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PAPER MANUFACTURE

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8 Claims. (Cl. 92—21)

The present invention relates to the manufacture of paper from cellulose fibers and more particularly to the production of paper containing ground wood fibers and an alkaline filler.

In the manufacture of paper from pulp containing ground wood fibers it has been known for some time that ordinary alkaline fillers such as calcium carbonate should not be used when a white sheet is desired. Ground wood, for example, containing all of the organic and inorganic materials of natural wood such as lignin, tannins, etc., darkens in color in an alkaline medium. Many suggestions have been made in the past, including means for stabilizing the pH value of alkaline fillers, in the search by previous investigators to find a satisfactory method which would permit the use of an alkaline filler with ground wood in the formation of a paper sheet of high brightness. The prior methods, however, have not been found to be generally satisfactory for in many instances, the alkaline filled ground wood papers prepared by the suggested processes still have a tendency towards discoloration or "yellowing" while in some cases the addition of acids have destroyed the desired characteristics of alkaline filled papers. In addition, many of the suggested processes were complex in nature, involving critical pH adjustments which are difficult to control, making the prior methods unsatisfactory for large scale commercial adaptation. In view of the above, ground wood has been considerably restricted in use, although ground wood which is obtained by mechanical grinding processes is relatively inexpensive compared to the cost of chemically treated wood such as sulphite pulp.

The principal object of my invention is to provide a method of making alkaline filled paper containing ground wood fibers which will be free from discoloration.

Another object of my invention is to provide a method of making paper sheets of high brightness or whiteness in which the sheets contain an alkaline filler and ground wood fibers.

A further object of my invention is to provide a method of using alkaline fillers and ground wood pulp in the manufacture of paper where a white sheet is desired that will be adaptable for large scale commercial utilization.

Other objects will be apparent as the description hereinafter proceeds.

I have discovered that alkaline fillers such as calcium carbonate may be satisfactorily used in the manufacture of paper containing ground wood pulp when the paper contains an aluminum phosphate pigment as an additional filler. I have also discovered that improved results are obtainable when the pulp or furnish containing ground wood fibers is first mixed with an aluminum phosphate pigment or an aluminum phosphate complex prior to the addition or use of the alkaline filler. The aluminum phosphate pigments have an unexpected "protective action" and exhaustive investigations have shown that the aluminum phosphate pigments, particularly when added to ground wood pulp prior to the addition of the alkaline filler, in accordance with my preferred process, keeps the ground wood from darkening in color in the presence of the alkaline filler.

Investigations have shown that the unexpected results obtained through the use of my invention are not due to pH adjustments, including the lowering of pH value. For example, the addition of free phosphoric acid to ground wood pulp followed by the addition of an alkaline filler such as calcium carbonate does not produce the desired results. Paper containing ground wood and made by using free phosphoric acid and an alkaline filler is subject to discoloration in manufacture and the final sheets possess a brightness substantially below sheets containing small amounts of an aluminum phosphate pigment used in accordance with the preferred embodiment of the present invention. Although the theory of my invention is not known, my investigations have shown the aluminum phosphate pigments to possess a peculiar action preventing discoloration of ground wood by alkaline fillers and particularly the calcium carbonate fillers.

The following examples will serve to illustrate the preparation of aluminum phosphate pigments for use in my invention:

Example I

About 600 pounds of sodium hydroxide and about 600 pounds of aluminum hydrate are added to about 290 gallons of water and boiled for approximately 20 to 30 minutes until the solution is clear. The solution is diluted to about 720 gallons with water and then added to about 720 gallons of water to which separately but at the same time about 1640 pounds of 75 percent phosphoric acid diluted to 720 gallons with water is being slowly added. To the above solution sufficient paper makers' alum (aluminum sulphate) is added so that the final solution has a pH value of approximately 5. The precipitate formed may be separated by filtration in the usual manner. In the above example the pro-

portions on a dry basis are approximately 2 parts alkali hydroxide, 2 parts aluminum hydrate, 4 parts phosphoric acid and 2 parts paper makers' alum.

