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PAPER IMPREGNATED WITH POTASSIUM ALUM AND PROCESS OF IMPREGNATING

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This invention relates to a process for strengthening fibrous material such as paper and rendering it water resistant and fire resistant, and to the products of such process. By way of example, the process may be used in the container and paperboard fields and the products may comprise impregnated fiber board, corrugated board or corrugating medium, any of which may be made of any of the usual fibrous materials such as virgin pulps from wood, sisal, straw, etc., or secondary pulps such as used in chipboard, newsboard and the like or mixtures of any of these pulps, as well as containers, container elements, tubes and the like made of such materials.

Considerable difficulty has been encountered in the art of stiffening paper by impregnating the same with various materials. Although several different substances are used for this purpose at the present time, none is satisfactory for one reason or another. Sulphur impregnation has been used for many years, but while this strengthens and renders the paper water resistant, its use is not altogether desirable because of the high degree of inflammability of sulphur impregnated paper. Furthermore, paper treated with sulphur becomes excessively brittle, and has a corrosive action on certain metals coming in contact with it. Another common impregnant is molten paraffin or other wax, but this is adequate only for waterproofing purposes as it provides very little additional strength; furthermore, it also presents a fire hazard. Various synthetic resins have also been used as impregnants, but even the cheapest of these is far too expensive for commercial use in the expendable container field, for example. Some plastics have been used but more as coatings than as impregnants; these require a very high ratio of plastic weight to paper weight and the resulting, necessarily expensive, product may be more properly described as a paper based plastic rather than as an impregnated paper.

It is an object of the present invention to provide an economical method whereby paper sheets or containers made of paper may be provided with increased stiffness, greater hardness, greater compression strength and greater tensile strength, and at the same time may be rendered more water resistant and more fire resistant. The present invention achieves all of these results and produces a new and superior product by immersing paper in an impregnating bath of water and potassium alum.

Potassium alum, $Al_2(SO_4)_3 \cdot K_2SO_4 \cdot 24H_2O$ melts at 92.0° C. and in its undiluted molten state has a specific gravity of about 1.70.

Best results are achieved according to the present invention, within certain preferred ranges of specific gravity and temperature. Preferably the potassium alum is melted and enough water is added to the molten alum to establish a specific gravity of from about 1.440 to 1.505. The molten potassium alum, with no water added, is of a thick and rather syrupy consistency. With the addition of water as specified above a viscosity is established which is much the same as that of pure water. This mixture may possibly constitute a higher hydrate of the alum than the formula given above, and indeed this is suggested by the fact that the amount of water actually necessary to establish a particular specific gravity is different from that which is indicated by calculation. However, no reference to such a higher hydrate has been found and it will therefore be identified herein as a mixture.

For best results the potassium alum-water mixture within the preferred specific gravity range set forth above should be maintained at a temperature of about 103° C. to about 104° C. and the fibrous article to be treated is immersed therein for a period of time long enough for the mixture to penetrate all the way through. Within the preferred ranges of specific gravity and temperature set forth above such penetration of the various paper and board products listed above is usually achieved in 30 seconds or less and adequate degrees of penetration for satisfactory results can be achieved in from 10 to 30 seconds. As water is always vaporizing at the temperature of the bath, it is necessary to add water continuously or from time to time at short intervals in order to keep the specific gravity within the above preferred range.

Although the preferred range of specific gravity set forth above for potassium alum gives the best results, it has been found that good results can be achieved with somewhat higher specific gravity within the range of about 1.505 to 1.535. Relatively poor results are obtained when the specific gravity is permitted to go much above 1.535 because the attendant increase in viscosity makes it difficult to secure adequate penetration. If the specific gravity is permitted to drop below 1.440 the results become erratic in that the alum shows a tendency, which becomes progressively greater, not to harden in spots, while at a specific gravity below about 1.420 the mixture will not harden sufficiently upon cooling to impart a desirable degree of strength to the paper. In a similar fashion, while the preferred temperature range is from 103° to 104° C., satisfactory results can be obtained at a temperature range of from about 102° C. to about 104° C. when the specific gravity lies within the preferred range of 1.440 to 1.505. Lower temperatures than about 102° C. give relatively poor results particularly as regards the extend of penetration. However, it should be noted that such poorly penetrated products exhibit increased strength. It is not possible, at atmospheric pressure to raise the temperature above about 104° C. because the mixture "boils" or at least partially volatilizes or decomposes at that temperature although, of course, this temperature will vary slightly with variations in specific gravity.

