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METHOD OF PREPARING WEBS FROM
CELLULOSE ESTERS

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This application relates to an improved method of preparing cellulose ester webs which includes the incorporation of staple fibers and plasticizer in the slurry of fibrous cellulose ester from which the web is formed. This invention is an improvement of the invention described in U.S. application Serial No. 533,526, of Charles D. Snead and Ralph W. Peters, filed September 9, 1955.

The Snead and Peters application describes a method for making webs and sheets from cellulose esters in which a fibrous flocculated precipitate of the cellulose ester is suspended in water and the resulting suspension is laid down on a foraminous surface such as the wire of a paper machine to form a sheet thereof. The sheets thus formed are useful for the preparation of cellulose ester sheeting of good properties.

One object of our invention is to provide a method of preparing sheets or webs of cellulose ester material. Another object of our invention is to provide a method of preparing cellulose ester sheeting in which the use of solvent is eliminated. A further object of our invention is to provide a method of making cellulose ester web in which the stock on the paper machine wire is freer and has better drainage than the web which has been formed heretofore from cellulose ester fibers. A still further object of our invention is to provide a method of preparing cellulose ester webs which possess considerably more internal strength than that of the webs which have been prepared heretofore by similar procedures. Other objects of our invention appear herein.

In the Snead and Peters method of preparing cellulose ester webs a cellulose ester in the form of a fibrous flocculated precipitate is dispersed in water and the dispersion so formed is laid down on the wire of the paper machine.

We have found that by incorporating, in the cellulose ester material, 5 to 10% of cellulose acetate staple fiber, based on the total weight of the fiber furnish prior to depositing it on the wire of the paper machine, additional advantages are obtained over those obtained when merely precipitated cellulose ester is employed for this purpose. By incorporating the cellulose acetate staple in the mass there is obtained a furnish which gives a much freer stock on the paper machine wire with much faster drainage and as a result the machine speed may be increased several fold. The staple fiber also acts as a bonding agent which gives the sheet good internal strength so that it may be stripped from the wire and moved to the press rolls without the necessity for a carrying felt. In addition, an emulsion of a liquid plasticizer is incorporated in the dispersion from which the paper is made. The plasticizer becomes intimately associated with the cellulose ester mass and thereby facilitates the conversion of the web into sheeting in subsequent operations.

The cellulose acetate staple fiber employed is desirably material of the conventional type, unplasticized and having a fiber length of $\frac{1}{4}$ to 1 inch. Cellulose acetate staple fiber is a well-known material and is described in texts such as "The New Fibers," by Sherman and Sher-

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man (1946), Van Nostrand, pp. 235-245, and "Rayon and Staple Fiber Handbook," by Mauersberger and Schwarz, 3d edition, page 42, Rayon Handbook Co. The cellulose acetate staple fiber can be varied from 5-10% of the total fiber furnish, approximately 8% being preferred. Also added is an emulsion of a liquid plasticizer in aqueous dispersion. Any of the liquid plasticizers compatible with the cellulose ester (particularly those of the ester type) may be added to the mass in this manner. For cellulose acetate plasticizers such as diethyl phthalate or dimethyl phthalate has been found to be eminently suitable while for other fatty acid esters of cellulose such as cellulose acetate butyrate, plasticizers such as dibutyl sebacate, dibutyl phthalate or dioctyl phthalate are useful. Plasticizers of the glycol type such as triethylene glycol dicaprylate are useful for the propionic acid esters of cellulose.

A slurry or suspension of cellulose ester fiber and staple is prepared in any suitable apparatus such as a hollander, hydropulper, turbomixer or the like wherein the slurry is formed without breaking up or otherwise altering the physical properties of the fibers.

In making the web, the aqueous suspension of fibrous cellulose ester, staple fiber and plasticizer emulsion is diluted to a low consistency such as on the order of 0.5-1% (if not already dilute) and is applied to the wire of the paper machine. The web after formation may be pressed to impart increased strength and compactness and may be dried at a moderately elevated temperature such as in an oven or a drying drum. The product may be marketed either in that form or after subjecting to a partial or complete fusion by the use of heat and pressure. The product is useful for forming into sheet material or shaped products by the use of elevated temperatures and pressures as desired.

We have found that in our operations it is often desirable to use a steam jet on the surface of the web which is formed on the wire of the paper machine shortly prior to the time the web is subjected to treatment in a pressure couch roll operation which acts to compact the sheet, makes it very much stronger and contributes to softening of the product prior to the pressing operation. Thereby the operating conditions in the pressing and drying ends of the machine are greatly facilitated. The following examples are illustrative of the invention:

Example 1

A slurry of 100 pounds of precipitated fibrous cellulose acetate in 1250 pounds of water was added to a conventional paper machine mixing chest having 1250 pounds of water therein. After this addition, 800 pounds more water were added.

