## UNITED STATES PATENT OFFICE

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## PAPER MANUFACTURE

No Drawing.

Application filed May 1, 1931. Serial No. 534,461.

This invention relates to the manufacture method by which the amount of sizing in the of paper filled with alkaline filler.

The principal object of this invention is to provide an improved method of manufac-5 turing paper filled with alkaline filler wherein size is employed.

An important object is to provide an improved method of manufacturing paper filled with alkaline filler wherein material later to 13 serve as a size precipitant is employed in the beater and alkaline filler and size are added at the dilute stage of the paper making

A further object is the use of a procedure 15 whereby the proportion of size used in an alkaline filler paper mix is susceptible to speedy adjustment.

Other objects and advantages of my invention will become apparent during the course

20 of the following description.

In my copending application Serial No. 304.167, filed September 5, 1928, now matured into Patent No. 1,803,642, issued May 5, 1931, have described a method of sizing paper 25 filled with an alkaline filler wherein an alkaline filler is added to a sized fibrous mix at the wet end of the paper machine. Moreover, in my copending application Serial No. 321,753, filed November 24, 1928, now matured into 30 Patent No. 1,808,070, issued June 2, 1931, I have described a procedure wherein substantially unsized paper filled with alkaline filler is made by adding alum, and if desired an amount of size insufficient to impart a sub-35 stantial degree of sizing to the resulting paper, in the beater and alkaline filler at the wet end of the paper machine.

I have now found that similar results may be obtained by a somewhat different method. 40 This method is by adding alum to the fibrous material in the beater and adding the size and the alkaline filler under conditions favoring the minimizing of the time and/or intimacy of contact of the constituents of the mix, e. g. at the wet end of the paper machine. Under certain conditions this method of operation is preferable to those disclosed in my cited copending applications. For example, as the size is added at the wet end of

furnish can be adjusted very much more quickly than where the size is added in the beater. Another advantage is that where unsized paper is made there are certain times, 55 when the stock is running very satisfactorily on the machine, where it appears to be unnecessary to continue the addition of a small amount of size, and in such case the size being added at the wet end of the paper machine 60 can be discontinued at once; and also can be introduced immediately if trouble begins without waiting for the time interval that would be required to elapse if the sizing were introduced in the beater.

In the preferred practice of my invention I add alum to fibrous material in a beater, which should contain substantially no alkaline filler either added as such or admixed with the fibre. Then after the mix has 70 passed through the subsequent chests, and refining machinery if desired, it is ready to be diluted at the mixing box preparatory to running off on the web-forming device of a paper machine. Preferably at this point 75 or subsequently, alkaline filler may be added to the diluted paper mix together with the size, or before or after the size. I prefer, however, that the size be added first and then the alkaline filler. I may, however, in 80 certain cases, previously mix the alkaline filler with the size and add the mixture at the wet end of the paper machine to the fibrous mix containing alum. In any case the alkaline filler is preferably added in a 95 continuous stream in aqueous suspension, suitably at a concentration of 2-3 lbs. per gallon, and the size in a dilute aqueous solution or emulsion conveniently at about 1-3 per cent. concentration. The broke and/or 90 machine effluents containing alkaline filler are preferably returned to the cycle at the wet end of the paper machine in order not to destroy the alum in the alum-fibre mix, as would occur if contact therewith was effected 95 in the beater or chests.

There is another modification of my invention which I find it convenient to practice at times, and that is a modification in some 30 the paper machine, there is thus provided a respects analogous to that disclosed in my 100

copending application Serial No. 440,645, filed March 31, 1930, wherein two separately prepared fibrous mixes, one containing sized fibre and the other containing unsized fibre 5 and alkaline filler, are mixed at the wet end of the paper machine. In the present instance two fibrous mixes may be separately prepared, one containing fibrous material and alum, and the other containing fibrous ma-10 terial, alkaline filler, and size, and the two fibrous mixes fed separately to the mixing box. There are possible variations of this paper making flow-sheet which I have treated in detail in my copending application just 15 referred to, and reference is made thereto concerning those variations, which are equally applicable in the present instance. The fibre-alum mix in the present invention corresponds to the sized mix in the previously re-20 ferred to application, whereas the size-alkaline filler-fibre mix in the present invention corresponds to the unsized mix in the previous invention. The alkaline filler mix is preferably the one to which the machine broke and/or effluents containing alkaline filler are returned.