Example II

About 558 pounds of 75 percent phosphoric acid diluted with 558 pounds of water are mixed with about 720 pounds of iron-free paper makers' alum dissolved in about 720 pounds of water. To this mixture is added an aqueous calcium hydroxide suspension containing approximately 100 grams of $Ca(OH)_2$ per liter of water until the pH value has been raised to about 5.0 to 5.2. The calcium aluminum phosphate complex precipitate obtained is an extremely good paper pigment for use in the present invention.

Example III

About 975 pounds of 75 percent phosphoric acid and about 780 pounds of 95 percent sulphuric acid are mixed with 5800 pounds of water. About 585 pounds of aluminum hydrate are added to the above acid mixture which is then heated to a boil for approximately 20 to 30 minutes until the aluminum hydrate dissolves. The pH value may be adjusted by adding the solution to about 7800 pounds of an aqueous suspension of calcium hydroxide containing about 100 grams of calcium hydroxide per liter of water. The use of the mixture of acids appears to form a phosphate-sulphate aluminum complex resulting in a pigment possessing unusual protective properties.

It will be apparent from the above examples that throughout the specification and claims the term "aluminum phosphate pigment" is intended to cover not only substantially insoluble aluminum phosphates, but also various other white physical and chemical combinations or complexes of aluminum and the phosphate radical which are substantially insoluble under paper making conditions and which might occur under varying conditions. Thus compounds suitable for use in the present invention may include acid or basic aluminum phosphates, double phosphates as, for example, the double phosphate of aluminum and calcium; and other modifications or compositions containing various basic or acid radicals which in view of the complex nature of the molecule are difficult to analyze and assign a definite chemical formula.

Reference is made to my co-pending application, Serial No. 151,456, filed July 1, 1937, which describes in detail the preparation of aluminum phosphate pigments. For purposes of the present invention it has been found preferable to precipitate the pigments at pH values of 5 to 7, although pigments obtained at lower or higher (e. g., 4 to 8) pH values have been found satisfactory.

The following example will serve to illustrate the present invention:

Example IV

Paper made up of 50 percent ground wood and 50 percent bleached sulphite fiber furnish and containing 20 percent by weight of inorganic filler material consisting of 90 percent calcium carbonate and 10 percent aluminum phosphate pigment prepared for example by the method of Example I, the aluminum phosphate pigment having been mixed with the ground wood furnish prior to the addition of the calcium carbonate, shows an improvement of approximately 9–10 points in brightness over a sheet made from the same furnish containing no filler. Although the paper above described contains only 2 percent by weight of aluminum phosphate pigment which is used in accordance with the preferred embodiment of my invention, the paper nevertheless shows an improvement of about 3 points in brightness over paper made with a similar fiber furnish containing 20 percent all calcium carbonate filler. The paper made in accordance with my process is free from discoloration ordinarily present in alkaline filled ground wood papers and the improvement in brightness, which is discernible to the eye, is unexpected.

My investigations have shown that in alkaline filled papers the aluminum phosphate pigment should make up at least about 5 percent of the total filler material employed although it is ordinarily preferred to use about 10 percent of aluminum phosphate pigment. The use of less than 5 percent aluminum phosphate pigment results in minor improvements which are not as a general rule of commercial importance. If desired, larger percentages, e. g., 20 to 30 percent, of aluminum phosphate may be used, although the increase in improvement is not proportional and is in most cases substantially the same as that obtained using my preferred percentage.

It will be understood that the aluminum phosphate pigments may be precipitated or formed in the presence of the ground wood fibers, in which case the protecting pigment will be deposited directly on the fibers, or may be separately prepared and added to the furnish. In either case it is preferred in order to obtain the improved brightness as well as freedom from discoloration that the ground wood fibers be thoroughly mixed with aluminum phosphate pigment prior to the introduction of the alkaline filler. It will also be understood by those skilled in the paper art that the common sizing compositions including alum, rosin, starch and the like, may be used in the manufacture of paper in conjunction with the present invention, and in addition that other non-alkaline filler materials such as calcium sulphite, which gives a particularly good result, may be employed with the alkaline and aluminum phosphate fillers, if desired.

It will be understood that although in my preferred process the alkaline filler is added after the addition of the aluminum phosphate pigment, the increase in brightness may be obtained by adding the alkaline filler and aluminum phosphate in admixture or simultaneously to the pulp containing the ground wood fibers. Discoloration may also be prevented or retarded when the aluminum phosphate pigment is added after the alkaline filler, although it is preferred in this case to add the aluminum phosphate complex immediately or shortly after the addition of the alkaline filler.

