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PAPER COMPOSITION AND PROCESS OF MAKING SAME

No Drawing.

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This invention relates to improved compositions and process of paper making, and relates more especially to certain improvements in the manufacture of transparent or translucent and waterproof papers, as well as to improvements in the manufacture of various other papers.

The object of this invention is to provide a pulp suitable for paper manufacture which will have greater absorptive qualities for sizing materials than such pulps as are now used, particularly for those sizing materials which contain substantial amounts of paraffin, carnauba, ceresin, japan, beeswax, or other waxes. Such improvement has for its object the production of more transparent or more translucent and waterproof papers and improved papers in general.

Ordinarily the various pulps now in use can carry only a rather limited amount of sizing material, particularly waxes, but I have been able to increase this absorptive capacity by either treating the wood pulp to convert a substantial portion of the cellulose to hydrocellulose, or by adding hydrocellulose to the usual pulp. The greater absorptive quality of hydrocellulose enables me to produce pulps and papers which will take larger quantities of various sizing materials, such as waxes, rosin, soaps, starch, etc. Accordingly more transparent and waterproof papers may be produced. The use of hydrocellulose is of particular value when aqueous emulsions of wax are employed. Hydrocellulose coming in contact with the dispersion of the substantially homogenized wax or other sizing material, through certain absorptive phenomena, causes its occlusive union with itself. This action apparently is quite different from that which occurs when a sheet of paper is passed through a bath of, for example, molten paraffin.

As previously stated, I may either treat the wood pulp so as to convert a portion thereof to hydrocellulose and utilize such mixture for making the improved paper, or, I may almost entirely convert the cellulose to hydrocellulose separately and add various amounts to the ordinary pulps employed. The conversion of a portion of the wood cel-

lulose to hydrocellulose may be accomplished in several ways. According to the method used I may almost completely convert the cellulose fibres to hydrocellulose, or only partially effect the conversion, and in some cases may react in such a manner that only the exterior surfaces of each cellulose fibre are converted to hydrocellulose.

It seems most advisable to maintain the pulp in a rather hydrated condition, not allowing same to become dry until the wax dispersion has been added and the sheet formed and calendered. The wax dispersion is preferably added to the pulp in the beater, substantially at the beginning of the beating operation in order to bring about a maximum absorption of wax by the hydrocellulose and cellulose fibres with the greatest degree of dispersion, although, in some cases, I may add the wax dispersion or size in various other stages of the beating operation. At the end of the operation rosin size may be added and set with alum in the usual manner. The emulsion is rapidly absorbed by the fibres, the sheet then being formed in the usual manner. When passing the sheet through the calender rolls, the latter preferably hot, the occluded material is forced under high pressure even more thoroughly throughout the sheet and such uniform distribution occurs as to render the sheets of papers unusually transparent. In some cases it is more desirable, especially when heavy thick paper is to be formed, to preheat the sheets to a temperature slightly above the melting point of the wax employed, and then calender with chilled rolls. Finely ground wood flour and similar fillers may be added to the pulp base if pressboard and other cheap opaque, waterproof papers are desired.

In carrying out my invention I may subject the usual sulphite or other pulp to a chemical treatment whereby hydrocellulose is formed in some degree. A rather rapid and very extensive conversion of the pulp to hydrocellulose may be effected by allowing say, 50 parts by weight of cellulose pulp to remain at room temperature with approximately 280 parts by weight of sulphuric acid having a specific gravity of from 1.52 to 1.54

and after 12 to 14 hours of such contact, washing well and rendering neutral. Conversion is so extensive that this material alone contains too much hydrocellulose for most papers and is best utilized by blending with untreated paper, or the pulp may be allowed shorter contact with the acid, whereby a less complete conversion to hydrocellulose will result and a more suitable paper stock formed—no exact percentage of hydrocellulose can be said to be suitable in all cases, the amount depending upon the kind of size to be employed and kind of paper desired and may be readily determined for any given paper by anyone skilled in the art.

In lieu of the sulphuric acid treatment I may convert a portion of the cellulose fibres to hydrocellulose in other ways as follows:—

To 20 parts by weight of sulphite pulp I may add 30 parts by weight of zinc chloride and 400 parts by weight of water. The mixture is heated at approximately 100° C., for 2 hours when the product may be diluted with approximately 2,000 parts by weight of water and washed free from zinc chloride. Another suitable method consists in treating, say, 20 parts by weight of sulphite pulp with 1 part by weight of oxalic acid and 400 parts by weight of water at 100° C. for 2 hours. The mixture is then diluted with about 400 parts by weight of water; washed free from acid and then used. This treatment also acts somewhat as a bleach. Another method consists in subjecting 300 parts by weight of a 1½ per cent glassine pulp dispersion with 10 parts by weight of sulphuric acid and 2½ parts by weight of sodium hydroxide or the equivalent amount of sodium bisulphate with 100 parts by weight of water. The mixture is heated for three hours at approximately 100° C. when it may be diluted with about 200 parts by weight of water and then washed free from the acid salts.

