Patented Oct. 4, 1932 1,881,419

UNITED STATES PATENT OFFICE

TREADWAY B. MUNROE AND FERGUS A. IRVINE, OF CHICAGO, ILLINOIS, ASSIGNORS TO THE CELOTEX COMPANY, OF CHICAGO, ILLINOIS, A CORPORATION OF DELAWARE

FIBER DERIVATIVE

No Drawing. Application filed June 29, 1929. Serial No. 374,944.

This invention relates to fiber derivatives and has for its object the production of a new product, fibrillæ, which will be of great value in the paper and related industries and the utilization of which will, it is believed, mark a revolutionary advance in certain phases of such industries.

With this and other objects in view the invention consists in the novel product and the steps and combinations of steps constituting the method of production of the fibrillæ as will be more fully hereinafter disclosed and particularly pointed out in the claims.

In order that the improvements and advantages of the invention may be understood a discussion of present and past practices of fiber refining will be given as follows, whereby this invention and the product thereof may be readily distinguished from certain seemingly analogous processes and products thereof as known in the industry. In the paper and board making arts the fibrous constituents employed comprise primarily wood which has been broken down into its individual fibers or into small fiber bundles comprising several unseparated fibers.

Inspection of the article "Esparto as a paper-making material", Paper Trade Journal of September 17, 1925, covering work done at the United States Bureau of Standards, shows that the average fiber diameter for nine fibrous materials used in the paper industry range from .030 m. m. to .012 m. m. with an average for the nine of .017 m. m. or .0007 inch. The paper maker works with these fibers, having a diameter of about .0007 inch whereas the board manufacturer usually works with material comprising such individual fibers admixed with fiber bundles, that is to say with unseparated groups of a small number of fiber bundles. The paper maker starts with fiber which has been subjected to a soda sulphate, sulphite, or other type of chemical digestion. The purpose of the digestion is to remove unwanted substances, lignin, mineral ash and the like, and to make the fibers flexible and to soften them. The fibers are not particularly separated by the digestion as a result of which fiber groups or fiber bundles are the usual product thereof. The paper makers problem is the formation of a satisfactory sheet from the fibers. For sheeting into paper the fibers are refined to the degree which experience indicates as necessary for production of the desired paper and these refined fibers are formed into a paper sheet.

Refining is almost universally carried out in a Hollander or other beater used usually in conjunction with a Claflin, Jordon, Bauer or some similar machine. The fiber is acted upon in these refiners, in fluid suspension, to open it out, to hydrate it, and at times to cut it and thus shorten the fiber. Brushing or combing out of fiber is the procedure accomplished primarily in the beater whereby individual fibers are separated one from another and fiber bundles are broken up. The beater man understands the proper beater setting for such action and can brush or open out the fiber with little or no cutting or hydration, but anything more than the lightest brushing out entails hydration of the fiber as a characteristic action and hydration of the fiber absorbs a large amount of work. Hydration of fiber has been very important in the manufacture of paper as it is by the use of properly hydrated stock that the finer and thinner papers are made. Fiber to felt properly, particularly for a thin sheet, must be well hydrated whereby the fibers are refined to a condition in which they properly form a sheet and adhere to one another.

There is apparently considerable confusion and uncertainty with regard to hydration of paper fiber but there seems to be a decided weight of opinion and conclusion that hydration takes place as follows. Mechanical hydration is generally considered to be an action whereby through a beating operation water is forced into the fiber accompanied by a gelatinizing of the surface of the fiber. By many mechanical hydration is considered to be a chemical action through which water and cellulose combine to form gelatinous hydrocellulose. It is at least known and evident, as the result of prolonged beating properly controlled, that the individual fibers swell considerably, take up water by chemical combination, adsorption, or in some other manner

and that the surface is gelatinized. This gelatinized fiber surface has been considered indispensable by the paper maker and as that which, on drying, cements together the fibers of the paper sheet to obtain strength thereof.

In the past it has been well known that cellulose fibers if sufficiently disintegrated and hydrated by mechanical action, could be brought to the form of a slimy gelatinous mass in which the fibrous structure has entirely disappeared. Such material formed into a mass and dried shrinks enormously and forms a dense, hard, bone or horn like substance of considerable strength. According to the prior patent art buttons and like articles have been made from dried masses of this gelatinous cellulose product. As distinguished from the prior known cellulose products above described, hydrated cellulose and gelatinized cellulose, the product of this invention is not merely a fractured gelatinized part of a fiber, nor is it a hydrated fiber. It is a product of the separation of a fiber into its constituent parts, designated fibrillæ. It has been known that some if not all wood fibers are composed of a plurality of distinct and smaller elements bound into a whole. These fiber elements have been designated as fibrillæ which, when viewed under the microscope, have an appearance similar to that of fiber as viewed by the unaided eye except having even a greater ratio of length to cross section.

