March 26, 1929. A. M. A. STRUBEN 1,706,468

TREATMENT OF CARBONACEOUS MATERIALS

Filed Jan. 15, 1926

Fig. 1.

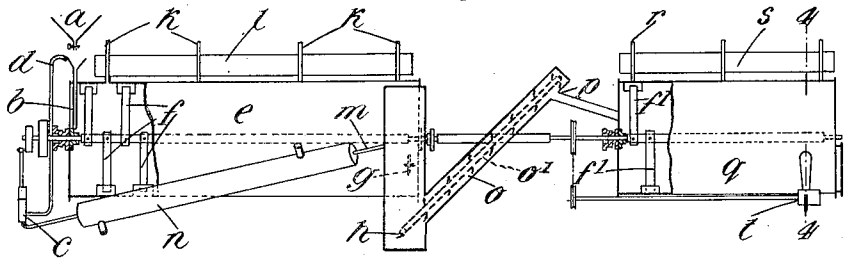


Fig. 2.

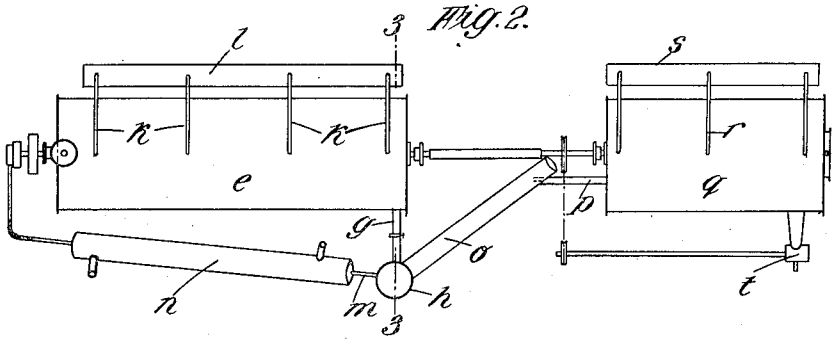


Fig. 3.

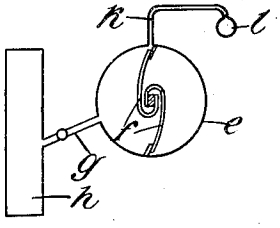
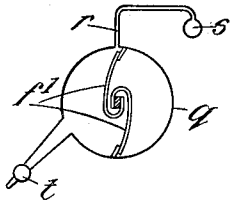


Fig. 4.



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UNITED STATES PATENT OFFICE.

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TREATMENT OF CARBONACEOUS MATERIALS.

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This invention relates to the treatment of solid carbonaceous materials and has for its object the production from such materials of products usually obtained as a result of the carbonization of the material in a retort under the ordinary dry distillation conditions, but without having resort to such usual carbonization treatment.

According to the present invention a process for the distillation of solid carbonaceous materials consists in subjecting to distillation a mixture of the carbonaceous material to be treated with a carrier comprising a substance having a higher boiling point than that of the products it is desired to obtain from the said material, whereby there is obtained a distillation product having a boiling point below that of the carrier.

As examples of the solid carbonaceous materials that may be treated by means of the present process there may be mentioned torbanite, oil shales, coal, brown-coal, peat, lignite, tar-sands, wood and woody-fibres, and the carrier added thereto before the mixture is subjected to distillation consists preferably of a body or bodies of generally similar character to one or other of the distillation products obtained by the ordinary processes of dry distillation of the carbonaceous material; the carrier is either a liquid at ordinary temperatures such as an oil or, when it is desired to produce from the carbonaceous material constituents having a very high boiling point, the carrier to be added may be solid at ordinary temperatures, such as a wax, being reduced to a liquid or viscous fluid in the course of heating it with the carbonaceous material.

Before treatment the carbonaceous material is crushed to a powder or broken to suitable sized lumps. It is then introduced into a still or other suitable distillation apparatus and a quantity of the liquid (or in certain circumstances solid) carrier of higher boiling point than that of the products to be distilled, which may amount to several times that of the carbonaceous material, is mixed therewith. In the case for example of the treatment of torbanite so as to obtain therefrom what ordinarily constitute the lighter fractions obtained in the usual distillation process, such as motor spirit, paraffin oil, and light and medium lubricating oils, the torbanite has added thereto as carrier a liquid comprising the so-called "heavy fractions" obtained from the distillation of carbonaceous materials, whereafter the mixture is heated and distilled until the last desired constituents boiling at a temperature below that of the carrier liquid have distilled off. One method of recovering the carrier which has been added to the carbonaceous material, after the constituents of lower boiling point have distilled off, is to carry the distillation further (with or without the aid of steam and preferably in a different apparatus from that in which the first distillation is carried out) until the carrier added together with the constituents of approximately the same boiling point derived from the carbonaceous material treated also distills off; the carrier together with any constituents derived from the carbonaceous material treated is of course then collected separately from the lower boiling constituents first obtained, and can be again used for treating a fresh batch of the carbonaceous material.

