July 2, 1963 W. U. DAY ETAL 3,096,228

MANUFACTURE OF CELLULOSIC PRODUCT

Filed Jan. 9, 1961 3 Sheets-Sheet 1

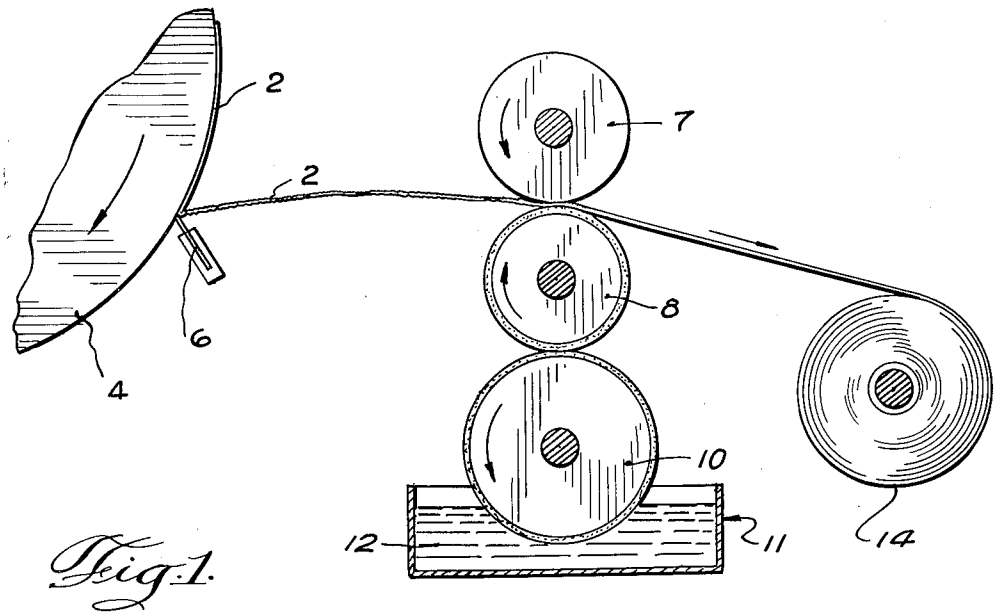
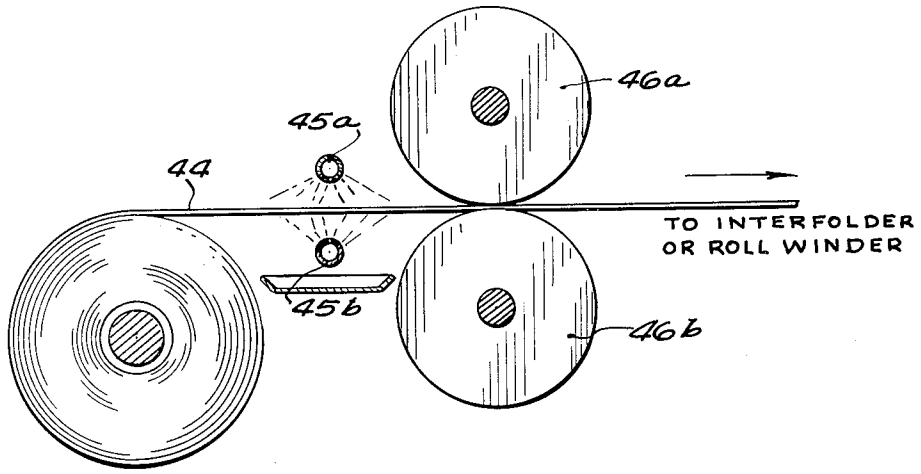


Fig. 1.

Fig. 4.



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MANUFACTURE OF CELLULOSIC PRODUCT

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Fig. 2.

