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ELECTRICALLY CONDUCTING FIBROUS CARBON

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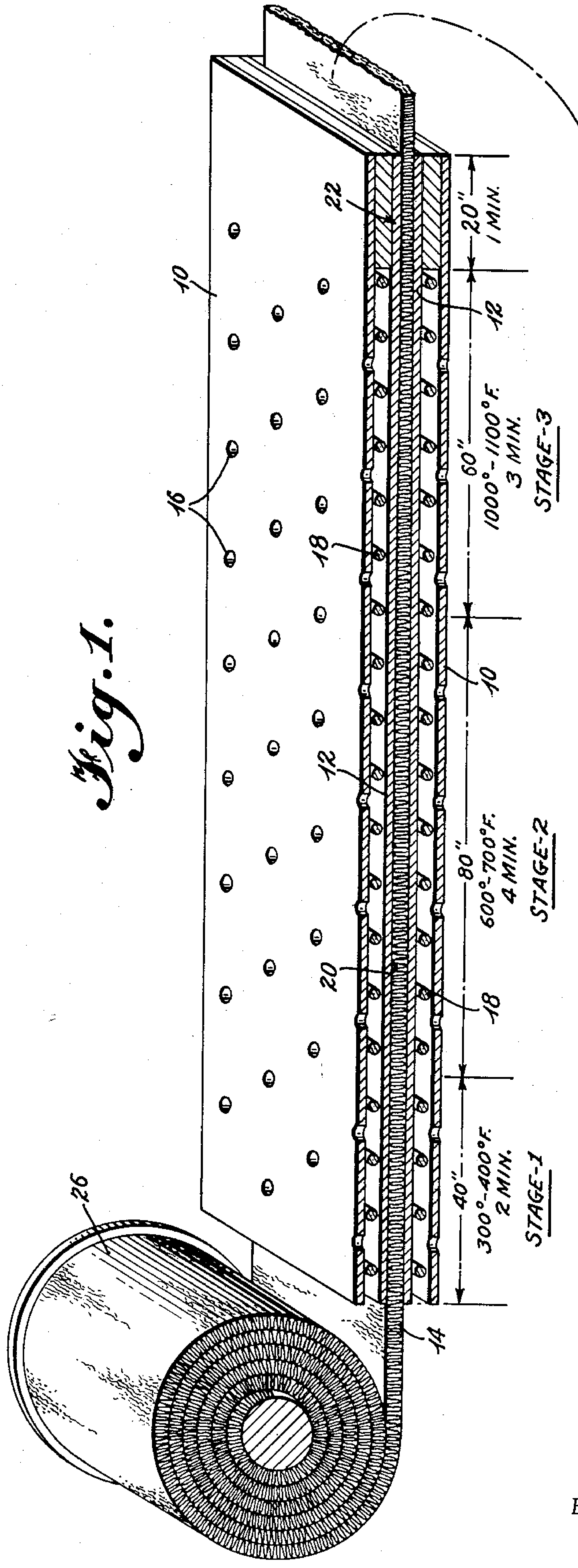
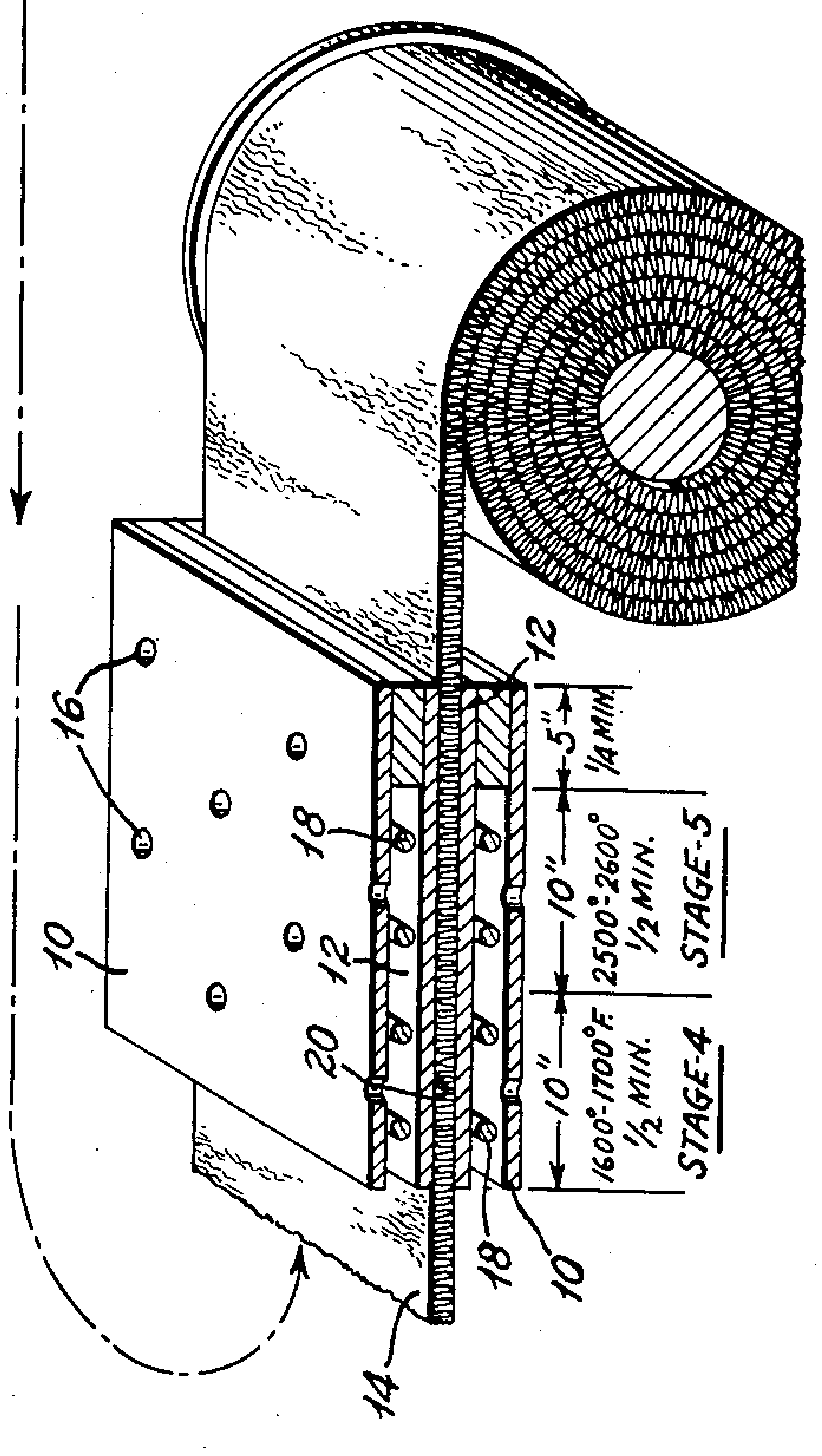


