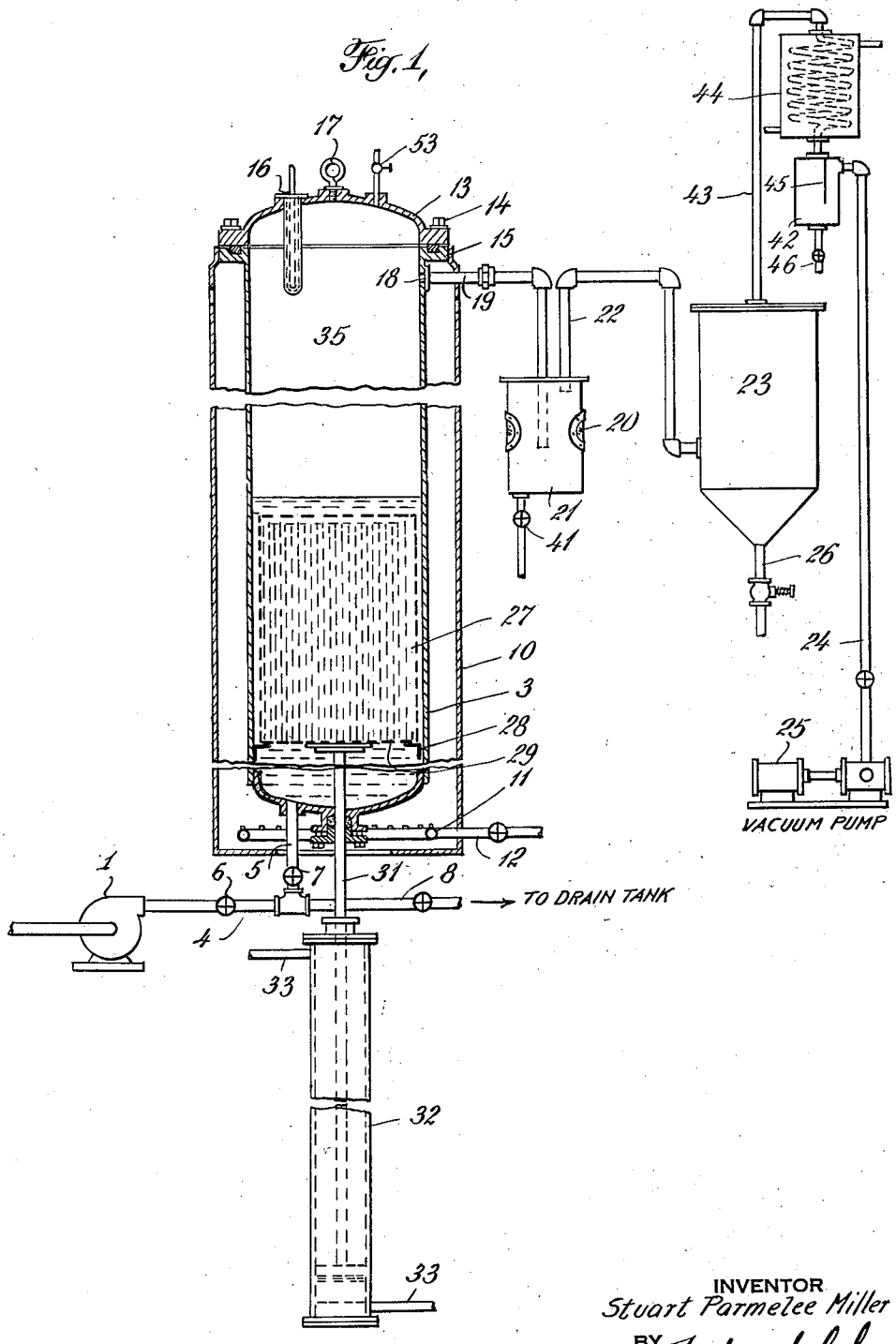
Sept. 3, 1935. S. P. MILLER 2,012,968

SATURATED FIBROUS CONDUIT AND PROCESS OF MAKING SAME

Filed May 27, 1932. 2 Sheets-Sheet 1

Fig. 1.



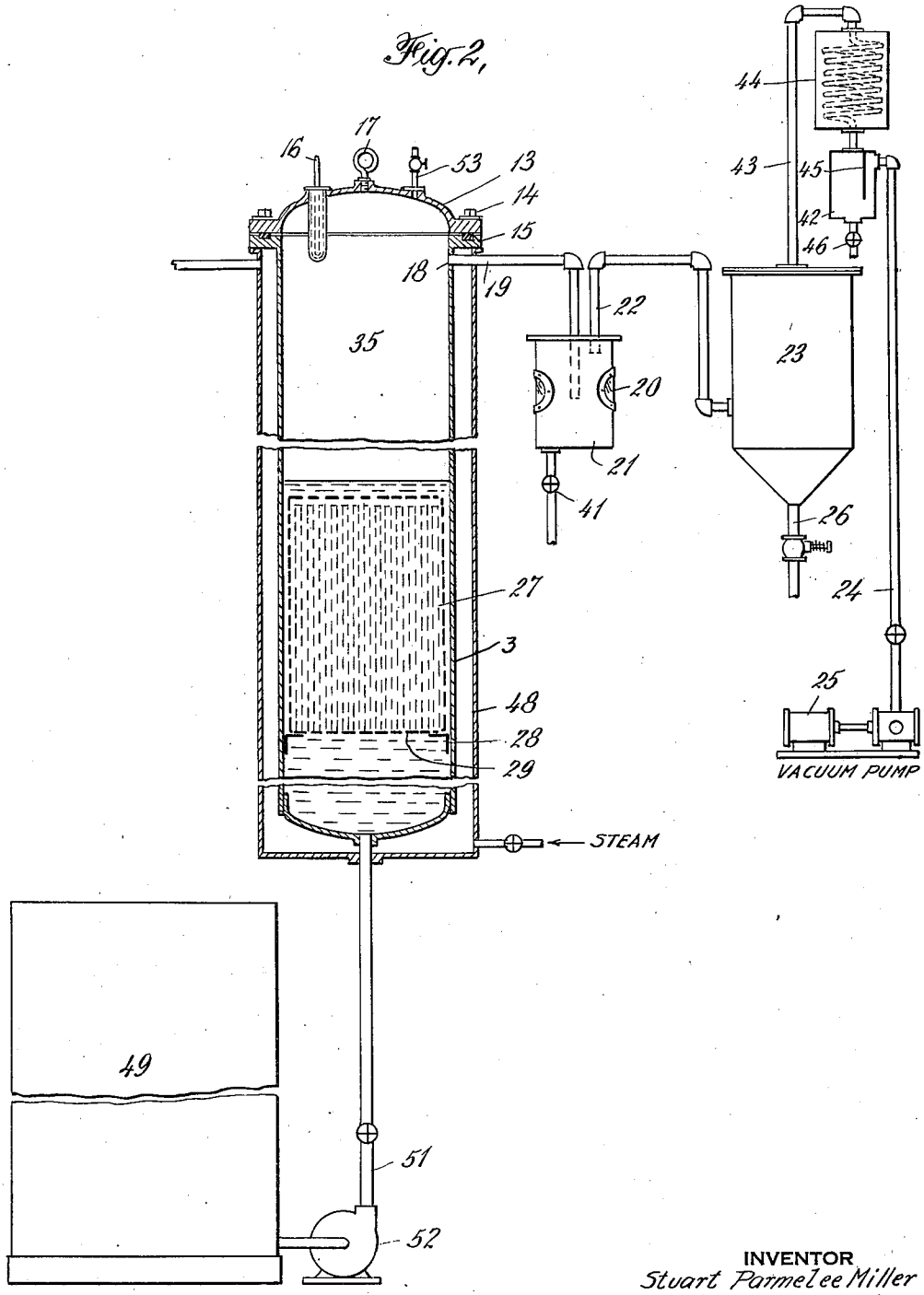
INVENTOR
Stuart Parmelee Miller
BY
ATTORNEY

