

UNITED STATES PATENT OFFICE

2,156,309

METHOD OF PRODUCING COMPRESSED PRODUCTS

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No Drawing. Application October 25, 1935, Serial No. 46,742. Renewed July 11, 1938

12 Claims. (Cl. 18—48)

This invention relates to compressed products and a method of producing the same, and more particularly to a new method for the production of light weight non-metallic products and to the products resulting therefrom.

Prior rigid thermal insulating materials may be divided into three classes, to wit:— (1) the porous type, such as blown molten limestone commercially known as "Rock cork", which is characterized by the presence of large visible voids, (2) the various fibre boards composed of large coarse wood, cane, and other fibre or cellulosic material matted and pressed together with or without binder, and (3) corkboard, either in its natural form or made from granulated cork baked under pressure with or without binder. Natural cork being the bark of a tree is obtained in sheets of limited thickness and of non-uniform size and shape and is an expensive product. It is therefore not used in the natural state to any extent for insulating board. Instead the trimmings from various trades employing natural cork and as may be otherwise obtained are granulated and this granulated cork is formed into boards usually one foot by three feet by two inches in thickness. It is to be noted that the granulated cork employed for this purpose consists largely of granules of considerable size, much of it being coarser than ten mesh. These granules may be formed into corkboard by mixing with an asphalt binder and pressing into shape. The use of a binder, however, greatly decreases the insulating efficiency and also increases the weight considerably. The discovery that these granules can be united into a board by simply baking under pressure was a very important invention commercially and practically all corkboard has been made by this process. The product, however, has been expensive.

In preparing cork for various uses in the trade, the exterior rough surface of the bark is ground off and in so doing a fine, reddish brown powder is obtained which naturally contains a small amount of impurities such as sand, etc. This is practically a waste product and can be purchased today at a very low price per ton. Heretofore, there has been no successful attempt to produce an insulating board from any such material as this. The general problem of producing a board, and particularly one of light weight, from a fine powdered material presents various serious obstacles. Although many attempts have been made to improve these materials, particularly those of group (3), none, as far as I am

aware, has been wholly satisfactory, practical and successful when carried into practice on an industrial scale.

I have discovered that it is possible to produce a desirable board or sheet of material from a fine powdered material without the use of any so-called binder material or with the use of such a small percentage of same as to make its presence entirely unobjectionable from the standpoint of increase in density, increase in thermal conductivity, and even increase in cost. To attain this object, I employ a process which is based upon a fundamentally new concept in the procedure for the production of this type of product.

The object of the invention is to obtain a non-metallic material of super light weight which has valuable thermal and acoustical properties.

A further object of the invention is to obtain a highly efficient insulating material of light weight and low cost.

It is also within the contemplation of the invention to obtain an efficient insulating material of light weight which has a fine uniform structure and is free from large visible voids and large coarse fibre such as occur in prior porous insulating materials and fibre board.

The invention likewise contemplates the provision of utilizing in the production of light weight products raw materials which are now practically waste products and very low in cost.

Other objects will become apparent from the following description of preferred embodiments of the invention.

In carrying my invention into practice, I prefer to employ a very dilute solution or dispersion containing (1) a binding agent, such as for example, a ½% latex or a ½% aqueous resinous dispersion, (2) a filter aid, and (3) what I call a medium of extension. The general procedure is to intimately mix these three fundamental components to form a dilute aqueous suspension, filter the same without disturbing the matrix as it is formed, then gradually subject the matrix to a predetermined pressure without breaking its continuous bonded structure, and finally dry the matrix. The operations are simple and easily carried into practice and are adaptable to large scale volume production resulting in low manufacturing cost.

By the expression "medium of extension", as referred to hereinabove, I mean a substance which when added to an aqueous dispersion of a powdered material will produce a very large bulk of practically non-settling precipitated substance

so that a slurry is obtained which when subjected to filtration will readily form a matrix of large volume maintaining the particles of powdered material in such relation to each other as to produce a characteristic orientation and physical structure. I have found that finely pulped fibrous material, such as finely pulped newspaper or wood pulp, in which the average individual fibre is less than approximately 0.08 of an inch in length, makes an excellent extension medium. The use of about 15% to about 25% of such a substance, together with about 75% to about 85% of the powdered material, if properly dispersed in an aqueous medium will, when subjected to filtration, begin to form a matrix at a remarkably low density of the dispersed material, in fact, values as low as 4 lbs. per cu. ft. have been obtained. Such a matrix is easily compressed and this makes possible a wide range of densities in the finished product. The important thing, however, is to have the minimum value of the densities obtainable as low as possible.

