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SHEETED CELLULOSE DERIVATIVE FIBERS

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ABSTRACT OF THE DISCLOSURE

Coherent sheets of at least partially water soluble and water swellable carboxy methyl cellulose fibers having requisite physical properties for handling and comminution are prepared by slurrying said carboxy methyl cellulose fibers in aqueous organic slurry media and forming fibrous sheets from the fibrous slurries. The fibrous sheets are then dried after displacement of additional amounts of water from the formed sheets, by successive stages of aqueous alcoholic solution displacement washing of decreasing water content, so that final drying proceeds from a moist fibrous sheet containing 0.02 to 0.5, preferably 0.02 to 0.3, parts by weight of water to carboxy methyl cellulose fiber.

FIELD OF THE INVENTION

This invention relates to fiber sheet formation techniques and more particularly to providing sheets of carboxy methyl cellulose fibers having superior physical attributes for handling in rolls and sheets. The fibrous sheets of this invention are also particularly adapted to comminution into their separated fibers for precision dispersive applications.

BACKGROUND OF THE INVENTION

At least partially water soluble and water swellable cellulose derivatives such as sodium carboxymethyl cellulose, wet epichlorohydrin cross-linked sodium carboxymethyl cellulose and self wet cross-linked sodium carboxymethyl cellulose are available in their fibrous forms. Such at least partially water soluble and water swellable sodium carboxymethyl cellulose fibers are disclosed in U.S. 3,347,855 issued to Russel Nelson on Oct. 17, 1967. Processes for making wet epichlorohydrin cross-linked cellulose derivative fibers and the products and utilization thereof are disclosed in U.S. 3,589,364 issued to Walter L. Dean and George N. Ferguson on June 29, 1971. Cellulose derivatives prepared and utilized according to the foregoing patent disclosure and teaching are highly absorbent and fluid retentive for use in specific products where these absorbent qualities contribute either enhanced product effectiveness or a hitherto unattainable result. Further examples of water soluble or partially water soluble fibers utilizable in the sheeting processes and products of the present invention are the self wet cross-linked carboxymethyl cellulose fibers produced according to the process disclosed in U.S. Ser. No. 82,797 filed by Howard L. Schoggen on Oct. 21, 1970, now issued as U.S. 3,678,031 on July 18, 1972.

Although cellulose derivative fibers have been available in bulk powder and, to some lesser extent, bulk fiber form, it has been noted that these physical forms can present problems where precision distribution and application of the cellulose derivative is desired. For example, continuous precision weight measurement of a bulk powder or a bulk fiber mass has proven to be difficult to achieve due to caking, bin bridging and weighing difficulties. These handling and weighing problems make difficult or impossible the precision dispersion and even application of precise amounts of cellulose derivative

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fibers on desired surfaces or in fibrous combinations in the preparation of hygienic, surgical, and personal use products such as baby diapers, surgical sponges, dressings and catamenial devices.

In order to alleviate the handling and dispersion problems attendant to the placement of cellulose derivatives of the stated types in the described usages, various devices utilizing several stages of increasing mechanical complexity have been proposed and utilized. Whereas these weighing devices and pieces of handling equipment aid in solving the problem, their use is expensive in equipment cost and generally requires meticulously skilled maintenance to insure continued accuracy.

In contrast, the cellulose derivatives provided in sheet form by the present invention are capable of roll or flat storage and, because of the accuracy of sheet weights per unit area, are susceptible of accurate weight feeding by sheet or calibrated roll unwind speed. Such area feeding provides excellent precision in feeding a process or a comminution device whose output is either distributed on or combined in a product.

Cellulose derivative fibers having specific physical properties, for example methyl cellulose fibers, have been prepared in sheet form by the process disclosed in U.S. 2,364,028 issued to Floyd C. Peterson on Nov. 28, 1944. Further, cellulose derivative fibers have been slurried in alcoholic solutions and formed into sheet structures according to the process of U.S. 2,648,635 issued to Russell Jacques Brown, John Downing and Walter Henry Groombridge on Aug. 11, 1953. Applicants have found, however, that these prior processes are not generally applicable and fail to provide sheeted cellulose derivative products which exhibit in combination physical properties for handling and comminution quality.

OBJECTS OF THE INVENTION

It is, therefore, an object of this invention to obviate the above problems.

It is another object of this invention to provide a process for the formation of at least partially water soluble and water swellable cellulose derivative fibers in coherent sheet form.

It is yet another object of this invention to provide sheeted partially water soluble and water swellable cellulose derivative fibers in a coherent sheet form having even formation and requisite physical characteristics for handling and comminution.