Sept. 3, 1935. S. P. MILLER 2,012,968

SATURATED FIBROUS CONDUIT AND PROCESS OF MAKING SAME

Filed May 27, 1932 2 Sheets-Sheet 2

Fig. 2,



INVENTOR
Stuart Parmelee Miller
BY John Sibley
ATTORNEY

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2,012,968

UNITED STATES PATENT OFFICE

2,012,968

SATURATING FIBROUS CONDUIT AND PROCESS OF MAKING SAME

Stuart P. Miller, Englewood, N. J., assignor to The Barrett Company, New York, N. Y., a corporation of New Jersey

Application May 27, 1932, Serial No. 613,939

12 Claims. (Cl. 91—70)

This invention is directed to the saturation of absorbent articles, and particularly, the saturation and impregnation of fibrous conduits employed to conduct and protect underground cables, telephone lines, electric light, and other conductors, and for the formation of water and drain pipes to render them substantially waterproof and resistant to abrasion, corrosion, and conditions encountered underground by fibrous conduits. While, as above stated, the invention is particularly adapted for the saturation of fibrous conduits, it will be understood that it may be employed to saturate small fibrous or other articles requiring impregnation, such as spools, coils, or other preformed articles, asbestos products, concrete pipes, flashing blocks, and to impregnate and saturate wood.

The articles to be saturated may, of course, be prepared in any known manner. In the case of fibrous bases for conduits, newsprint and paper pulp, or other fibrous material, may be beaten in the usual paper beaters and the resultant stock screened and then pumped to paper cylinder machines and there formed into wet felts or paper. The wet paper may be wound about a cylindrical tube or mandrel to produce a wet conduit of proper thickness. This conduit may be dried in kilns to remove the major portion of its moisture content. The fibrous base may, of course, be prepared in any other known manner. Such conduits are made in several sizes, commonly varying from two inches to six inches or larger in internal diameter, have an average wall thickness of approximately 3/8 of an inch and are usually produced in 5, 6, and 8 foot lengths. The wall thickness of the conduits may, of course, be greater or less than 3/8 of an inch and other dimensions may vary. As they come from the drying kilns, they usually contain from 5 to 8 per cent moisture, although considerably more than 8 per cent moisture may, on occasion, be found present.

The porosity or absorptive characteristics of the walls of fibrous conduits may vary within wide limits from a very dense wall of a density corresponding to hard wood and exceedingly difficult to saturate with low carbon water gas tar pitch or even with other waterproofing material, such as asphalt, to a porous open wall similar in density to that of roofing felt, which will readily absorb bituminous saturants. Samples of present commercial fibrous conduits have been tested and found to vary from substantially less than 1 cc. of voids per gram of tube wall to 2.8 or more cc. of voids per gram of tube wall. To determine the cubic centimeters of voids per gram of tube wall, a small sample of tube wall (say 2" x 5") is dried for one hour at 100° C., cooled, dessicated, and thereafter weighed. It is then immersed in kerosene, maintained at a temperature of 25° C., for 18 hours. Thereafter, the sample is removed from the kerosene, drained in vertical position for 30 seconds, and again weighed. The difference in weight represents the kerosene absorbed. This difference in weight in grams, divided by the specific gravity of the kerosene, corresponds to the cubic centimeters of kerosene absorbed. This volume in cubic centimeters, divided by the original weight in grams, gives the voids in cubic centimeters per gram.

The fibrous conduits, as they come from the drying kiln, containing from 5 to 8 per cent of moisture, have heretofore commonly been impregnated with water gas tar pitch by immersing them in tanks of molten pitch maintained at atmospheric pressure or pressure above atmospheric and at a temperature of 275° to 300° F. or upwards. The conduits have in many cases been allowed to soak in the molten pitch for from 7 to 12 hours, then withdrawn, and permitted to drain to remove excess saturant therefrom.

This process of saturating fibrous conduits, it will be noted, requires the soaking of the conduits for long periods of time to obtain satisfactory penetration of the saturant throughout the walls of the conduit. In the practice of the process, numerous difficulties are encountered, among the most important of which may be mentioned—

(1) Foaming of the pitch during the saturation of the conduits with consequent loss of volatile oils from the pitch and restriction of the amount of pitch absorbed by the fibrous walls. This, I have found, may largely be attributed to the moisture content of the fibrous tubes. In the case of prior processes involving the maintenance of pitch in open tanks loss of volatile oils also occurs due to evaporation even without foaming.

(2) Building up of the "free carbon" content of the pitch due to the prolonged maintenance of large bodies of pitch in which the conduits are immersed under heat, resulting in decomposition of the pitch. Increase in the "free carbon" content of the pitch decreases the penetrability of the saturant and also results in carbon deposits on the walls of the conduit, preventing entrance of the saturant thereinto and resulting in defectively saturated conduits, i. e., conduits having "white spots" within the walls. The term "free carbon" is used in the sense common in the coal tar industry to indicate material contained in the

bituminous saturant which is insoluble in benzol or carbon bisulfide. It is commonly determined as described by Weiss (Journal of Industrial and Engineering Chemistry, vol. 10, 1918, pages 736 and 820, Test D5). It comprises high-molecular weight carbon compounds of low solubility, carbon, and other insoluble material;

(3) Unsatisfactory saturation of the conduits due to the fibrous walls not absorbing sufficient saturant to render them substantially waterproof. Conduits which are not adequately saturated do not attain maximum strength and resistance to deflection or deformation;

(4) In the case of saturation of conduits under substantially atmospheric pressure, or under higher pressures, a substantial fire hazard may exist. This is especially the case where increased pressure is produced by air pressure on the saturant and where inadequately covered and protected saturating tanks are used; and

(5) Due to long time immersion of the organic or fibrous bases in hot saturant, charring or burning of the organic bases frequently takes place.