In regard to the modification of my invention mentioned immediately above, it may be also practiced by mixing alum with fibre to constitute one fibrous mix, mixing alkaline filler with fibre to constitute a second fibrous mix, and adding the two independent fibrous mixes, together with size in a separate stream. at the mixing box of a paper machine.

Relative to the amounts of materials to be used, the alkaline filler may be used in any amount customarily employed in filled papers. For example, in sized papers it is customary to use 8-15 per cent. by weight in the furnish, whereas in unsized papers the amount may be substantially higher, for example 25 to 30 per cent. In regard to alum, the amount will vary as will be apparent according to conditions but I have found that where substantially unsized paper is to be made, approximately 1 to 2 per cent. on the total furnish is usually a satisfactory amount. The amount of size in such case may be suitably approximately .5 per cent. or there-50 abouts on the total furnish. Where sized paper is to be made, as will be readily understood the amount of size will vary with the type of paper to be made, machine conditions, and the like, but ordinarily it will be found 55 that approximately 2 per cent. size will be ample, and alum in a proportion of 3 to 2 as regards size, is a convenient amount to use. It will be understood of course that the above figures are merely illustrative, and in no way 60 limiting as I may practice my invention with proportions of ingredients varying widely from the above, and still obtain satisfactory

In my various copending applications re-65 ferred to herein as well as in others, I have application Serial No. 533,872, filed April 29, 130

discussed in full the principle on which the expression "minimizing the time and/or intimacy of contact of the constituents of the mix" is based. Briefly it is that alkaline filler, which always is present in the stock 70 from which paper filled with alkaline filler is made in great stoichiometrical excess over the acidic constituent, acts upon the acidic constituent to the ultimate destruction thereof and/or destruc- 75 the tion of the effect maintained by such constituent. (The acidic constituent socalled may be one which is actually acid, and/or one which such as a size precipitate is capable of being affected or deteriorated by an alkaline material.) The action just referred to has been found to depend upon the time of contact of the acidic and alkaline constituents and also upon the intimacy of contact of these constituents. Agitation such as 85 normally occurs in papermaking procedure in stuff chests or the like accelerates the rate of reaction, but agitation at low concentrations while having some effect is not nearly so deleterious. Based on this principle a prac- 96 tical application has been developed whereby, by minimizing the time and/or intimacy of contact of the acidic and alkaline constituents, the acidic constituent and/or the effect produced thereby, is substantially preserved in an alkaline filler mix. With the present types of paper machines and the present arrangement of apparatus usually employed in paper machine operation, the contact of the acidic material and the alkaline 100 constituent at any time prior to the passage of the stock from the last stuff chest, which is commonly called the "machine chest", results in great deterioration or complete destruction of the acidic material. Addition 105 of one or both of these constituents at a point subsequent to the passage of the stock from the machine chest, after which point according to present day operating practice the stock remains in concentrated condition only for a very brief interval of time, or addition preferably at or subsequent to the point of dilution, i. e. at the wet end of the paper machine, serves to substantially conserve the acidic material and/or effect thereof in the 115 fibrous mix. It is the application of this principle which I utilize herein in connection with the precipitation of the rosin size under conditions favoring the minimizing of the time and/or intimacy of contact of the con- 120 stituents of the mix, e. g. at the wet end of the paper machine, and as shown by the above discussion this precipitate would be seriously deteriorated if not completely destroyed if the contact thereof with alkaline filler took 12. place under other conditions, such as when the stock was in the concentrated condition, e. g. in the beater or stuff chests.