Fig. 1.

Fig. 2.



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ELECTRICALLY CONDUCTING FIBROUS CARBON

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This invention relates to new products and to new methods for their production. More particularly, the present invention relates to new products which, chemically speaking, are composed substantially of pure carbon, and which are derived from cellulosic material in its broadest sense. Still more particularly speaking, the invention is concerned with new methods and new and novel carbonized products which results from heat treatment of cellulosic fibrous materials. Such fibrous materials, whether natural products or synthetic as, for example, in the case of rayon, may be of substantially any variety of source, although preference is for those fibrous materials which are highest in cellulosic content. Additionally, the invention contemplates the production of new products from, and the application of the new methods to products made from cellulosic fibers, e.g., strands, skeins, ropes, fabrics, batting pads, and the like.

Cellulosic materials, generally stated, may be divided into several classes based upon the source of the fibrous material. For convenience in setting forth the scope of the invention particularly as regards the materials which may be employed in producing the new product according to the described method, reference is made to Hackh's Chemical Dictionary, third edition, published by The Blakiston Company, Inc., New York, 1953. This dictionary sets forth four classes of materials as follows:

(1) Seed fibers (87–91% cellulose); as, cotton kapok and other flosses.
(2) Woody fibers (58–62% cellulose); as, pine.
(3) Bast fibers (32–37% cellulose); as, straw, flax, hemp, ramie.
(4) Leaf fibers; as, sisal and manila hemp.

According to this invention, valuable products may be produced from fibrous materials falling in any of the above-listed categories, although as indicated above, preference within the invention is for those fibrous materials high in cellulosic content, such as cotton. It may be said that synthetic materials such as rayon may be equally suitable from the standpoint of their cellulosic content, but such synthetic materials suffer from the disadvantage of their much higher cost, thereby increasing the cost of the resulting products. It will be understood nonetheless that such synthetic materials are contemplated by the invention and may be employed to produce the new products according to the new method to be described in detail presently.

One of the objects of the present invention is to provide a carbon product which has high absorptivity, and which is made from a vegetable product, as differentiated from activated charcoal which is made from wood products.

Another object of the present invention is to provide a carbon product of vegetable origin which has high flexibility and electrical conductivity.

Yet another object is to provide a flexible carbon product which is free from sulphur and substantially free from ash content so that the carbon product may be employed as a filter material for cigarettes and for liquids such as alcoholic beverages and the like without any danger of sulphur contamination of the liquid being filtered.

Other and further objects of the present invention will be apparent from the following description thereof and from the claims appended thereto.