SUMMARY OF THE INVENTION

Briefly stated, in accordance with one aspect of the invention, there are provided sheet forms of at least partially water soluble and water swellable cellulose derivative fibers by preferably slurrying same in aqueous methyl and isopropyl alcohol solutions and combinations thereof, containing about 32% to 50% by weight of water to preserve fibrous integrity, and thereafter sheet forming the fibers from said slurry. Other organic slurry media conventionally used in the production of such fibers, including acetone, can be used to prepare the slurries. In order to achieve a pliable, windable and comminutable sheet having the prerequisite strength for handling, the aqueous content of the formed fibrous sheet is decreased after sheet formation so that the sheet is finally dried from a condition wherein it contains about 0.02 to 0.5, preferably 0.02 to 0.3, parts by weight of water per part of cellulose derivative fiber. In general, the cellulose derivative fibers are displacement washed in several stages after sheeting, with alcoholic solutions containing lesser amounts of water in the successive stages prior to final drying. Cellulose derivative sheets dried directly from sheet formation, and not accorded the alcoholic washing of the present invention, have been found to exhibit a comminution quality, as hereinafter defined, of about 50 to 80. It has been discovered preferable in the execution of the present sheeting process to reduce the water content of the cellulose derivative fiber sheet gradually in the successive stages of alcoholic solution displacement washing so that the first alcoholic displacement wash should contain at least 10% by weight of water so that the free water content of the cellulose derivative fibrous sheet is gradually reduced.

Subsequent to methyl alcohol countercurrent washing to adjust the water content of the sheet to about 0.02 to 0.5, preferably 0.02 to 0.3, parts by weight of water to cellulose derivative fiber in free contact with the water swollen fibers, as a means of adjusting the physical properties of the sheet product, the washed sheet is dried. In another means of reducing the free water in contact with the fibers, additional isopropyl alcohol can be added in the final stage of countercurrent washing so that free water is taken up in azeotropic solution with the isopropyl alcohol.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

In the production by slurry processes of partially water soluble and water swellable cellulose derivative fibers, such as those identified heretofore, the fibers are generally available in a moist fibrous cake form. In processes where the slurry reaction to produce the fibers was carried out in an isopropyl alcohol medium to prevent dissolving the cellulose derivative product in the water present as formed, the moist fibrous cake generally contains about 30% to 60% by weight of soft, pliable water swollen fibers. The percentage is dependent upon the efficiency of operation of the centrifuge or other device used to expel the reaction media from the fibrous products. The remaining about 40% to 70% by weight in the moist cake of fibers is isopropyl alcohol and water. In the 30% to 60% by weight of solids contained in the moist fibrous cake, about 50% to 70% by weight comprises the partially water soluble and water swellable cellulose derivative fibers from which the present superior cellulose derivative fiber sheets are produced. The remainder of the solids content of the moist fiber cakes are by-product salts of the cellulose derivative fiber formation reactions, usually sodium chloride since the preferred cellulose derivative fibers are commonly produced in the forms of their sodium salts. The process of the present invention is additionally advantageous in insuring the complete removal of these by-product salts from the cellulose derivative fibers during the displacement washing occurring after sheet formation.

In order to advantageously carry out the sheeting process of the present invention, the preferably moist cake of cellulose derivative fibers or cellulose derivative fibers in other available forms including dried cellulose derivative fibers, must be separated and dispersed in slurry form. In an embodiment of the invention wherein the slurrying proceeds from the moist cake state and salts of reaction, normally sodium chloride, are present, it is preferable that the slurrying operation also leach the salts of reaction from and remove same from the cellulose derivative fibers.

Organic slurry media conventionally used in the production of potentially water soluble and water swellable cellulose derivative fibers, including acetone, can be used in aqueous solutions to accomplish dispersion slurrying.

Both methyl and isopropyl alcohol and combinations thereof are preferably employed in aqueous solutions to accomplish dispersion slurrying of the cellulose derivative fibers in the present invention and the present process is further described herein in terms of their use. Methyl alcohol is the preferable slurrying media for salt removal in that the usual salt of reaction, sodium chloride, is more soluble in aqueous solutions of methyl alcohol. As will be more fully discussed below, however, isopropyl alcohol used alone or in combination with methyl alcohol has an advantage in producing an extremely soft and comminutable sheet product where these properties are desired.