Cross reference is made to my copending

1,897,145

1931, wherein in one modification of the invention disclosed therein, the procedure set forth herein, in the case where a non-acid compound of aluminum is additionally present, is described but not claimed.

For size I may use rosin size, or any size which may be used as a substitute therefor in the paper industry, such as saponified fatty acids or the like, or emulsions of various materials such as paraffin, or emulsions of other materials which may be substantially unaffected by alkaline filler.

Instead of alum, with a certain measure of success, I may use alone or in combination acidic materials, such as a mineral or other acids, particularly metallic salts such as compounds of aluminum, zinc, tin, or the like.

The term "paper mix" is used herein is intended to mean a mix or various constituents including fibre from which paper may be made, either alone or after the addition of

other ingredients.

By the term "alkaline filler" I mean substantially water insoluble filler which when 25 agitated in contact with freshly boiled distilled water, say for an hour, will impart a pH value to such water greater than 7.0, that is, which will be on the alkaline side of the neutral point. Among fillers included in this group may be mentioned calcium carbonate, of which lime mud from the causticizing process is one form; calcium carbonate magnesium basic carbonate employed in the paper disclosed in my U.S. Patent No. 1,595,416 35 of August 10, 1926; calcium carbonate magnesium hydroxide disclosed in my U.S. Patent No. 1,415,391 of May 9, 1922; and other substantially water insoluble normal or basic carbonates of alkaline earth metals, (which expression is herein intended to include magnesium), or compounds, double salts, or physically associated mixtures of these with one or more other acid soluble materials of a substantially water insoluble nature.

When I use the word "paper" herein, I use it in the broad sense to include products of manufacture of all types and of all weights and thicknesses, which contain as an essential constituent a considerable amount of prepared fibre and which are capable of being produced on a Fourdrinier, cylinder, or other forming, felting, shaping or molding

machine :

55. By the term "wet end of the paper machine", I intend to include those instrumentalities employed in paper manufacture by which and/or in which a relatively concentrated paper mix is diluted, and treated, conveyed or fed up to the point of web-formation, such as the mixing box, regulating and proportioning devices, riffers, troughs, screens, head boxes, inlets and the like, including also instrumentalities used in the white water cycle.

It is to be understood that my invention is not dependent upon or limited by any

theory expressed herein.

While I have described in detail the preferred embodiments of my invention, it is to 70 be understood that the details of procedure, the proportions of ingredients, and the arrangement of steps may be widely varied without departing from the spirit of my invention or the scope of the subjoined claims. 75

I claim

1. The method of manufacturing paper filled with alkaline filler comprising mixing fibrous material and size precipitant, and adding to the resulting mix size and alkaline filler under conditions favoring the minimizing of the time of contact of the constituents of the mix, and thereafter making paper therefrom.

2. The method of manufacturing paper 85 filled with alkaline filler comprising mixing fibrous material and size precipitant, and adding to the resulting mix size, in an amount insufficient to impart a substantial degree of sizing to the resulting paper, and alkaline filler under conditions favoring the minimizing of the time of contact of the constituents of the mix, and thereafter making

paper therefrom.

3. The method of manufacturing paper 95 filled with alkaline filler comprising mixing fibrous material and size precipitant, and

adding to the resulting mix size and alkaline filler under conditions favoring the minimizing of the time and intimacy of contact of the constituents of the mix, and thereafter

making paper therefrom.

4. The method of manufacturing paper filled with alkaline filler comprising mixing fibrous material and size precipitant, and 105 adding to the resulting mix at the wet end of the paper machine size and alkaline filler, and thereafter making paper therefrom.

