UNITED STATES PATENT OFFICE.

RUSSELL WILLIAM MUMFORD, OF NEW YORK, N. Y., ASSIGNOR, BY MESNE ASSIGNMENTS, TO DARCO CORPORATION, OF WILMINGTON, DELAWARE, A CORPORATION OF DELAWARE.

DECOLORIZING CARBON AND PROCESS OF MAKING SAME.

1,402,007. Specification of Letters Patent. Patented Jan. 3, 1922.

No Drawing. Application filed March 19, 1918. Serial No. 223,428.

To all whom it may concern:

Be it known that I, RUSSELL WILLIAM MUMFORD, a citizen of the United States, residing at New York, in the county of New York and State of New York, have invented certain new and useful Improvements in Decolorizing Carbons and Processes of Making Same, of which the following is a specification.

This invention relates to decolorizing carbons and processes of making same; and it comprises as a new material a very porous clarifying, purifying and decolorizing carbon of a high degree of efficiency, having the microscopic structure of carbonized fibrous open cane cells but containing somewhat more calcareous ash than is normal to cane charcoal; and it also comprises a method of making clarifying, purifying and decolorizing carbon wherein bagasse from cane mills is finely comminuted and mixed with a calcareous mineral matter and the mixture subjected to carbonization at a temperature gradually increasing to above 600° C.; all as more fully hereinafter set forth and as claimed.

In the usual process of making sugar from sorghum, sugar cane, etc., the cane is submitted to a high degree of pressure by means of rollers and crushers to express the juice, this pressing being repeated a number of times with intermediate additions of water. The final fibrous residue is bagasse or megasse and is nearly free of soluble matter. Its only use is for burning under the boilers. The sugar juice which has been expressed contains many things other than sugar, the so-called "non-sugars", albuminoids, amids, salts, coloring matter, gummy and viscous matter, etc. In part these non-sugars are in solution and in part they are in a state of suspension. It is necessary to remove the non-sugars so far as possible in order to give an efficient and economic manufacture of the sugar from the cane juice. Coloring matters must be removed as far as possible and so must the finely subdivided materials in a state of suspension or semi-suspension. It is necessary in order to produce a high grade sugar not only to have juice as free as possible of coloring matters but also of suspended matters; a juice which is "bright". But these suspended matters are frequently very difficult to remove, many of them being colloidal. The purer the juice that can be made, the better is the quality of sugar and the less is the danger of scaling and clogging of the multiple effects. Cane juice as it comes from the mills is a turbid liquid of more or less acid nature. The turbidity is due to the suspended matters previously mentioned. Included in these suspended matters is usually a relatively large amount of fine cane fragments. To the juice caustic lime, usually in the form of milk of lime is added in amount sufficient to make the juice neutral or nearly so. The limed juice is then heated with or without pressure. Under the influence of heat and lime there is a copious separation which includes and carries with it much of the mechanically suspended matter of the juice. The point where this separation takes place is known as the cracking point. At this point the impurities separate, part going to the bottom as mud and part going to the top as scum. The scums are usually brushed off and the juice allowed to settle, when the clarified juice is drawn off from the settled impurities. The scum and mud are sent through mud presses, these presses forming the mud and scum into press cakes by removal of juice, the removed juice being added to that previously obtained.

This heating treatment of the juice which is known as defecation removes most but not all of the mechanically suspended impurities. The juice is rarely left absolutely clear and bright. The coloring matter is not affected and indeed the defecation operation may add somewhat to the amount. Unless the gummy and coloring matters are removed from the sugar containing liquid by a treatment with boneblack or other efficient decolorant, the sugar finally recovered is colored and of a relatively low grade of purity. However, treatment with boneblack is impractical in a plantation mill, partly because of the great volume of juice containing more or less suspended matter which must be handled and partly because of the usual climatic conditions.

Bagasse contains a relatively large amount of mineral ash and a part of this ash is of such character as to slag or clinker rather readily.

I have found that a cheap and excellent decolorizing agent for clarifying, purifying

and decolorizing raw sugar juices may be made by treating the bagasse with various mineral matters which diminish or impede the slagging or clinkering tendency of the ash constituents; and then charring under carefully regulated conditions. It is difficult to obtain any efficient article of decolorizing carbon by charring bagasse without some such addition of mineral matter; possibly because the natural ash constitutents at the temperatures necessary in carbonizing tend to soften and to clog or glaze the pores. As I have found, and elsewhere described, see Serial No. 167,971, (Patent No. 1,286,187, Nov. 26, 1918), highly efficient chars for decolorizing purposes may be obtained by slowly heating vegetable matters through a wide range of temperatures ending above 600° C. In charring any vegetable material the first result is the production of a charred residue with evolution of tars, gases, and vapors, and these latter again char, giving what may be termed secondary carbon. In other words, in charring any vegetable material there is a primary carbon which represents the structure of the original material more or less nearly; and there is also a production of secondary carbon from primary products. In making charcoal, where it is desirable to obtain a dense material it is the effort to push charring as fast as possible so that the secondary carbon may be formed and remain in the pores of the primary carbon; that is, it is the effort to carbonize the volatile materials in the pores before they have a chance to escape; thereby filling up the pores to that extent. But for a decolorizing carbon which depends for its activity largely upon area of surface it offers, this deposition of secondary carbon in the pores of the primary carbon is highly undesirable. I have found that I can obviate this clogging of the pores of the primary carbon by a slow charring, under certain specified and well regulated conditions as described. By the use of suction or by sweeping a vapor or gas through the charring zone the removal of these volatile products prior to their decomposition in the pores is facilitated.

