

UNITED STATES PATENT OFFICE.

CARLETON ELLIS, OF MONTCLAIR, NEW JERSEY, ASSIGNOR TO ELLIS-FOSTER COMPANY, A CORPORATION OF NEW JERSEY.

PROCESS OF TREATING PAPER PULP.

No Drawing.

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This invention relates to a process of treating paper pulp for use in paper making and involves a new and novel method for increasing the absorptive capacity of the pulp for wax emulsions or dispersions and other sizes. The treatment is of particular value in the preparation of waterproof and transparent or translucent paper.

Various sizes and methods of sizing for paper and paper products have been suggested, and in many cases, used. The most generally used method for waterproofing paper consists in preparing a sheet of paper, immersing it in a bath of molten wax, oil or resin. In some cases, the emulsions containing waxes have been incorporated with the pulp prior to paper manufacture. In general considerable difficulty has been experienced in securing proper absorption and dispersion.

In carrying out my invention I prepare an emulsion of the wax, or mixtures thereof with oils, resins, starches, and the like, employing water as a vehicle, and preferably using a colloid mill to convert the waterproofing agent into a state of extreme fineness or dispersion. It is advantageous, but not essentially necessary to previously dissolve in the water a small amount of a soluble soap, such as sodium, potassium or ammonium stearate, palmitate, resinate, and the like. In other cases, a small amount of an alkaline salt such as sodium phosphate, soda ash, or of an alkali, such as caustic soda or potash, may be employed instead of the soap. The use of such salts is particularly desirable when free rosin or fatty acids have been added to the mixture.

Having prepared the emulsion by means of a powerful emulsifying means such as the aforesaid colloid mill, I introduce said emulsion into the beater with the pulp. I find it highly desirable to add the emulsion to the pulp substantially at the beginning of the beating operation, as this accomplishes a very much better dispersion and absorption than by adding the emulsion at a later stage. The dispersion is rapidly absorbed by the fibres (the speed and extent of absorption depending to a large extent on the propor-

tion of hydrocellulose present) and after sufficient time has elapsed, the sheet is formed in the usual manner.

In order to obtain the maximum amount of absorption, I find it occasionally desirable to add the wax dispersion to the pulp in the beater at a temperature substantially above the melting point of the wax employed. In most cases the temperature of boiling water is about right, although temperatures as low as 90° C. are in some cases suitable.

Such waxes as paraffin, Montan, ceresin, carnauba, japan, beeswax, etc., are suitable for the purpose of my invention, although, in most cases, I employ paraffin, particularly on account of its low cost.

In order to obtain the maximum absorption I have found that it is necessary to use a pulp containing a substantial amount of hydrocellulose. While hydrocellulose may be formed by a very prolonged action of pulp in the beater engine, I find it more economical, in most cases, to accelerate the formation of hydrocellulose by means of acid hydrolysis. In general, it is desirable to conduct this acid hydrolysis separately and previous to the introduction of the wax dispersion, although, in some cases, the acid hydrolysis may be employed in the beater simultaneously with the incorporation of the wax emulsion. Since the various acids and salts which may be employed for this treatment are rather corrosive, it is, however, more preferable to treat the pulp previous to the treatment in the beater. In order to accelerate the formation of hydrocellulose, I may add to the pulp such substances as sulphuric, hydrochloric, phosphoric and oxalic acids, or acid salts such as sodium or potassium bisulphates, zinc chloride, and such other salts as potassium iodide, calcium thiocyanide and various other salts which are found to accelerate the formation or production of hydrocellulose. These substances generally exert the most pronounced effect when used at an elevated temperature, i. e., over 80° C. and it is usually necessary to heat the pulp with the concentrated salt solutions above 80° C. for proper modification of the fibres with formation of hydro-

cellulose. Stronger chemicals such as sulphuric acid, when used, usually require no heating.

In order to obtain maximum absorption of wax sizes or emulsions by the pulp I have found not only that the presence of hydro-cellulose is of particular value, but that in order to obtain maximum absorption of the wax, it is highly desirable to bring the pulp to a definite hydrogen ion concentration prior to the incorporation with the wax emulsion. It would seem that the pulp should be maintained slightly on the acid side in order to bring about the greater, more uniform and rapid absorption of the wax emulsion or size employed.