In order to satisfactorily protect cables and other electrical conductors, it is important that the fibrous conduit be substantially waterproof, resistant to abrasion and soil pressures, and capable of withstanding underground conditions and not punctured or broken by the soil and débris surrounding it. One of the tests to which conduits are subjected to determine their waterproofness is to immerse them in distilled water, maintained at a temperature of approximately 77° F. for 48 hours, then ascertain the percentage increase in the weight of the conduit and thus arrive at the amount of water absorbed thereby. Present conduits, saturated with water gas tar pitch, have been tested in this manner and many have been found to absorb in the neighborhood of 12 to 14 per cent of water. At the present time, it is generally recognized that a conduit which, upon immersion for 48 hours in distilled water maintained at a temperature of approximately 77° F., will absorb not more than 6 per cent moisture represents a satisfactorily waterproof conduit; a conduit absorbing not more than 4 per cent moisture under these conditions is regarded as eminently satisfactory. It should be noted that as indicated hereinabove, many of the saturated fibrous conduits now made, when subjected to the waterproofness test briefly described above, show an absorption of moisture substantially in excess of 4 per cent, many of them absorbing 12 per cent and more moisture. In general, the lower the water absorption, the more satisfactory is the product.

It is an object of the present invention to provide a process of saturating porous articles, and particularly fibrous conduits, which results in substantially uniform saturation and impregnation of conduits or other articles. In the invention disclosed and claimed in application of George Emberg Serial No. 613,937, filed May 27, 1932, involving the saturation of fibrous conduits by a vacuum process with the conduits in vertical position, while the waterproofing properties of the conduits are greatly improved as compared with prior processes of saturating the conduits, involving the soaking in saturant at atmospheric pressure or pressures above atmospheric, the portion of the conduit occupying the base portion of the tank was more thoroughly saturated, and consequently, more resistant to absorption of water than the opposite end of the conduit. The process of this invention results in substantially more uniform saturation of the conduits.

Another object of this invention is to provide a process that is simple and inexpensive to operate, requires comparatively inexpensive apparatus for its practice, the handling of comparatively small quantities of saturant, and a comparatively short time of contact between the saturant and the fibrous bases. Hence, substantially all tendencies toward charring or burning the organic bases are eliminated. The apparatus may be compact so as to occupy a small amount of floor space. In a well known method of saturating conduits, very large amounts of pitch were recirculated in contact with the conduit, the pitch being maintained in contact with the conduits for a long period of time. As hereinafter indicated, the process of this invention may be practiced in simple, compact apparatus which requires no pumping equipment for recirculating material within the tank and in which only a relatively small quantity of saturant is employed, consequently materially reducing the cost involved in saturating the conduits.

In the impregnation of absorbent articles with bituminous saturant, two of the factors which tend to prevent satisfactory saturation are—

1. Air in the pores or voids, and

2. Moisture on the fibers or other solid surfaces and as vapor or even liquid in the pores.

Air and water vapor in the pores of the material tend to prevent entrance of the saturant, and water on the inner or outer material surfaces tends to prevent wetting of the surfaces by the saturant.

When saturation at atmospheric pressure is attempted, the major portion of the water may be removed by long soaking in the hot saturant at a temperature above the boiling point of water. The escaping steam carries with it some air. The removal of water by this means is time-consuming and air is ordinarily not completely removed. By the heretofore commonly practiced methods of saturation, relatively poor degree of waterproofness has been attained either because the voids of the saturated article are incompletely filled with saturant, or the fibers are incompletely surrounded by and wet with saturant.

If it is attempted to improve the degree of saturation by applying pressure, some improvement is found, but saturation is still imperfect. The effect of pressure is, by forcing additional saturant into the material, to compress residual air and water vapor in the pores, even to the extent of somewhat compressing, densifying, and deforming the walls of the saturated article. On release of pressure, the compressed air and water vapor expand, forcing saturant out from the material, thereby reducing the degree and uniformity of saturation.

In accordance with a preferred embodiment of this invention, occluded gas and moisture are removed from the conduit walls by placing them in a saturating tank and evacuating the tank while heating it. In the preferred procedure of drying, I subject the conduits to be saturated to a vacuum gradually increasing to a maximum, say 28 to 29 inches of mercury, and hold them at this vacuum while heating them long enough to remove substantially all water, water vapor, and air from the voids, without allowing them to contact with the pitch saturant. When thus dehydrated and deaerated, I permit pitch to come into contact with them. Even while the pressure head on any given point of the tube due to the level of pitch

in the tank is low, the capillarity of the pores causes a considerable amount of pitch to be absorbed. Increase of pressure head of the pitch forces additional amounts into the vapor- and air-free voids, effecting substantially complete saturation.

Slow increase of head of pitch has the added advantage of permitting satisfactory saturation with saturants containing fine suspended or colloidal matter like the "free carbon" of coal tar pitch. Slow increase in pressure (as is well known in the filtering art) tends to eliminate the building up of impenetrable layers of such suspended matter (e. g. "free carbon") on the surface of the fiber conduit, and by avoiding such impenetrable layers, assures greater penetration and improved saturation.

The higher the vacuum attained, the more effective is the removal of air and water from the voids and the more complete is the absorption of saturant.

In impregnating with other saturants such as aqueous of alcoholic solutions or the like, the removal of volatile liquids immiscible with the saturant from the porous or absorbent articles can be accomplished as herein described thereby facilitating the wetting of the porous article by the saturant.

Instead of drying the conduits in the preferred manner hereinabove described, the conduits may be soaked at atmospheric pressure but preferably under vacuum in hot saturant, such as coal tar pitch or other bituminous waterproofing material, until foaming of the pitch stops and substantially all moisture is removed from the conduit walls. The pitch is maintained at a temperature materially above the boiling point of water effective during the drying of the conduits; for example, at a temperature of about 275° F. to 300° F. if atmospheric pressure be employed. If this procedure of drying the conduits is followed, the present equipment for saturating them, involving the soaking of the conduits in the hot saturant, may be employed to dry the conduits. After the conduits have been dried, they may be saturated under vacuum, as hereinafter more fully pointed out. The saturant employed may be the pitch in which the conduits were immersed to effect the drying of fresh waterproofing saturant may be utilized.