Generally stated, this invention contemplates the treatment of the above-described fibrous materials by a process involving the application of heat in the substantial absence of oxygen and moisture within the temperature range of about 300° F. up to approximately the melting point of substantially pure carbon, namely, about 7200° F. As will be appreciated immediately by those skilled in the art the present process is in extreme contrast to those heretofore employed in producing materials such as activated carbons, which are used as absorbing mediums in various applications, wherein it is seldom that temperatures above 600 or 700° F. are involved and in which cases employ oxygen for the purpose of burning out impurities and forming a desired structural condition within the characoal or activated carbon product. Depending upon the thickness of the material undergoing treatment, which material, for example, may be a textile fabric of any description, the residence time of the fibrous material within the areas of heat application is varied. Thus, for a very thick mat of fibrous material, for example, ½ to 1 inch in thickness of a loosely woven cotton batting the residence time is longer than for a cotton cloth of a few mils thickness. Generally stated, time is in direct proportion to thickness and may, in general, be decreased in particular stages as the temperature therein is increased.

The carbon products produced according to the method described generally above possess entirely new qualities not heretofore present in other carbonized structures. For example, such products are excellent conductors of electricity in contrast to known carbonized products which do not conduct electricity, they have enhanced properties of absorption in that for a given volume they absorb many times the quantity that other activated products absorb. A still further important attribute of such products is that they are flexible in contrast to other carbonized products which are brittle, readily fracturable in their crystalline form and have no tendency at all to remain intact under working stresses. For example, according to this invention a relatively loosely woven mat of fibrous material derived from any of the aforementioned sources and having dimensions something of the order of ½ inch thick by 4 inches in diameter by 10 inches in length may be carbonized according to this invention and the resulting carbonized products will have substantially the same characteristics of flexibility as had the original mat of material, although its strength will be in some instances reduced considerably. Nevertheless, the product may be employed over long periods of time under conditions where it is required to flex without breaking down and falling to pieces. These new flexible products may be bent or shaped into any desired configuration without seriously breaking up cell structure or impairing electrical properties.

In describing the various aspects of the process of this invention, cotton will be used as an exemplary material; however, it will be understood that all of such detail as is described in relation to cotton is equally applicable to fibrous materials of the various sources mentioned hereinbefore and that the invention is not limited to cotton.

According to this invention, it has been discovered that when fibrous materials of the nature mentioned above are heat treated relatively slowly, preferably in stages, and under conditions involving gradual increase of temperature up to a very high value, substantially all of the impurities contained in the original material are removed and a substantially pure carbon product is obtained which retains the original fibrous structure and which, very surprisingly, is a conductor of electricity equal to the finest known and which also may be bent, curved, or flexed while remaining intact as if it were the original fibrous material prior to carbonization. Additionally, the product is highly absorptive to any substance for which activated carbons have been employed heretofore, and, in fact, is much more highly absorptive than known materials compared volume for volume.

Generally stated, the invention involves treating cellulosic fibrous material at a temperature of from approximately 300° F. up to its melting point. During the initial stages of heating when the heat applied is of the lower intensity range, for example, from about 300° F. to about 1500° F., the time of exposure of the material to the hot atmosphere is quite long in relation to the time of exposure of the material to higher temperatures, for example, from about 1500° F. upwards. Thus, in the early stages of heating, the material has an opportunity to carbonize substantially throughout its entire mass under relatively low temperature conditions so that the entire fibrous mass is not destroyed, yet under conditions that a large portion of the volatilizable impurities and materials which naturally go to ash under the conditions employed are acted upon so as to effect their removal or condition them for removal in later washing operations. Strangely enough, it is found that while the carbonized material produced in the early stages of the present process involving temperatures up to about 1450° F. are materials having excellent absorptive qualities, though not as good as are produced by the application of the complete process herein described, such intermediate products cannot be made to conduct electricity to a significant extent. About 1450° F. appears to be the point at which the products begin to conduct electricity and conductivity increases as processing temperature increases. However, the products so produced have the appearance and the flexible characteristics of those products which are produced as the result of the complete process involving the use of much higher temperatures. It is not entirely understood what takes place in the higher temperature carbonization stages although it is known that still further impurities are removed from the product thereby rendering the obtaining of a more pure carbon. However, just what imparts the properties of electrical conductivity to the final products is not entirely understood.

According to the invention, it has been found to be quite important to begin the carbonization in the early stages at a very low temperature and, thereafter, in succeeding steps to increase the temperature up to a value generally within the range of about 1000° F. to about 1500° F., the exact temperature not being particularly critical. The duration of time within which the material is exposed to various increasing levels of heat intensity, similarly, is not highly critical although, as will appear more fully hereinafter, it is permissible to allocate the time of exposure more or less equally between the different heat levels. Carbonization operations involving temperatures up to about 1500° F. are herein referred to as the "first carbonization operation" and reference is made to a "first carbonization zone." Where higher temperatures are involved, reference is made to a "second carbonization operation" or "second carbonization zone." However, there may be a greater number of zones if desired.

The heating zones may be interconnecting and may comprise a unitary structure, the material being treated passing directly from the zones of lower heating to the higher zones without interruption; similarly, the zone may be separated, as will hereinafter appear.