It will also be understood that my invention includes paper containing various percentages of ground wood, e. g., 10 to 90 percent ground wood. I have found that paper containing about equal proportions of ground wood and sulphite pulp (pulp cooked with bisulphite, sulphur dioxide, etc.) to be particularly adaptable for use in my invention. It will likewise be understood by those skilled in the art that the term "alkaline filler" includes calcium carbonate, the cal-

cium carbonate-magnesium carbonate and hydroxide complexes, etc.

Numerous other applications of my invention will doubtless suggest themselves to those skilled in the art without departing from the spirit of my invention. Hence, I do not wish to be limited to the specific details or embodiments disclosed herein, but intend that the scope of my invention shall be determined from the appended claims, which should be interpreted as broadly as the state of the art will permit.

I claim:

1. In a method of making paper from cellulose fibers which includes substantial amounts of ground wood fibers and an alkaline filler as two of its essential ingredients, the improvement which consists in adding an insoluble aluminum phosphate pigment to said cellulose fibers prior to the use of the alkaline filler in making said paper, the proportions of ingredients employed in said method being such that said pigment makes up at least about 5 per cent of the total filler material in said paper.

2. In a method of making paper from cellulose fibers which includes not less than about 10 per cent ground wood fibers and a substantial amount of calcium carbonate filler as two of its essential ingredients, the improvement which consists in mixing an insoluble aluminum phosphate pigment with said ground wood fibers prior to the addition of the calcium carbonate filler to said cellulose fibers, the proportions of ingredients employed in said method being such that said paper contains at least 10 per cent filler material and said pigment makes up at least about 5 per cent of the total filler material in said paper.

3. In a method of making an alkaline filled paper from a pulp furnish containing 10 to 90 percent ground wood fibers, the improvement which consists in adding an aluminum phosphate pigment to said ground wood fibers prior to the addition of the alkaline filler to said furnish, the proportions of ingredients employed in said method being such that said paper contains at least 10 per cent filler material and said pigment makes up at least about 5 per cent of the total filler material in said paper.

4. The step in the method of making paper from a furnish containing substantial amounts of ground wood fibers, sulphite fibers, and an alkaline filler as three of its essential ingredients, the improvement which consists in mixing an aluminum phosphate pigment with said ground wood fibers prior to the addition of said sulphite fibers and said alkaline filler to the furnish containing said ground wood fibers, the proportions of ingredients employed in said method being such that said paper contains at least 10 per cent filler material and said pigment makes up at least about 5 per cent of the total filler material in said paper.

5. Paper containing a substantial amount of ground wood fibers and an alkaline filler as essential ingredients and an aluminum phosphate pigment, the total filler in said paper being made up of at least about 5 percent of said aluminum phosphate pigment, said paper being free from discoloration ordinarily present in alkaline filled papers containing ground wood fibers.

6. Paper containing sulphite pulp and not less than about 10 percent ground wood fibers and filled with calcium carbonate and an aluminum phosphate pigment, the total filler in said paper being at least 10 percent of the weight of said paper and being made up of at least about 5 to 10 percent of said aluminum phosphate pigment.

7. Paper containing sulphite pulp and 10 to 90 percent ground wood fibers and filler material consisting essentially of an alkaline filler, and an aluminum phosphate pigment, the total filler material being at least about 10 percent of the weight of said paper and the aluminum phosphate pigment being at least about 5 percent of the total filler material.

8. Paper containing about equal proportions of chemical pulp and ground wood fibers and filled with filler material containing a major proportion of calcium carbonate and a minor proportion of an aluminum phosphate pigment, said paper at least 10 percent filler material containing at least about 1 percent based on the weight of the paper of said aluminum phosphate pigment and said pigment making up at least about 5 per cent of the total filler material in said paper.

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CERTIFICATE OF CORRECTION.

Patent No. 2,208,574. July 23, 1940.

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It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 1, second column, line 46, after the word "added" insert --slowly--; page 3, second column, line 42, claim 8, after "paper" insert --containing--; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 1st day of October, A. D. 1940.

(Seal)

Henry Van Arsdale,
Acting Commissioner of Patents.