My invention also contemplates the drying of the conduits by heating them at atmospheric pressure, or higher pressures. Thereafter the conduits, while hot, are subjected to vacuum, with heating if desired, to remove moisture and occluded gas therefrom, the sensible heat of the conduits aiding in, or effecting, the evaporation under the reduced pressure to which they are subjected.

In the preferred method of saturation after the conduits are dried, saturant, which may be coal tar pitch, water gas tar pitch, asphalt, cutback pitches, blended pitches, or other waterproofing material, is progressively introduced into the tank while the vacuum is maintained therein. The vacuum in the tank may be utilized to cause the saturant to enter thereinto. As the saturant progressively rises, it contacts with the walls of the material to be saturated, filling the evacuated pores or voids thereof. The rate of flow of the saturant into the tank is controlled so that a substantially constant vacuum, preferably from 23 to 29 inches of mercury, is maintained during the entire period required for the saturant to rise to completely immerse the material being saturated. Adequate vacuum generating capacity must be provided since otherwise, as the pitch rises, the vacuum would become less and less until, under the most extreme conditions, the voids at the top of the tube would become substantially full of air and hence resistant to penetration of the saturant thereinto. The saturating tank is preferably made of such height as to leave a substantial space above the body of saturant introduced thereinto and in which the fibrous tubes are immersed. Preferably, the fibrous tubes are permitted to soak in the saturant for from 15 minutes to two hours or longer while the vacuum is maintained therein.

Due to the maintenance of substantially constant vacuum in the container during the entire period required for the introduction of the saturant into the tank or container and the maintenance of the vacuum during the soaking period, the voids at the top end of the fibrous tubes when the tubes are being treated in vertical position will be as well filled with saturant as those at the bottom end and a uniformly saturated tube of improved waterproof properties and strength will result.

As the saturant for fibrous conduits, a coal tar pitch having a melting point of from 140 to 180° F. may employed. Pitch of a melting point as high as 225° F. may be used in special cases, e. g., where conduits are to be exposed to temperatures which would soften the lower melting point pitches. Preferred pitches having melting points of from 140 to 180° F. or higher will have a "free carbon" content of from 5 to 9 per cent, but pitches of higher "free carbon" content up to from 15 to 25 per cent, or even higher may be used to give commercially satisfactory saturation under favorable conditions, e. g., in saturating products having comparatively open wall structure.

Preferably, coal tar pitch made by a vacuum distillation process, such for example as disclosed in Patent No. 1,759,816, granted May 20, 1930, or other processes minimizing decomposition of the tar undergoing distillation, is employed as the saturant. Pitches made by a low temperature vacuum distillation process are characterized by low "free carbon" content as compared with pitches made by higher temperature processes from the same tar, and I have found that such pitches are particularly suitable as the saturant for fibrous conduits. For example, such pitch of 149° F. melting point (cube in water) when tested had a "free carbon" content of from 5 to 9 per cent; such pitch of 158° F. melting point (cube in water) when tested had a "free carbon" content of from 5 to 9 per cent; such pitch of 226° F. melting point (cube in air) had a "free carbon" content of from 11 to 15 per cent; and such pitch of 291° F. melting point (cube in air) when tested had a "free carbon" content of from 16 to 21 per cent.

Pitches produced by distilling tar not under vacuum but by continuously heating the tar and then passing the heater tar into a vapor box where the vapors separate from the pitch have considerably higher "free carbon" contents for the same melting point pitch, e. g., pitch produced in this manner having a melting point of 149° F. (cube in water) when tested had a "free carbon" content of from 11 to 13 per cent; a pitch of 158° F. melting point (cube in water), had a "free carbon" content of from 10 to 14 per cent; a pitch of 226° F. (cube in air) had a "free carbon" content of from 19 to 23 per cent; and a pitch of 291° F. melting point had a "free carbon" content of

from 33 to 37 per cent. The "free carbon" content will depend largely on the temperature treatment during distillation as well as on the melting point of the pitch and the "free carbon" content of the tar used to make the pitch. Coke oven tar generally will not run much above 5 per cent "free carbon"; 1 per cent "free carbon" content is relatively low for such tars.

The use of a pitch made by a vacuum distillation process for saturation has a two-fold advantage:

(1) In its manufacture, this pitch is distilled under vacuum and consequently at a lower temperature than would be needed to produce a pitch of the same melting point by the usual methods of distillation. This low temperature causes minimum decomposition and hence minimum "free carbon" formation in the pitch. Low "free carbon" content is an aid in effecting complete penetration and saturation of porous articles.

(2) In its manufacture, the pitch has been subjected to temperature and vacuum conditions such as to insure the substantially complete removal of oils which would be volatile under the conditions maintained in the saturating operation of my invention. Such pitches will be substantially free from oils volatile under the temperature and vacuum conditions encountered in the saturating operation, and consequently, will suffer minimum or at least only nominal losses from volatilization during saturation. Owing to low volatilization losses, concentration of "free carbon" resulting from oil losses as well as increases in melting point during saturation will be reduced to a minimum.

Other methods of manufacture, such as steam- or inert-gas-distillation, which effect distillation at relatively low temperatures, will yield desirable pitches for my process for the reasons outlined above.

While my invention is particularly adapted to saturating absorbent articles with waterproofing material in a molten condition, it is likewise adapted to saturating with waterproofing compounds dissolved to form liquid solutions in solvents by observing the proper temperature, pressure and time conditions. Such materials may be natural resins (e. g. rosin), drying oils (e. g., linseed, tung oil, etc.), synthetic resins (e. g., paracoumarone resin, phenol-formaldehyde resin, etc.); plasticizers and modifying agents may be added. The solvents used may be chosen to meet requirements for the water-proofing material used, the temperature of saturation and vacuum and other working conditions; such solvents may be benzol, toluol, solvent naphthas, petroleum naphtha, or other fractions, alcohols, chlorinated hydrocarbons, natural oils, etc.

Besides waterproofing absorbent articles this invention may be employed in fireproofing, oil proofing, gas proofing, insulating, painting and coloring such articles. For example, in fireproofing the article it may be impregnated with an aqueous solution of inorganic salts, e. g., solutions comprising phosphates, in oil proofing a resin or cellulose solution may be used, in gas proofing and insulating the article may be saturated with resin, cellulose or rubber mixture and in painting and coloring the article may be impregnated with paints, lacquers, enamels, or varnish. The invention may also be applied to the preserving of wood by impregnation of the wood with creosote or aqueous solution of fungicidal salts.