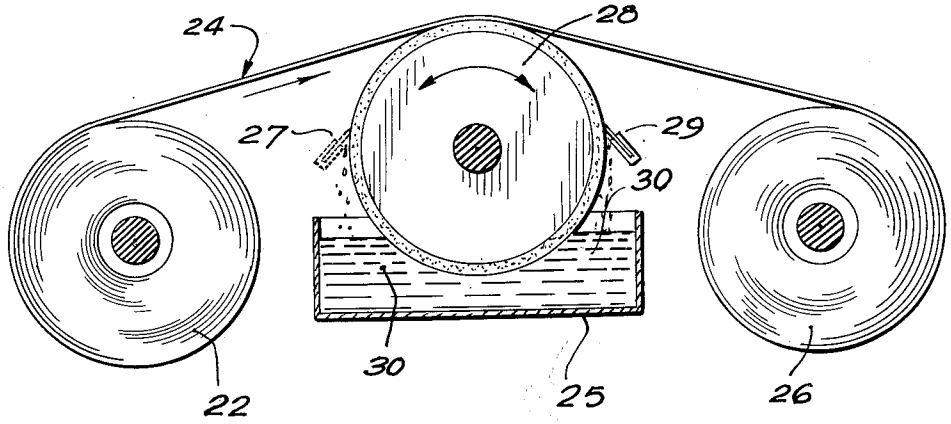
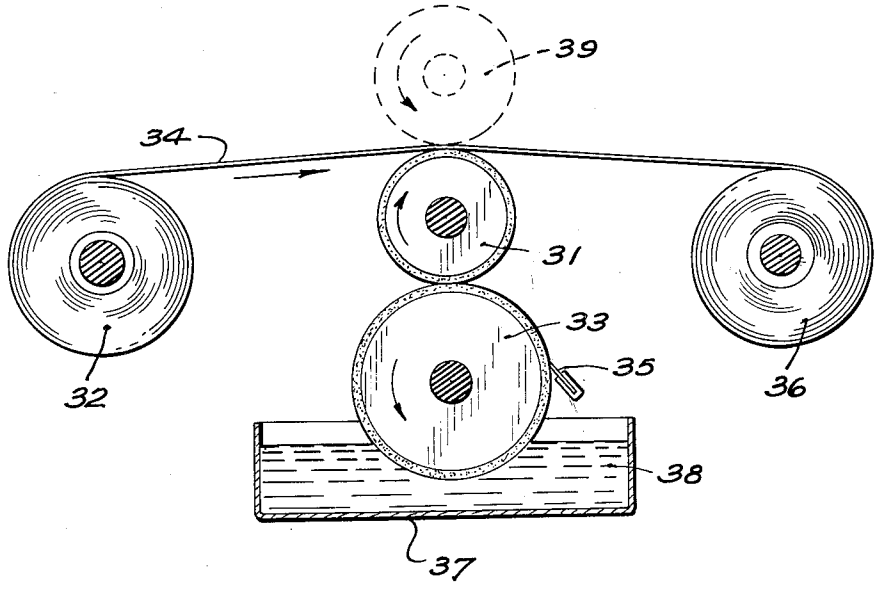


Fig. 3.