10 pounds of $\frac{3}{4}$ inch cellulose acetate staple fiber of the conventional type was dispersed in 450 pounds of water in a 35 pound Valley beater for 10 minutes at minimum roll pressure. The staple fiber dispersion thus formed was added to the machine mixing chest together with 200 pounds of water.

150 pounds of an emulsion of dimethyl phthalate in equal parts of water prepared by using Triton N-100 (or any oil-in-water emulsifying agent) was prepared and was added to the mass in the mixing chest. The stock slurry was allowed to stand for 45 minutes. The slurry was then formed into a web on the wire of a conventional Fourdrinier paper machine and was dried in the normal manner employed in making paper. The addition of the cellulose acetate staple permitted much faster drainage on the wire of the paper machine and the web obtained could be handled without the necessity of using felt.

Example 2

A procedure as described in the preceding example was carried out but a shower of steam of sufficient width to cover the span of the web was applied to the web surface at a point from two to six inches ahead of the nip of the pressure couch employed in the web making operation. The steam had a softening effect on the plasticized cellulose acetate web which was sufficient to permit the pressure from the couch rolls to partially fuse the acetate fibers of the web. The sheet thus obtained exhibited extraordinary high wet strength and could be readily handled.

The plasticizers used in cellulose dispersions in carrying out our invention are first emulsified to form an aqueous emulsion thereof by means of a surface active agent which will retain the plasticizer in the water in dispersed form. The surface active agent used may be any compound which is commonly known as suitable for use in preparing oil-in-water emulsions. It may be of the non-ionic type, for instance, an alkyl aryl polyether alcohol, such as is marketed as Triton N-100, or it may be of the cationic type, for instance stearyl dimethyl benzyl ammonium chloride, such as is marketed as Triton X-400.

We claim:

1. A method of making sheets adapted to be formed into products which comprises forming a dilute suspension in water of a fibrous flocculated precipitate of a lower fatty acid ester of cellulose and 5-10% of cellulose ester staple fiber, laying down the suspension onto a foraminous surface to form a sheet therefrom and drying the so formed sheet.

2. A method of forming sheets adapted to be formed into desired products which comprises forming a dilute suspension in water of a fibrous flocculated precipitate of a lower fatty acid ester of cellulose and 5-10% of cellulose ester staple fiber, adding thereto an aqueous emulsion of a plasticizer for the cellulose esters, laying down the suspension onto a foraminous surface to form a sheet therefrom and drying the so formed sheet.

3. A method of making colloidized cellulose ester sheeting which comprises forming a suspension in water of a fibrous flocculated precipitate of a lower fatty acid ester of cellulose and 5-10% of cellulose ester staple fiber, adding thereto an aqueous emulsion of a cellulose

ester plasticizer, laying down the suspension onto a foraminous sheet and subjecting to sufficient heat and pressure to at least partially fuse the material.

4. A method of making sheets adapted to be formed into desired products which comprises forming a dilute suspension in water of a fibrous flocculated precipitate of a lower fatty acid ester of cellulose and 5-10% of cellulose ester staple fiber, adding thereto an aqueous emulsion of a plasticizer, laying down the suspension onto a foraminous surface to form a sheet therefrom, subjecting the sheet thus formed to a jet of steam followed by subjecting the sheet to pressure sufficient to at least partially fuse the fibers thereof.

5. A method of making sheets adapted to be formed into desired products which comprises forming a dilute suspension in water of a fibrous flocculated precipitate of a lower fatty acid ester of cellulose and 5-10% of cellulose ester staple fiber, laying down the suspension thus formed onto the wire of a paper machine and drying the thus formed product.

6. A method of making sheets adapted to be formed into desired products which comprises forming a dilute suspension in water of a fibrous flocculated precipitate of a lower fatty acid ester of cellulose and 5-10% of cellulose acetate staple fiber, laying down the suspension onto a foraminous surface to form a sheet therefrom and drying the thus formed sheet.

7. A method of making sheets adapted to be formed into desired products which comprises forming a dilute suspension in water of a fibrous flocculated precipitate of cellulose acetate and 5-10% of cellulose acetate staple fiber, adding thereto an aqueous emulsion of a cellulose acetate plasticizer, laying down the suspension onto a foraminous surface to form a sheet therefrom and drying the thus formed sheet.

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