This process may be directly applied to bagasse, but in so doing the char formed is not as desirable as that formed by treating the bagasse so as to incorporate more mineral matter of a calcareous character in it. The ash then no longer has an injurious effect on the carbon; and highly active decolorizing carbons may be produced which are particularly applicable to raw cane sugar juices. The convenience of this is obvious since it enables the sugar manufacturer to use one of his waste products for purifying his liquors.

In practice I finely comminute the bagasse in any convenient way, comminuting dry or wet as the case may be. The comminuted bagasse, either in a dry state or a moist state, is mixed with a mineral matter capable of preventing the injurious effects of the naturally contained ash constituents of the cane.

The mineral matter to be used with the bagasse may vary but is best of calcareous character. Milk of lime is a useful addition. Another useful addition is a mixture of milk of lime with powdered soluble calcium phosphate (monocalcium phosphate). The two react to form finely divided bicalcium phosphate and tricalcium phosphate. The presence of this phosphate of lime for some reason adds to the decolorizing power. Another material which may be mixed with bagasse is crude wood vinegar (pyroligneous acid containing acetic acid, tars, etc.) mixed with milk of lime. Or commercial brown acetate of lime may be used. A mixture of fine ordinary ground phosphate rock with milk of lime may also be employed.

In using milk of lime alone with bagasse there is the added advantage that the final product or decolorizing carbon may, if the extra lime be not subsequently removed, be used directly not only for decolorizing and purifying but also for neutralizing the cane juice. All cane juice is, as stated, somewhat acid and requires an addition of lime in defecation. In making a decolorizing char from bagasse with an addition of lime, this lime so added remains with the carbon and most of it is available for neutralizing purposes. The lime and the bagasse are partly in a state of mechanical admixture before and after the charring and if lime has been used in making the char in greater proportion than it is desired to have remain in the finished product, the excess in the form of the coarser portion of intermingled lime may be removed by sedimentation in water. Any desired amount of lime can be removed by carefully washing and sedimentation, leaving a carbon with any desired percentage of lime in it. The tendency is for the finer parts of lime to adhere to the carbon.

In using the black composition of carbon and lime produced by the present process for purifying sugar juice, it may be added to the juice before defecation; and in so doing the lime is available for neutralization purposes. With a carbon containing a given amount of lime, enough carbon may be added to effect neutralization. Or a small amount of carbon may be used in conjunction with the milk of lime. It is however ordinarily better in this use of my material to use enough of the carbon to furnish the lime for neutralizing purposes In so adding the carbon to the juice, it may be previously made into a milk or cream with water as is done in making milk of lime and with the same apparatus ordinarily used,

In other uses of my material, I add it to the juice after the first defecation and separation of muds and scums. In so doing, it is better to extract the lime from the carbon as completely as may be.

Decolorizing carbon made by the addition of milk of lime to the bagasse and charring the mixture is so inexpensive that the lime-containing carbon may be simply added to the juice to effect purification, decolorization and neutralization simultaneously, and after filtering the juice to remove the carbon mingled with impurities of defecation, the mud and scum coming from the presses and containing carbon may be thrown away. However, very advantageously, the mud and scum so produced may be added to more bagasse in making up new material thereby in effect recovering the carbon and the lime as well as making a rather better char. There is more or less nitrogen in the scums and in charring a mixture of scums and bagasse, a char is obtained containing more nitrogen than is normal to the char from the same bagasse. The presence of this additional nitrogen in the char is beneficial to its decolorizing properties. The slimes, scums and mud from the defecating tanks in and of themselves give a good decolorizing carbon with or without admixture of bagasse. As a matter of fact they always contain some bagasse anyhow. In grinding cane, more or less fine bagasse always goes forward with the juice, becoming mixed with these scums or muds.

Various other cheap or waste materials, products of the sugar mill capable of carbonizing, may also be mixed with bagasse as, for instance, final molasses, but the addition of milk of lime, etc., is equally advisable.