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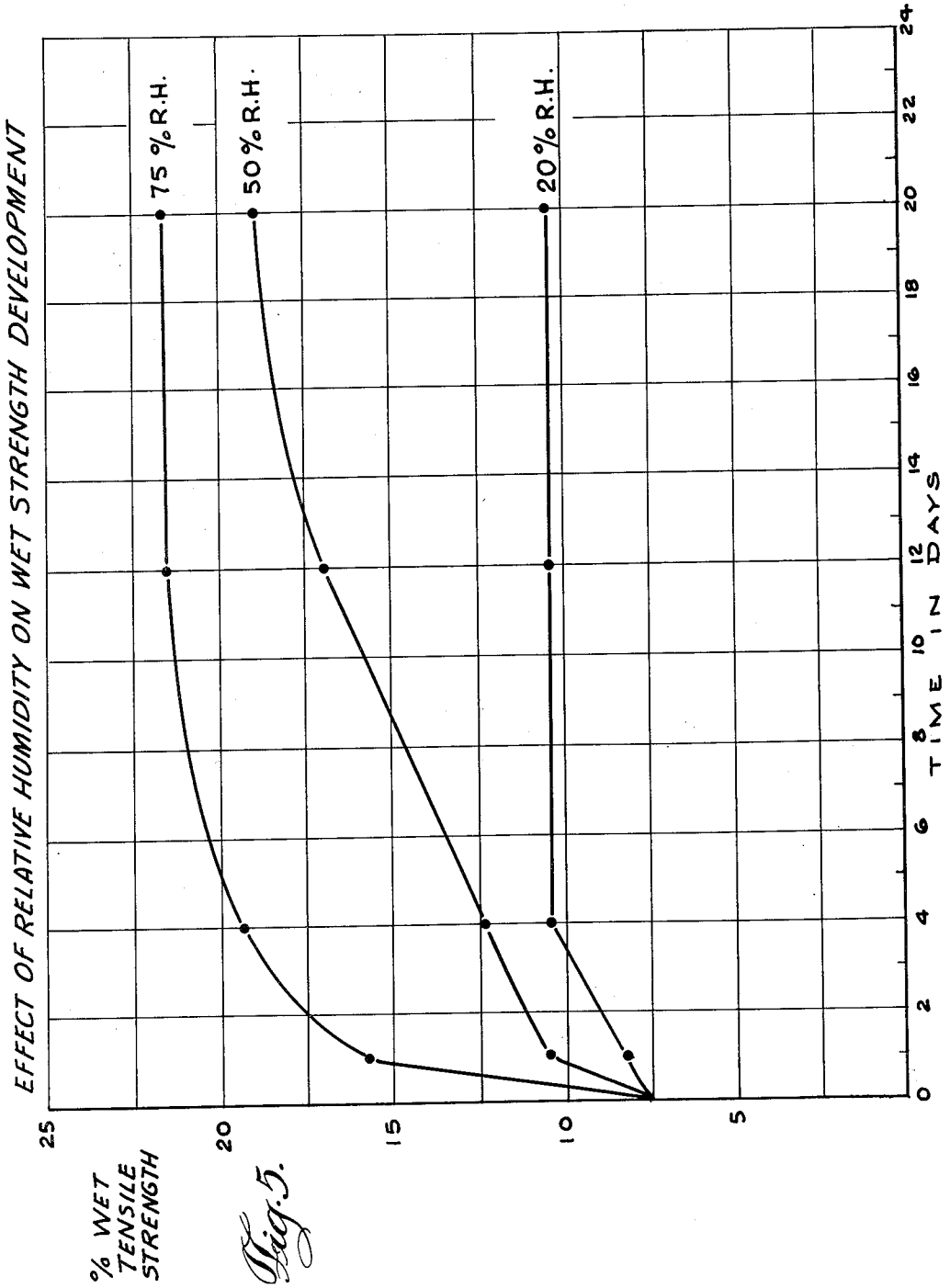


Fig. 5.

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MANUFACTURE OF CELLULOSIC PRODUCT

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This invention relates to a method of treating absorbent paper webs to impart wet strength thereto. The invention is particularly directed to a method for imparting temporary wet strength to absorbent paper webs which are subsequently fabricated into disposable products such as toilet tissure, facial cleansing tissue, absorbent towels, and the like.

A major object of the invention is to provide a method of treating absorbent paper webs with a wet strength agent in a manner to impart temporary wet strength to the treated web, without loss of other desirable physical characteristics of the web.

An equally important object is to provide an improved facial tissue or the like which has the physical characteristic of resisting wet rupture for a short period of time after being wetted, but thereafter rapidly disintegrating to prevent clogging of sanitary disposal systems during passage therethrough.

Other objects and advantages will become readily apparent to persons skilled in the art upon examination of the description and drawings, as will various modifications, without departure from the inventive concepts as defined in the appended claims.

In the drawings:

FIG. 1 is a fragmentary schematic view illustrating a method for applying wet strength material to the paper web, after the web is creped off from the surface of the drier on a creped wadding paper machine.