Proceeding with the step of dispersion slurrying it is advantageous to the production of even sheet formation that the alcoholic solution used have a water concentration sufficiently high to produce fiber swelling and reduce fiber floccing and sufficiently low to prevent fiber solubilization. In order to achieve these dual results the aqueous alcoholic solution for dispersion is adjusted to contain about 32% to 50%, preferably about 40%, by weight of water, prior to slurrying the cellulose derivative fibers therein.

The amount by weight of alcoholic solution employed as a slurrying media is adjusted relative to the amount of fibers dispersed and slurried therein so that a fiber concentration of about 0.5% to 3%, preferably about 2%, by weight in the aqueous alcoholic slurry media is obtained. The above noted fiber concentrations in the slurry media result in a stirred and agitated fibrous slurry wherein the fibers are swollen and well separated for subsequent sheet formation. As an additional aid to fiber dispersion in the slurry media it has been discovered that moderate increases in slurry temperature are conducive to improved dispersion in the presently employed aqueous alcoholic slurry media; temperatures of about 20° C. to 50° C., preferably 50° C., have been found particularly advantageous in promoting the desired fibrous dispersion.

Subsequent to preparation of the dispersed fibrous slurries in the aqueous alcoholic slurry media the fibers are formed into sheets on a level belt filter. A 60 by 40 mesh woven filter media formed from polypropylene monofilaments has been found efficacious in the present process. Other filter media suitable for the formation of fiber sheets and commensurate with the drainage rates of the present cellulose derivative fiber dispersions will be found suitable for use in the process.

In forming the fibrous sheets, the fiber dispersions as first made can be adjusted to fiber consistencies of about 0.5% to 2.0% by weight with aqueous alcoholic solutions, preferably from the recycled alcohol wash solutions described below, and introduced into a headbox of the conventional papermaking type. The fibrous slurry is then flowed from the headbox out onto the filter media at papermaking slurry velocities of about 4 to 100, preferably 25 to 50, feet per minute. Higher formation speeds in the papermaking range are possible. As an example, filter media travel speeds of about 4 to 100, preferably 25 to 50, feet per minute and higher are preferable in the formation of the fiber sheets having a dry basis weight of about 110 to 250 pounds, preferably about 210, pounds on a 19 by 24 inch-500 sheet ream weight basis. Fibrous sheets having other basis weights can be produced by the process of the present invention.

Although the present invention is described herein in terms of a level belt filter similar to the conventional Fourdrinier forming wire used in papermaking, the cellulose derivative fibrous sheets can be formed advantageously on other types of conventional paper forming equipment, for example, laboratory handsheet equipment, cylinder machines and slanted wire Fourdrinier sheet formation equipment. It is specifically noted that any sheet forming equipment selected for use in carrying out the present process must be operated at speeds in accordance with drainage characteristics of the dispersed slurries, as will be readily understood by those skilled in the art.

After sheet formation the formed fibrous sheets travel forward on the filter media and are displacement washed with aqueous alcoholic solutions of decreasing water content applied by sprays or other conventional distributive equipment. In carrying out the displacement washing step critical to the present process and product, the objectives are to displace and gradually decrease the water remaining in the sheet as first formed. The displacement washing serves the purpose of insuring that the fibrous sheet, prior to drying, contains about 0.02 to 0.5 (preferably 0.02 to

0.3, parts by weight of free water to fiber. Vacuum pressure differentials of, for example, about 5 inches of Hg, applied through the sheet and filter media, have been found useful in promoting thoroughness of displacement in the washing stages.

In the most preferred embodiment of the invention, the displacement washing is carried out by countercurrent stages of displacement washing using about 10 to about 50, preferably about 30, parts by weight of methyl alcohol per part by weight of fiber.

In other embodiments of the invention an ultra soft sheet and comminutable sheet is produced by adding at least 6.9 parts by weight of isopropyl alcohol per part of free water, that is water between the fibers, remaining in the fibrous sheet in the last countercurrent displacement wash to form an azeotropic solution with said water. Such application of isopropyl alcohol can be made either in a separate final washing stage or in combination with the final methyl alcohol washing stage. Although the mechanism of such ultra soft sheet formation is not fully understood, applicants theorize that the use of such an amount of isopropyl alcohol in the final countercurrent displacment washing stage combines any remaining free water in an azeotropic solution. The fibers of the sheet are then bonded by the water in the swollen fibers.

Applicants have found that the displacement washing of the formed fibrous sheet to adjust water content prior to final drying can be most advantageously applied in 2 to 5, preferably 3 to 4 stages. It has been found further advantageous, in producing the present product sheets of desirable physical characteristics and comminution quality, that the water content of the formed sheet be reduced gradually, i.e., it has been found harmful to produce sheet quality to wash the first formed sheet with substantially non-aqueous methyl alcohol in the first washing stage after sheet formation, and aqueous alcoholic solutions containing at least 10% by weight of water are preferred in this stage.