The hydrogen ion concentration which is an exact measure of acidity, particularly when in low concentrations, is usually stated on what is known as a p_H scale. 7.0 is the neutral point. Higher values of p_H above 7, indicate increase in alkalinity. The lower values below 7 indicate increase in acidity.

Accordingly, I prefer to employ a pulp which has been brought substantially on the acid side, that is, having a p_H value of less than 7, but not substantially less than 1.5, since too great an acidity is harmful since it is apt to break down the emulsion and cause coagulation of the colloiddally dispersed wax particles, whereby the proper absorption will not occur.

Ordinary sulphite pulp was found to have a p_H value of approximately 6.8, and it was found that when this pulp was subjected to a beating treatment over a period of twenty-four hours, or so, or merely heated in boiling water for several hours, that the acidity increased. My invention includes the treatment of pulp in such manner to bring about a change of the hydrogen ion concentration, or, in case the p_H value of any given pulp needs adjustment, I may readily do so by the addition of a proper amount of acid or alkali, as the case may require.

I prefer to employ wax dispersions or emulsions containing a minimum of soap since soaps tend to make the emulsion too alkaline and are apt to cause foaming in the beater engine when the emulsion and pulp are blended.

A suitable emulsion may be made in a colloid mill, employing, say, paraffin wax of 135° F. melting point. This was melted in a steam jacketed container and introduced into the mill with an aqueous solution of potassium oleate in equal volumes, the water containing one per cent of potassium oleate. This calculated to give a concentration of $\frac{1}{2}$ per cent of potassium oleate in the finished emulsion. Emulsions prepared by the use of a colloid mill are stable with the presence of a very materially less quantity of stabilizing agent (as the potassium oleate

in this case) than emulsions prepared by other means. The dispersion of the wax is in a state of much finer division than emulsions prepared by other methods. This particular emulsion exhibits Brownian movement, which is common in emulsions prepared by the use of a colloid mill. This is not characteristic of the usual emulsions prepared by other means.

$17\frac{1}{2}$ parts by weight of "glassine" or other pulp, may be incorporated with 10 parts by weight of paraffin wax emulsion containing 5.75 parts by weight of water, $4\frac{1}{4}$ parts by weight of paraffin and $\frac{1}{10}$ part by weight of potassium oleate. Many other emulsions and various proportions may be used for the production of various papers, and I do not wish to limit myself to any specific proportion, the exact proportion for any given paper being readily determined by anyone skilled in the art.

The novel feature of my invention consists in the use of a pulp, either with or without, the presence of hydrocellulose, with the adjustment of the hydrogen ion concentration, in order to promote sizing efficiency.

There are several ways in which I may treat the paper or paper pulp to accelerate the formation of hydrocellulose, but in the preferred form I use sulphuric acid of specific gravity of approximately 1.52 and treat the pulp with this acid for several hours at room temperature, then wash and adjust hydrogen ion concentration. Hydrocellulose is formed in substantial amount. Various other acids or salts may be used in lieu of this treatment, as previously described.

In order to produce a more flexible waterproof paper suitable for the manufacture of milk bottle caps and waterproof papers suitable for use on food containers, I prefer to use a wax dispersion containing a substantial amount of a colorless paraffin oil, such as is known to the trade as "marcol" and whose boiling point is substantially over 200° C. and specific gravity .850 at 17° C. The use of this oil confers added flexibility upon the waterproof product and is therefore highly advantageous especially where product is to be drawn through dies. In certain cases various other petroleum oils may be used, although the pure, almost medicinal grade of oil, known to the trade as "marcol" is to be preferred for the production of waterproof papers which are to be used on food containers.

What I claim is:—

1. In the process of making paper, the step which comprises the adjustment of the hydrogen ion concentration of the pulp so as to increase the absorption of a wax emulsion.

2. In the process of making paper, the step which consists in bringing the pulp to a p_H value of between 1.5 and 7, and incor-

porating therewith a colloidal dispersion of a wax in an aqueous medium.

3. In the process of making paper, the step which consists in bringing the pulp to a p_H value of between 1.5 and 7, and incorporating therewith a colloidal dispersion of a wax and a size.

4. A process for treating paper pulp which contains hydrocellulose comprising the adjustment of the hydrogen ion concentration of said pulp so as to increase the absorption of a wax emulsion.

CARLETON ELLIS.