Preferably, after the tubes have been allowed to soak for 15 minutes or more, while the body of saturant is maintained under vacuum, the tubes are removed from the saturant and moved into the evacuated space thereabove. This permits saturant to drain from the tubes and the removal of residual gas or moisture in the tube walls. The fibrous tubes may be subjected to vacuum momentarily while in this evacuated space and then again immersed in the saturant or they may be maintained in the evacuated space for a predetermined period of time which may be as short as one or two minutes, and may, if desired, be for longer periods e. g., for one or more hours. In this way, removal of a maximum proportion of occluded gas and moisture and the absorption of a maximum amount of saturant by the fibrous tubes is insured; a uniform and thoroughly saturated tube results. Instead of moving the tubes up into the evacuated space, the saturant may be passed out of the tank while maintaining vacuum therein and the tubes thus subjected to vacuum.

As a modification of the procedure hereinabove described, after the initial soaking of the fibrous tubes under vacuum, the vacuum may be broken and pitch drained out of the tank. The saturated tubes are left in the tank subjected to the atmospheric pressure then prevailing therein for a period of one or two minutes or longer. The tank is then again evacuated and then saturant passed thereinto while holding the vacuum substantially constant. The saturated tubes are permitted to soak while maintaining the vacuum for any desired period, for example 15 minutes or longer, and then the vacuum again broken. After the tubes are soaked in the saturant under atmospheric pressure for a brief period, they are removed, allowed to drain, cooled, trimmed and then stacked for shipment to the consumer.

If desired, after the saturation of the fibrous conduits under vacuum, as hereinabove described, the vacuum may be broken and pressure which may be up to 100 pounds per square inch built up on the saturant by introducing pitch or other saturant or inert gas into the saturating tank. The pitch or inert gas is introduced slowly into the tank so that the pressure builds up gradually. Operation in this manner is particularly suited to the saturation of very dense conduits. The pressure on the saturant in which the conduits are immersed after the conduits have been subjected to vacuum saturation as hereinabove described, causes the introduction of maximum amounts of saturant into the conduit walls.

In the preferred embodiment illustrated in the drawings, the invention is shown incorporated in the production of a fibrous conduit saturated with a bituminous material and the present disclosure will be confined to the present illustrated embodiment of the invention. It will be understood, however, that the novel features and improvements are susceptible to other applications, such, for example, as the saturation of absorbent articles generally. Included in the scope of my invention is the saturation of conduits, piping and other conductors made of cement, concrete, cement-asbestos mixture, or other porous or absorbent articles capable of being saturated. The conduits produced by my process are not only resistant to external pressures, such as soil pressures, but by suitable choice of materials and saturating conditions, may be made resistant to high internal pressures and hence suitable for pipe lines conveying corrosive waters or the like. Hence, the scope of this invention is

not confined to the specific embodiment herein described.

In the drawings, Figure 1 is a side elevation, partly in section, illustrating a preferred form of apparatus for saturating conduits in accordance with the process of this invention, and Figure 2 illustrates a modified form of apparatus for practicing this invention.

In the drawings, reference numeral 1 indicates a pump communicating with a source of supply of saturant, and a pipe 4. This pipe connects with pipe 5 leading into the base of a vertical tank 3. Pipes 4 and 5 have valves 6 and 7 therein. A valve-controlled discharge pipe 8 communicates with pipe 4 and leads to a storage tank (not shown) or other point of disposal of residual pitch or other saturant in tank 3. The tank 3 is preferably made of a height such as to accommodate a maximum barometric column of the saturant. In the case of coal tar pitch of 1.2 specific gravity, the tank is made approximately 38 feet high so that a maximum barometric column of the saturant, approximately 28 feet high, may rise therein, and a considerable evacuated space provided above the column of saturant.

Tank 3 may be heated directly by burners 11 disposed about the base and sides of tank 3 or may be steam heated, e. g., by a steam jacket or coils. Gaseous or liquid fuel may be fed to these burners through valve-controlled pipe 12, the products of combustion passing through a jacket 10 surrounding the tank. Cover 13 provided with a valve-controlled vent 53 for venting the tank to the atmosphere seals the top of the tank. In the drawings the cover is shown as bolted to the tank, as indicated by reference numeral 14, a gasket being inserted between the cover and flange 15 of the tank to give a gas-tight joint. A thermometer-well 16 is formed in the top of the cover. The top of the cover is also provided with an eye bolt 17 permitting ready removal of the cover from and replacement on the tank. A port 18 near the upper edge of tank 3 communicates by means of pipe 19 with the receptacle 21, having sight glasses 20 permitting visual inspection of any vapors and foam which may come over from the saturating tank 3. A valve-controlled drain pipe 41 leads from receptacle 21, permitting removal of any liquid which may collect in the receptacle.

Pipe 22 connects the receptacle 21 with a safety tank 23. This tank functions to collect any foam coming from the saturating tank and prevents the entrance of this foam into the vacuum line 24, leading from trap 42 to vacuum pump 25. Trap 42 is provided with a valve-controlled draw-off 46. Discharge of the contents of tank 23 may be effected through valve-controlled pipe 26. A pipe 43 passing through condenser 44 connects the safety tank 23 with trap 42. A baffle plate 45 in trap 42 prevents flow of entrained condensate into the vacuum line 24.

The conduits may be supported in a perforated basket or other device 27 resting on suitable brackets or supports 28 bolted or otherwise secured to the sides of tank 3. A grid 29 rests on supports 28. This grid carries the weight of the basket 27. Secured to grid 29 is the piston rod 31 of a hydraulic or pneumatic cylinder 32 provided with inlet and outlet ports 33. Operation of the cylinder 32 by the admission and discharge of pressure fluid through ports 33 causes elevation of the piston 31 with consequent elevation of the basket 27 into the space 35 in the top of tank 3. When the piston rod 31 descends, the basket 27 descends therewith into the lower portion of the tank 3.

Parts of the apparatus of Fig. 2 corresponding to those of Fig. 1 have been given like reference numerals. The apparatus of Fig. 2 differs from that of Fig. 1 chiefly in that the mechanism for reciprocating the basket of tubes within the tank has been eliminated, the tank is provided with a steam jacket 48 for heating the contents thereof and a kettle 49 of a capacity sufficient to hold the saturant fed to tank 3 communicates with the tank by means of a line 51 leading into the base thereof. In the apparatus of Fig. 2, the basket of tubes is maintained at a fixed point within the tank during saturation and is not moved up and down as in the case of the apparatus of Fig. 1. The level of saturant within the tank may be changed during the saturation so that the tubes are alternately contacted with saturant and subjected to vacuum or pressure. While the saturant may be fed into the tank 3 from kettle 49 disposed therebeneath by the vacuum created in tank 3 preferably a pump 52 is placed in valve-controlled line 51 to pump the saturant into the tank.

