

UNITED STATES PATENT OFFICE.

MILTON B. PUNNETT AND RAYMOND A. WHITAKER, OF ROCHESTER, NEW YORK,
ASSIGNORS TO EASTMAN KODAK COMPANY, OF ROCHESTER, NEW YORK, A
CORPORATION OF NEW YORK.

PROCESS OF PRODUCING DECOLORIZING-CARBON.

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To all whom it may concern:

Be it known that we, MILTON B. PUNNETT and RAYMOND A. WHITAKER, citizens of the United States of America, residing at Rochester, in the county of Monroe and State of New York, have invented certain new and useful Improvements in Processes of Producing Decolorizing-Carbon, of which the following is a full, clear, and exact specification.

This invention relates to a process of making decolorizing carbons. One object of this invention is to produce highly efficient decolorizing carbons by relatively simple and inexpensive operations from cheap ingredients. Another object of this invention is to produce carbon which will have a strong decolorizing effect on the usual liquids requiring purification, such as sugar solutions, glue, oils, organic reducing substances like photographic developers, etc., yet will not interfere with the usual filtering and the like subsequent operations. Other objects will hereinafter appear.

We have discovered that decolorizing carbons having the necessary qualities can be made by charring carbonizable material (preferably comminuted) such as straw, sawdust, vegetable ivory nuts, cocoanut shells, cohune nuts, residue of nut-gall-pulp, paper, etc., in the presence or admixture of sulfate salts of which the alkali metal sulfates, like sodium or potassium sulfate, (Na_2SO_4 , K_2SO_4) and sodium or potassium acid sulfate, (NaHSO_4 , KHSO_4) are typical. The range of sulfates available includes many different compounds, but for the sake of cheapness crude forms are used, such as niter-cake containing NaHSO_4 and some free sulfuric acid H_2SO_4 . After charring, the charred material is separated from the residue of the salt and subjected to a second heat treatment at higher temperatures while substantially protected from burning.

By way of example we shall now describe one of the ways in which our process may be carried out. For the carbonizable material we may use comminuted vegetable matter of a woody nature, such as hardwood sawdust. To approximately 100 parts of this by weight there is added 100 parts of an aqueous solution containing 75% niter-cake. The latter is composed chiefly of sodium hydrogen sulfate together with some

free sulfuric acid which is ordinarily left in the cake during the manufacture of nitric acid, in order to make the cake readily removable. This sulfuric acid does no harm and in fact may take a distinct part in the preferred process. Since the niter-cake and the sawdust are both normally waste products, it will be seen that the ingredients are very inexpensive.

The sawdust should take up practically all of the solution, the amount of solution being regulated accordingly and the above proportions being merely illustrative. This mixture is allowed to stand for several hours, two hours being sufficient in some instances, although the time may be prolonged to twenty-four hours without injurious effect.

After standing, the mixture is heated in an open retort in contact with the air to a temperature between 300°C . and 500°C . until the mass is completely charred. Such charring will ordinarily take place in from one-half to one hour, and the end point is conveniently indicated by the fact that the evolution of sulfurous fumes is diminished or substantially ended. During the charring operation the mixture may be in thin layers and should preferably be stirred at frequent intervals.

The charred mass, when sufficiently cooled to permit of such operation, is stirred into the least amount of water that will dissolve the sodium sulfate or other salts resulting from the original niter-cake. This watery mass is then heated to boiling and filtered, thereby separating the charred material from the salts. If desired, the filtrate may be evaporated and the salts recovered by crystallization, but such an auxiliary step is, of course, not necessary.

The charred and purified material is then dried in any preferred way and then finely ground.

Finally the purified and ground, dried, charred material is heated in a closed retort to a temperature above 700°C ., the preferable range of temperature being from 800°C . to 1000°C . This second heating under non-oxidizing conditions or in substantial freedom from contact with the air is generally completed in from 20 to 30 minutes. The material should be sufficiently cooled before the retort is opened and the

carbon exposed to the air, to prevent ignition thereof. The product is then finely ground, although for many purposes this final grinding step is not necessary. The resulting decolorizing carbon has a most excellent and powerful action and does not interfere with subsequent operations, such as filtering, etc.

When straw is used as the comminuted vegetable material, which is carbonizable, it is cut into pieces, say one inch in length. It is then put into a sulfate solution made up with a weight of niter-cake equal to seven times the weight of straw in an equal weight of water. This may stand over night and the straw then be filtered off. The resulting mass is then treated in the same way as the sawdust mixture hereinabove described. This product is excellent.

By a generally similar method useful carbons having varying degrees of decolorizing power may also be made from cocoanut shells, cohune nuts, vegetable ivory nuts, paper, the nut-gall-pulp resulting from the manufacture of pyrogallic acid, etc.

While we have hereinabove disclosed certain ingredients and steps by way of exemplifying our process, our invention is not limited thereto nor to the precise proportions given therein, our experiments indicating that the proportions and temperature ranges may be considerably varied and equivalent ingredients may be substituted without departing from the principle of our invention as defined in the appended claims.

Having thus described our invention, what we claim as new and desire to secure by Letters Patent is:

1. In the process of producing decolorizing carbon the step of charring by heat carbonizable but uncarbonized material in the presence of an acid sulfate salt present in substantial quantity when said charring begins.

2. In the process of producing decolorizing carbon the step of heating a mixture of carbonizable but uncarbonized material and an acid sulfate salt open to the air to a temperature between 300° C. and 500° C.