Investigation of the structure of fibers has disclosed that each fiber is composed of a large number of fibrillæ of dimensions approximately one tenth to one twentieth the diameter and length dimensions of the fiber which they comprise. According to the apparent dimensions of the fibrillæ it is evident that each average fiber is composed of some 1,500 to 3,000 or more fibrillæ. It will be realized that the fibrillæ resulting from the separation of a fiber into such elements will present surfaces totaling much more in area than the superficial surface of the fiber which they originally formed, and that such fibrillæ due to their thinness will be, relatively, extremely soft and flexible and long in comparison to their diameter. Due to the facts just stated the fibrillæ will mat and felt very thoroughly and a sheet made therefrom will be dense and have a high degree of strength.

For preparation of fibrillated stock, fiber digested by any of the known chemical cooks apparently is satisfactory, for instance kraft pulp has been found to be quite satisfactory. Kraft is produced primarily from spruce and Southern pine by the regular kraft digestion, as is well known, which digestion removes substantially all the lignin, resins, gums, minerals and the like leaving only relatively pure cellulose, an essential for the practice of this invention. For fibrillation the kraft should preferably be obtained as a wet lap since it seems that after the fiber has once dried it cannot be brought back to its former state by water saturation. Some constitutional change of the fiber apparently takes place on drying whereby fiber once dried cannot be fibrillated, or this can be only incompletely accomplished and with difficulty. Wet kraft lap is the preferred raw material for the processes of this invention, but it is to be understood that more or less satisfactory results may be obtained with pulps prepared other than by a kraft digestion. Apparently the digestion producing the pulp must be such that a relative pure cellulose pulp is produced without appreciable weakening of the fiber in the chemical processes of digestion.

It has been determined that fibers are composed of concentric sheaths and in fact such sheaths have been separated by appropriate chemical manipulation. Each of the individual sheaths of the fiber is composed of many minutes fibrillæ and it appears that the fibrillæ in each sheath lie in somewhat different arrangement. In general those of the outside sheath extend in a direction more or less circumferential of the fiber whereas those of the inner sheaths are arranged more or less in spiral arrangement relative to the fiber axis, in some cases being arranged substantially parallel with the axis.

On consideration of the arrangement of the fibrillæ of fibers it was considered that, could these fibrillæ somehow be unwrapped or untwisted and separated without injury thereto, they would be extremely valuable in the manufacture of articles made from fibers. In other words, due to their fineness they would felt and intertangle to an extent not hitherto thought of and in fact impossible to conceive of in the consideration of ordinary paper fiber. As the result of investigations brought about in view of this possibility the following method of separating fibers into constituent fibrillæ has been discovered, which method is simple in operation and which produces from the fibers a mass of substantially uninjured fibrillæ.

The desired result is achieved through mechanically working the fibers between plate surfaces which revolve, one relative to another, at a high speed, said surfaces being set up practically in contact. The thoroughly saturated fiber, or fiber which has been digested as previously described, is fed between the relatively revolving plates and subjected therebetween to a rolling and twisting action. This action, to which the fiber is subjected with attenuated heavy pressure applied to the plates, apparently loosens the fiber structure and untwists or unwinds it whereby, probably, the sheaths comprising the fiber are successively stripped therefrom thus liberating the individual fibrillæ. The action on the fiber, when thus subjected to the relatively revolving plates, may be considered as some-

what analogous to the action (upon a relatively fixed roll of paper) such as would result from applying to the surface of said roll oppositely revolving rolls with their axes parallel and parallel to the paper roll axis. The paper would be rapidly unwound from such roll and, if in layers thereon, would be rapidly removed in successive layers.

Suppose the revolving rolls just mentioned are replaced by tapered rolls in contact with the paper roll and are forced thereagainst with very heavy pressure. In such case not only will the rolls tend to strip paper from the paper roll but each roller will exert a tension on the paper tending to tear it along lines at right angles to the axis of the paper roll. This action as just described is just about that which acts to separate fibers into fibrillæ when subjected under pressure to a rolling and twisting action between closely adjacent relatively revolving plates.

So far as has been discovered up to the present time fibrillation can best be accomplished in a Bauer refiner. The Bauer is a commercially available machine which comprises two parallel plates which relatively revolve, the plates having on their faces slightly raised tooth-like projections. These projections do not appear to be necessary to fibrillation and most probably plain surfaced plates would be at least equally and probably more satisfactory. The distance between the plates is adjustable and they have a slight taper towards their centers whereby they approach closer together toward their edges.

According to this invention the material is supplied to the refiner from wet laps, broken up or shredded, with a small stream of water just sufficient to float it from the feed opening to the plates. As an alternative to the use of shredded wet laps a fluid suspension of the material may be fed to the machine, but such suspension should be of the highest density possible to handle since, apparently to prevent cutting of the material, it should be refined with a minimum of fluid.

The plates of the refiner are set up with practically metal to metal contact whereby it is possible to strip apart the sheaths of the fibers being refined and to separate these sheaths into their constituent fibrillæ. A finer operating at 1200 R. P. M., or relatively at 2400 R. P. M., has been found satisfactory. It is not to be inferred that fibrillation can not be accomplished at other speeds, the previous statement covering only what has, up to the present time, been apparently the most satisfactory speed for the most complete fibrillation relative to power consumed.