In operation of the apparatus of Fig. 1, the fibrous conduits to be saturated, preferably in vertical position, are placed in perforated basket 27, and the basket then inserted in the saturating tank 3, resting on grid 29. Thereafter, cover 13 is suitably sealed onto the saturating tank. Cylinder 32 is operated so that the tubes are maintained in space 35 at the top of the tank. The vacuum pump 25 is then started, causing the vacuum to gradually build up in the container and the container or saturating tank heated by means of burners 11 simultaneously with the building up of the vacuum. Preferably, before heating the container, a limited amount of saturant is admitted thereinto. The saturant in the base of the container served to prevent local overheating of the container walls during the heating to drive out the moisture and occluded gas from the fibrous conduits, and prevents overheating of the fibrous conduits.

A vacuum of from 17 to 29 inches of mercury, preferably above 26 inches of mercury, may be maintained during the drying of the conduits. The heating of the conduits under vacuum is carried on until moisture and occluded gas are removed therefrom to the maximum possible degree. Just how long this should take depends upon the porosity of the conduits, their initial moisture content, etc. For a fibrous conduit having about 1.6 cc. of voids per gram of tube wall and having a moisture content of approximately 5 to 6 per cent, heating from about 45 minutes to 2 hours should suffice. The optimum temperature in the container or saturating tank during the drying of the conduits will depend upon the percentage of water, size of voids, density of the material, time cycle, and other factors. In one case, 140° F. was found to be satisfactory with a vacuum of 27 inches of mercury when drying conduits containing approximately from 5 to 8 per cent moisture and having 1.6 cc. of voids per gram of tube wall.

After the moisture and occluded gas have been removed, the basket of fibrous tubes is lowered to the position indicated in Fig. 1 and the saturant gradually pumped through pipes 4, 5, while the vacuum is maintained in tank 3. The flow of saturant is controlled by regulating valves 6 and 7, the speed of the vacuum pump 25 being if need be increased proportionately to the rise in

level of the saturant in the tank 3 so that a substantially constant vacuum is maintained on the exposed portions of the fibrous conduits in the basket 27. The flow may be so regulated that the saturant rises to a level above the tops of the conduits, as indicated on the drawings, during a predetermined time interval. With conduits having relatively open wall structure, satisfactory results have been obtained when the flow of saturant was regulated so that 6 to 10 minutes were consumed in submerging the tubes. With exceptionally dense structures an hour or more may be consumed in submerging the tubes.

Utilizing coal tar pitch having a melting point of about 158° F., the pitch is maintained in the saturating kettle at a temperature of from 275° to 350° F., preferably about 320° F. The conduits are permitted to soak in the saturant while the vacuum is maintained thereon for a suitable period of time, for example from 2 to 4 hours, and thereafter cylinder 32 actuated to elevate the basket of conduits into the space 35 above the saturant. Excess saturant drains from the conduits and the vacuum to which the conduits are subjected effects the removal of any residual moisture or occluded gas from the conduits. The basket of conduits is then again immersed in the saturant by lowering basket 27 into the position indicated on the drawings. The basket may be quickly moved from its position immersed in the saturant into the evacuated space 35 and then returned into the saturant so that the conduits are disposed only momentarily in the space 35, or the conduits may be maintained in the evacuated space for a considerable period of time, one or two minutes, or if desired, for a period of an hour or longer. After the soak period, the vacuum may be released, cover 13 removed, and the basket of saturated tubes or conduits taken out of the tank. They may then be allowed to drain for from 2 to 6 minutes or longer, e. g., 15 to 20 minutes, cooled, trimmed and stored for shipment to the consumer. If desired, the conduits prior to final draining may be given a wash treatment with bituminous saturant at a temperature higher than the temperature of the saturant in tank 3 to remove any foreign matter, such as lint, carbon, etc., adhering to the saturated tube walls.

If desired, the basket of conduits may be maintained at the base of the tank during the vacuum drying, and after the drying the saturant gradually introduced while maintaining a vacuum in the tank. Further the basket of tubes may be dried while supported in the top of the tank, saturant run into the tank to a level beneath the base of the tubes and then the tubes gradually immersed by lowering the basket of tubes while maintaining vacuum in the tank.

In the apparatus of Fig. 2, the basket of tubes is inserted in the tank, the cover of the tank sealed thereon and vacuum gradually built up to a maximum and maintained until the tubes are dried. Thereafter pump 52 progressively introduces the saturant into the tank while the vacuum is maintained in the tank. The tubes are then permitted to soak while the vacuum is maintained. Thereafter the saturant is drained from the chamber 35 through pump 52 by opening the valve in line 51, the height of the saturant in chamber 35 above the level of saturant in tank 49 being greater than a barometric column. The tubes are then subjected to vacuum for the desired period of time out of contact with the saturant. Saturant is then again progressively introduced into the tank. After the desired soak period, the vacuum may be broken, the saturant returned to kettle 49 and the saturated tubes withdrawn.

In the case of the apparatus of Fig. 1, after saturation, the saturant remaining in tank 3 is permitted to return to its source by breaking the vacuum in tank 3 and opening valves 7 and 6 in lines 5 and 4, respectively. If desired, the pitch remaining in the saturating tank may be drained to a suitable point of disposal through pipe 8 by closing valve 6 in line 4 and opening the valves in lines 5 and 8. Vapor coming off from the saturant in the tank 3 during the drying of the conduits and the saturation operation is condensed in condenser 44, collected in trap 42 and may be conserved.

In a modified procedure of saturating fibrous conduits, the tank 3 is evacuated and coal tar pitch or other saturant run in at a controlled rate of flow while the vacuum is maintained substantially constant at from 23 to 29 inches of mercury. After the saturant has reached a level such that the conduits are completely immersed, the fibrous conduits are permitted to soak for a period of from 15 minutes to an hour or more while the vacuum is maintained at from 23 to 29 inches. Thereafter, the vacuum is broken and the pitch or other saturant drained from the tank into kettle 1. The conduits remain in tank 3 subjected to the atmospheric pressure therein. The tank is then again evacuated and while maintaining a vacuum of from 23 to 29 inches, pitch is progressively introduced thereinto from kettle 1 until the level of the pitch in the tank 3 rises to a point such that the conduits are completely immersed in the saturant. The vacuum is then again released, the conduits permitted to soak for a brief period in the saturant and thereafter the saturant removed. The saturated conduits are then removed from the tank, permitted to drain, cooled, trimmed and stored for shipment to the consumer.