The following example is given by way of illustration of one suitable method for the distillation of torbanite or oil shale in accordance with the process of the present invention.

The torbanite or oil shale is crushed to a coarse powder and together with a carrier comprising a heavy oil or paraffin wax having a boiling point of about 300° C. to 350° C. introduced into a still, the relative proportions of the torbanite or oil shale and carrier being about one part by weight of the former to two to three parts by weight of the latter. The mixture is then subjected to distillation by heating it in a suitable apparatus and a mixture of oils ranging from light motor spirit to heavy oils with a boiling point of about 330° C. and containing a little of the carrier is obtained; this mixture can be regarded as approximating in composition to an ordinary crude oil obtained by distillation according to the ordinary methods. By subjecting this mixture to fractional distillation, its separation into various light and heavy oils can be effected, or instead of collecting the products of distillation from the mixed torbanite or oil shale and carrier in one fraction, the fractions with boiling point between certain limiting ranges can be separately collected, and each such fraction thereupon subjected to any further treatment desired. By this method there can be obtained a yield of mixed crude oils amounting to about 90 gallons per ton of torbanite, whereas by the ordinary dry distillation method the yield is only about 70 to 80 gallons of crude oils per ton, which latter however are considerably

inferior in quality, are of a somewhat higher specific gravity and contain more free carbon than the products obtained by the present process. In carrying out the distillation of the mixed torbanite or oil shale and carrier, it is preferred to collect separately the last portions of the heavy oils which distill off, namely those having a boiling point above about 290° C., and to return these portions to the still (together with the carrier which may be recovered from the residual solid material in a manner referred to hereinafter) where they act similarly to the original carrier in facilitating the distillation of the crude oils from the torbanite or oil shale, and also at the same time undergo a fractionation to some extent so that their lighter constituents distill off and are recovered with the other distillation products. When the temperature of distillation reaches a point, say about 320° C., at which the crude oils have been distilled off as far as practicable, the residual material can be subjected to a further heating (either in the same still but preferably after withdrawal therefrom and in a separate apparatus) up to a temperature of about 360° C. to 370° C., whereby there is distilled off both the carrier originally added to the torbanite or oil shale and the heavy oil or wax obtained from its final heating, a residue of fine carbonaceous material being left. The carrier and products of a similar high boiling point so recovered can be utilized in the distillation of a fresh batch of torbanite, oil shale, or other carbonaceous material, or for any other purpose desired. Instead of subjecting the whole of the residual material to a further heating as just mentioned, it can be withdrawn from the still and allowed to settle, the lighter portions being then returned to the same still for use in the process, whilst only the heavier portions are introduced into a second still wherein they are heated to considerably higher temperatures. Alternatively, instead of distilling the heavier portions, they can be split up into various lighter constituents by any suitable cracking process, or they can be submitted to a suitable separation treatment whereby the oils and solid substances are separated from one another, the former of which may be again employed as carrier and the residual solids treated as a by-product or used as a fuel for supplying heat required in carrying out the process.

One form of apparatus suitable for carrying out the process according to the present invention is illustrated by way of example in the accompanying diagrammatic drawings in which:—

Figure 1 is a part sectional elevation,

Figure 2 is a plan,

Figure 3 is a section on the line 3—3 of Figure 2 and

Figure 4 is a section on the line 4—4 of Figure 1.

The crushed carbonaceous material, for example torbanite or oil shale, to be subjected to the distillation treatment is fed through the hopper *a* into the mixing pipe *b* in which it meets and is mixed with the carrier consisting of hot oil or liquid wax delivered from the pump *c* through the pipe *d*; from the bottom of the mixing pipe *b* the mixture of carbonaceous material and carrier passes into the still *e*, heated in any suitable manner, in which it is continuously agitated by the rotatable blades or paddles *f*. The vapours produced during the distillation pass away through the various off-take pipes *k* and thence to a condensing plant *l*; the various distillates may however be condensed and collected separately if desired. The residual materials consisting of solid carbonaceous materials suspended in the liquid carrier are delivered through the pipe *g* to the settling tank or stand pipe *h* wherein the residual solid carbonaceous material settles out at the bottom and the liquid oil above it is drawn off through the pipe *m* and, after if desired passing through a heat interchanger *n*, returns to the pump *c* whence it is delivered again to the still, the passage of the carrier and solid carbonaceous material suspended therein from the still *e* through the pipe *g* taking place as an ordinary hydraulic flow under the circulating action of the pump *c*. The solid material from the stand pipe is lifted through the elevator shaft *o* by a conveying device *o'* such as a bucket conveyor of any suitable construction whence it is delivered through the pipe *p* to a second still *q*; whilst passing through the elevator shaft a considerable amount of the liquid adhering to the solid material runs back into stand pipe. The still *q*, wherein there are also provided rotatable blades or paddles *f'* for the agitation of the material therein, is heated to a considerably higher temperature than the still *e*, so as to drive off the carrier originally added to the carbonaceous material in the still *e* and products of a similar high boiling point obtained from the said carbonaceous material under the action of the high temperature. The distillates from the still *q* are taken off through the off-take pipes *r* whence they pass to a condenser *s* from which they can be delivered to the pump *c* for reuse in the process, or utilized for any other desired purpose. The residue is discharged from the still *q* through a delivery valve *t*.

