

## UNITED STATES PATENT OFFICE

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## ALKALINE EARTH FILLER

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7 Claims. (Cl. 134—58)

Our invention relates to the sizing of paper and has to do more particularly with the manufacture of an alkaline earth filler for rosin-sized paper.

The present invention is a division of our co-pending application, Serial No. 9,015, filed March 2, 1935, and assigned to the same assignee as the present invention.

Heretofore, it has been considered inexpedient to produce, by standard paper-making processes, a rosin-sized paper containing an alkaline earth filler. While such papers have been produced, it has been only by employing inconvenient and uneconomical processes, and the results have been far from satisfactory. The principal difficulty lies in the fact that rosin sizing requires acid conditions, in view of the use of alum for precipitating the size, and alkaline earth filler reacts with the alum, producing an alkaline condition and thus preventing completion of the sizing reaction. The only efforts to solve this condition in the past have required deviation from standard paper-making operations and thus have not been satisfactory, as stated above.

The use of alkaline earth fillers is highly desirable in the manufacture of book, printing, and writing papers for several reasons. In the first place, such fillers produce a high white color; furthermore, an alkaline earth sludge is usually available in most paper plants as a by-product from the production of caustic soda from burnt lime or dolomite, in connection with the manufacture of soda pulp.

After considerable experimentation, we have found that it is possible to use an alkaline earth filler in connection with rosin-sized pulp, employing the usual standard plant practice, if the filler is first sized or coated with a substance which is inert to the rosin-size reagents. We have found that by employing a colloidal suspension of inert material, each particle of alkaline earth filler may be provided with a protective film of such inert material which will be quite stable under all the conditions of standard plant operation, preventing reaction of the filler with the alum or other ingredients of the pulp sizing bath and producing no undesirable effect at any point of the operation, and at the same time yielding the maximum benefit as a filler. We have found paraffin to be the most satisfactory size for the filler, but may use, in lieu thereof, various other inert wax-like substances.

*Sizing the filler*

According to our preferred embodiment, we in-

troduce the inert material in the form of a colloidal emulsion of paraffin, such as we describe in our co-pending application Serial No. 3,807, filed January 28, 1935, now Patent No. 2,058,085, patented Oct. 20, 1936. This emulsion may, for example, consist of 10 parts of a protein material such as a soya bean or milk casein, which is dissolved with 0.7 parts of soda ash in about 60 parts of water at about 65° C. To this warm solution are then added 5 parts of a suitable emulsifying agent, such as ammonium oleate, which may have previously been dissolved in 5 parts of water. When solution of these ingredients has occurred, 40 parts of paraffin having a melting point of approximately 124° F. are slowly run in in a molten condition with agitation. The agitation is continued in any suitable apparatus until complete emulsification has occurred. The size thus produced is diluted to about 5 percent paraffin with warm water at approximately 65° C.

The filler may be lime mud, recovered, as stated above, from the causticizing of burnt limestone or dolomite, although it will be apparent that our invention is applicable to various other types of filler which may be susceptible to undesirable reaction with other elements of the furnish. The filler and the emulsion prepared as described above are thoroughly mixed and from 3 to 5 percent of an acidic substance, such as alum, is run in gradually while the agitation continues, for the purpose of breaking the emulsion. As a result, each particle of filler is enveloped with a sub-microscopic film of paraffin, said film of paraffin being in turn coated with a film of protein material. We have demonstrated with special microscopic equipment that such films actually are produced on the particles of filler.

The presence of these protective coatings is of great importance in the sizing and paper-making operations, inasmuch as the filler is protected from reaction with the pulp-sizing ingredients and, at the same time, the paraffin is protected by the protein film, thus preventing agglomeration of the paraffin to form large particles thereof which might produce wax spots on the finished paper.