The preferred countercurrent displacement washing stages are arranged in countercurrent sequence along the forward traveling filter media such that the aqueous alcoholic wash liquid containing the least amounts of water is applied in the wash stage adjacent to the subsequent drying step. In one preferred embodiment of the invention, according to the above general mode of operation, there are provided three stages of aqeuous alcoholic displacement washing using in each washing stage 20 to 40, preferably 30 parts by weight of alcohol per part by weight of fiber in the fibrous sheet. In the stage of displacement washing adjacent the dryer the aqueous alcoholic wash liquid is substantially dry methyl alcohol. The second stage of displacement washing uses the countercurrently recycled wash liquid from the first stage of displacement washing and contains about 10% by weight of water picked up by the displacement wash in the first stage. The third stage of displacement washing, the stage immediately subsequent to sheet formation, employs the recycled aqueous methyl alcohol wash liquid from the second displacement wash stage and contains about 20% by weight of water.

Two, four and five stage displacement wash operation is also advantageous and is operated in a similar manner with the resulting balanced differences in the alcoholic wash liquid water content in the individual stages of displacement washing.

All modes of preferred displacement washing operation accomplish the result of avoiding initial washing of the formed sheet, immediately after sheet formation, with alcoholic wash liquid containing less than about 10% by weight of water to insure that the sheet does not become glazed, although such operation is within the scope of the invention. It is noted that close physical placement of the washing stage sprays or other distributive means is preferred to avoid the passage of air through the sheet between displacement washing stages; such air passage has been found to reintroduce water from the atmosphere air into the sheet.

It is important that the fibrous sheet product physical and comminution characteristics be controlled by adjustment of the displacement wash rate or addition of water to the stage of displacement washing adjacent the drying stage to insure a fibrous sheet water content immediately prior to drying of about 0.02 to 0.5, preferably about 0.02 to 0.3 parts by weight of water per part of cellulose derivative fiber. Higher final water contents in the fibrous sheet, immediately prior to drying, tend to stiffen the product sheet and lessen comminution quality. Lower final water contents in the fibrous sheet, immediately prior to drying, tend to decrease the strength properties of the fibrous sheet product and increase comminution quality.

In operation of the present process, particular attention is essential to achieving the low ranges of parts by weight of water to fiber specified. It is theorized, although applicants do not wish to be bound by such theory, that the displacement washing of the present process affects largely the free water between the water swollen fibers and not the water retained within the fibers themselves. As stated above, the addition of amounts of isopropyl alcohol to the displacement wash liquid in the methyl alcohol displacement wash stage adjacent the drying stage in amounts to azeotrope remaining water is advantageous. Such isopropyl alcohol can also be added to the fibrous sheet in a separate washing stage immediately prior to drying. These additions of isopropyl alcohol result in a softer fibrous sheet product having an increased comminution quality.

Subsequent to final water content adjustment in the fibrous sheet for desired sheet property characteristics, the fibrous sheet is dried. Drying can be accomplished by conventional drying equipment in which provision for inflammable solvents is provided. Drum dryers, through tunnel dryers and air dryers in which heated air or inert gas passes through the fibrous sheet can all be used to accomplish drying. In one preferred embodiment of the process, the sheet is dried by passing inert gas, e.g. nitrogen, at about 250° F. through the fibrous sheet at the rate of about 80 to 120 $ft.^3$/min./square foot of sheet to be dried. Under such drying conditions a drying time of about 4 minutes has been found efficacious. The dried fibrous sheet product can be slitted, cut to length, sheeted and wound, as desired, on conventional equipment for storage and handling.

The preferred dried coherent fibrous sheet product of the present process has, as stated heretofore, a basis weight of about 110 to 250 pounds, preferably about 210 pounds on a 19 by 24 inch-500 sheet ream weight basis. Such product sheets will have a thickness of about 0.050 inch to about 0.120 inch, preferably about 0.060 inch to 0.080 inch, depending upon roll pressures applied to the sheet during formation and drying. As stated heretofore, fibrous sheets having other basis weights can be produced by the process of the present invention. Such fibrous sheets will have commensurate thicknesses and exhibit the present comminution quality. In addition to the foregoing physical attributes, the fibrous sheets of the present invention exhibit a comminution quality as defined in the following paragraph of about 0.1 to 30, preferably about 0.1 to 20. Applicants find the achievement of such excellent comminution quality in a coherent fibrous sheet of the present fibers surprising and contributive to the art in that such sheets, formed and dried directly from fibrous slurries, exhibit comminution qualities of about 80.