Whereas it has been stated that the stock fed to the refiner should have the minimum possible amount of water, it is questioned whether such is an absolute requirement. Available machines are provided with spring pressure relief means whereby, should a hard foreign substance be inadvertently fed to the refiner, one of the plates can give slightly to allow such substance to pass through without wrecking the machine. It is thought that when an excess of water, as in a fluid suspension of less than about 5%, is fed through the refiner the water is present in sufficient quantity to slightly open up the plates whereby the proper rolling and twisting action is not imparted to the material being refined. It is believed and indicated that, with a machine having a very heavy and rigid frame and without the plate relief means, the desired fibrillation may be secured regardless of the fiber concentration of the fluid suspension fed thereto.

Whereas certain reasons have been advanced to account for the results obtained by this invention it is to be distinctly understood that the theories set out are statements made only on belief and opinion and that such theories are not advanced as hard and fast, and necessarily accurate causes of the results produced. The refining is carried on in a massive and totally enclosed refining machine, the raw material is treated as a mass, and the product comprises innumerable and minutely subdivided material, all to the end that the individual processes and forces acting to produce fibrillation of one fiber have been impossible of accurate quantitative and qualitative analysis.

A particular advantage of the product of this invention lies in the fact that there is produced the fibrillæ, unhydrated or at least substantially so, which fibrillæ constitute such minutely fine subdivisions of the original fibers, yet with relatively great length in proportion to section, that they interangle and interlock to a degree heretofore unknown in the paper art.

This product, even though so finely subdivided, is found not to be in the least hydrated, or at least for all practical purposes to be not hydrated, and it is therefore apparent that the work expended in its refining is utilized in the mechanical work of separating the fibrillæ and therefore is not dissipated in hydrating the fiber.

Regardless of the theories advanced and reasons given, it is evident that by this invention there has been created a product which, in the foregoing description, has been differentiated from prior products and with respect to which full details of production have been adequately set forth. This fibrillated vegetative product will have many uses in, and will be of great benefit to, the paper and related industries. In fact it is now known that, by the intelligent utilization of this fibrillated product, present products of the paper industry may be improved and that new and desirable articles and products may be produced.

What is claimed is:—

1. A product derived from wood fibers, the major portion of the fibers mechanically disintegrated into their fibrillæ constituents.

2. A product mechanically derived from digested wood fibers the major portion whereof is resolved into its fibrillæ constituents.

3. A product mechanically derived from wood fibers and composed of relatively minute subdivisions of the fibers, the subdivision comprising separation of distinct elements of the fiber as distinguished from subdivision of the fiber by fracture.

4. A product mechanically derived from wood fiber, such product composed primarily of unfractured subdivisions of individual fibers.

5. A product mechanically derived from wood fiber, such product composed primarily of unfractured subdivisions of individual fibers the subdivisions of the individual fibers comprising fibrillæ which are in size about one one-thousandths part of an average fiber from which derived.

6. A product prepared from wood fibers mechanically disintegrated to a composition comprising a major portion of unfractured fibrillæ together with a minor portion composed of fractured and incompletely fibrillated fibers.

7. A product derived from chemically digested vegetable fibers and composed of relatively minute subdivisions of the fibers, the subdivision comprising separation of distinct elements of the fiber accomplished substantially instantaneously through a rolling action imparted to the fiber at high speed while under heavy pressure.

8. The method of disintegration of digested fiber into separated fibrillæ through a twisting and rolling action under heavy pressure, the fibers being delivered for disintegration in a fluid solution having a fiber content greater than 5%.

9. The method of fibrillating fiber wherein fibrous material, suitably digested for thorough softening and removal of substantially all its mineral and lignin content, is separated into fiber elements and fiber bundles and fed, in fluid suspension containing over 5% fiber content, to and between relatively moving surfaces whereby it is subjected under pressure to a combination of twisting and rolling actions, the fiber sheath thus laid open and separated into individual fibrillæ elements through forces acting in compression and longitudinal shear whereby there are produced unfractured fibrillæ as distinguished from fiber produced by mere fracture and breaking up thereof.

10. The method of producing fibrillæ constituents of fiber wherein chemically digested fiber, while under heavy pressure is rolled under the action of relatively revolving plates travelling at a relative speed in excess of 88 feet per second.

11. A new article of manufacture comprising a chemically digested pulp mechanically disintegrated into its fibrillæ elements.

12. An article of manufacture comprising a kraft cooked pulp mechanically disintegrated into its constituent elements through subjection to a combined twisting and rolling action while subjected to heavy pressure.

13. An article of manufacture comprising a chemically digested pulp disintegrated into its constituent elements by passage through a Bauer type refiner in which the plates are in substantially face to face contact.

14. An article of manufacture comprising a chemically digested pulp disintegrated into its constituent elements by passage through a refining machine wherein the pulp is disintegrated between the faces of relatively revolving plates arranged in substantially face to face contact.

In testimony whereof we affix our signatures.

TREADWAY B. MUNROE.
FERGUS A. IRVINE.