Still another method consists in subjecting, say, 20 parts by weight of a pulp with 50 parts by weight of concentrated hydrochloric acid and 100 parts by weight of water to a temperature of approximately 100° C. for one hour. It may then be diluted with approximately 2,000 parts by weight of water and then washed. To 20 grams of sulphite pulp I may add 100 parts by weight of potassium iodide and 200 parts by weight of water, heating the mixture for two hours at approximately 100° C. The product may then be diluted with approximately 2,000 parts by weight of water, and washed free from salt.

Another method using sulphuric acid may be conducted substantially as follows:—

To 20 parts by weight of sulphite pulp dispersed in 400 parts by weight of water I may add approximately 12 parts by weight of sulphuric acid. Within a few minutes I may filter this material and press out the excess

acid and dry at 40° C., for approximately 12 hours and then raise the temperature to 70° C. for a period of 3 hours. The product is then washed free from acid and is then ready for use.

Various other treatments may be given to the pulp in order to accelerate the formation of hydrocellulose. Such other acids as for example, phosphoric and acetic; acid salts such as aluminum chloride, potassium bisulphate, etc., and salts such as sodium chloride, calcium thiocyanate and various other substances may be used to induce the rapid formation of hydrocellulose, whereby I obtain pulps having greater absorptive capacities for wax dispersions and sizing materials.

Various types of sizing materials and wax dispersions may be employed, but in general, I prefer to use a wax dispersion which has been made by means of a colloid mill. Paraffin, ceresin, cernauba, montan, japan, beeswax, or other waxes, alone or admixed with rosin, various oils, starches or soaps, fed in a liquid condition with water, which preferably is hot, to a colloid mill, whereby a very great dispersion or emulsification of wax in water is obtained. In many cases I prefer to use mixtures of waxes and oils in order to produce more flexible paper than otherwise would result. In particular, especially where papers intended for contact with food products, such as milk bottle discs, ice cream containers, etc., are to be made, I prefer to use a water-white grade of medicinal petroleum oil, such as for example as is known generally throughout the trade as "Marcol". This improves the flexibility so that the paper may be drawn more readily through dies, improves the texture, and adds considerably to the waterproofing value. Various proportions of oil and wax may be used and as much as two parts by weight of oil to one part by weight of wax, may, in some cases, be employed.

Various amounts of hydrocellulose containing pulp may be blended with wax dispersions and with sizes and, in some cases, fillers such as ground wood or wood flour, according to the type of paper desired. One composition for a paper stock may be made substantially as follows:—

To 200 parts by weight of a five per cent sulphite pulp dispersion in water which has been subjected to any chemical treatment whereby hydrocellulose is formed in substantial degree, I may add in the beater engine, 20 parts by weight of a 50 per cent paraffin in water emulsion and 200 parts by weight of a 10 per cent wood flour in water dispersion. After the beating operation I may add 5 parts by weight of a rosin size and may then set with alum in the usual manner. A composition suitable as a paper stock for use in contact with food products may be made substantially as follows:

To 250 parts by weight of a pulp dispersion

in water containing $12\frac{1}{2}$ parts by weight of actual pulp, I may add an equal amount of wood flour and may incorporate a wax dispersion with this material in the beater, containing approximately 5 parts by weight of paraffin wax dispersion containing 1 part of "Marcol" to 10 parts of wax, 1 part by weight of stearic acid and enough ammonia to emulsify. The wax dispersion may be separately prepared in an emulsifier or colloid mill and may then be incorporated with the rest of the mixture in the beater. It is important that the pulp used in this procedure be chemically treated to convert a substantial proportion thereof to hydrocellulose in order to secure the proper absorption of the wax.

In some cases it is desirable to obtain special effects by using a very small proportion of soap or other emulsifying agent. One illustrative procedure is as follows:

A dilute soap solution is prepared (in proportion of $1\frac{1}{4}$ ounces avoirdupois to one gallon of water) paraffin wax (melting point 135° F.) is melted. The soap solution and the melted wax, each at a temperature of about 140 – 150° F.) are fed, in approximately equal proportions by volume, into a colloid mill. This mill, with fairly free flow, has an output of over 200 gallons per hour, but to produce a very fine dispersion, as is employed in the present illustration, the adjustment of the mill gives an output of 80–90 gallons per hour. This yields a dispersion exhibiting the Brownian movement in notable degree and although containing about 50 per cent of wax, the dispersion is a fairly thin milk-white liquid. In this form, dilution with water may be made as required without materially altering the utility of the product.