The comminution quality test results set forth referred to above and set forth in the following examples were obtained by the Quick Disintegration test method, carried out as follows. Dried fibrous sheet samples are conditioned over night at 50 percent relative humidity and 72° F. The conditioned dried fibrous sheet samples are then cut into 1 inch by 1 inch squares. A whole number of squares weighing 10 to 12 grams is weighed and the weight recorded to constitute a sample. The weighed sample of 1 inch by 1 inch squares is placed into a Waring Blender (Model No. CB–15) with dull blades and whizzed at the lowest speed (16,500 r.p.m.) for 15 seconds. The resulting fibers are placed, without densification on the top of a standard 14 mesh screen placed between two airtight chambers. Compressed air is introduced into the top chamber to violently agitate, but not further comminute the fibers, while the lower chamber is provided with a vacuum system for removing fibers which pass through the screen. The compressed air and vacuum system acting in conjunction provide a 52 mm. Hg differential through the screen, which pressure differential is applied for 2 minutes. The fraction of fiber remaining on the top of the 14 mesh screen and the fraction falling through are then recovered and weighed. The percentage of total fiber weight remaining on the 14 mesh screen is recorded. At least two, and preferably 10, replicate Quick Disintegration tests are made, and their mathematical average is recorded as the test result. Higher Quick Disintegration test numbers indicate a lower comminution quality while lower Quick Disintegration test numbers denote easier comminution and higher comminution quality. The Mullen and tensile strength test results noted in the examples were obtained by conventional test methods common in the paper industry.

Having described the process and product of this invention, the following examples are intended to illustrate advantageous modes of operation within the process to produce sheeted cellulose derivative fiber products having desirable physical properties. It will be understood that those skilled in the art will immediately be aware of other advantages stemming from the herein disclosed inventive concept. It is understood, therefore, that the examples are intended to be illustrative and not limiting, and the scope of the invention is only to be construed by the scope of the appended claims.

Examples I–IV

Laboratory handsheets were prepared by slurrying bibulous fibers, prepared according to the process disclosed in U.S. 3,589,364 and referred to therein as CLD fibers, in an alcoholic solution containing 60% methyl alcohol and 40% water by weight. The fibrous slurry was prepared at 50° C. and contained 0.5% by weight of the fibers. After dispersion of the fibers in the slurry by stirring, handsheets having a basis weight of 220 pounds on a 19 by 24 inch-500 sheet ream weight basis were formed on conventional laboratory handsheet equipment. The formed fibrous sheets were washed in three stages of displacement washing with aqueous methyl alcohol solutions using in each washing stage 20 parts by weight of aqueous methyl alcohol solution per part of fiber weight in the formed fibrous sheets.

The first washing stage after sheet formation was carried out with the stated amount of aqueous methyl alcohol solution containing an amount of water such that the aqueous alcoholic solution contained 80% by weight of methyl alcohol and 20% by weight of water.

The second washing stage was carried out with the same stated amount of aqueous methyl alcohol solution per part of fiber weight in the formed fibrous sheets. The aqueous methyl alcohol solution in the second washing stage contained an amount of water such that the alcoholic solution contained 90% by weight of methyl alcohol and 10% by weight of water.

The third stage of washing prior to sheet drying was carried out with the same amount of aqueous methyl alcohol solution wherein the water content was varied for Examples I–IV. The water contents of the aqueous alcoholic solutions used in the foregoing washing stages simulated the water contents of aqueous alcoholic washing solutions employed in continuous operation of the present process. In Example I, the third stage wash contained 0% water, while the third stage wash in Example II contained 2.5% by weight of water. Similarly, Example III had 5.0% by weight of water in the final washing stage, and Example IV had 10% by weight of water. Examples I–IV illustrate formation of the present fibrous sheets by laboratory handsheet methods and the preferred mode of operation wherein the formed fibrous sheet is subjected to displacement washing in stages with aqueous alcoholic solutions of gradually decreasing water content.

Subsequent to the third stage of displacement washing, the fibrous sheets of Examples I–IV were dried on a laboratory drum dryer and tested for comminution quality. The comminution quality test results together with the parts by weight of water per part by weight of fiber in the fibrous sheets prior to drying are tabulated in Table I below.