FIG. 2 is another schematic view illustrating a method for applying wet strength material to a paper web in a converting operation separate from the paper machine.

FIG. 3 illustrates an alternate method of applying the wet strength material in a separate operation.

FIG. 4 illustrates still another method of applying the wet strength material to both sides of a multiply paper web in a converting operation separate from the paper machine.

FIG. 5 is a chart illustrating the effect of relative humidity conditions during aging on the development of the wet strength in treated papers.

While it is well known that absorbent papers may be treated with various agents to impart permanent wet strength properties thereto, no satisfactory treatment has heretofore been developed which imparts relatively transient wet strength properties to the paper while retaining other desirable properties. Ideally, absorbent paper products such as toilet tissue, facial cleansing tissue, and toweling should have some degree of wet strength while retaining the desirable qualities of high absorptive ability and softness, but still must be readily disposable in conventional sanitary systems. Absorbent papers treated by prior art wet strength processes tend to lose some of their original softness and absorbent properties, and do not readily disintegrate in water after use without the aid

of alkali or other chemicals. Thus, they often contribute to, or are the cause of, clogging in wet sanitary systems.

Wet strength generally is desirable in absorbent papers used for sanitary purposes to compensate for an inherent low tensile strength in the cross direction, i.e., transverse of the sheet, resulting from the limitations imposed by high speed forming and subsequent creping. Low cross direction tensile strength is the most common cause for failure when the papers are used in contact with moisture. Machine direction tensile strength in these papers is usually more than adequate, even for wet use. By increasing overall wet strength, the inherent deficiency in cross direction tensile strength is minimized and the potential usefulness of these papers greatly increased.

Thus, it is obvious that wet strengthened papers, in general, have increased functional uses over papers with no wet strength and can be employed where ordinary papers fail. However, when conventional wet strength treated papers are disposed of after use, care must be taken not to deposit them in toilets or other wet sanitary systems because they do not disintegrate readily. Domestic sanitary systems are thus constantly in danger of becoming clogged or stopped-up from careless disposition of wet strength papers. Such stoppages result in considerable inconvenience and expense to the user.

The present invention is directed to eliminating the disadvantages attendant the non-disintegration of ordinary wet strengthened papers after disposal, by treating the paper with a wet strength agent in a manner to insure initial, but short-lived, resistance to wet rupture, without inhibiting subsequent rapid disintegration of the web when immersed in water. The above objective is accomplished by treating absorbent webs with an aqueous solution of glyoxal in a manner to control the amount of moisture added to the web during such treatment and thereafter aging the treated web at non-elevated room temperatures and under controlled humidity conditions to develop wet strength. The wet strength developed in webs thus treated remains adequate to resist wet rupture for a period of about one minute after immersion in water, but thereafter rapidly decays to a degree which permits disintegration of the web, and substantially disappears about 3 minutes after wetting. Thus, the paper treated in accordance with this invention disintegrates quickly in water, similar to absorbent paper products having no wet strength treatment.

While it is known to use glyoxal per se as a wet strengthening agent for paper webs, and in the textile industry for modification of textile properties to improve crease resistance, the known treatments require the application of extrinsic heat, or cooperation with other chemicals, to develop wet strength properties. None of the prior art teachings recognize, however, that by applying aqueous solutions of glyoxal to absorbent papers under carefully controlled conditions of moisture addition and retention, it is possible to develop a highly fugitive wet strength, without employing extrinsic heat, which permits the treated papers to disintegrate rapidly after about one minute of immersion in water, while retaining wet strength properties when slightly moist as a result of use.

