

## UNITED STATES PATENT OFFICE

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## CELLULOSE PRODUCT

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My invention relates to improvements in the manufacture of cellulose products and relates more particularly to absorbent cellulose products such as paper towels, tissue and the like.

It has been found in practice that it is undesirable for absorbent cellulosic sheets to possess too low a wet strength, because, if such is the case, the sheet disintegrates too quickly when moistened. Thus in the case of highly absorbent paper toweling it is found that the sheet pulls apart or crumbles in the hands when used for drying. It has accordingly been the contemplated practice to strengthen the cellulosic sheets by delaying absorbency through the use of materials such as the ordinary rosin-alum size and to counteract the loss of absorbency caused by the aging of the size through the use of wetting agents such as described in U. S. patent to Youtz, No. 2,032,645 of March 3, 1936, and specifically a material known in the trade as "bea-tex," e. g., alkylated, sulphonated naphthalene. It has also been the contemplated practice to strengthen the paper sheets by establishing a water resistant bond between the cellulosic fibers by application of compositions containing gum arabic, viscose, glue-formaldehyde, etc.

It has been found, however, that the presence of the size and suggested compositions, with or without the wetting agents, greatly accelerates the natural aging of the cellulosic sheet and in addition invariably involves an excessive loss of absorbency. Some of the size and suggested compositions also harden the sheet and materially decrease its softness. The paper industry has long sought an absorbent sheet having a satisfactory temporary wet strength.

It is an object of my invention to produce an absorbent cellulose product having a high temporary wet strength that will retain its absorbency.

Other objects will be apparent as the description hereinafter proceeds.

I have discovered a method of preparing absorbent cellulose products having the necessary wet strength that are free from the objections noted above. My products, for example, retain their absorbency on aging and have an extremely high wet strength making them particularly adapted for use as toweling and the like. My products in addition have an increased dry strength.

In accordance with my invention I incorporate locust bean gum with the cellulose fibers. My invention includes the use of locust bean gum with unsized and sized cellulose fibers and has particular reference to the use of locust bean gum which has been hardened after incorporation by treatment with alkaline materials in-

cluding alkali and alkaline earth hydroxides and alkaline salts, as well as certain organic compounds having an alkaline reaction.

The preferred method of preparing locust bean gum for use in my invention is to soak locust bean gum powder, e. g., 100 mesh, in cold water for at least 5 minutes and then heat it to a temperature of about 70° to 100° C. so as to cause rapid hydration and gelatinization of the gum. The preferred concentration of the gum ranges from about 1 to 2 percent, at which consistency it forms a viscous paste-like dispersion in the water.

The gum dispersion should be added to the pulp furnish either at the beater or the head box or at some convenient place between these two positions. To insure complete dispersion of the gum in the furnish, however, the gum should be added to the beaten pulp before sheet formation is completed on the wire.

The amount of gum added to the furnish will depend upon the results desired. I have found for example that 1 percent of gum based on the dry weight of the furnish is a satisfactory amount in the manufacture of paper toweling. The use of larger percentages and particularly over 5 percent while showing a somewhat higher wet strength, has the disadvantage in the case of paper toweling of slightly increasing the stiffness of the sheet. On the other hand, 0.5 percent concentration has been found to give a somewhat smaller effect and my results indicate that as we go below this amount the effect will be correspondingly lessened and be in most instances of little practical value.

In my preferred method I add to the cellulosic sheet containing the locust bean gum a small amount of alkaline material that will react with the gum in the sheet in such a way as to harden the gum or "develop" or "set up" the wet strength. The alkaline material may comprise alkali and alkaline earth hydroxides and inorganic alkaline salts, i. e., salts having a basic reaction in water solution, or an alkaline organic material such as triethanolamine, and may be applied to the sheet during its formation, for example, on the wire of a Fourdrinier machine or subsequently in the paper making process. A water solution of the alkaline material is generally employed in order to insure a thorough and even distribution throughout the sheet.