TABLE I

	Parts water per part fiber to drying (by weight)	Comminution quality
Example:		
I	0.125	2.3
II	0.144	9.8
III	0.216	7.9
IV	0.384	14.3

The fibrous sheet products of Examples I–IV were coherent and of excellent comminution quality well suited for precision weight dispersion by comminution into fiber combinations for use in hygienic, surgical and personal use products. The processes of Examples I–IV have been used to prepare similar fibrous sheet products from sodium carboxymethyl cellulose fibers.

Examples V–VII

Additional laboratory handsheets were prepared according to the method of Example I above with the exception that in Examples V and VII the water present in the aqueous alcohols solution used to slurry the bibulous fibers prior to sheet formation was varied. In Example V, the alcoholic slurrying solution contained 36% by weight of water, while in Example VII the alcoholic slurrying solution contained 44% by weight of water. The alcohlic slurrying solution used in Example VI contained the 40% by weight of water used in Example I. Examples V–VII were prepared to show the effect on sheet formation quality due to varying water content in the alcoholic slurrying solution. The dried fibrous sheet products of Examples V–VII were visually examined for formation quality, i.e evenness of fiber distribution in the sheet, and tested for comminution quality; the results are tabulated in Table II below.

TABLE II

	Percent water in alcoholic slurrying solution (by weight)	Formation quality	Comminution quality
Example:			
V	36	Poor	2.3
VI	40	Good	1.8
VII	44	Best	3.2

The coherent fibrous sheet products of Examples V–VII are useful for precise weight distribution by comminution onto the surfaces of baby diapers. Similar fibrous sheet products can be prepared according to the procedure of Example I with the exception that the initial fiber slurry dispersion is conducted at 20° C.

Examples VIII and IX

Coherent fibrous sheets were prepared, in a continuous sheet formation operation, from self wet crosslinked carboxymethyl cellulose fibers. The fibers were first slurried and dispersed by agitation in an alcoholic solution containing 57% by weight of methyl alcohol and 43% by weight of water. The slurry dispersion contained 3% by weight of fibers and was at a temperature of 50° C. Prior to introduction into a conventional headbox for continuous sheet formation, sufficient aqueous methyl alcohol was added to the slurry so that the headbox slurry fiber consistency was reduced to 2% by weight.

The fibrous slurry was then run out through the headbox slice onto a moving continuous belt filter consisting of a 60 by 40 mesh woven cloth filter media formed from polypropylene monofilaments to form a continuous sheet having a dry basis weight of 210 pounds on a 19 by 24 inch-500 sheet ream weight basis.

The moving sheet was then countercurrently displacement washed in Example VIII by four stages of displacement washing using in each washing stage, as washing liquid, an alcoholic solution containing 30 parts by weight of methyl alcohol per part by weight of fiber in the sheet. The procedure of countercurrent washing was such that the moving sheet was displacement washed with the stated quantity of methyl alcohol containing 0% by weight of water just prior to drying. The remaining three stages of countercurrent washing were applied successively and countercurrently to the continuous advance of the fibrous sheet in the direction of the headbox. Taken in direction countercurrent to the direction of fibrous sheet advancement, each displacement washing stage was effected with the stated quantity of methyl alcohol together with water taken from the fibrous sheet in the preceding displacement wash. In the four successive countercurrent displacement washing stages the water content of the aqueous alcoholic wash liquid was 0%, 3–7%, 7–12% and 19–24% as determined by individual samples during continuous operation. Similar continuous sheet formation operation can be carried out using 2 and 5 stages of countercurrent displacement washing, although in the operation with 2 stage countercurrent washing particular attention must be paid to achieving efficient displacement in the limited number of washing stages.

Subsequent to countercurrent displacement washing, the fibrous sheet was dried by passing through it nitrogen at 250° F. at the rate of 100 $ft.^3$/min./square foot of continuously advancing fibrous sheet for four minutes. Similar drying operation can be carried at different temperatures and rates of nitrogen passage through the sheet; the times for drying will, of course, vary.

The fibrous sheet of Example IX was prepared in the same manner as that of Example VIII with the exception that just prior to drying and subsequent to the displacement wash with methyl alcohol containing 0% by weight of water, there was applied to the fibrous sheet isopropyl alcohol an amount equal to 6.9 parts by weight of isopropyl alcohol per part of water remaining in the fibrous sheet. It is noted that a similar sheet product will result if the isopropyl alcohol is applied together with the pure methyl alcohol used in the preceding stage of countercurrent displacement washing.