So prepared, it is preferably added to the paper stock or pulp in the beater engine at the beginning of the beating operation or in the early stage of beating, to better effect uniform dissemination through the cellulose stock or mixture. The addition of the dispersion at or near the close of the beating operation is not precluded, but if added preferably well in advance of the addition of rosin size (when the latter is required) the segregation of the two steps—waterproofing and sizing—is desirably accomplished.

Using so small a proportion of soap as indicated, foaming in the beater is minimized.

A suitable stock is made of equal parts by weight of ground wood (or "exploded" wood) and sulphite pulp. This mixture preferably is beaten together for a time in about 20 times its weight of water. To approximately 80 volume of the above 1 volume of the wax dispersion is added, and the mixture warmed and agitated for one-half hour or more. One volume of ordinary rosin size is added and the agitation continued for 1 hour. Then a solution of alum is added to set the size. This is followed by a final agitation for

15 minutes. This pulp composition is sheeted and calendered. A smooth waterproof sheet results.

In the illustration last given the amount of hydrocellulose is comparatively small and the fixation of the wax is not as complete as when a substantial proportion of hydrocellulose is produced chemically or by mechanical treatment or is added in any suitable manner. Thus, for example, the hydrocellulose obtained by the reversion of viscose or other hydrolyzed celluloses may be introduced into a mixture of ground wood, and sulphite pulp, or any other analogous paper pulp composition.

While the illustrations have been mainly directed to the employment of paraffin wax, with or without a mineral oil softening agent, it should be understood that other waxes may be employed in admixture with paraffin wax, or by themselves, especially in the form of a highly dispersed emulsion showing Brownian movement. By introducing such emulsions or dispersions of waxes or mixtures of waxes well in advance of the addition of rosin size, especially in the presence of a substantial proportion of hydrocellulose, the full effect of the waxy material as a waterproofing agent is secured. The increase in flexibility secured by also incorporating a dispersion of a mineral oil such as lubricating oil or medicinal oils such as "Nujol" or "Marcol" is desirable in the case of papers which have to be drawn through dies to obtain specific shapes.

Paraffin wax emulsions made with a substantial proportion of ordinary soap are liable to cause a serious degree of foaming in the beater engine. By the employment of colloid mill emulsions made with, say, less than one per cent of soap, difficulties from foaming are overcome in most cases. The colloid mill product therefore has a specific utility in the waterproofing of paper. Aside from checking the difficulties of foaming as when a large proportion of soap is present, the extreme dispersion of wax (exhibiting the Brownian movement) made in the colloid mill as aforesaid, appears to have a peculiar colloidal affinity for hydrolyzed cellulose.

What I claim is:—

1. The process of making paper intended for use with food products, which comprises subjecting paper pulp of a fibrous character to a chemical treatment in which some hydrocellulose is formed, and incorporating with this fibrous pulp a wax dispersion comprising a wax and a light-colored petroleum oil.

2. In the process of producing a flexible waterproof paper from paper pulp containing hydrocellulose, the step of incorporating with said pulp, a wax dispersion containing mineral oil.

3. A water-resistant paper product made

from a paper pulp containing an incorporated wax-petroleum oil emulsion.

4. In the process of producing water-resistant paper from paper pulp, the step
5 which comprises incorporating with said pulp a wax dispersion carrying mineral oil.

5. In the process of producing water-proof paper from paper pulp containing hydrocel-
lulose, the step of incorporating with said
10 pulp a paraffin wax-petroleum oil emulsion.

6. A paper product made from a paper pulp containing an incorporated wax emul-
sion including a flexibilizing oil.

7. In the process of producing water-proof
15 paper from paper pulp, the step which com-
prises incorporating with said pulp, a wax
dispersion carrying a flexibilizing oil.

8. In the process of producing water-proof
paper from paper pulp, the step which com-
20 prises incorporating with said pulp, a milled
wax dispersion carrying a flexibilizing oil.

9. A paper product made from a paper
pulp containing an incorporated wax emul-
sion including a flexibilizing oil, and a small
25 amount of a soap.

10. In the process of producing water-
proof paper from paper pulp, the step which
comprises incorporating with said pulp, a
wax dispersion carrying a flexibilizing oil
30 and a soap in an amount insufficient to pro-
duce foaming during the incorporation step.

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