*Pulp treatment*

The filler produced as described above may be employed, for example, in the following manner:

There are introduced into the beater about 500 pounds of bleached soda pulp and about 500



pounds of sulphite pulp. After this material has been mechanically prepared in the usual manner, there is added to this pulp in the beater about one percent by weight of rosin as ordinary  
 5 rosin size, and this size is set in the usual manner with alum to produce a mixture with a pH of about 4.5 to 5.0. To the beater are then added about 400 pounds of pre-sized alkaline earth  
 10 filler, prepared as described above, the amount of this filler varying according to the amount of ash required in the finished sheet. To secure adequate sizing, it is desirable that the beater be furnished with fresh water, but ordinary white  
 15 water can be used for subsequent operation, such as the dropping of the beater, dilution of the stock for the paper machine, showers, etc. The resulting mixture on the paper machine will be slightly alkaline with a pH range of about 7.2 to 7.4.

20 The finished paper made from this stock will be fully sized, will offer no mechanical troubles on the paper machine, such as clogging of the wire, sticking, foaming, etc., and will be free from wax spots and all possible sizing defects.

25 Pulp mixtures of stock containing alkaline earth fillers pre-sized in accordance with our invention described above are stable for long periods of time, even when subjected to constant agitation and high temperatures, and will show no  
 30 tendency for the paraffin to separate.

Modifications of our invention will no doubt suggest themselves to those skilled in the art without departing from the spirit of our invention. Hence, we do not wish to be restricted to  
 35 the specific embodiments disclosed above or uses mentioned, but intend that the scope of our invention shall be determined from the appended claims, which should be interpreted as broadly as the state of the art will permit.

40 We claim as our invention:

1. A method of preparing an alkaline earth filler for use in paper making, comprising admixing with a sludge of alkaline earth solids in  
 45 finely divided condition an emulsion of substantially inert wax material, stabilized with an acid precipitable, proteinaceous, protective colloid and adding an acidic reagent to said emulsion whereby the particles of alkaline earth material are  
 50 coated with a film of said wax-like material and the latter is in turn protected by means of said colloid, the ratio of the proteinaceous colloid to the wax material being at least about 1:4.

2. A method of preparing an alkaline earth filler for use in paper making, comprising admix-  
 55 ing with a sludge of alkaline earth solids in finely

divided form an emulsion of paraffin stabilized with an acid precipitable protein and adding an acid reagent to said emulsion whereby the particles of alkaline earth material are coated with  
 5 a film of paraffin and the latter is in turn protected by means of said protein, the ratio of protein to paraffin being at least about 1:4.

3. A method of preparing an alkaline earth filler for use in paper making, comprising admixing with a sludge of alkaline earth solids  
 10 in finely divided condition an emulsion of paraffin stabilized by a substantial proportion of acid precipitable protein in colloidal form, and adding an acidic reagent to set the emulsion, whereby the particles of alkaline earth material are coat-  
 15 ed with a film of said paraffin and the latter is in turn protected from agglomeration by said protein, the ratio of protein to paraffin being about 1:4.

4. A method of preparing an alkaline earth  
 20 filler for use in paper making, comprising admixing with a sludge of alkaline earth solids in finely divided condition an emulsion of paraffin stabilized by a substantial proportion of casein in colloidal form, and adding an acidic reagent to  
 25 set the emulsion, whereby the particles of alkaline earth material are coated with a film of paraffin and the latter is in turn protected by means of said casein, the ratio of casein to paraffin being at least about 1:4.

5. An improved filler for use in paper making, comprising alkaline earth material in finely divided particles, said particles being coated with  
 35 a film of substantially inert wax material and the latter being in turn coated with an acid precipitated, proteinaceous, protective colloid, the ratio of proteinaceous colloid to wax material being at least about 1:4.

6. An improved filler for use in paper making, comprising alkaline earth material in finely divided particles, said particles being coated with  
 40 a film of paraffin and the latter being protected from agglomeration by a film of an alum precipitated protein, the ratio of protein to paraffin being at least about 1:4.

7. An improved filler for use in paper making, comprising alkaline earth material in finely divided particles, said particles being coated with  
 45 a film of paraffin and the latter in turn being protected with a casein in substantial quantity  
 50 with relation to said paraffin, the ratio of casein to paraffin being at least about 1:4.

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