The dried coherent fibrous sheets of Examples VIII and IX were tested for comminution quality with the result that Example VIII had a comminution quality of 5.5. The comminution quality of Example IX was 0.5. These results illustrate the improvement obtained in comminution quality obtained by the inclusion of amounts of isopropyl alcohol in the fibrous sheet just prior to drying, according to the present invention.

In the continuous sheet formation procedure of Example VIII, inefficient displacement in the washing stages can yield a coherent fibrous sheet product with a comminution quality of 30. Comminution qualities of 0.1 can be obtained by applying 15 parts of isopropyl alcohol instead of 6.9 parts in the procedure of Example IX. The coherent fibrous sheet products are well suited for rolling and subsequent comminution dispersion into hygienic and surgical products.

Examples X–XVII

In order to further illustrate the sheet property effects obtained by variance in the parts by weight of water per part by weight of fiber in the fibrous sheets of the present invention just prior to drying, Examples X–XVII were prepared according to the procedures of Examples I–IV with the exception that the water content in the third stage of alcoholic washing immediately prior to sheet drying was varied as tabulated in Table III below. The parts by weight of water per part by weight of fiber in the fibrous sheets prior to final drying are tabulated in relation to the fibrous sheet characteristics as illustrated by Mullen tests, for sheet elasticity and bursting properties, and tensile strength tests.

TABLE III

	Percent water in final displacement wash (by weight)	Parts water per part fiber to drying (by weight)	Mullen (lbs./in.²)	Tensile strength (lbs./in.)
Example:				
X	0	0.079	52	1.1
XI	2	0.094	50	1.8
XII	4	0.167	40	3.6
XIII	6	0.238	40	7.9
XIV	8	0.274	58	12.4
XV	10	0.352	59	19.7
XVI	12	0.413	73	39.2
XVII	14	0.442	111	58.7

The fibrous sheet products of Examples X–XVII had comminution qualities of 5 to 30 for comminution ease and their physical properties were suitable for handling and comminution according to the present invention.

Many modifications of the above invention may be used and it is not intended to hereby limit it to the particular embodiments shown or described. In particular those skilled in the art will readily understand that while the invention has been described in terms of particular fibers, alcohols and operational ranges, that other fibers, organic slurry media and displacement washing alcohols having similar physical properties can be utilized within the broad scope of the invention. Likewise, the principles of the invention are utilizable to produce fibrous sheets having a broad range of physical properties according to the desires of the practitioner. The terms used in describing the invention are, therefore, used in their descriptive sense and not as terms of limitation, it being intended that all equivalents thereof be included within the scope of the appended claims.

What is claimed is:

1. A process for the production of coherent fibrous sheets from fibers consisting essentially of at least partially water soluble and water swellable carboxymethyl cellulose fibers, which process comprises the steps of:

(a) forming a fibrous slurry dispersion of said fibers at a fiber consistency of about 0.5% to 3% by weight in an aqueous organic slurry media, containing about 32% to 50% by weight of water,

(b) forming a fibrous sheet from the slurry dispersion on a filter media,

(c) washing the fibrous sheet in 2 to 5 stages of alcoholic displacement washing, using about 10 to about 50 parts by weight of alcohol per part of carboxymethyl cellulose fiber in the fibrous sheet in each washing stage, to gradually decrease the water content of the fibrous sheet to about 0.02 to 0.5 part by weight of water per part by weight of carboxymethyl cellulose fiber, and

(d) drying the washed fibrous sheet to produce a coherent fibrous sheet having a comminution quality of about 0.1 to 30.

2. A process for the production of coherent fibrous sheets from fibers consisting essentially of at least partially water soluble and water swellable carboxymethyl cellulose fibers, which process comprises the steps of:

(a) forming a fibrous slurry dispersion of said fibers at a fiber consistency of about 0.5% to 3% by weight in an aqueous alcoholic solution, containing about 32% to 50% by weight of water,

(b) forming a fibrous sheet from the slurry dispersion on a filter media,

(c) washing the fibrous sheet in 2 to 5 stages of alcoholic displacement washing, using about 10 to about 50 parts by weight of alcohol per part of carboxymethyl cellulose fiber in the fibrous sheet in each washing stage, to gradually decrease the water content of the fibrous sheet to about 0.02 to 0.5 part by weight of water per part by weight of carboxymethyl cellulose fiber, and

(d) drying the washed fibrous sheet to produce a coherent fibrous sheet having a comminution quality of about 0.1 to 30.