Presuming a mixture of, say, 70 parts finely comminuted bagasse and 30 parts of lime, as milk of lime, be made as a dough-like mass, and then slowly carbonized in a closed retort vented to allow the free and easy escape of vapors and gases as fast as they are formed, heating is continued until the temperature reaches a point somewhat above 600° C., finishing the carbonization and causticizing the calcium carbonate to obtain lime. The dough-like mass may be formed into lumps or briquets. As stated, suction may be applied to facilitate escape of vapors and gases. Or various vapors or gases may be passed through the retort to expedite the removal of tarry or other harmful vapors which tend to clog the pores during carbonization. Dry steam, products of combustion, etc., may be so employed. It is a useful, expedient to admix more or less ammonia with vapors so passed in. This gives a somewhat more effective carbon; possibly from the retention of nitrogen compounds. Much the same effect may be obtained by mixing tarry liquids from gas works etc., with the bagasse in making the original mixture. In charring, the temperature is brought from 100° C., up to 200° C., producing a progressive drying and carbonization. In this carbonization, water vapor is developed and operates to sweep out other vapors and gases from the pores and the primary carbon absorbs and adsorbs the moisture more or less. As the temperature goes up to a high point, this water or vapor begins to oxidize the carbon to some extent, helping in cleaning out any secondary carbon which may have formed in the pores. The carbon dioxid liberated from the calcium carbonate formed by the milk of lime from the products of distillation does the same thing but is only liberated to exercise this action at a higher temperature. In this reliberation of carbon dioxid caustic lime, or quick lime, is left. The carbon, when drawn from the retort, because of its extensive surface, is apt to take fire unless carefully cooled with exclusion of air. This may be done by blowing through cooled products of combustion, gases or vapors. A useful expedient is to blow in a little steam prior to emptying. The carbon may be dropped into water under exclusion of air. The lime and mineral matters present may be sedimented out as far as desirable. Or the carbon may be washed without sedimenting away much or any of the admixed lime. Where the removal of lime is desired, the bulk of the lime may be removed by sedimenting and the rest removed by any suitable acid, such as hydrochloric acid. The washed product I usually place in retort tubes or the like and thoroughly dry under the influence of heat. The excess gases coming from the carbonizing furnace may be used for this purpose. In furnishing heat for the carbonizing operation, it is convenient to use producer gas made from bagasse. The exact control of temperatures in the charring operation is easier to effect with gas than with coal or other fuel and producer gas made from bagasse furnishes a fuel which is particularly good for this purpose.

The final product obtained by the described process is a more or less coarse powder, the size of granule of course depending largely upon the degree to which the comminution of the bagasse was carried. It is a carbon having open pores and a texture in this respect analogous to the cellular structure of the original bagasse. It has a high degree of purifying and decolorizing power. It is particularly noteworthy in its power of absorbing and adsorbing viscous non-sugars from cane juice. Unlike many of the decolorizing carbons, it has the power of attracting and agglomerating the very fine solid matters in suspension and semi-suspension in sugar juices and giving a

bright or brilliant liquid. It does not itself go into suspension to cause increased turbidity, as is the case with many carbons of otherwise advantageous properties. No subsequent treatment with kieselguhr or the like is necessary to remove suspended matters; either the suspended matters not collected by the carbon or the suspended matters furnished by the carbon itself. It filter presses readily to give brilliant juices. And it may be used in any ordinary modern filter press.

In the employment of material containing lime for purifying raw cane juice, the decolorizing carbon is presented to the juice first while the juice is somewhat acid and continues its action while the juice becomes neutral. The carbon being in the form of porous fibrous cells, forms a filter layer which filters with unusual rapidity.

Where a cane bagasse gas producer is used for furnishing heating gas, there is the production of a considerable amount of tar which may be used in lieu of the gas works tar mentioned in forming decolorizing carbon. It is a useful agglutinant and contributes to the formation of an active carbon.

What I claim is:—

1. A decolorizing and purifying carbon having the microscopic structure of cane bagasse and having a high degree of purifying and decolorizing activity; such carbon containing more free lime than is normal to carbon from said bagasse and not being glazed by sintered ash; and also containing more nitrogen than is normal to carbon from said bagasse.

2. The process of making decolorizing and purifying carbon which comprises carbonizing a mixture of finely comminuted bagasse with calcareous material in a vented retort through which a suitable draft current is passed to facilitate removal of vapors and slowly charring the mixture through a range of temperatures ending above 600°C.

3. The process of making decolorizing and purifying carbon which comprises carbonizing a mixture of finely comminuted bagasse with calcareous material in a vented retort through which a draft current of steam is passed to facilitate removal of vapors and slowly charring the mixture through a range of temperatures ending above 600° C.

4. The process of making decolorizing and purifying carbon which comprises carbonizing a mixture comprising finely comminuted bagasse, calcareous material and defecation residues from cane juice and slowly charring the mixture through a range of temperatures ending above 600° C.

5. The process of making decolorizing and purifying carbon which comprises carbonizing a mixture of comminuted bagasse with defecation residues from sugar cane, such defecation residues comprising carbon from a previous operation and slowly charring the mixture through a range of temperatures ending above 600° C.

6. The process of producing a material adapted for simultaneous decolorization, neutralization and defecation of cane juice which comprises slowly charring a mixture containing cane bagasse and a relatively large amount of caustic lime through a range of temperatures ending above 600° and at a temperature sufficient to causticize calcium carbonate.

7. The process of producing a material adapted for simultaneous decolorization, neutralization and defecation of cane juice which comprises slowly charring a mixture containing cane bagasse, defecation residues and a relatively large amount of caustic lime through a range of temperatures ending above 600° and at a temperature sufficient to causticize calcium carbonate.

In testimony whereof, I affix my signature hereto.

RUSSELL WILLIAM MUMFORD.