The manner of conducting the carbonization operation is subject to a great deal of variation and it is difficult, if not impossible, to set forth a general statement which will include the many possible variations of temperature, time, type of material being treated, etc., that are workable. It is found possible to impart at least some of the desired electrical conductivity and good absorptivity properties to the object products by exposing the intermediate products of the first carbonization operation to a single zone of heating wherein the temperature may be as low as, for example, 1500° F. and the time of exposure may be quite short, for example, one quarter of a second if the material treated is quite thin. For the same time exposure, temperatures of, for example, 2500° F. produce a product of still better electrical and absorptivity qualities, that is to say, the product would possess a lower resistivity value. Also, heating in several stages in each of which stages the temperatures would increase by stages, for example, from 1500° F. to 3000° F., a product still better in all respects would be obtained. An intermediate carbonized product may be heat treated in the second carbonization zone beginning at 2500° F. and the temperature may thereafter gradually increase up to almost the melting point or lower, with resulting production of excellent products. Still further, one may heat an intermediate product in several stages beginning, for example, in a first stage where the temperature is 1600° F.; the product may then be passed to a second stage where the temperature, for example, may be 1800° F. and still to a third and fourth and fifth zone involving similar increases in temperature. The temperature changes may be much less or more than a 200° difference. For example, the temperature may increase gradually by a few degrees as the product being treated moves through the furnace so that at the end of the second carbonization operation the temperature may be as high as 3000° F. or higher. In other words, the second carbonization may be carried out at a fixed temperature or it may be carried out at a gradually increasing temperature. In all of these variations, the time of exposure similarly may vary, for example, from a fraction of a second to several minutes producing products of the general nature herein desired but varying in quality from fair in various respects to excellent in all respects.

Thus, it will be understood that the phase of the invention designated as the second carbonization operation cannot readily be defined except in a very broad manner; however, as will more readily appear hereinafter, specific examples are provided which will teach interested persons the preferred and various manners of practicing the invention.

Generally, therefore, it may be stated the invention contemplates that intermediate products from the first carbonization zone will be subjected to a further heating operation involving temperatures of at least about 1500° F. extending over a period of time sufficient to impart electrical properties to the end products. The specification of 1500° F. as the starting point for the second carbonization operation is based upon the discovery that only products which have been heated at this temperature or above for a significant time will conduct electricity to a significant extent. According to the invention, it has further been found that, in general, higher temperatures in the second carbonization tend to increase electrical conductivity. Of course, minimum time of exposure must be observed but it is not reasonably possible to establish a minimum time inasmuch as the thickness and density of material being treated is a factor which obviously may be varied at will and which is largely determinative of residence time. However, it may be stated that at suitable temperatures materials of a few mils in thickness may be carbonized in one second or less.

More specifically, in treating, for example, a cotton batting material of about ½ inch in thickness which is a loosely woven material averaging only a few ounces per square foot, carbonization is begun by advancing the material into the interior of a multi-zone furnace affording in a first zone a temperature within the range of about 300° to 400° F. then to a second zone within the range of about 500° to 700°, a third zone affording temperatures within the range of about 800° to about 1500° F., preferably, about 1000° to about 1100° F., and, if desired, additional zones beginning at about 1500° and increasing upwardly in order to cover the high temperature described above as the second carbonization operation. Alternatively, the furnace may be in two or more sections, one section affording temperatures not higher than about 1100° or 1200° F. and the other section providing for the upper temperature ranges of the second carbonization. In the first and lower temperature zone, the material may suitably be exposed for from about 1 to about 4 minutes, preferably about 2 minutes after which it is delivered to the second stage affording temperatures in the range of about 400° to 700° F. where suitably it may reside for a somewhat longer period of time, for example, from about 2 minute to about a 6 minute exposure, about 4 minutes being found excellent after which the material is passed to the third zone affording temperatures in the range of about 700° to about 1100° F. where a similar residence time is found to be appropriate, for example, from about 2 to about 6 minutes, generally about 3 minutes being satisfactory. From this stage, the material may pass directly into the higher temperature sections of the furnace, or, if desired, the thus carbonized material may be quenched in a water bath, preferably a distilled water bath maintained at about room temperature, although the temperature is not critical, wherein it may be soaked for a period of time sufficient to remove any soluble impurities and gentle agitation may be employed to dislodge ash material so that the product is as free as possible of undesired content prior to the time it goes into the final carbonization stages. If desired, without notably adverse effects, this washing or soaking step may be delayed and carried out immediately after the final carbonization operation although it is believed to be preferable to conduct the soaking or washing step in the intermediate stages of the process. If the products are treated in the water bath at an intermediate point, then the products should be dried prior to the introduction into succeeding heating zones so as to avoid the presence of steam in the furnace.

In any event, having completed the steps of the first carbonization operation, the material undergoing treatment is then delivered into the higher temperature furnaces employed in the second carbonization operation. A suitable temperature for initial exposure is of the order of 1500° F. to 1800° F. in which the residence time may vary, usually 15 to 60 seconds being appropriate, suitably about 30 seconds. The material then passes to the second heating zone of the higher carbonization operation where the temperature may be of the order of 2200° F. upwardly as high as desired. Residence time in this zone is similar to that in the first heating zone. Thereafter, the hot fully carbonized material may be passed into a third zone which is not heated where it is allowed to cool for a short while, for example, 15 seconds to a minute usually being sufficient to lower the temperature to a point that the hot carbon will not burst into flames upon being exposed to the atmosphere.