For example, it is known that wet strength may be

significantly improved in absorbent paper by saturating a preformed sheet by immersion or spraying thereof with a dilute aqueouse solution containing glyoxal as its sole active ingredient and subsequently heating the treated sheet to a temperature of at least 212° F. for a limited period to react the glyoxal with the cellulose. While this known method results in a high degree of wet strength when employed in the manufacture of heavier absorbent toweling, it has several disadvantages when employed in the manufacture of toilet paper, facial-type tissues, or light weight towels from creped cellulose wadding. Applying aqueous solutions of glyoxal to webs of the latter by saturation followed by drying with heat, as taught in the prior art, tends to embrittle the fibers and also causes an undesirable loss in tear strength of the web rendering subsequent converting steps difficult and expensive. Webs treated thus, break frequently when stretched as required during subsequent conversion, and if stretched to a lesser degree to avoid breaks, result in an undesirably harsh product.

The concept herein taught avoids the prior art disadvantages in part by applying an aqueous solution of glyoxal to a dry web in an amount such that the amount of moisture added to the web during such application is controlled relative to total moisture content, and in part by aging the glyoxal-treated web to develop wet strength at room temperatures and under controlled conditions of moisture content, instead of thereafter subjecting the treated web to elevated temperatures. The absorbent paper, thus treated, is characterized by an acceptable degree of temporary wet strength effective for normal use, while rapidly disintegrating within a few minutes after immersion in ordinary tap water, thus eliminating disposal problems.

Although this process is generally applicable to all types of absorbent paper, it is particularly applicable to creped cellulose wadding such as toilet tissue, facial-type tissue and light weight toweling. These webs are customarily made on Yankee cylinder or Fourdrinier machines, and creped off a Yankee drier at a moisture content of from about 3 to 7% based on the weight of the dry web. Drier basis weight of the web, i.e., basis weight before creping, usually is in the range of 7 to 20 pounds per 3000 square foot ream. The web is creped from the drier to a crepe ratio within the range between about 1.5 and 2.6. A crepe ratio of 1.5 means that one unit length of creped web will pull out to measure 1.5 unit lengths of substantially flat web. Pulps used for forming the web may include the usual bleached and unbleached commercial pulps, such as sulfite, kraft, soda and groundwood.

Generally, the process of the present invention comprises the following steps: A preformed web of "dry" absorbent paper, i.e., having a moisture content of about 3 to 7% by weight based on the weight of the bone dry paper, is treated by applying thereto an aqueous solution of glyoxal in an amount such that the moisture added to the web by the applied solution brings the total moisture content of the web to more than 4% and not more than 20% by weight. The treated web, without intermediate drying, may be wound into large storage rolls for transfer to further converting operations, or alternatively, may be converted and packaged directly after the glyoxal solution is applied. These converting operations usually include interfolding the treated web and inserting the interfolded packs in dispenser cartons, or rolling the treated web into small rolls and wrapping the rolls to obtain packages for commercial distribution. In either case, it is essential to maintain in the web a specified minimum amount of the moisture, after the glyoxal application, in order for wet strength to develop. Usually, the sealed protection offered packaged items by the wrapper or carton under ordinary storage conditions serves to retain the moisture long enough for the full temporary wet strength to develop. In the case where unwrapped rolls are aged, or where abnormally dry conditions exist in storage, it is preferred that relative humidity be controlled at between 50% to 75%, to prevent excessive moisture loss, for at least 24 hours and preferably longer. If the treated web is allowed to dry out under ambient conditions without taking steps to control moisture content, wet strength development is inadequate and not of commercial significance.

The glyoxal used in the process may be any of the commercial or technical grades now available. It is preferred that the purer grades be used. Most commercial grades are supplied in a 30% solution and are strongly acid, having a pH range of 1 to 2. It is preferred to adjust the pH of the solution to between about 4 to 6 with a suitable alkali, such as soda ash, before application to the web. The glyoxal may be applied in the 30% concentration as commercially supplied, or further diluted. No matter what concentration is employed, the amount of moisture added to the web during application of the solution, must be controlled within the range specified.

The ultimate extent of wet strength developed is proportional to the amount of glyoxal present in the web. About 0.3% by weight, based on the bone dry weight of the web, appears to be a practical lower limit and about 5% by weight, an economically feasible upper limit. Amounts in excess of 5% produce additional wet strength but at disproportionate cost.