5. The method of manufacturing paper filled with alkaline filler comprising mixing fibrous material and size precipitant, and adding to the resulting mix at the wet end of the paper machine alkaline filler previously mixed with other fibrous material, and size, and thereafter making paper therefrom. 115

6. The method of manufacturing paper filled with alkaline filler comprising mixing fibrous material and size precipitant, and adding to the resulting mix at the wet end of the paper machine alkaline filler and size, previously mixed with other fibrous material, and thereafter making paper therefrom.

7. The method of manufacturing paper filled with alkaline filler comprising mixing fibrous material and size precipitant in a beater, and adding to the resulting mix alkaline filler and size at the wet end of the paper machine, and thereafter making paper therefrom

8. The method of manufacturing paper 130

filled with alkaline filler comprising mixing machine, and thereafter making paper therefibrous material and size precipitant, and adding to the resulting mix alkaline filler and size, in an amount insufficient to impart a substantial degree of sizing to the resulting paper, at the wet end of the paper machine, and thereafter making paper therefrom.

9. The method of manufacturing paper filled with alkaline filler comprising mixing 10 fibrous material and acidic material, and adding to the resulting mix alkaline filler and size at the wet end of the paper machine, and

thereafter making paper therefrom.

10. The method of manufacturing paper filled with alkaline filler comprising mixing fibrous material and metallic salt, and adding

to the resulting mix alkaline filler and size at the wet end of the paper machine, and thereafter making paper therefrom.

11. The method of manufacturing paper filled with alkaline filler comprising mixing fibrous material and salt of aluminum, and adding to the resulting mix alkaline filler and size at the wet end of the paper machine, and thereafter making paper therefrom.

12. The method of manufacturing paper filled with alkaline filler comprising mixing fibrous material and alum, and adding to the resulting mix alkaline filler and size at the wet end of the paper machine, and thereafter

making paper therefrom. 13. The method of manufacturing paper filled with alkaline filler comprising mixing fibrous material and size precipitant, and 35 adding to the resulting mix alkaline filler and rosin size at the wet end of the paper machine, and thereafter making paper there-

14. The method of manufacturing paper 40 filled with alkaline filler comprising mixing fibrous material and alum, and adding to the resulting mix alkaline filler and rosin size at the wet end of the paper machine,

and thereafter making paper therefrom.

15. The method of manufacturing paper filled with alkaline filler comprising calcium carbonate, comprising mixing fibrous material and size precipitant, and adding to the resulting mix alkaline filler and size at the wet end of the paper machine, and thereafter making paper therefrom.

16. The method of manufacturing paper filled with alkaline filler comprising calcium carbonate and magnesium compound, comprising mixing fibrous material and size precipitant, and adding to the resulting mix alkaline filler and size at the wet end of the paper machine, and thereafter making paper therefrom.

17. The method of manufacturing paper filled with alkaline filler comprising calcium carbonate magnesium hydroxide, comprising mixing fibrous material and size precipitant, and adding to the resulting mix alkaline 65 filler and size at the wet end of the paper

from.

18. That improvement in the manufacture of paper filled with alkaline filler comprising the addition of alkaline filler and size to 70 a paper mix under conditions favoring the minimizing of the time of contact of the constituents of the mix.

19. That improvement in the manufacture of paper filled with alkaline filler comprising 75 the addition of alkaline filler and size to a paper mix under conditions favoring the minimizing of the time and intimacy of contact of the constituents of the mix.

20. That improvement in the manufacture 80 of paper filled with alkaline filler comprising the addition of alkaline filler and size to a paper mix at the wet end of the paper

21. That improvement in the manufacture 85 of paper filled with alkaline filler comprising the addition of alkaline filler and rosin size to a paper mix at the wet end of the paper machine.

22. That improvement in the manufacture 90 of paper filled with alkaline filler comprising the addition of alkaline filler and size to a previously acidified paper mix at the wet end of the paper machine.

In testimony whereof I affix my signature. 95 HAROLD ROBERT RAFTON.

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