As will be understood, the foregoing heating operations may be carried out in any suitable type furnace. The drawings, which are appended hereto, illustrate the heating portions of a furnace which is found suitable for processing the various forms of cellulosic material according to the foregoing described process. Two figures of drawings are presented, namely, FIG. 1 and FIG. 2 (enlarged for clarity) which are longitudinal sectional views illustrating the furnace in two zones, namely, the first and second carbonization zones, and showing the internal portions in relationship to the material undergoing treatment according to one embodiment of the invention. The structure illustrated in FIGS. 1 and 2, as certainly will be understood, is in practice enclosed in a substantially gas-tight hood except for an inlet and outlet which permit inert gases to be introduced whereby the furnace may be flushed free of air and maintained so during the entire process. The illustration of the drawings is also schematic in other respects which are not important to an understanding of the invention and it is believed that no difficulty will be encountered by those skilled in the art in understanding the operation of the furnace and in constructing one for use in operating according to the process.

Referring to the drawings, numeral **10** denotes outer plates which may be of steel or other suitably heat-resistant material. Numeral **12** denotes inner heating plates between which heating plates a layer of cellulosic material is shown in position as when it is undergoing treatment. Similarly, plates **12** are of steel although this is not critical and they may be supplanted by other suitable material considering the heat that is involved. The material undergoing treatment is indicated by numeral **14**, which material, for purposes of illustration, is a layer of cotton batting having a thickness of the order of about 187.5 mils. Numeral **16** denotes a plurality of gas vents or openings which extend through upper plates **10** and upper plate **12**. These vents are nothing more than small passageways which permit gases generated by the application of heat to the cellulosic material from the heating chamber proper to pass therefrom into a suitably mounted plenum chamber and on out to the atmosphere through the same vent or outlet which permits the escape of air at the time of flushing the furnace free of air. Similarly, gas vents or openings through the lower plates **10** and **12** may be provided, although is not found to be necessary.

Numeral **18** denotes electrical resistance elements located between both pairs of upper and lower plates **10** and **12**. These resistance elements are, of course, connected to a suitable voltage supply and provide heat for the furnace according to that desired for the particular furnace section with which they are associated.

As can be seen from the drawings, upper and lower plates **12** form a heating chamber between their surfaces which chamber is denoted by numeral **20**. In the drawings, the walls of plate **12** are shown as closely confining the cellulosic material which is under treatment; in fact, the walls of plates **12** in this embodiment of the invention are shown to be in contact with the cellulosic material. While physical contact between the material undergoing treatment and the walls of the heating chamber is not necessary, depending upon the type of furnace employed such contact may or may not be desirable. In employing the type of furnace illustrated in the drawings, it is found that somewhat better heating of the material and therefore better uniformity of carbonization throughout the entire mass is achieved. Also, it will be noted that heating chamber **20** of FIG. 1 is somewhat deeper from the entrance end to about the middle of the furnace than it is at the discharge end thereof. This construction is employed so as to maintain the walls of the heating chamber in contact with material as it passes therethrough and undergoes shrinkage as a higher and higher degree of carbonization is accomplished in the first carbonization zone. Shrinkage is generally insignificant in the second zone. It is found that shrinkage takes place in proportion to the actual carbonaceous content of the cellulosic material. For example, in the cotton material shown undergoing treatment, shrinkage may be to the extent of about 25% of the original thickness of the material.

As shown in the drawings, the furnace is broken into two sections, like reference numerals referring to similar parts, FIG. 1 corresponding to the first carbonization zone and FIG. 2 being that corresponding to the second carbonization zone wherein the heretofore mentioned higher temperatures are employed. In practice, the two zones may be made continuous and the material to be treated may pass from the entrance end of the first zone to the discharge end of the second zone without interruption. On the other hand, if desired and as illustrated, the carbonization may take place in a two-zone furnace wherein a washing operation is interposed between the first carbonization zone and the second carbonization zone. If this latter method is employed, then it is desirable to provide the first carbonization zone with a cooling section such as that illustrated in FIG. 2 at numeral **22**. This portion of the furnace is merely an unheated space which permits the material to cool substantially before it is discharged. Means for washing or soaking the material from the first carbonization zone is not illustrated in the drawings; however, as will be obvious, this may include nothing more than a suitable water tank, or a series thereof, if desired, into which the material from the cooling zone of the furnace is discharged immediately upon its exit from the furnace. If a continuous two-zone furnace is employed, suitable washing or soaking means may be provided at the discharge end of the higher temperature portion thereof.