"Best results" as used hereinabove take into account the degree of impregnation, the time required to obtain complete impregnation, as well as the development of optimum strength. Thus, within the preferred range of temperature and specific gravity it is possible to fill, largely and uniformly, the interstices of material such as that described in the specific example set forth below in about thirty seconds. The resulting product exhibits the excellent strength characteristics set forth in that example. The impregnation time may be shortened, with an attendant decrease in ultimate strength, when less strength is required. Operation outside the preferred ranges may produce a product which is not completely penetrated or which, if completely penetrated, is not as strong as the best example but such products may be entirely satisfactory or even preferred for specific purposes.

The present invention offers many advantages over the impregnation of paper with sulphur both from the standpoint of the process involved and the desirability of the product. According to this invention paper and paper products such as those listed above may be vastly strengthened and stiffened by an impregnation at a temperature only a few degrees above the boiling point of water and for a period of thirty seconds or less whereas the widely practiced sulphur impregnation process is carried out at a considerably higher temperature and requires immersion of the paper for much greater periods of time.

As a specific example of the preferred process and as an illustration of the desirable characteristics of the product the following is presented:

A sheet of laminated corrugated kraft board about ⅜" thick and made up of three plies of nine-point A and B flute corrugations with sixteen-point liners was immersed for 30 seconds in a bath of potassium alum and water having a specific gravity maintained as closely as possible within the range of from 1.440 to 1.505 and being maintained at a temperature between 103° C. and 104° C. The sheet was then removed from the bath and permitted to cool. The resulting sheet was a hard, stiff product suitable for use in many fields where such a product is desirable. For test purposes, strips 2 inches wide and 12 inches long were cut from this sheet.

The test strips made in accordance with the foregoing example were positioned with B flute up and supported only near opposite ends on horizontal cylindrical supports 10 inches apart and subjected to uniform and positioned loading tests. These strips supported a uniform loading of 10 lbs. of shot plus a positioned loading, applied equally at the third points of the strips, of 32 lbs., making a total load of 42 lbs. The failure indicated was a compression failure. In tension the specimens did not fail at 80 lbs., the limit of the apparatus used. Identical test strips from unimpregnated board of the same construction failed in compression with only a uniform loading of 6 lbs. of shot.

The amount of impregnant contained in the product of the specific example, expressed in percentage to represent the ratio of weight of impregnant to the weight of the unimpregnated paper was approximately 140%. This figure was arrived at by subtracting the weight of the unimpregnated paper from the weight of the impregnated paper and dividing this difference by the weight of the unimpregnated paper. For convenience, this figure is referred to as the percentage pickup.

For the development of maximum strength in the product without wasting alum in the form of superficial coating upon the surfaces or upon the fibres of the paper, it has been found to be particularly advantageous to work within the preferred ranges of specific gravity and temperature set forth above. Within these ranges rapid penetration can be achieved and the impregnated product will readily drain so as to leave the product thoroughly impregnated without excessive superficial coating. Obviously, the percentage pickup of a given product processed within the preferred ranges of specific gravity and temperature may be varied by variations in the time of impregnation up to such period of time as may be required to secure penetration all the way through the product. For papers of the weight and characteristics used in the specific example, approximately 30 seconds or less may be required to achieve complete penetration. In such a case immersion for periods of substantially less than 30 seconds will bring about less complete penetration and a proportionately lower percentage pickup. However, for the amount of impregnant thus added to the product, the maximum favorable strength improvement will be achieved. A useful improvement in strength can be achieved with products of the type involved in the specific example by reducing the impregnation time so as to bring about a percentage pickup of from about 50% up to the 150% achieved in 30 seconds. Furthermore, if the product is not thoroughly drained, the percentage pickup may range as high as 190% but this is not considered to be the best practice from the standpoint of economy.

In a general sense in the application of this invention to products of various types, it has been observed that

the product should achieve a percentage pickup of at least about 50% when operations are carried out under the preferred conditions of specific gravity and temperature. When operating outside the preferred ranges but still within the ranges which will produce an acceptable product as explained above, it has been found that the percentage pickup due to impregnation will tend to be quite high. For example, when operations are conducted at relatively high specific gravities between 1.505 and 1.535, the percentage pickup will run from 190% to 400%. Under these conditions a great deal of the alum is probably wasted in the form of superficial coating which detracts from the appearance and contributes a disproportionately small amount to the ultimate strength of the product.

