material and also to hydrate the fibers, thus swelling the fibers and producing a fibrillation of the ends of the fibers. The term "beater" has also been and is defined in this art, to designate the batch or charge of cellulosic material and water. As used in the claim herein, the term "beater" designates the mixture of the cellulosic material and water. According to this disclosure, the solution of the ammonium carbonate in said beater is very dilute.

The ratio of the dry mixture to the starting material depends on the condition and composi-

tion of the starting material and the desired gloss and hardness of the paper end-product. The use of more sulfur results in higher gloss and hardness and resistance in the end-product.

A temperature of 29° C. is a good working temperature of the water in the hydrator.

The 1200 pounds of paper starting material are beaten in the hydrator or hydro-pulper with the 60,000 pounds of water, to which the dry mixture is gradually added. After all the dry mixture has been added, the beating and hydration period may be 40 minutes. As above noted, the fibrils or tendrils of the cellulose fiber which are formed in the hydrator, take up the negatively charged suspensoidal particles during this beating period.

After the beating and hydration have been completed, the initial pulp web is formed by means of forming screens, in which a good working temperature of the water is 29° C.–35° C.

The initial pulp web is then pressed between heated rolls, which condense the fibrils or tendrils of the cellulose fibers, without forming sulfur dioxide or hydrogen sulfide. The sulfur acts as a binder, in addition to being a sizing and hardening agent.

The pulp takes up at least 90% of the clay and sulfur and di-octyl sulfo-succinate in the water of the beater. As one example, the finished paper product may contain an average of substantially 1.8% by weight of the clay, and sulfur and di-octyl sulfo-succinate.

A sufficient proportion of clay is used in the dispersion or suspension in the form of fine, negatively charged particles in order to maintain a stable suspension or dispersion of the sulfur particles in the aqueous phase, and also to prevent or minimize the agglomeration of the sulfur particles on the cellulose fibers. The weight of the clay particles is preferably at least substantially 2% of the weight of the sulfur particles.

The proportion of clay or other filler may be varied.

The complete process may include other steps which are well known per se, such as bleaching, de-inking, etc., which require no description.

The invention includes the individual steps and respective sub-combinations of the complete process disclosed herein. The sulfur and clay remain in fine particle size in the finished end-product.

All the operations disclosed herein may be performed under standard atmospheric pressure of 760 millimeters of mercury.

I have disclosed a preferred embodiment of my invention, but numerous changes, substitutions and omissions and additions may be made without departing from its scope.

I claim:

A combination method which consists in milling a mixture which consists substantially of 94.3% of sulfur and 2.5% of di-octyl sulfo-succinate and 2.45% of clay and 0.5% to 1.0% of ammonium carbonate, said mixture being milled dry to produce a final milled mixture which has a particle size of substantially 3.5 microns to 5.9 microns, adding said final milled mixture to a beater to dissolve said ammonium carbonate in said beater, the weight of the dry milled mixture being substantially 1.7% to 2.2% of the weight of the paper starting material in the beater, the proportion of ammonium carbonate being selected to produce a pH of substantially 7.4 to 7.8 in said beater, fibrillizing the cellulose fiber in said beater at substantially 29° C.–35° C. to form fibrils and to incorporate suspended particles of sulfur and clay and di-octyl sulfo-succinate from said beater into said fibrils, separating said fibrillized fiber and said incorporated particles from said beater at substantially 29° C.–35° C. to form an initial pulp web, and heat-pressing said initial pulp web to condense its cellulose fibers.

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