The material to be carbonized in the furnace may be transported therethrough in any suitable manner. In the drawings, a collecting reel denoted by numeral **24** which reel is suitably power driven by means not illustrated. Similarly, a storage reel for fresh material is illustrated in numeral **26** located at the entrance to the first carbonization zone.

As is indicated in the drawings, the first carbonization zone is divided into three sections or stages of successively increasing temperature conditions. Ranging from about 300° up to about 1100°, each temperature stage merges directly with the next succeeding stage. Of course, it will be understood that the resistance elements are so chosen as to provide the desired temperature and that the furnace may be provided with suitable thermometers whereby the temperature may be verified and maintained within the desired bounds at all times. The indications of time on the drawings refer to the residence time of a particular increment of material undergoing treatment and it will be apparent from the indicated approximate length of each stage of each zone of the furnace and the corresponding time relationship that, in the embodiment of the invention illustrated in the drawings, the material is being moved through the entire furnace at a speed of about 20 inches per minute. Of course, it will be understood that the operating characteristics shown in the drawings are merely illustrative of a single embodiment of the invention which has been found to provide a product of outstanding qualities in all respects. The indicated time spans and lengths of each stage of each zone of the furnace is by no means critical and may be altered so as to better suit the invention to the treatment of various types of materials leading to products of particular specifications.

It is believed that the operation of the process of the invention in relation to the furnace will be readily apparent to all interested persons but for the sake of completeness, it may be stated that as exemplary in processing a cotton material according to the embodiment of the invention illustrated in the drawings, and with the heating sections of the furnace in place within a suitable enclosure and having been flushed free of air and maintained under inert atmospheric conditions, the collecting reel is energized and its speed is regulated to draw the material through the furnace at a rate of about 20 inches per minute; the furnace in all of its stages, of course, having been brought up to the temperature indicated in the drawings prior thereto, namely, 300–400° F. in stage one, 600–700° F. in stage two, 1000–1100° F. in stage three, 1600–1700° F. in stage four, and 2500–2600° F. in stage five. The length of each stage in the furnace is correlated with the reel speed so as to provide a residence time in each stage of about two, four, three one-half and one-half minutes respectively. The cooling zone provides about one-fourth minute residence time. Assuming that the final washing and drying operation is not to be conducted prior to collecting upon the collecting reel, the material moves steadily through the furnace and by the time it has reached the final stages of the second carbonization zone, it is fully possessed of the characteristics hereinbefore indicated and the results of the invention are fully accomplished. The material, preferably, is allowed to cool somewhat within the furnace after the power has been shut off prior to the exposure of the atmosphere; however, in material which is not too dense, cooling is quite rapid.

Carbonization products from cotton produced in this manner, and particularly according to the conditions indicated on the drawings, are found to have excellent properties, for example, electrical resistivity of the particular product, when under pressure, of the specific illustration is found to be of the order of 0.827 ohm-cms. as compared with commercial annealed copper at 1.7241 ohm-cms. at 20° C. Flexibility and absorptivity is excellent and the product is substantially free of impurities, notably, without any sulfur content.

Rayon, hemp, flax, and other similar materials are treated in a manner identical with the foregoing example except as to materials other than rayon, the residence time in each of the heating stages is increased by about 50%. Results entirely comparable to those achieved in treating cotton are obtained. Instead of altering residence time, the temperature levels in the several stages, particularly stages three, four and five, may be operated somewhat higher, for example, about 100° F. It is not preferred to apply much higher temperatures in the early stages as this leads to undesired rapid searing in some materials.

Where the washing and final drying operation take place prior to collection upon the collecting reel, the said reel may be located outside of the furnace enclosure and the material may be drawn over suitably mounted rollers from the furnace into a quenching and washing vat, also preferably located outside of the hood of the furnace so as to avoid the presence of possibly deleterious moisture in the furnace.

It is desired to refer once again to the washing or soaking operation in order that its proper place in the operation of the process of the invention will be fully understood. It is found that in treating various materials that the final products will contain different percentages of impurity content which bears a more or less direct relation to the percentage of cellulosic material in the originally chosen material. This being so, in order to remove these impurities to whatever extent may be desirable, the washing or soaking operation may be varied. For example, a hemp or flax material contains a much higher percentage of impurity than does cotton. Accordingly, these materials require more intensive treatment to bring about a desired removal of impurity content. Thus, it will be understood that these materials may be transported through several water baths prior to collection upon the final collecting reel. Alternatively, the materials may be collected upon the reel first and, at a later time, subjected to the desired washing operation and then stored in whatever manner desired. Some materials may be substantially completely free of impurities after a matter of a few moments of soaking or washing and other materials may require as much as 24 hours. Also, the degree of impurity removal will in large measure be dictated by the desired characteristics of the end product. Thus, if a material having highly desirable electrical conductivity and high absorbtivity is the object of the process, it may be found desirable to conduct suitable testing operations on the products whereby to obtain information concerning the impurity content and, thereafter, to carry out such impurity removing washing operations as may be required.