Saturation of the conduits may be effected by placing them in the top portion of the saturating tank, sealing the cover, opening vent 53 (Fig. 2) and then pumping saturant into the tank until it is filled, the air in the tank escaping through vent 53. Thereafter the vent 53 is closed and the level of the saturant permitted to fall to its barometric height, the bottom of the column being sealed by the liquid in tank 49. Thus a vacuum will be created in the top portion of the tank. The vacuum pump may be operated to maintain a substantially constant vacuum during the evacuation of the pores or voids of the conduits. The conduits may be placed in the base portion of the tank which is filled with saturant while vented to the atmosphere, the vent closed and the column of saturant permitted to fall to create a vacuum in the top portion of the tank. The conduits may then be moved into the evacuated space or permitted to soak in the saturant while vacuum is maintained thereon.

It will be noted that the position of the conduits may be maintained fixed during saturation and the level of the saturant changed so that the conduits are subjected to different pressures of saturant followed by periods during which the conduits out of contact with the saturant are subjected to vacuum or atmospheric pressure, or the conduits may be moved while the level of saturant may be maintained constant, or both the level of saturant and the conduits may be moved relatively to each other. In all procedures, the combination of long time of immersion of the fibrous

conduits in the saturant and the pulsation of the saturant into and out of the side walls of the conduits due to the change in pressure to which the conduit walls are subjected by immersing them in the saturant, subjecting them to vacuum or atmospheric pressure, and then again immersing them in the saturant under vacuum progresses through the cycle of saturating operations and results in thoroughly saturated conduits.

In the specification and drawings the saturation of conduits while maintained in a vertical position has been described and shown. Such position of the conduit has several advantages and is therefore preferred, but it will be understood that the conduits during drying and saturation may be maintained in a horizontal position or a position inclined to the horizontal. Among the advantages of maintaining the conduits in vertical position may be mentioned, the necessary apparatus occupies smaller floor space, ready egress of air from the wall of the conduit is permitted, contact of the saturant with the conduit walls is facilitated, and tendency of the conduit to bend or deform is lessened. Settling of "free carbon" on horizontal surfaces is also obviated.

In accordance with one example of practicing this invention, a conduit containing about 9 per cent moisture and having a porosity of about 1.6 cc. of voids per gram of conduit wall was placed vertically in a saturating tank and dried while a vacuum of 29 inches was maintained in the tank. During the drying, a body of pitch at a temperature of about 320° F. was disposed approximately 2 inches below the bottom of the conduit. The conduit was maintained under vacuum for approximately 2 hours. Thereafter, coal tar pitch of a specific gravity of 1.23 at 60° F., a melting point of 158° F. (cube in water), and having a "free carbon" content of 6.4 per cent, was gradually introduced into the tank by the vacuum maintained in the tank, this vacuum being maintained substantially constant at 29 inches. Thirty minutes were required for the introduction of the pitch into the tank. The conduit was permitted to soak in this pitch maintained at a temperature of approximately 320° F. for four hours and the vacuum was then broken, the pitch drained out of the saturating tank, the conduit drained and cooled.

The conduit on a bone dry basis absorbed 230 per cent saturant. Sections were cut from the top, middle and bottom of the conduit and immersed in distilled water for periods of 24 hours and 48 hours and the following results obtained:

Period of immersion 24 hours	Percentage of absorption of distilled water
	Percent
Section through top	2.37
Section through middle	2.29
Section through bottom	2.08

Period of immersion 48 hours	Percentage of absorption of distilled water
	Percent
Section through top	2.92
Section through middle	2.98
Section through bottom	2.82

In another example, the conduit was slowly immersed vertically during a period of 6 minutes in coal tar pitch of 158° F. melting point, specific gravity of 1.23 at 60° F., maintained at a temperature of 320° F., while maintaining a vacuum of 29 inches of mercury in the tank. The conduit was permitted to soak in the pitch under vacuum for approximately one and one-half hours. Thereafter, it was moved from the saturant into the vacuum space above the body of saturant and maintained under vacuum for about 40 minutes. It was then slowly immersed in the pitch so that it took approximately 12 minutes for the conduit to be completely immersed. After the conduit was thus immersed, the vacuum was gradually reduced to atmospheric pressure, a period of 50 minutes elapsing during the reduction of the vacuum to atmospheric. The conduit permitted to soak at atmospheric pressure for 40 minutes, then withdrawn, drained, and cooled. The conduit prepared in this manner was found to absorb 258 per cent pitch. Sections cut from the top and bottom of the conduits and immersed in distilled water maintained at a temperature of 78° F. for 24 and 48 hours showed water absorption of—

Period of immersion 24 hours	Percentage of absorption of distilled water
	Percent
Section through top	0.48
Section through bottom	0.14

Period of immersion 48 hours	Percentage of absorption of distilled water
	Percent
Section through top	0.78
Section through bottom	0.40

Conduits having approximately 8 per cent moisture and about 1.3 cc. of voids per gram of tube wall were saturated in accordance with this invention and absorbed from about 180 to 190 per cent saturant, based on the bone dry weight of the fibrous conduit. Sections through the top and bottom of the conduit were immersed for 24 hours in distilled water maintained at a temperature of 77° F. with the following results: The section through the top of the conduit absorbed 0.23 per cent of water and the section through the bottom absorbed 0.19 per cent of water.

Conduits having approximately 7 per cent moisture and less than 1 cc. of voids per gram of tube wall (very dense conduits) were saturated in accordance with this invention and absorbed 100 per cent saturant, based on the bone dry weight of the conduit.

In Figs. 1 and 2, the tops of the tubes are indicated as lying just below the surface of the saturant. The upper ends of the tubes are, therefore, subjected to a liquid pressure corresponding to only a few inches depth of saturant, whereas the bottom ends are subjected to a liquid pressure corresponding to approximately nine feet depth of saturant (assuming a tube length of eight feet). In some cases it may be desirable to subject the tubes to considerably greater liquid pressures. This may be accomplished by locating the supports 28 at the bottom or at an intermediate

point in tank 3 and lowering the tubes to that level. In the case in which saturant is allowed to rise to the maximum barometric level (approximately 28 feet depth for pitch of 1.2 specific gravity) the lower ends of the tubes would be subjected to pressure corresponding to approximately 27 feet depth of saturant; the upper ends (assuming 8 foot tube lengths) would be subjected to a pressure of approximately 19 feet of saturant. Pressures considerably greater can, of course, be attained by making tank 3 much deeper, e. g., 60 feet, and pumping in saturant to a depth of approximately 50 feet. Ordinarily, however, it will be more convenient to attain such pressures by pumping saturant or inert gas into the tank 3 until the desired pressure is obtained.