FIG. 1 illustrates the application of glyoxal to a web at the dry end of a Yankee Fourdrinier or cylinder creped wadding paper machine. The formed tissue web **2** is creped off Yankee drier **4** by doctor blade **6**. An aqueous solution of glyoxal **12**, in supply tray **11** is picked up by roll **10** and applied in a thin film to metering roll **8** which applies a controlled amount of glyoxal solution to the bottom surface of creped web **2**. A pressure roll **7** is lightly pressed against the web **2** as it passes over metering roll **8** to aid penetration of the applied solution into the web. The treated web is then wound into roll **14** which may be stored under controlled humidity conditions, or immediately converted into consumer products. The amount of glyoxal applied is controlled by metering roll **8**, which may be a plain rubber roll, a metal or rubber intaglio roll, or a metal or rubber roll with a textured or patterned surface.

In FIG. 2, web **24**, is unwound from supply roll **22** and passes over patterned applicator roll **28** which dips directly into a glyoxal solution **30** in vat **25** and applies the solution to the underside of web **24**. Applicator roll **28** may turn in either direction depending upon how much solution it is desired to apply to the web. When rotated in the same direction and speed as the moving web, less less solution is applied than when rotated counter to the direction of web movement. Doctor blade **27**, or **29**, depending on direction of rotation of roll **28**, wipes off excess solution leaving a controlled amount to be transferred to the web by applicator roll **28**. The treated web is rolled up into storage roll **26** and allowed to develop wet strength under conditions which substantially maintain in the web the amount of moisture added thereto during the application of the glyoxal solution.

In FIG. 3, web **34** is unwound from supply roll **32** and passes over transfer roll **31** which applies glyoxal solution to the underside of said web **34**. The solution **38** is picked up from vat **37** by metering roll **33** which in this case is of the intaglio type. Excess solution is doctored off by blade **35** and the remaining amount transferred to roll **31** which applies the solution in a controlled amount to the underside of web **34**. Rubber covered pressure roll **39** coacting with transfer roll **31** is optional, but is a useful means for accelerating penetration of the applied solution. The treated web then is maintained under controlled humidity conditions for a period sufficient to permit wet strength to develop in the presence of moisture added on during application of the glyoxal solution.

FIG. 4 shows an alternate method of spray application to a multiply paper web 44 supplied from roll 42. Spray nozzle 45*a* directs a fine patterned spray of minute drops of glyoxal solution onto the top surface of the upper ply while spray nozzle 45*b* directs a similar spray of solution onto the underside of the lower ply. Interacting pressure rolls 46*a* and 46*b* aid in pressing the solution into the web. The treated web then proceeds directly to an interfolder or roll winder, not shown, for conversion into suitable end products, which are stored for at least 24 hours under controlled humidity conditions. It will be understood that other methods of controlled spray application, such as by a brush roll, may also be used. While this embodiment shows spraying the glyoxal solution onto both surfaces of the multiply web, adequate wet strength may be obtained by spraying, or otherwise applying, more solution on only one side thereof.

When the treated sheet is subsequently subjected to a converting operation which requires stretching and ironing, as is done to singly ply bulk rolls obtained from the paper machine, the preferred amount of moisture add-on is about 2%, providing a total moisture content in the sheet of between 5 to 9%. At this rate of moisture add-on, the treated sheet appears to be and feels dry, and tear strength and feel properties, or hand, are not adversely affected.

When the sheet to be treated has already been stretched and ironed, as is customary in the production of absorbent facial-type cleaning and toilet tissue, as much as 13% moisture may be added to the web, but total moisture content in the treated web should not exceed 20%.