I have found in practice that the application of the alkaline "fixing solutions" may be easily accomplished by means of a conventional regulated spray system and that the point of spray, as indicated above, is not a critical factor. Excellent results have been obtained, for example,

by application on the Fourdrinier wire, on the pick-up felt and also after the creping doctor. I have also applied the alkaline fixing solutions to the sheet at the press roll, the solution being poured into the trough made by the angled position of the scraping doctor blade as it presses against the roll, the revolving motion of the roll carrying the solution into the sheet. The above examples illustrate the addition of the fixing solution to the sheet. Other methods of adding the solution, such as at the size press, etc., will readily suggest themselves to those skilled in the art.

The amount of alkaline fixing chemical required to develop the maximum wet strength varies, of course, with the particular chemical used and with the amount of locust bean gum employed. The rate of spray or other method of application and the concentration of the aqueous solution may easily be regulated so as to give the desired or optimum wet strength. When a strong alkaline material, such as caustic soda, is employed, a 0.25 percent solution ordinarily produces a satisfactory wet strength. The use of large quantities of strong alkalis has been found undesirable particularly when the paper is to be used for toilet purposes. In addition, a high percentage of strong alkalis should be avoided as they often discolor the cellulosic sheet making it commercially unsatisfactory.

When an alkaline salt such as borax or sodium tetraborate, the preferred alkaline fixing agent, is used, the concentration based on a dry basis in the sheet should ordinarily not fall below 1 percent as quantities less than this amount will not produce the most effective response on the locust bean gum. Concentrations somewhat higher than 1 percent, i. e. 2 to 4 percent, are desired in practice as they allow a factor of safety. A concentration of borax over 4 percent on the dry weight of the pulp on the other hand has been found of no practical advantage as the larger percentages do not give added wet strength when the usual amount of locust bean gum is employed.

The following examples, all of which are aqueous solutions, will serve to illustrate the preferred alkaline materials that may be used to harden the locust bean gum in accordance with my invention.

- 5 percent borax ( $\text{Na}_2\text{B}_4\text{O}_7$ )
- 10 percent borax
- 20 percent borax
- 10 percent borax and 10 percent tri-sodium phosphate
- 10 percent borax in saturated lime water
- 10 percent borax and 0.25 percent caustic soda
- 10 percent borax and 2.5 percent rosin size
- 10 percent borax and 5 percent triethanolamine.

The following examples of paper made with and without the addition of locust bean gum will serve for illustrative purposes.

#### EXAMPLE I

Furnish—

	Pounds
Unbleached sulphite pulp, spruce and hemlock	1000
Unbleached sulphite pulp, poplar	600
Ground wood	400
Locust bean gum	20

The locust bean gum used in the above furnish is stirred in about 25 gallons of cold water and then diluted with water to about 120 gal-

lons. Steam is next passed into the gum-water mixture until a temperature of approximately 80° C. is attained. The gump dispersion is then added to the pulp furnish in the beater and immediately after the formation of the paper sheet in the usual manner on the Fourdrinier wire, it is sprayed with a 10 percent solution of borax made up in saturated lime water. Tests on the finished paper gave the following results.

Table I

Absorbency (0.1 c. c. water)----- 24.4 seconds.

	Machine direction	Cross direction
Wet tensile (Schopper tester)-----	4.33 units (433 grams)	2.07 units (207 grams)

#### EXAMPLE II

Furnish—

	Pounds
Unbleached sulphite pulp, spruce and hemlock	1000
Unbleached sulphite pulp, poplar	600
Ground wood	400
Alum	10
Rosin size	2
Beatex wetting agent	1

Regular paper toweling made from the above furnish containing rosin-alum size but containing no locust bean gum gave the following results.

Table II

Absorbency (0.1 c. c. water)----- 25.0 seconds.

