

PATENT SPECIFICATION

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- (21) Application No. 46861/76 (22) Filed 10 November 1976
 (23) Complete Specification Filed 17 November 1977
 (44) Complete Specification published 2 May 1979
 (51) INT. CL.² C01B 31/08
 (52) Index at acceptance
 C1A J243 J244 J245 J361 J385 J391 J392
 J393 J394 J3 J424 J453 J454 J461
 J462 J500 J502 J604 J632 J634 J685
 J868

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(54) ACTIVATED CARBON

(71) We, THE UNIVERSITY OF MALAYA, a Body Corporate of Malaysia of Kuala Lumpur, Malaysia, do hereby declare the invention, for which we pray that a Patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to activated carbon and more specifically to the production of activated carbon from nut shells of the oil palm *Elaeis Guineensis*.

Whilst it has previously been suggested that activated carbon could be prepared from oil palm nut shells, see for example M. Alibert, Bull. mat. grasses inst. Colonial Marseille, 1940, 24, 146—14D; and L. Margaillan and J. Bonnefoi Compt. rend. trav. faculte' sci Marseille, 1941, 1, 7—8, no method for the production of activated carbon from oil palm nut shells is known.

The present invention accordingly provides a method for the production of activated carbon and the recovery of volatile substances from the nut shells of the oil palm *Elaeis Guineensis* comprising:—

- (i) subjecting the shells to pyrolytic distillation to produce carbon and to cause volatilisation of the volatile substances,
- (ii) recovering the volatile substances, and
- (iii) activating the carbon at a temperature of at least 800°C.

The production of the activated carbon in two stages viz pyrolysis (carbonisation) followed by activation is advantageous in that the large amount of volatile material present in the oil palm nut shells, which would impart undesirable properties to the carbon if it remained, is removed from the carbon. Further, the process allows the recovery of the volatile material which is itself of value.

Pyrolysis of the shells may suitably be carried out in stainless steel, brass or Pyrex (registered Trade Mark) glass retorts and the charcoal is obtained in a yield of about 33%. The pyrolysis is preferably carried out at 300—

400°C for 1—2 hours.

It is essential that the temperature at which activation of the carbon is carried out must be at least 800°C as at temperatures below this significant activation does not occur.

Activation of the carbon may be carried out by one of the following methods. For all three methods the carbon may conveniently be ground, e.g. either to 5—10 mesh (British Standard Specification series), i.e. granular; or to 100—300 mesh (British Standard Specification series), i.e. powder. In the first method the ground carbon is activated in the absence of air by passing a stream of nitrogen, carbon dioxide or other inert gas preheated to a temperature of above 250°C over the carbon in a furnace at a temperature of at least 800°C for a period of at least 30 minutes. Preferably the temperature of the furnace is from 850° to 900°C and the treatment is carried out for from 60—90 mins. Temperature and time of heating are interdependent, lower temperatures requiring longer heating times.

The second method of activation is carried out by passing a slow stream of steam, preheated to at least 250°C, over a bed of the ground carbon at a temperature of at least 800°C during at least 30 minutes. Preferably a temperature of from 850°—900°C is used during 60—90 mins. although reducing the treatment time to 30 mins. whilst increasing the temperature to 900—950°C produces carbon having no significant reduction in activity. Activation of the carbon at temperatures below 800°C requires extended activation times and results in insufficient activation.

The third alternative activation method consists of soaking the ground carbon in an aqueous solution of zinc chloride, preferably having a concentration of about 1kg/litre, preferably for from 12 to 100 hrs., e.g. 48 hours, followed by heating at a temperature of at least 800°C, preferably from 800° to 850°C, for at least 30 minutes, preferably from 1 to 2 hours. The activated carbon is then washed

TABLE 1

Activating Agent	Temperature	Time	Yield	Specific surface area m ² /g
Steam	800°C	1½ hrs.	60%	1360
Steam	850°C	1½ hrs.	30%	1600
Zinc Chloride	850°C	1½ hrs.	30%	1860

with hydrochloric acid and water and dried at a temperature above 100°C, preferably 150°C.

Under the preferred conditions specified in the three methods described above the following characteristics of the activated carbon products (granular and powder) are obtained:

Yield of carbon from shell charcoal: 30–60%

Bulk density: 0.35–0.45 g/ml

Methylene Blue Absorptivity at 25°C:

200–450 mg/g

Specific Surface area: 800–2000 m²/g

Iodine Absorptivity (25°C) 800–1400 mg/g

Of the three methods of activation the one using steam is most convenient, simple and inexpensive. The above activation processes are conducted batchwise using a muffler furnace or tube furnace but can readily be adapted for a fluidized bed process.

The present invention is advantageous in that it uses readily available oil palm nut shells and the activated carbon produced there from is of high quality.

The activated carbon so produced can be applied to the removal of toxic vapours and gases (including vinyl chloride), the production of high vacuum, the recovery of solvents from air or gases, the absorption of coloured compounds, the absorption of coloured impurities from water, the removal of odours from air and aqueous solutions. The activated carbon was also specially useful for gas or vapour absorption. The activated carbon was also effective in the low temperature (below 100°C) bleaching of palm oil and the deodorisation of palm and other oils, the non-destructive absorption and recovery of carotenes from palm oil and the treatment of oil palm mill effluent.

After use the carbon can be reactivated by steam activation or by heat activation so that it can be reused.