Paper impregnated with potassium alum in accordance with the present invention cannot be ignited to start combustion without the presence of other fuel and it therefore presents a very great advantage over the readily inflammable sulphur impregnated paper heretofore used. Burning sulphur impregnated paper gives off large quantities of sulphur dioxide which, while not toxic, is highly irritating and renders fire-fighting difficult. When such paper is destroyed in an incinerator, the sulphur dioxide adversely affects the neighboring atmosphere. Even with other fuel present the impregnated paper of the present invention will not burn with active flaming. However, in a hot flame with other fuel present, it will glow and char and be consumed. This is the optimum degree of fire resistance; the material does not constitute a fire hazard yet it can be consumed in an incinerator, thus solving the ultimate disposal problem.

The alum impregnated paper of this invention is nonhygroscopic and quite water resistant. For special purposes it can be made waterproof by a paraffin or other wax spray after the alum impregnation. In particular, it has been found that dipping the impregnated product in a mixture of 50% paraffin and 50% montan wax gives excellent waterproofing while at the same time the fire-resistant qualities of the alum impregnated paper are largely retained. If desired, the two processes can be combined by floating the wax melt on the surface of the alum-water bath so that each sheet or container of paper picks up a coating of the selected wax or wax mixture as it leaves the impregnating bath.

If desired, a greater surface hardness of the paper can be achieved, without impairing the overall strength, by the addition of inert materials to the alum-water mixtures described above. Such enhanced surface hardness is particularly desirable in the case of paper containers which are to be secured by steel straps for it renders the container more impervious to the cutting action of the steel straps. The preferred inert material for this purpose is kaolin, although fuller's earth, spent fuller's earth and nepheline syenite also produce good results. The preferred quantity of inert material to be used, by weight, is about 10% of the weight of the alum but good results can be obtained by adding anywhere from 5% to 25% by weight of such material. Besides producing increased surface hardness, the addition of such inert material to the impregnating bath gives a uniform opaque color to the paper that is quite pleasing in appearance.

Paper can be treated by the process of this invention in sheet form for building and construction purposes or in the form of finished containers which will be utilized for storage or shipping purposes. If desired, paper can be impregnated in sheet form and then formed into containers before it has cooled. Also, corrugating medium may be impregnated and thereafter corrugated while still hot or it may be impregnated and cooled in smooth condition, rewound for storage and/or shipment and corrugated by the usual heated corrugating rolls prior to assembly into corrugated board. In the latter case the liner board may be untreated or treated in any suitable manner or it may be impregnated in accordance with this invention if so desired.

The process of this invention can be operated economically to produce a very greatly strengthened paper which is highly fire-resistant and which, furthermore has no harmful corrosive action on common metals. Even in the case of pure copper it has only the effect of slightly blackening the surface and it does this to an extent far less than that of sulphur impregnated paper or, for that matter, ordinary unimpregnated kraft paper.

Although there is shown and described herein preferred processes to be used in accordance with the invention, and improved products resulting therefrom, it is to be understood that it is not desired to limit the application of the invention thereto except as may be required by the scope of the subjoined claims and various modifications are possible without, however, departing from the general principles and spirit of my invention.

I claim:

1. The process of treating felted cellulose fibrous material to strengthen the same comprising impregnating fibrous material in a bath of potassium alum and water while said bath is maintained at a specific gravity within the range of from about 1.440 to about 1.535 and at a temperature within the range of from about 102° C. to about 104° C.

2. The process of strengthening paper and paper products wherein said paper is composed principally of cellulose fiber, comprising impregnating the same in a bath of potassium alum and water while said bath is maintained at a specific gravity within the range of from about 1.440 to about 1.535 and at a temperature within the range of from about 102° C. to about 104° C.

3. The process of treating felted cellulose fibrous material to strengthen the same comprising melting potassium alum, adding sufficient water to make a potassium alum-water mix having a specific gravity within the range of from about 1.440 to about 1.535, heating said mix to maintain a temperature within the range of from about 102° C. to about 104° C., impregnating fibrous material by immersing the same in said mix while maintaining said mix within said ranges of specific gravity and temperature, withdrawing said fibrous material from said mix, and cooling the same.