	Machine direction	Cross direction
Wet tensile (Schopper tester)-----	0.63 unit (63 grams)	0.35 unit (35 grams)

The tables under the above examples give the results of tests carried out on my improved sheets containing locust bean gum and the results of tests carried out on the ordinary sheet containing rosin-alum size. The treated stock containing locust bean gum shows a wet tensile strength of 4.33 units in the machine direction as compared to 0.63 unit for the untreated stock containing no locust bean gum. This is an improvement of about 587 percent. In the cross direction the wet tensile strength goes from 0.35 unit for the untreated stock to 2.07 units for the treated stock, which represents a 462 percent improvement in this direction.

The following additional examples, all employing the same pulp furnish, will serve to illustrate the improved absorbent and wet tensile characteristics of paper sheets containing locust bean gum in accordance with my invention. In Example III the furnish contains rosin-alum size and a wetting agent (beatex), but does not contain any locust bean gum; in Example IV the furnish contains locust bean gum, but the sheets made therefrom were not treated with an alkaline hardening agent; and in Example V the furnish contains locust bean gum and the sheets made therefrom were subjected to an alkaline fixing reagent. The results of the tests carried out on paper sheets are given in the tables under the examples.

## EXAMPLE III

Table V

Furnish—

	Pounds
Unbleached spruce.....	1100
Unbleached hemlock.....	300
Unbleached poplar.....	200
Groundwood.....	200
Broke (paper trimmings).....	200
Alum.....	10
Rosin size.....	2
Beatex wetting agent.....	1

Paper sheets made up from this furnish in the usual manner gave the following results.

Table III

	Before aging	After aging
	Seconds	Seconds
Absorbency (0.1 c. c. of water).....	40	54.6
	Machine direction	Cross direction
	Units	Units
Wet tensile (Schopper test).....	0.74	0.61

## EXAMPLE IV

Furnish—

	Pounds
Unbleached spruce.....	1100
Unbleached hemlock.....	300
Unbleached poplar.....	200
Groundwood.....	200
Broke (paper trimmings).....	200
Locust bean gum.....	20

Paper sheets made up from this furnish in the usual manner, the locust bean gum added as described in Example I, gave the following results.

Table IV

	Before aging	After aging
	Seconds	Seconds
Absorbency (0.1 c. c. of water).....	28	36.2
	Machine direction	Cross direction
	Units	Units
Wet tensile (Schopper test).....	1.57	1.07

## EXAMPLE V

Furnish—

	Pounds
Unbleached spruce.....	1100
Unbleached hemlock.....	300
Unbleached poplar.....	200
Groundwood.....	200
Broke (paper trimmings).....	200
Locust bean gum.....	20

Paper sheets made up from this furnish in the usual manner, the sheets being subjected to a lime water spray containing 10 percent borax as described in Example I, gave the following results.

	Before aging	After aging
	Seconds	Seconds
Absorbency (0.1 c. c. of water).....	18.9	32.2
	Machine direction	Cross direction
	Units	Units
Wet tensile (Schopper test).....	5.31	3.39

15 From a study of the above tables it will be seen that the sheet containing no locust bean gum has a low absorbency and low wet tensile strength, that the sheet containing locust bean gum not treated with an alkaline agent possesses a higher absorbency and a higher wet strength and that the sheet containing locust bean gum treated with an alkaline hardening agent shows a still higher absorbency and an extremely high wet tensile strength. Similarly, the tables show that the sheets containing locust bean gum retain their absorbency after aging much better than the rosin-alum sheet even with the addition of the age resisting wetting agent.

30 The chemical action which takes place when the locust bean gum is treated with the alkaline fixing material is not known. Results have shown, however, that the gum appears to harden or develop resulting in an enormous increase in the wet tensile strength.

35 It will be understood by those skilled in the art that my invention is not limited to the specific proportions of locust bean gum employed in the above examples. The optimum amount of locust bean gum depending on the characteristics desired in the finished sheet may be found by carrying out simple experimental tests. It will also be understood by those skilled in the art that other fixing agents or alkalis and alkaline earth hydroxides such as potassium hydroxide, calcium hydroxide, barium hydroxide, and other alkaline salts such as sodium carbonate, sodium bicarbonate, trisodium phosphate, and organic compounds of an alkaline nature such as the amines, may be used in place of the preferred alkaline materials without departing from the spirit of my invention.