3. In the process of producing decolorizing carbon the step of charring by heat comminuted vegetable matter in admixture with added sodium acid sulfate.

4. In the process of producing decolorizing carbon the step of heating an intimate mixture of comminuted vegetable matter and sodium acid sulfate to a temperature between 300° C. and 500° C. until there is a substantial cessation in the evolution of fumes.

5. In the process of producing decolorizing carbon the step of heating in thin layers open to the air with stirring an admixture of hardwood sawdust and niter-cake to a

temperature of from 300° C. to 500° C. until the evolution of sulfurous fumes substantially ceases.

6. In the process of producing decolorizing carbon the steps of mixing carbonizable but uncarbonized material with a solution of an acid sulfate salt and drying and charring the carbonizable material in the mixture.

7. In the process of producing decolorizing carbon the steps of soaking comminuted vegetable matter in a strong solution of an acid sulfate salt, drying said matter with the salt admixed therein, and heating the mixture to a temperature between 300° C. and 500° C.

8. In the process of producing decolorizing carbon the steps of absorbing in comminuted vegetable matter a concentrated solution of sodium acid sulfate, drying, and heating to a temperature between 300° C. and 500° C. until the evolution of fumes substantially ends.

9. In the process of producing decolorizing carbon the steps of treating hardwood sawdust with a strong solution of niter-cake, drying, and heating, while open to the air, to a temperature below 500° C. until the evolution of sulfurous fumes substantially ends.

10. The process of producing decolorizing carbon which comprises charring carbonizable material in the presence of an acid sulfate salt, separating the charred material from said salt, and heating the charred material out of contact with the air to a temperature above 700° C.

11. The process of producing decolorizing carbon which comprises mixing the carbonizable material with an acid sulfate salt, heating the mixture between 300° C. and 500° C. to carbonize the material, separating the carbonized material from the salt and heating the carbonized material substantially out of contact with the air, to a temperature above 700° C.

12. The process of producing decolorizing carbon which comprises intimately mixing comminuted vegetable matter and sodium acid sulfate, heating the mixture open to the air to a temperature between 300° C. and 500° C. to carbonize the matter until the evolution of fumes substantially ceases, separating the carbonized matter from the residue of said sulfate and heating said carbonized matter to a temperature between 700° C. and 1000° C. substantially out of contact with the air.

13. The process of producing decolorizing carbon which comprises mixing carbonizable material with a solution of an acid sulfate salt, drying the mixture, charring the carbonizable material in the mixture, washing the residue of said acid sulfate from said charred material, drying said charred ma-

terial and heating said charred material under substantial non-oxidizing conditions to a temperature above 700° C.

14. The process of producing decolorizing carbon which comprises absorbing in comminuted vegetable matter a strong solution of sodium acid sulfate, drying and heating the mixture to a temperature between 300° C. and 500°. to char said matter, washing from the mixture the residue of said sulfate, drying and heating the charred matter to a temperature between 800 C. and 1000° C. substantially out of contact with the air.

15. The process of producing decolorizing carbon which comprises impregnating comminuted vegetable matter with a strong solution of niter-cake, drying and heating the impregnated matter until the diminution of fume evolution indicates that the matter is charred, dissolving out the salts from the charred matter, and heating said charred matter under substantially non-oxidizing conditions to a temperature above 700° C.

16. The process of producing decolorizing carbon which comprises adding to 100 parts of hardwood sawdust 100 parts of a 75 per cent. aqueous solution of niter-cake, allowing the mass to stand from two to twenty-four hours, drying and charring the mass by heating it open to the air in thin layers with stirring to a temperature between 300° C. and 500° C. for one-half to one hour, cooling, adding a small enough amount of water to dissolve the sulfate salts, boiling and filtering, drying the charred residue, grinding the charred residue, heating the charred residue to a temperature between 800° C. and 1000° C. in a closed retort, cooling until said charred residue will not ignite in the air and grinding said residue.

17. The process of producing decolorizing carbon which comprises charring the carbonizable material in the presence of an acid sulfate salt, separating the charred material from said salt, grinding the charred mate-

rial, and heating the charred material substantially out of contact with the air to a temperature over 700° C.

18. The process of producing decolorizing carbon which comprises intimately mixing comminuted vegetable matter and sodium acid sulfate, heating the mixture to char the matter, separating the charred matter from the residue of said sulfate, grinding the charred matter, heating the charred matter to a temperature between 800° C. and 1000° C., and grinding said charred matter.

19. In the process of producing decolorizing carbon the step of charring by heat carbonizable but uncarbonized material while open to the air in the presence of an initially present sulfate salt.

20. In the process of producing decolorizing carbon the steps of mixing carbonizable but uncarbonized material with a solution of a sulfate salt and drying and charring the carbonizable material in the mixture while open to the air.

21. The process of producing decolorizing carbon which comprises charring carbonizable material in the presence of a sulfate salt while open to the air, separating the charred material from said salt and heating the charred material substantially out of contact with the air to a temperature above 700° C.

22. The process of producing decolorizing carbon which comprises mixing carbonizable material with a solution of a sulfate salt, drying the mixture, charring the carbonizable material in the mixture while open to the air, washing the residue of said sulfate from said charred material, drying said charred material and heating said charred material under substantially non-oxidizing conditions to a temperature above 700° C.

Signed at Rochester, New York, this 9th day of May, 1919.

MILTON B. PUNNETT.
RAYMOND A. WHITAKER.