3. A process for the production of coherent fibrous sheets from fibers consisting essentially of at least partially water soluble and water swellable carboxymethyl cellulose fibers, which process comprises the steps of:

(a) forming a fibrous slurry dispersion of said fibers at a fiber consistency of about 0.5% to 3% by weight in an aqueous methyl alcohol solution, containing about 32% to 50% by weight of water, at about 20° C. to 50° C.,

(b) forming a fibrous sheet having a basis weight of about 110 to 250 pounds on a 19 by 24 inch-500 sheet ream weight basis from the slurry dispersion on a filter media travelling at a speed of about 4 to 100 feet per minute,

(c) washing the fibrous sheets in 2 to 5 stages of countercurrent alcoholic displacement washing using about 10 to about 50 parts by weight of methyl alcohol per part of carboxymethyl cellulose fiber in the fibrous sheet in each washing stage, to gradually decrease the water content of the fibrous sheet to about 0.02 to 0.5 part by weight of water per part by weight of carboxymethyl cellulose fiber, and

(d) drying the washed fibrous sheet to produce a coherent fibrous sheet having a comminution quality of about 0.1 to 30.

4. The process for the production of coherent fibrous sheets from fibers consisting essentially of at least partially water soluble and water swellable carboxymethyl cellulose fibers of Claim **3** wherein:

(a) the fibrous slurry dispersion of said fibers is formed in step (a) at a fiber consistency of about 2% by weight in an aqueous methyl alcohol solution, containing about 40% by weight of water, at 50° C.,

(b) a fibrous sheet having a basis weight of about 210 pounds on a 19 by 24 inch-500 sheet ream weight basis is formed in step (b) from the slurry dispersion on a filter media traveling at a speed of about 25 to 50 feet per minute,

(c) the fibrous sheet is washed in 3 to 4 stages of countercurrent alcoholic displacement washing stages in step (c) using about 30 parts by weight of methyl alcohol per part of carboxymethyl cellulose fiber in the fibrous sheet in each washing stage, to gradually decrease the water content of the fibrous sheet to about 0.02 to 0.3 part by weight of water per part by weight of carboxymethyl cellulose fiber, and

(d) the washed fibrous sheet is dried in step (d).

5. The process for the production of coherent fibrous sheets from fibers consisting essentially of at least partially water soluble and water swellable carboxymethyl cellulose fibers of Claim **3** wherein at least 6.9 parts by weight of isopropyl alcohol per part of water remaining in the washed fibrous sheet is applied to the washed fibrous sheet in the final stage of methyl alcohol displacement washing in step (c) prior to drying.

6. The process for the production of coherent fibrous sheets from fibers consisting essentially of at least partially water soluble and water swellable carboxymethyl cellulose fibers of Claim **3** wherein at least 6.9 to 15 parts by weight of isopropyl alcohol per part of water remaining in the washed fibrous sheet is applied to the washed fibrous sheet in a separate final washing stage in step (c) prior to drying.

7. The process for the production of coherent fibrous sheets from fibers consisting essentially of at least partially water soluble and water swellable carboxymethyl cellulose fibers of Claim **3** wherein drying step (d) is carried out by passing inert gas at about 250° F. through the washed fibrous sheet at the rate of about 80 to 120 ft.^{3}/min./square foot of sheet to be dried for about 4 minutes.

8. A coherent fibrous sheet product formed from an aqueous alcoholic fibrous slurry and consisting essentially of at least partially water soluble and water swellable carboxymethyl cellulose fibers, which coherent sheet product has a comminution quality of about 0.1 to 30.

9. A coherent fibrous sheet product formed from an aqueous alcoholic fibrous slurry and consisting essentially of at least partially water soluble and water swellable carboxymethyl cellulose fibers, which coherent sheet product has a basis weight of about 110 to 250 pounds on a 19 by 24 inch-500 sheet ream weight basis, a thickness of about 0.050 inch to about 0.120 inch and a comminution quality of about 0.1 to 30.

10. The coherent fibrous sheet product of Claim **9** which has a thickness of about 0.060 inch to about 0.080 inch and a comminution quality of about 0.1 to 20.

11. The coherent fibrous sheet product of Claim **9** which has a basis weight of about 210 pounds on a 19 by 24 inch-500 sheet ream weight basis.

12. The coherent fibrous sheet product of Claim **9** which is formed from at least partially water soluble and water swellable sodium carboxymethyl cellulose fibers.

13. The coherent fibrous sheet product of Claim **9** which is formed from at least partially water soluble and water swellable wet epichlorohydrin cross-linked carboxymethyl celluose fibers.

14. The coherent fibrous sheet product of Claim **9** which is formed from self wet cross-linked carboxymethyl cellulose fibers.

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