In the converting process, two or more plies of the creped tissue as received from the paper machine are supplied from a multi-roll backstand to a conventional stretching and ironing apparatus. The juxtaposed plies are stretched until the crepe ratio is about 1.2 and then ironed between unheated rotating pressure rolls. This stretching and ironing operation contributes to the characteristic softness and cosmetic feel desired in tissues of this type. If total moisture in the treated web is allowed to exceed about 20% there are several undesirable results. Excess moisture tends to release the crepe and results in a weak, undesirably stiff sheet with poor drape characteristics. An excessively wet web also compacts easily, and such compaction adversely affects absorbent properties and desirable softness and feel characteristics. When total moisture is considerably in excess of 20%, the web is weakened in the area of moisture application to a point where frequent and costly breaks occur.

Applying the glyoxal in a manner to control the amount of water simutlaneously applied to the web, and aging at specific moisture content and non-elevated temperature are critical to the invention.

Non-elevated temperatures refer to room temperature, or temperatures normally maintained in manufacturing and storage areas, which may range from about 60° F. to about 80° F.

In the early stages of this development, attempts were made to apply glyoxal to webs without saturation in order to retain softness and feel characteristics, followed immediately by heat treating, which, in accordance with prior art teachings, was believed essential to the development of wet strength. Surprisingly, although the amount of glyoxal retained in the sheet and the temperature used to cure the sheet were in the range taught in the prior art, it was found that little, if any, wet strength developed. It was only when the moisture content of the web, during treatment with glyoxal solution, was controlled at between 4% and 20% by weight, followed by aging at non-elevated temperature while maintaining total moisture content as taught herein, that the desired fugitive wet strength was achieved.

Retention of the moisture added as a result of applying the glyoxal solution aids in uniform migration of the glyoxal throughout the sheet after application. This uniform migration, after controlled application without saturation, is believed important to the development of temporary wet strength while retaining the desirable characteristics of softness and hand.

To determine the effect of migration, a two-ply facial tissue web was treated with a surface application of a 30% glyoxal solution to deposit thereon about 1.5% glyoxal by weight (total moisture add-on of about 5%). Various samples were then: (1) heat dried immediately, (2) air dried overnight, and (3) kept moist in a polyethylene wrapper overnight. Results were as follows:

	Cross Direction Tensile, Dry	CD Tensile, Wet	Percent Wet (Wet/dry×100)
1. Heat Dried	355	44	12.4
2. Air Dried	368	59	16.0
3. Polyethylene wrapped	340	60	17.7

(Tensile strength is given in grams per 3 inches of width. Samples were immersed between blotters in tap water and tested for wet tensile after 45 seconds of soaking.)

The samples in which substantially all of the moisture added during the glyoxal treatment was retained developed the best wet strength overnight while the heat dried sample developed least, and was, in fact, below the minimum cross direction wet tensile strength necessary for it to be classified as a wet strength facial tissue product.

FIG. 5 further demonstrates the effect which aging glyoxal treated webs at controlled humidity and moisture content has on wet strength development. Two-ply facial tisue having an initial moisture content of about 4% was sprayed with a 30% solution of glyoxal adjusted to a pH of about 4.0 with soda ash. Moisture added on by the solution was about 2% by weight, bringing the total content to about 6%. Total glyoxal retained was about 0.65% by weight. Selected samples were aged in storage areas under conditions where relative humidity was controlled at 20%, 50%, and 75% respectively. The samples stored at 20% R.H. (about 4% moisture in air) developed negligible wet strength. Those stored at 50% R.H. (about 6% moisture) showed satisfactory development of about 18% wet strength, while those stored at 75% R.H. (about 10% moisture) showed a more rapid and slightly higher development, leveling off at about 25% wet strength. In each case, the largest increment in wet strength developed in the first 24 hours.

At 20 %relative humidity, the total moisture content of the web dropped to about 4%, indicating that the total moisture content must be maintained in excess of 4% and preferably at 6% or over, but not exceeding 20%, to achieve satisfactory results.