4. The process of treating felted cellulose fibrous material to strength the same comprising melting potassium alum, adding sufficient water to make a potassium alum-water mix having a specific gravity within the range of from about 1.440 to about 1.535, heating said mix to maintain a temperature within the range of from about 102° C. to about 104° C., adding finely divided inert filling material to said mix in an amount constituting from about 5% to 25% by weight of the weight of alum in said mix, immersing fibrous material in said mix containing said inert material while maintaining the relative amounts of water and alum therein substantially constant, withdrawing said fibrous material from said mix, and cooling the same.

5. The process of treating felted cellulose fibrous material to strengthen the same comprising impregnating fibrous material in a bath of potassium alum and water while said bath is maintained at a specific gravity within the range from about 1.440 to about 1.535 and at a temperature within the range from about 102° C. to about 104° C., withdrawing said fibrous material from said bath and applying thereto a waterproof coating.

6. The process of treating felted cellulose fibrous material to strengthen the same comprising impregnating fibrous material in a bath of potassium alum and water while said bath is maintained at a specific gravity within the range of from about 1.440 to about 1.535 and at a temperature within the range of from about 102° C. to about 104° C., said bath having a layer of molten wax floated upon the surface thereof whereby upon withdrawal of said fibrous material from said bath through said layer of molten wax said fibrous material will have applied thereto a coating of said molten wax.

7. The process of treating felted cellulose fibrous products to strengthen the same comprising impregnating fibrous material in a bath of potassium alum and water while said bath is maintained at a specific gravity within the range of from about 1.440 to about 1.505 and at a temperature within the range of from about 103° C. to about 104° C.

8. The process of treating felted cellulose fibrous material to strengthen the same comprising impregnating fibrous material in a bath of potassium alum and water while said bath is maintained at a specific gravity within the range of from about 1.440 to about 1.505 and at a temperature within the range of from about 103° C. to about 104° C., withdrawing the impregnated product from said bath, and applying thereto a waterproof coating.

9. A strengthened paper comprising paper composed principally of cellulose fiber and impregnated throughout a substantial fraction of the thickness thereof with potassium alum which is a solid at room temperature and which has been impregnated into the paper in molten condition and solidified by cooling in situ, said potassium alum being substantially uniformly distributed throughout those portions of said paper which are impregnated thereby, and said potassium alum impregnated in said paper having a ratio by weight to the weight of the unimpregnated paper of at least 50%.

10. A strengthened paper comprising paper composed principally of cellulose fiber and impregnated throughout a substantial fraction of the thickness thereof with potassium alum which is a solid at room temperature and which has been impregnated into the paper in molten condition and solidified by cooling in situ, said potassium alum being substantially uniformly distributed throughout those portions of said paper which are impregnated thereby, and said potassium alum impregnated in said paper having a ratio by weight to the weight of the unimpregnated paper lying within the range of from about 50% to 190%.

11. A strengthened paper comprising paper composed principally of cellulose fiber and impregnated throughout a substantial fraction of the thickness thereof with potassium alum which is a solid at room temperature and which has been impregnated into the paper in molten condition and solidified by cooling in situ, said potassium alum being substantially uniformly distributed throughout those portions of said paper which are impregnated thereby, said potassium alum impregnated in said paper having a ratio by weight to the weight of the unimpregnated paper of at least 50%, and said impregnated paper having a coating of waterproof material on the surfaces thereof.

12. A strengthened paper comprising paper composed principally of cellulose fiber and impregnated throughout a substantial fraction of the thickness thereof with an impregnant comprising potassium alum which is a solid at room temperature and which has been impregnated into the paper in molten condition and solidified by cooling in situ, said potassium alum having distributed therein from 5% to 25% by weight of said potassium alum of a finely divided inert material, said impregnant being substantially uniformly distributed throughout those portions of said paper which are impregnated thereby, and said impregnant having a ratio by weight to the weight of the unimpregnated paper of at least 50%.

13. A strengthened paper comprising paper composed principally of cellulose fiber and impregnated throughout a substantial fraction of the thickness thereof with an impregnant comprising potassium alum, which is a solid at room temperature and which has been impregnated into the paper in molten condition and solidified by cooling in situ, said potassium alum having distributed there-

in from 5% to 25% by weight of said potassium alum of a finely divided inert material, said impregnant being substantially uniformly distributed throughout those portions of said paper which are impregnated thereby, said impregnant having a ratio by weight to the weight of the unimpregnated paper of at least 50%, and said impregnated paper having a coating of waterproof material on the surfaces thereof.

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