I have found that a properly processed fibrous material as described hereinabove in connection with its use as a medium of extension, likewise acts as an excellent filter aid. This material therefore performs the dual function of acting as a filter aid and medium of extension.

It is to be observed that merely mixing the three basic components mentioned hereinabove, filtering, and drying does not necessarily produce the desired result. Several problems and difficulties arise which I have discovered can only be overcome by using these components within certain critical ranges of composition. For example, it has been found that if too large a percentage of medium of extension is employed, the shrinkage and warping during drying is excessive, which is a serious problem. On the other hand, too small a percentage results in a product too high in density. Then again, an inadequate percentage of filter aid or medium of extension acting as such, causes filtration difficulties and makes it impossible to retain the matrix in its shape and form while pressure is applied. Moreover, the presence of small quantities of substances in aqueous solution, which may be advantageous for certain reasons, will often act as colloidal stabilizers and entirely prevent the filtration process from being carried out. Thus, the use of 3% sodium silicate or glue or other commonly used materials as binding agents when used herein will in many cases make filtration practically impossible to carry out. The use of the foregoing proportions is to be avoided.

It is also to be noted that the present invention embodies not only the establishment of certain critical ranges of composition but moreover sets forth certain important factors of control and embodies a procedure whereby a predetermined result can be obtained and any such obstacles as mentioned hereinabove entirely avoided. This is of considerable practical importance because of the variety of products which are possible to produce and the exactness with which it becomes possible to meet the requirements in any given product. For example, in standard corkboard used in refrigeration high thermal efficiency is extremely important but high tensile strength is not, whereas in insulating wallboard for building construction high tensile strength is required and the thermal efficiency is not so critically important. Furthermore by appropriate compression of the matrix during

processing a comparatively high strength material may be obtained which might serve as a substitute for linoleum or even leather. The invention therefore does not contemplate simply the production of a corkboard substitute or a linoleum substitute, or a leather substitute, etc., but rather contemplates a process in which certain factors of control are set up and a general basic procedure followed whereby the properties of the finished product are kept under control and can be varied at will to produce, for example, a high degree of flexibility, a rigid or even brittle product, a super light weight product, a medium weight product, high or low tensile strength, a high or low moisture absorption product, etc., and in each case the density of the product, comparatively speaking, is low due to the novel "Microcell" structure of the material. Moreover, the "Microcell" structure is preserved at all densities and unless the matrix has been greatly condensed, the volume of non-visible voids will be at least 40% of the total volume of the structure.

For the purpose of giving those skilled in the art a better understanding of the invention, the following illustrative examples will be given.

Example No. 1

To about 1000 pounds of a ½% latex dispersion, about 20 pounds of newspaper are added and subjected to high speed mechanical distintegration until the average individual fibre is about or less than approximately 0.08 inch in length. To this medium of extension about 40 pounds of finely ground regranulated cork are added with high speed mixing. The mixture is run into molds which in their simplest form are rectangular boxes the bottoms of which are filter screens of approximately 16 mesh. It is essential to fill the form quickly and agitate the mixture while still very dilute so as to obtain a uniform suspension in the mold and, once the matrix is formed, it must not be disturbed by further mixing. This latter point is important because in filling a form with a dilute aqueous mixture as used herein to the depth required, a considerable washing effect is encountered tending to disturb and wash out the matrix which is formed immediately adjacent to the filter screen during the filling process. If such local washing out is allowed to occur during filling, the slabs will have holes and regions of decreased thickness when finished even though the slabs are apparently absolutely evenly filled at the end of the filling operation. By noting the above precaution, an even and uniform slab can be made.

After the major portion of the liquid has filtered off, a cover made of 16 mesh screen and fitting accurately inside the mold, is placed on the material and a slight pressure is applied. It has been found that a pressure as little as about 2 to about 4 pounds per square inch is sufficient to produce the result required in this present case. The pressed slabs will still contain 400% to 600% aqueous liquid calculated on the dry weight of the material used. The slabs are then removed from the forms by inverting them onto another screen and are placed in a dryer. The filtrate is, of course, recovered and used for subsequent batches. The dried material will have a density of approximately 7 to 9 pounds per cubic foot which is lower than corkboard itself and a thermal conductivity of approximately 0.24. Those skilled in the art will readily understand

how unit operations of the above type may be conducted on a continuous scale.