Obviously, for commercial production reasons, a single furnace unifying all of the various temperature zones from the lowest to the highest is desirable in order to facilitate continuous production. However, it is desired to point out that in some instances, it has been found preferable to carry out the impurity removal operation prior to delivering the product of the first carbonization zone to the higher temperature second carbonization zone. Accordingly, where extremely fine products are required, it is recommended that the raw materials be subjected to a first carbonization operation wherein they are heated successively to temperatures of the order of about 1100° to about 1500° F., and then quenched and subjected to the washing operation and finally delivered to a second carbonization zone wherein temperatures conducive to highly desirable electrical conductivity and other properties are present. It has been found that as the temperature in the final stage in the second carbonization zone is increased, the electrical resistivity of the end products are decreased. For example, a product treated otherwise in accordance with the foregoing specific example of the invention but otherwise being subjected to a temperature in the final carbonization zone of the order of about 3000° F., an end product is obtained having electrical conductivity properties fully comparable to those of the best copper conductors. Still higher temperature conditions and careful observance of the impurity factor leads to a substantially perfect conductor.

Reference has been made herein regarding the difficulty of setting precise ranges for temperature and time factors involved in the process of the invention. The matter of residence time is exceedingly difficult to define considering all of the various factors that are involved. It will be appreciated, at this point in the description of the invention, that regardless of how the process is applied, that is to say, whether in the several stages or in a two-step operation involving separate heating zones, residence time is related to the temperatures which are being applied, the thickness of material undergoing treatment, the degree of actual cellulosic content of the fibrous material and, finally, the density of the material which is being processed. In the foregoing paragraphs, it has been indicated in a general way that residence time ordinarily is shorter as the temperatures are higher and, also, that as the material undergoing treatment is thicker, residence time should be longer in order to effect uniform carbonization throughout the material. It will be appreciated that the same is true where one is treating a very dense material, that is to say, that as the material is more dense, residence time needs to be longer. Additionally, where the material undergoing treatment contains a relatively low percentage of cellulosic material, it is desirable to remove other extraneous matter, that is to say, material other than cellulosic content, by volatilization or other processes which take place during the course of the heat treating steps. Thus, where a particular material is high in impurity content, in general, it is advantageous to increase the residence time where it is desired to maintain the temperature constant.

More or less in summary of the matter of residence time in relation to factors of temperature etc., having first established a particular temperature range within which it is desirable to operate, residence time should be sufficient to effect substantially complete carbonization of the fibrous content of the cellulosic material within the first carbonization operation. As to residence time in the second carbonization operation, time there should be sufficient to produce a product having significant electrical conductivity. Also, it may be said that the residence time is sufficient to volatilize a substantial proportion of the impurity content of the material undergoing treatment; however, this accomplishment ordinarily follows and is coincident with the carbonization operation which takes place largely in the first carbonization zone and considerable impurity removal by volatilization may take place in the second carbonization zone although at this point in the process the majority of removable material has been acted upon and reduced to ash or gases. It has been indicated heretofore that the ash content, for the most part, is removed in washing operations.

In the foregoing discussion, considerable emphasis has been placed upon the production of products having desirable electrical properties. It is gratifying to state that while producing products having such electrical characteristics these same products have many other valuable and important applications. For example, it is found that products produced according to the invention have the ability to absorb gases to a remarkable extent. The products are adapted to the fabrication of filtration materials suitable for use in cigarettes which are far superior to any presently known filter material. As a matter of fact, from the standpoint of their use as cigarette filters, some of the new products are found to be somewhat overactive in that they absorb all of the tars, nicotine, and the like present in cigarette smoke and, therefore, remove all taste characteristics of the cigarette. In producing products suitable for use in such environments, it may be found desirable to employ less drastic conditions than those employed in the production of electrical conductors. For example, a greatly superior filter is provided by operating in accordance with the conditions heretofore indicated in connection with the specific embodiment of the invention. In producing such filter materials, rope-like cotton raw material may be delivered to a furnace providing suitably formed cylindrical channels. The rope-like material as it emerges from the furnace and after final processing steps including the washing and drying operation will be in a form suitable for separation into filters of the exact size for use in the cigarette forming operation.

If desired, carbonized fibers produced according to this invention in whatever form may be spun into threads and used as such or they may be woven into fabric.

Where fire is an element for consideration in the use of the new products, it is important to bear in mind that they do not burn under high temperature and do not ignite upon application of flame.

One of the most outstanding characteristics of the products of this invention is flexibility. If a flexible product is delivered to the furnace for treatment, a flexible product will be obtained as the end result. It is found possible to treat material of any preformed characteristics and preserve them in the end product. This facet of the invention provides products of remarkable adaptability to particular requirements. Thus, in employing the products of this invention in electrical applications, the carbonized products may be bent, curved or installed in any electrical circuitry with ease. Inasmuch as the materials have remarkable tensile strength, they may be subjected to considerable flexing and load without deleterious effects. Where the products are employed in audio circuits, for example, high fidelity applications, great advantage is achieved because of the substantial absence of background phenomena normally present.