It will be noted that the process of this invention results in a more thorough saturation of absorbent articles than prior processes, requires materially less time for efficient saturation and impregnation, and involves comparatively simple and compact apparatus for its practice. Further, the process of this invention results in substantially uniform saturation from end to end of the article with a material improvement in the waterproofness properties of the fibrous conduits or other porous article saturated. From the examples of saturated conduits given above, it will be noted that the variation in the amount of water absorbed by the top and bottom portions of the conduits, when subjected to the waterproofness test, is from 0.04 per cent to 0.38 per cent, less than half of one per cent variation. It is evident, therefore, that the conduits are substantially uniformly saturated throughout their length. The conduit of this invention has its voids or pores substantially completely filled with coal tar pitch or other saturant employed; has the coal tar pitch or other saturant covering the fibers and bonding with the pitch in the voids. Hence, the fibrous base is effectively waterproofed and protected against corrosion, abrasion, and soil pressures. This is evidenced by the water-absorption tests described above.

The conduit of this invention, saturated with coal tar pitch, absorbs materially less moisture; is more resistant to corrosion, abrasion, and soil pressures; and is of greater tensile and compressive strength than the saturated conduits heretofore known to me. It is also much more resistant to attack by molds, fungi, and all cellulose-destroying organisms.

It is to be understood that this invention is not restricted to the present disclosure otherwise than as defined by the appended claims.

I claim:

1. The process of saturating conduits which comprises immersing the conduits in a body of saturant maintained under vacuum, and while continuously maintaining said vacuum during the impregnation removing the conduits from the body of saturant, subjecting them to vacuum and then again immersing them into the body of saturant.

2. The process of saturating absorbent articles which comprises immersing the articles in a body of saturant maintained in a container having an evacuated space above the body of the saturant, soaking the articles in the saturant while maintaining them stationary within the body of saturant, moving the articles from the body of saturant into the space thereabove and thereafter again immersing the articles into the body of the saturant.

3. The process of saturating porous articles which comprises drying the articles, immersing them in a body of saturant maintained in a container having an evacuated space above the body of the saturant, moving the articles from the body of the saturant into the space thereabove, and thereafter again immersing the articles into the body of the saturant.

4. The process of saturating absorbent material which comprises placing the material in a container, evacuating the container to remove moisture and occluded gas from the absorbent material while maintaining vacuum thereon, progressively introducing the saturant into the container until the material is completely immersed and an evacuated space above the material is provided, moving said material from the saturant into the evacuated space and again immersing the material into the saturant in the container.

5. The process of saturating absorbent material which comprises placing the material in a saturating tank, evacuating the tank while heating it and thus removing moisture and occluded gas from the material, progressively introducing the saturant until the material is immersed and while maintaining vacuum in the tank, soaking the material in the saturant after the introduction of the saturant into the tank and while maintaining the vacuum in said tank, subjecting the material to vacuum conditions out of contact with the saturant without interrupting the vacuum in the tank and then again immersing the material in the saturant.

6. The process of saturating absorbent articles which comprises placing the articles to be saturated in a saturating tank, evacuating said tank to remove occluded gas from said tank, progressively introducing saturant into said tank at a controlled rate of flow while regulating the vacuum so that a substantially constant vacuum is maintained on the articles during the introduction of saturant into the tank, the amount of saturant introduced being sufficient to completely immerse said articles and leave an evacuated space above the level of the saturant, moving the articles from the saturant into the evacuated space thereabove and thereafter again immersing the articles in the body of the saturant.

7. A process of saturating fibrous conduits with bituminous saturant which comprises evacuating the voids of said conduits, progressively introducing bituminous saturant into said voids while maintaining the fibrous conduits under vacuum, permitting the conduits to soak in the saturant maintaining vacuum on the saturant while the conduits soak therein, then subjecting the conduits to vacuum conditions out of contact with the saturant without interrupting the vacuum under which the fibrous conduits are maintained during their soaking and thereafter continuing the introduction of saturant into said voids.

8. A process of saturating fibrous conduits with coal tar pitch which comprises drying the conduits, immersing them in a body of coal tar pitch maintained in a container having an evacuated space above the body of pitch, moving the conduits from the body of pitch into the evacuated space there above, to subject them to vacuum conditions in said space and then again immersing the conduits in said body of coal tar pitch.

9. The process of saturating fibrous conduits with bituminous saturant which comprises heating the fibrous conduits, thereafter while still hot subjecting the fibrous conduits to vacuum thereby removing moisture and occluded gas from the

conduits, thereafter contacting the conduits with bituminous saturant while maintaining vacuum in the saturating zone, without interrupting the vacuum to which the fibrous conduits are subjected while contacted with the saturant, subjecting the partially saturated conduits to vacuum conditions out of contact with the saturant, thereafter again contacting the conduits with the saturant and maintaining them in contact with said saturant until the voids are substantially completely filled with saturant.

10. The process of saturating porous material which comprises progressively contacting the material with liquid saturant while the portions not contacted are subjected to vacuum, after all portions to be saturated have been contacted with saturant and without interrupting said vacuum, moving the partially saturated material out of contact with the saturant, and subjecting it to vacuum maintaining the material out of contact with the saturant while subjecting it to vacuum and thereafter again contacting the material with saturant.

11. The process of saturating fibrous tubes with bituminous saturant which comprises gradually immersing the fibrous tubes in the bituminous saturant, maintaining vacuum on the body of saturant in which the tubes are immersed while continuously maintaining said vacuum, removing the tubes from the body of saturant and subjecting them to vacuum conditions out of contact with the saturant, and thereafter again immersing the tubes in the body of saturant while subjecting to vacuum said body of saturant and the tube walls as they are gradually immersed in the saturant.

12. The process of saturating fibrous conduits which comprises immersing the conduits in a body of saturant maintained in a container having an evacuated space above the body of saturant, soaking the conduits in the saturant while maintaining them within the body of the saturant, moving the conduits from the body of saturant into the evacuated space thereabove, thereafter again immersing the conduits into the body of saturant and removing the saturated conduits from the body of saturant.

STUART P. MILLER.

CERTIFICATE OF CORRECTION.

Patent No. 2,012,968. September 3, 1935.

STUART P. MILLER.

It is hereby certified that error appears in the above numbered patent requiring correction as follows: In the grant and heading to the printed specification, title of invention, for "SATURATING FIBROUS CONDUITS AND PROCESSES OF MAKING SAME" read SATURATED FIBROUS CONDUITS AND PROCESSES OF MAKING SAME; page 3, first column, line 23, for "of" read or; and second column, line 28, after "may" insert the word be; and line 63, for "heater" read heated; page 7, first column, line 73, right section of table, for "2.98" read 2.92; line 74, same section, for "2.82" read 2.88; and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 8th day of October, A. D. 1935.

(Seal)

Leslie Frazer
Acting Commissioner of Patents.