In the above illustration, it is advisable to have the cork as finely powdered as practicable, certainly less than about ten mesh and preferably about 50 to about 200 mesh. This is contrary to prior methods of corkboard manufacture wherein large granules are used. The use of an extension medium as herein described in the manufacture of a corkboard is a novel feature of the process. The establishment of a critical range of composition, namely, about 20 to about 50% medium of extension, with the optimum value established at about 25%, is an important factor of control. The formation of a greatly extended matrix under filtration and its gradual compression without interrupting its continuous physical structure, is another important principle of the invention.

Example No. 2

The procedure used in Example No. 1 is followed but different materials are used. Thus, about 1000 pounds of about 1 to about 3% sodium silicate solution, about 48 pounds of a medium of extension (dry weight), and about 62 pounds of finely powdered exterior portion of corkboard are used. These boards are usually thinner than for refrigeration use, say about $\frac{1}{2}$ " to about 1", and a greater compression, say approximately 5 to 20 pounds per square inch is used. The finished material will have a density of approximately 12 to 20 pounds per cubic foot, and a thermal conductivity of approximately 0.29 to 0.35. The tensile strength is approximately 150 to 400 pounds per square inch. This makes an excellent low cost insulating building board. Moreover, the obvious advantage of using a cork base in an insulating board is extremely important from the point of view of the public.

Example No. 3

The same general procedure as described in connection with Example No. 1 is followed. The materials used are about 1000 pounds of 15% latex dispersion, about 60 pounds of a medium of extension (dry weight), and about 60 pounds of finely powdered exterior bark portion of corkboard. The boards are made still thinner, about $\frac{1}{8}$ of an inch for say sole leather substitute or flooring material, and a still higher compression is used, sufficient to reduce the aqueous content to about 100 to 150%. A strong, flexible, physically uniform structured material having a fine texture, free from visible voids, or cork granules, or visible fibre, is obtained. For certain purposes, hardening agents, such as sodium silicate, various resins, etc., may be added. Of course, in using latex, it may be compounded according to well known principles with the use of anti-oxidants, accelerators, etc.

I have found that the addition of a small percentage of dispersed wax to the medium of extension will entirely eliminate any capillary action in the finished product and will render it internally waterproof. For many products, this is extremely important. I have found that this wax may be satisfactorily incorporated by slowly pouring the melted wax into the hot medium of extension under high speed agitation.

In waterproofing my new light weight products, I have made an important discovery. I have found that an extremely effective paraffin preparation can be made which is both very practical and very economical to prepare and to use in a form in which it is obtained. In making my

paraffin preparation, I pour hot, melted paraffin into boiling water containing a dispersing agent, preferably a small amount of soap and then subject the mixture to high speed mixing while making the addition. The agitation is continued for several minutes while the mixture is cooled and a thick white paste is obtained comprising dispersed particles of paraffin and an aqueous vehicle containing a dispersing agent, preferably soap. Less than about 10% of soluble soap stock can be used to the weight of the paraffin. From about 100% to about 150% of water may be used to the weight of the paraffin. From a commercial point of view, it is preferred to use a cheap soluble soap stock such as rosin laundry soap stock and the like. This paraffin paste distributes itself in practically a dispersed form in a dilute aqueous mixture even when cold. Only about 4% to about 6% of this paraffin paste is required to destroy all capillary action in my new light weight products and render them water repellent. It is also to be pointed out that various resinous materials rosin, shellac, gums, and natural and synthetic resins can likewise be made into a similar paste for use in these light weight products.

While in each of the foregoing examples, powdered cork is employed as a basic material, it is possible to substitute other powdered materials such as wood flour or the like in this general process and obtain new materials of practical commercial importance, all of which have a similar physical "Microcell" structure, and a resultant comparative low density. This new "Microcell" structure is produced when and only when the number of particles of powdered material bears a certain relation to the volume of extension medium present. I have found a simple, practical empirical expression of this relation to be as follows:—per cent of extension medium, (per cent of total dry weight of composition), equals 900 divided by the density of the powdered material. Thus, if the density of the powdered material is 90 (as for certain rock flour) the per cent of extension medium used should be 10%. Using wood flour having a density of 30 pounds per cubic foot, 30% extension medium should be used. For a mixture of various flours having a density of 60, 15% medium of extension should be used. Using the waste product cork flour herein referred to having a density of 25 pounds per cubic foot, 36% extension medium should be used. Naturally, however, the percentage of extension medium used may be varied over a small range before the desired structure will show signs of breaking down and sometimes it becomes necessary to shift somewhat away from the calculated percentage. Thus, in Example No. 3, 45% extension medium is used instead of 36%, but the extension medium is also acting as a filter aid and unless at least 45% is used in this case filtration and compression is practically impossible to carry out. In general, the range of allowable variation might be expressed as follows:—percent extension medium should fall between 450 divided by the density of the powdered material and 1800 divided by that density. When this allowable variation is exceeded the breakdown in physical structure is evidenced by the formation of layers of cleavage perpendicular to the direction of compression. Likewise, a parallel matting of the material takes place similar to that found, for example, in cardboard. The greater the compression the more serious the breakdown. Thus, again, it is important to work