Additionally, the new products of this invention make excellent filtration materials for liquids, e.g., in processing liquors, and air, e.g., air filters for internal combustion engines; also as filters for fish tanks for removal of cigarette smoke content of the incoming air. For example, engine filters are effective for long periods of time, e.g., many months in salt air, the salt being removed. Such filters may be washed free of the entrapped salty material and reused. They make fine thermal and sound insulators. Many other uses will be noted by those skilled in the art.

One of the very nice attributes of the products herein described is that they are not dirty to the touch as other carbons are. In other words, there is no carbon-black smudge from these products and, therefore, use in products where this is a factor is under no disadvantage.

It is desired to emphasize that the present invention is herein described in its more generally useful forms and that many variations are permissible without departing from the invention. As regards the products herein, useful products possessing the ability to conduct electricity may be obtained by heat treating cellulosic material at a temperature above about 1500° F. sufficiently long to obtain substantially pure carbon and to effect removal of impurities to whatever extent desired, if such are present. Such carbon, however, will not be found to be flexible and they generally require grinding and then compacting to a useful form of electrical conductor. Such products are prepared under the same conditions as other products described herein except that the initial heat at temperatures below 1500° F. is eliminated. Moreover, the new products herein need not be produced from cellulose fibers but may be produced from extracted or other forms of solid cellulose or solid regenerated cellulose in whatever form obtainable.

It should be understood that the present invention extends to the new products described herein and it is intended to claim them as such without regard to methods other than those described herein by which they may be obtained. The present invention extends also to the apparatus herein disclosed in which the new methods are practiced.

Although the present invention has been described with reference to specific embodiments and particular manipulative steps, various changes and modifications obvious to those skilled in the art are deemed to be within the spirit, scope and contemplation of the invention.

This application is a continuation-in-part of application Serial No. 535,750, filed September 21, 1955, now abandoned.

What is claimed is:

1. A process for producing electrically conductive fibrous products from fibrous and substantially pure cellulosic materials selected from the group consisting of strands, skeins, ropes, fabrics and batting pads of said material consisting essentially of the steps of subjecting the said materials in the substantial absence of oxygen to a first heat treatment within the temperature range of about 300° F. to about 1500° F. for a time sufficient to effect substantially complete carbonization of the contained fibers and to volatilize at least a substantial portion of the extraneous matter contained in the said material which are volatilizable at temperatures within the said range; subjecting said material to a second heat treatment at a temperature above about 1500° F. for a time sufficient to render said material electrically conductive.

2. A process as defined in claim 1 wherein said first heat treatment involves a series of stages, each stage affording a higher temperature than the preceding stage.

3. A process as defined in claim 1 wherein the said material is cotton.

4. A process as defined in claim 2 wherein there are five stages in said heat-treating operations, the first of said stages involving temperatures of from about 300° F. to about 400° F., the second of said stages involving temperatures of from about 500° F. to about 800° F., the third said stage involving temperatures of from about 800° F. to about 1500° F., the fourth and fifth of said stages involving temperatures above 1500° F.

5. A process as defined in claim 4 wherein the temperature involved in said fourth stage is within the range of about 1500° F. to about 2500° F. and the fifth said stage is above about 2500° F.

6. A process as defined in claim 5 wherein said material is subjected to a washing operation between the third and fourth said heating stages.

7. A process as defined in claim 5 wherein said material is subjected to a washing operation after the last said heating stage.

8. An electrically conductive fibrous product of substantially pure carbon produced by the method which consists essentially of the steps of subjecting a fibrous and substantially pure cellulosic material selected from the group consisting of strands, skeins, ropes, fabrics and batting pads of said material in the substantial absence of oxygen to a first heat treatment within the temperature range of about 300° F. to about 1500° F. for a time sufficient to effect substantially complete carbonization of the contained fibers and to volatilize at least a substantial portion of the extraneous matter contained in the said material which are volatilizable at temperatures within the said range; subjecting said material to a second heat treatment at a temperature above about 1500° F. for a time sufficient to render said material electrically conductive, said product retaining substantially the original fiber form and flexible characteristics of said fibrous cellulosic material.

9. A product as defined in claim 8 wherein said first heat treatment involves a series of stages, each stage affording a higher temperature than the preceding stage.

10. A product as defined in claim 9 wherein there are five stages in said heat-treating operations, the first of said stages involving temperatures of from about 300° F. to about 400° F., the second of said stages involving temperatures of from about 500° F. to about 800° F., the third said stage involving temperatures of from about 800° F. to about 1500° F., the fourth and fifth of said stages involving temperatures above 1500° F.

11. A product as defined in claim 10 wherein the temperature involved in said fourth stage is within the range of about 1500° F. to about 2500° F. and the fifth said stage is above about 2500° F.

12. A product as defined in claim 11 wherein said material is subjected to a washing operation between the third and fourth said heating stages.

13. A product as defined in claim 11 wherein said material is subjected to a washing operation after the last said heating stage.

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