It will be understood that in carrying out the present process the distillates are collected and condensed in any usual manner, and they may be subjected to any desired process for further purification or fractionation, although such distillates are generally purer and not contaminated by other products to the same extent as those produced by the ordinary retorting methods.

The process according to the present in-

vention possesses the further advantages over the methods heretofore in use for the production of distillation products from carbonaceous materials that the distillation can be effected at uniform and easily controllable temperatures, whilst by a suitable choice of the carrier added to the carbonaceous material distillates having a boiling point below a predetermined figure can readily be obtained.

What I claim and desire to secure by Letters Patent of the United States is:—

1. A continuous process for the distillation of solid carbonaceous material, such for example as torbanite, oil shales, coal, brown coal, peat, lignite, and tar sands, which comprises suspending the solid carbonaceous material to be treated in a comminuted form in a bath of a hydrocarbon carrier material maintained in bulk in an enlarged zone and having a higher boiling point than that of the desired distillate of the carbonaceous material, the relative amount of the hydrocarbon carrier being sufficient to maintain the solid material in mechanical suspension at the temperature of the distillation and to maintain such suspension in mobile condition during the operation of the process, supplying heat directly to said zone and thereby subjecting the suspension without substantial alteration of the distillate or the carrier to free distillation at a temperature as high as possible while being below that at which the bulk of the carrier normally distills, thereby obtaining a maximum yield of the desired distillation products having a boiling point lower than that of the carrier while distilling over a minimum amount of the carrier, agitating the suspension, removing the distillate, subjecting the solid carbonaceous material remaining undistilled together with associated hydrocarbon carrier to a settling treatment in a separate zone, thereby effecting separation of the hydrocarbon carrier, and returning the carrier to the bath.

2. A continuous process for the distillation of solid carbonaceous material, such for example as torbanite, oil shales, coal, brown coal, peat, lignite, and tar sands, which comprises suspending the solid carbonaceous material to be treated in comminuted form in a bath of hydrocarbon carrier material maintained in bulk in an enlarged zone and having a higher boiling point than that of the desired distillate of the carbonaceous material, the relative amount of the hydrocarbon carrier being sufficient to maintain the solid material in mechanical suspension at the temperature of the distillation and to maintain such suspension in mobile condition during the operation of the process, supplying heat directly to said zone and thereby subjecting the suspension without substantial alteration of the distillate or carrier to free distillation at a temperature as high as possible while being below that at which the bulk of the carrier normally distills, thereby obtaining a maximum yield of the desired distillation products having a boiling point lower than that of the carrier while distilling over a minimum amount of the carrier, agitating the suspension, removing the distillate, subjecting the solid carbonaceous material remaining undistilled together with associated hydrocarbon carrier to a settling treatment in a separate zone, thereby effecting separation of the hydrocarbon carrier, continuously returning the carrier to the bath, and subjecting the residual solid carbonaceous material from the settling zone to further treatment for the recovery therefrom of hydrocarbon carrier still retained by such residual material.

3. A continuous process for the distillation of solid carbonaceous material, such for example as torbanite, oil shales, coal, brown coal, peat, lignite, and tar sands, which comprises suspending the solid carbonaceous material to be treated in comminuted form in a bath of hydrocarbon carrier material maintained in bulk in an enlarged zone and having a higher boiling point than that of the desired distillate of the carbonaceous material, the relative amount of the hydrocarbon carrier being sufficient to maintain the solid material in mechanical suspension at the temperature of the distillation and to maintain such suspension in mobile condition during the operation of the process, supplying heat directly to said zone and thereby subjecting the suspension without substantial alteration of the distillate or carrier to free distillation at a temperature as high as possible while being below that at which the bulk of the carrier normally distills, thereby obtaining a maximum yield of the desired distillation products having a boiling point lower than that of the carrier while distilling over a minimum amount of the carrier, agitating the suspension, removing the distillate, subjecting the solid carbonaceous material remaining undistilled together with associated hydrocarbon carrier to a settling treatment in a separate zone, thereby effecting separation of the hydrocarbon carrier, continuously returning the carrier to the bath, withdrawing the solid carbonaceous residue from the settling zone, draining off the associated hydrocarbon carrier, returning the carrier so drained off to the settling zone, and subjecting the solid carbonaceous material to a second distillation at a higher temperature than that employed for the initial distillation.