under the large dilution described herein and filter gradually without disturbing the matrix, otherwise a non-uniform, weak, inferior structure is obtained comprising large nodules of material, loosely connected together with resultant cleavages around these aggregates.

It is to be noted that natural cork itself has a "Microcell" structure, hence its low density. Finely ground regranulated cork has a density of approximately only 6 pounds per cubic foot. In using this material as in Example No. 1, a special case is encountered regarding the application of the empirical formula given above and a correction must be made due to the exceptional structure of these cork particles. The values given in the example have been found to work out in practice. This exception does not hold for the waste cork flour referred to in Example No. 2, which has a density of 25 pounds per cubic foot. The present invention embodies the use of latex as a binding agent in this particular combination and method of procedure, when the finished product is desired to have a high degree of flexibility. The invention contemplates the use of sodium silicate as a binding agent in this combination and process, when low cost is of vital importance and also where additional stiffness is required. This latter quality may also be obtained by use of appropriate resins. The invention likewise contemplates the use of wax to destroy capillary action in the finished product, as herein specified. Other well known waterproofing agents, such as soaps, oxychloride of zinc, etc., may of course be added without departing from the spirit of the invention.

Although the present invention has been described in connection with certain preferred embodiments, it is to be understood that variations and modifications can be resorted to as those skilled in the art will readily understand. These variations and modifications are to be within the purview of the spirit of the present invention and within the scope of the appended claims. Thus in the specification and claims, the term powdered materials may include such powdered materials as cork, wood flour, bark, limestone, diatomaceous earth, pumice stone, tripoli, fuller's earth, resins, sulphur, asphaltum, stone, marble, alumina, etc. The substances forming the medium of extension may comprise such fibrous materials as cellulose fibre, cotton, wool, paper, wood, cane fibre, straw, jute, rags, felt, kopak, asbestos, mineral wool, etc. The binding agents may comprise latex, glue, sodium silicate, and dispersed resins including prepared resinous pastes such as the prepared resins referred to in connection with the especially prepared paraffin herein described.

I claim:

1. The process of forming light weight substantially non-metallic products which comprises intimately mixing finely ground regranulated cork, a filter aid, and extension medium and a dispersed binding agent in a liquid, said regranulated cork being about 60 to 90% of the total dry weight of said mixture exclusive of the said binding agent, filtering said mixture without breaking the matrix previously formed, compressing said matrix to a desired density without breaking the continuous bonded structure thereof and including further expression of the liquid phase, and subsequently drying said matrix to form a light weight product.

2. The process of forming light weight, compressed, substantially non-metallic products

which comprises intimately mixing finely ground exterior portions of natural cork bark, a filter aid, an extension medium, and a dispersed binding agent in a liquid, said natural cork bark being about 50 to 90% of the total dry weight of said mixture exclusive of the said binding agent, filtering said mixture without breaking the matrix previously formed, compressing said matrix to a desired density without breaking the continuous bonded structure thereof and including further expression of the liquid phase, and subsequently drying said matrix to form a light weight product.

3. The process of forming light weight, compressed, substantially non-metallic products which comprises intimately mixing wood flour, a filter aid, an extension medium, and a dispersed binding agent in a liquid, said wood flour being about 50 to 90% of the total dry weight of said mixture exclusive of the said binding agent, filtering said mixture without breaking the matrix previously formed, compressing said matrix to a desired density without breaking the continuous bonded structure thereof and including further expression of the liquid phase, and subsequently drying said matrix to form a light weight product.

4. The process of forming light weight products which comprises intimately mixing in water a dispersed binding agent, a finely powdered, light weight, water insoluble material substantially inert with respect to said binding agent, and a finely pulped fibrous material of the group consisting of newspaper stock, wood pulp stock and asbestos pulp stock having individual fibres less than about 0.08 inch in length, said fibrous material being adapted to perform the dual function of a filter aid and of a medium of extension and constituting about 10 to 50% of the total dry weight exclusive of the binding agent, filtering said mixture without breaking the matrix previously formed, compressing said matrix to a desired density without breaking the continuous bonded structure thereof including further expression of the liquid phase, and subsequently drying said matrix to form a light weight product.