45 My improved paper is, of course, prepared in accordance with the standard paper making process and a detailed discussion is not deemed necessary for a clear understanding of my invention. The aging test used comprises heating the specimen sheet for about one hour at 100° C. and measuring the changes in absorbency due to heating. It is considered, in the paper industry, that this artificial aging test is the equivalent of about one year of natural aging. The other tests carried out on the various paper sheets, the results of which are given in the above tables, were also made in accordance with standard paper testing practices and are familiar to those skilled in the art.

70 The tree from which the locust bean is taken has the botanical name "*Ceratonia siliqua*" and is a tree of the Mediterranean region having ever-green pinnate leaves and apetalous flowers in small red racemes. This tree is also known by the name of Carob tree and has long pods containing seeds which are sometimes called St. John's bread. It is these seeds from which the

gum powder used in the process and product of the present application is obtained.

Various modifications and variations of my invention may suggest themselves to those skilled in the art. Hence, I do not wish to be limited to the specific embodiments disclosed herein, but intend that the scope of my invention be determined from the appended claims which are to be interpreted as broadly as the state of the art will permit.

I claim:

1. The steps in the method of preparing an improved absorbent paper sheet product of high temporary wet strength, increased dry strength and substantially free from age hardening and particularly adaptable for use as a paper towel, which comprises adding an aqueous dispersion consisting of gelatinized locust bean gum to a paper making furnish consisting essentially of wood cellulose fibers and forming the paper sheet containing locust bean gum dispersed therein from said furnish, the locust bean gum being present in amounts between 0.5 to 5.0 percent based on the dry weight of the furnish.

2. An absorbent paper sheet product adaptable for use as a paper towel, consisting essentially of sulphite pulp and ground wood fibers having dispersed, distributed and incorporated therein to produce high absorptive power an aqueous dispersion consisting of locust bean gum, said locust bean gum being present in amounts between approximately 0.5 to 5.0 percent based on the dry weight of the fiber, said product having a high temporary wet strength and being substantially free from age hardening.

3. An absorbent paper sheet adaptable for use as a paper towel, consisting essentially of sulphite pulp and ground wood fibers having dispersed, distributed and incorporated therein to produce high absorptive power, the reaction mixture of a locust bean gum dispersion and an alkaline treatment agent, said gum being present in amounts of about 0.5 to 5.0 percent based on the dry weight of the fiber, said sheet having a high temporary

wet strength and being substantially free from age hardening.

4. The steps in the method of preparing an improved absorbent paper sheet product of high temporary wet strength, increased dry strength and substantially free from age hardening and particularly adaptable for use as a paper towel, which comprises adding an aqueous dispersion consisting of gelatinized locust bean gum to a paper making furnish consisting essentially of sulphite pulp and ground wood fibers and forming the paper sheet containing locust bean gum dispersed therein from said furnish, the locust bean gum being present in amounts around 1 per cent based on the dry weight of the furnish.

5. The steps in the method of preparing an improved absorbent paper sheet product of high temporary wet strength, increased dry strength and substantially free from age hardening and particularly adaptable for use as paper toweling, which comprises adding an aqueous dispersion consisting of gelatinized locust bean gum to a paper making furnish consisting essentially of wood cellulose fibers, forming a paper sheet from said furnish and hardening the locust bean gum dispersed in said sheet by treating the sheet with a dilute alkaline solution, the locust bean gum being present in amounts between 0.5 to 5.0 per cent based on the dry weight of the furnish.

6. The steps in the method of preparing an improved absorbent paper sheet product of high temporary wet strength, increased dry strength and substantially free from age hardening and particularly adaptable for use as paper toweling, which comprises adding an aqueous dispersion consisting of gelatinized locust bean gum to a paper making furnish consisting essentially of sulphite pulp and ground wood fibers, forming a paper sheet from said furnish and hardening the locust bean gum dispersed in said sheet by treating the sheet with a dilute alkaline solution, the locust bean gum being present in amounts around 1 per cent based on the dry weight of the furnish.

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