4. A continuous process for the distillation of solid carbonaceous materials as in claim 1, in which there is employed a carrier consisting of a liquid hydrocarbon.

5. A continuous process for the distillation

of solid carbonaceous materials as in claim 2, in which there is employed a carrier consisting of a liquid hydrocarbon.

6. A continuous process for the distillation of solid carbonaceous materials as in claim 3, in which there is employed a carrier consisting of a liquid hydrocarbon.

7. A continuous process for the distillation of solid carbonaceous materials as in claim 1, in which there is subjected to distillation about one part by weight of crushed oil shale suspended in two or three parts by weight of a carrier comprising a liquid hydrocarbon having a boiling point of about 300° C. to 350° C. so as to obtain distillation products consisting of a mixture of oils ranging from light motor spirit to heavy oil.

8. A continuous process for the distillation of solid carbonaceous materials as in claim 2, in which there is subjected to distillation about one part of crushed oil shale suspended in two to three parts by weight of a carrier comprising a liquid hydrocarbon having a boiling point of about 300° C. to 350° C. so as to obtain distillation products consisting of a mixture of oils ranging from light motor spirit to heavy oil.

9. A continuous process for the distillation of solid carbonaceous materials as in claim 3, in which there is subjected to the initial distillation in the first distillation apparatus about one part by weight of crushed oil shale suspended in two to three parts by weight of a carrier comprising a liquid hydrocarbon having a boiling point of about 300° C. to 350° C. so as to obtain distillation products consisting of a mixture of oils ranging from light motor spirit to heavy oil.

10. A continuous process for the distillation of solid carbonaceous materials as in claim 1, in which the heavier oils produced during the last stages of the distillation of the carbonaceous material in the bath of carrier are collected separately and returned for re-use.

11. A continuous process for the distillation of solid carbonaceous materials as in claim 2, in which the heavier oils produced during the last stages of the distillation of the carbonaceous material in the bath of carrier are collected separately and returned for re-use.

12. A continuous process for the distillation of solid carbonaceous materials as in claim 3, in which the heavier oils produced during the last stages of the distillation of the carbonaceous material in the bath of carrier in the first distillation zone are collected separately and returned to the aforesaid first distillation zone.

13. A continuous process for the distillation of solid carbonaceous materials as in claim 3, in which the distillates from the second distillation zone wherein the drained solid carbonaceous material is subjected to distillation are condensed and returned to the first distillation zone for re-use as carrier in the process.

14. A continuous process for the distillation of solid carbonaceous material, such for example as torbanite, oil shales, coal, brown coal, peat, lignite, and tar sands, which comprises suspending the solid carbonaceous material to be treated in comminuted form in a bath of a hydrocarbon carrier material maintained in bulk in an enlarged zone and having a higher boiling point than that of the desired distillate of the carbonaceous material, the relative amount of the hydrocarbon carrier being sufficient to maintain the solid material in mechanical suspension at the temperature of the distillation and to maintain such suspension in mobile condition during the operation of the process, supplying heat directly to said zone and thereby subjecting the suspension without substantial alteration of the distillate or carrier to free distillation at a temperature as high as possible while being below that at which the bulk of the carrier normally distills, thereby obtaining a maximum yield of the desired distillation products having a boiling point lower than that of the carrier while distilling over a minimum amount of the carrier, agitating the suspension, removing the distillate, collecting separately the heavier oils produced during the last stages of the distillation of the carbonaceous material in the bath of carrier and returning the said heavier oils for re-use, withdrawing the residue whilst the said residue is in the heated condition and without having been previously cooled, subjecting the said residue so withdrawn to a settling operation in a separate zone so as to separate it into a lighter portion consisting of the liquid carrier and a heavier portion consisting of residual solid carbonaceous materials admixed with carrier, continuously returning the said lighter portion to the bath for re-use, draining the said heavier portion so as to remove therefrom as much of the adherent liquid carrier as possible and returning the liquid carrier so drained off for re-use, subjecting the drained heavier portion to a second distillation and heating it, whilst continuously agitating it, to a considerably higher temperature than that employed for the initial distillation so as to distill off the remaining carrier and any products of approximately the same boiling point as that of the carrier derived from the carbonaceous material, and returning the carrier so recovered and other products of about the same boiling point to the first distillation zone for re-use in the process.

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