5. The process of forming light weight compressed products which comprises intimately mixing in water a filter aid, an extension medium, dispersed rubber in the form of latex as a binding agent constituting about 1/2% to 5% of the mixture in the aqueous phase and finely powdered, light weight, water insoluble material substantially inert with respect to said binding agent, filtering said mixture without breaking the matrix previously formed, compressing said matrix to a desired density without breaking the continuous bonded structure thereof including further expression of the liquid phase, and subsequently drying said matrix whereby a low strength light weight compressed product is obtained.

6. The process of forming light weight compressed products which comprises intimately mixing in water a filter aid, an extension medium, dispersed rubber in the form of latex as a binding agent constituting about 15% to 25% of the mixture in the aqueous phase and finely powdered, light weight, water insoluble material substantially inert with respect to said binding agent, filtering said mixture without breaking the matrix previously formed, compressing said matrix to a desired density without breaking the continuous bonded structure thereof including further expression of the liquid phase, and subsequently drying said matrix whereby a high strength light weight compressed product is obtained.

7. The process of forming light weight compressed products which comprises intimately mixing in water a filter aid, an extension medium, finely ground dispersed resinous material as a binding agent, and finely powdered, light weight, water insoluble material substantially inert with respect to said binding agent, said binding agent constituting about $\frac{1}{2}\%$ to 5% of the mixture in the aqueous phase for obtaining low strength products and 10% to 25% of the mixture for obtaining high strength products, filtering said mixture without breaking the matrix previously formed, compressing said matrix to a desired density without breaking the continuous bonded structure thereof including further expression of the liquid phase, and subsequently drying said matrix to form a light weight compressed product.

8. The process of forming light weight compressed products which comprises intimately mixing in water a finely pulped fibrous material for filter aid and extension medium constituting about 40% to 60% of the mixture exclusive of a binding agent, a binding agent of sodium silicate, and finely powdered, light weight, water insoluble material substantially inert with respect to said binding agent, said binding agent constituting about $\frac{1}{2}$ to 5% of the mixture in the aqueous phase for obtaining low strength products and 15% to 25% of the mixture for obtaining high strength products, filtering said mixture without breaking the matrix previously formed, compressing said matrix to a desired density without breaking the continuous bonded structure thereof including further expression of the liquid phase, and subsequently drying said matrix to form a light weight compressed product.

9. In the production of light weight materials, the improvement which comprises embodying an extension medium in an aqueous mass containing a finely powdered, light weight, substantially water-insoluble material for producing an enlarged uniform dispersion of said material in an aqueous mixture, whereby such abnormally large volumetric dispersion of said powdered material will be maintained during filtration and compression and also during subsequent drying of

said mixture to produce light weight materials.

10. The process of forming light weight products which comprises intimately mixing in a liquid an extension medium, a dispersed binding agent, a finely powdered, light weight, water insoluble material substantially inert with respect to said binding agent, filtering said mixture without breaking the matrix as formed, compressing said matrix to a desired density without breaking the continuous bonded structure thereof including further expression of the liquid phase, and subsequently drying said matrix to form a light weight product.

11. The process of forming light weight products which comprises forming an intimate aqueous mixture containing a dispersed binding agent, a finely powdered, light weight, water insoluble material substantially inert with respect to said binding agent, and a finely pulped fibrous material having individual fibers less than about 0.08 inch in length, said fibrous material being adapted to perform the dual function of a filter aid and of a medium of extension and constituting about 10 to 50% of the total dry weight exclusive of the binding agent, filtering said mixture without breaking the matrix as formed, compressing said matrix to a desired density without breaking the continuous bonded structure thereof including further expression of the liquid phase, and subsequently drying said compressed matrix to form a light weight product.

12. The process of forming light weight products which comprises intimately mixing an extension medium and a dispersed binding agent in a hot liquid, slowly pouring melted wax into said hot mixture under high speed agitation, adding a finely powdered, light weight, water insoluble material substantially inert with respect to said binding agent, filtering said mixture without breaking the matrix as formed, compressing said matrix to a desired density without breaking the continuous bonded structure thereof including further expression of the liquid phase, and subsequently drying said matrix to form a light weight and substantially waterproof product.

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