The pyrolytic distillation of oil palm nut shells according to the present invention also provides pyroligneous acid (37% of shell), of which the principal component is crude acetic acid (5%), and phenolic tar (11% of shell). Pure phenol can be obtained from the tar. The phenolic tar was found to be applicable directly or when made alkaline for the preservation of timber wood. It was also found to be a good carbon remover when diluted with one part of dichloromethane and one part of water and the pH adjusted by sodium hydroxide to the range of 9–10. The following Example illustrates the invention.

EXAMPLE

Oil palm nut shells were carbonised by pyrolysis at 300–400°C for a period of 1 to 2 hours. The carbon so obtained was then activated by either the steam or zinc chloride method described above. The conditions of the activation process and the results obtained are given in Table 1.

WHAT WE CLAIM IS:

1. A method for the production of activated carbon and the recovery of volatile substances from the nut shells of the oil palm *Elaeis Guineensis* comprising:—

- subjecting the shells to pyrolytic distillation to produce carbon and to cause volatilisation of the volatile substances,
- recovering the volatile substances, and
- activating the carbon at a temperature of least 800°C.

2. A method as claimed in claim 1 wherein the carbon is ground before activation.

3. A method as claimed in claim 2 wherein the ground carbon is activated in the absence of air by passing a stream of nitrogen, carbon dioxide or other inert gas preheated to a temperature of above 250°C over the carbon in a furnace at a temperature of at least 800°C for at least 30 minutes.

4. A method as claimed in claim 2 wherein the activation is brought about by passing a slow stream of steam, preheated to at least 250°C over a bed of ground carbon at a temperature of at least 800°C for at least 30 minutes.

5. A method as claimed in claim 2, wherein the activation is brought about by soaking the ground carbon in an aqueous solution of zinc chloride followed by heating at a temperature of 800°C to 850°C for at least 30 minutes and then washing with hydrochloric acid and water and dried at 150°C.

6. A method as claimed in claim 3 wherein the carbon is activated at 850° to 900°C for 60 to 90 minutes.

7. A method as claimed in claim 4 wherein the carbon is activated at 850° to 950°C for 30 to 90 minutes, lower temperature requiring longer heating times.

8. A method as claimed in claim 5 wherein the aqueous solution of zinc chloride has a concentration of about 1 Kg/litre.

9. A method as claimed in any one of claims 1 to 8 wherein the pyrolytic distillation is carried out at 300° to 400°C for from 1–2 hours.



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The present invention is advantageous in that it uses readily available oil palm nut shells and the activated carbon produced there from is of high quality.

The activated carbon so produced can be applied to the removal of toxic vapours and gases (including vinyl chloride), the production of high vacuum, the recovery of solvents from air or gases, the absorption of coloured compounds, the absorption of coloured impurities from water, the removal of odours from air and aqueous solutions. The activated carbon was also specially useful for gas or vapour absorption. The activated carbon was also effective in the low temperature (below 100°C) bleaching of palm oil and the deodorisation of palm and other oils, the non-destructive absorption and recovery of carotenes from palm oil and the treatment of oil palm mill effluent.

After use the carbon can be reactivated by steam activation or by heat activation so that it can be reused.

The pyrolytic distillation of oil palm nut shells according to the present invention also provides pyroligneous acid (37% of shell), of which the principal component is crude acetic acid (5%), and phenolic tar (11% of shell).

Pure phenol can be obtained from the tar. The phenolic tar was found to be applicable directly or when made alkaline for the preservation of timber wood. It was also found to be a good carbon remover when diluted with one part of dichloromethane and one part of water and the pH adjusted by sodium hydroxide to the range of 9–10. The following Example illustrates the invention.

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(i) subjecting the shells to pyrolytic distillation to produce carbon and to cause volatilisation of the volatile substances, (ii) recovering the volatile substances, and (iii) activating the carbon at a temperature of at least 800°C.

2. A method as claimed in claim 1 wherein the carbon is ground before activation.

3. A method as claimed in claim 2 wherein the ground carbon is activated in the absence of air by passing a stream of nitrogen, carbon dioxide or other inert gas preheated to a temperature of above 250°C over the carbon in a furnace at a temperature of at least 800°C for at least 30 minutes.

4. A method as claimed in claim 2 wherein the activation is brought about by passing a slow stream of steam, preheated to at least 250°C over a bed of ground carbon at a temperature of at least 800°C for at least 30 minutes.

5. A method as claimed in claim 2, wherein the activation is brought about by soaking the ground carbon in an aqueous solution of zinc chloride followed by heating at a temperature of 800°C to 850°C for at least 30 minutes and then washing with hydrochloric acid and water and dried at 150°C.

6. A method as claimed in claim 3 wherein the carbon is activated at 850° to 900°C for 60 to 90 minutes.

7. A method as claimed in claim 4 wherein the carbon is activated at 850° to 950°C for 30 to 90 minutes, lower temperature requiring longer heating times.

8. A method as claimed in claim 5 wherein the aqueous solution of zinc chloride has a concentration of about 1 Kg/litre.

9. A method as claimed in any one of claims 1 to 8 wherein the pyrolytic distillation is carried out at 300° to 400°C for from 1–2 hours.



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10. A method for the production of activated carbon and recovery of volatile substances from oil palm nut shells substantially as hereinbefore described.

claimed in claims 1 to 10.

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5 11. The activated carbon product whenever prepared by the methods claimed in claims 1 to 10.

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15

12. The volatile substances from oil palm nut shells whenever obtained by the method

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