Example

A conventional two-ply facial tissue sheet had glyoxal applied as shown in FIG. 2. The starting web had a moisture content of about 5% and the moisture added during the glyoxal application amounted to about 5% by weight. An aqueous solution containing 30% glyoxal by weight(with pH adjusted to about 4, was used. Thus, the total amount of glyoxal added was about 1.5% by weight and total moisture content of the treated web was about 10%. The treated web was immediately interfolded, inserted in cartons, and aged for 24 hours under humidity conditions which maintained the total moisture content in the web at about 10%. Cross direction tensile strengths were then measured on four random samples with the following results:

Sample No.	Dry Tensile, CD	Orig. Wet Tensile, CD	Wet Tensile after 3 min. of wetting	Percent Wet Strength (Wet/Dry × 100)	Percent Wet Strength after 3 min.
1	332	75	24	22.6	7.2
2	372	94	30	25.3	8.1
3	327	84	26	22.6	8.0
4	355	74	20	20.8	6.0

(Tensile strength given in gm./3" strip.)

Wet tensile of two-ply facial tissue, with no wet strength treatment, is almost negligible and difficult to measure, however, it is estimated to be less than about 20 gm./3" strip, or less than 7% of the original dry tensile strength. Thus, the facial tissue treated in the manner taught herein shows an increase in initial wet strength, followed by a rapid decrease of wet strength 3 minutes after saturation with ordinary tap water. A tissue of this type is strong enough to be "sneeze-proof" or satisfactory for short time use in the presence of water, yet disintegrates readily when disposed of in a wet sanitary system.

Other methods and means of applying the principles of this invention may be used without departing from its spirit or scope as specifically pointed out and described in the above specification and the appended claims.

What is claimed is:

1. The process of producing an absorbent paper having temporary wet strength which comprises providing a substantially dry web of absorbent paper having an initial moisture content of about 3 to 7% by weight, applying to said web an aqueous solution of glyoxal in an amount to obtain a total moisture content in said web after application of more than 4% and not more than 20% by weight of said web, and substantially maintaining said moisture content in said web while aging said web at room temperature to develop wet strength therein.

2. As a new article of manufacture, an improved absorbent paper having temporary wet strength and produced in accordance with the process of claim 1.

3. In a process of producing an absorbent paper having temporary wet strength, the steps of transferring to a substantially dry absorbent paper having an initial moisture content of about 3 to 7% by weight a metered amount of an aqueous solution of glyoxal to obtain a total moisture content in said paper of more than 4% and not more than 20% based on the bone dry weight of said paper, and aging the paper at room temperature for at least 24 hours under conditions wherein relative humidity is controlled at between 50% to 75%, whereby said total moisture content is retained in said paper to develop wet strength therein.

4. The process of producing an absorbent paper having temporary wet strength which comprises forming, drying, and creping a web of absorbent cellulose wadding at a moisture content of from 3 to 7%, applying to said web an aqueous solution of glyoxal in an amount to obtain a total moisture content in said web of more than 4% and not more than 20% by weight, and substantially maintaining said moisture content while aging the treated web at about room temperature to develop wet strength therein.

5. The process of producing an absorbent paper having temporary wet strength which comprises providing a preformed creped web of substantially dry absorbent paper having a drier basis weight of between 7 and 20 pounds per 3000 square feet and an initial moisture content of from 3 to 7% by weight, transferring to said web a metered amount of a solution consisting essentially of glyoxal and water in a manner such that the glyoxal applied to said web amounts to from 0.3% to 5% based on the dry weight of said web while the water applied to said web from said solution provides a total moisture content in said web of more than 4% and not more than 20% by weight, and thereafter aging said web for at least 24 hours at room temperatures and under conditions which maintain said total moisture content in said web.

6. In a method of imparting water-dispersible wet strength to a two-ply web of absorbent facial-type tissue having an initial moisture content of 3 to 7% by weight, the steps of applying to at least one surface of said two-ply web an aqueous solution of glyoxal in an amount to deposit in said web from about 0.3% to 5% glyoxal by weight and enough moisture to bring the total moisture content of said two-ply web to more than 4% and less than 20% by weight, and thereafter aging the treated web for at least 24 hours at room temperature and under conditions which substantially maintain said total moisture content in said treated web.

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