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COMPLETE SPECIFICATION

Improvements in or relating to processes for the removal of carbon black particles from suspension in an aqueous liquid

We, N.V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ, a company organised under the laws of The Netherlands, of 30 Carel van Bylandtlaan, The Hague, The Netherlands, 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 processes for the removal of carbon black particles from suspension in an aqueous liquid.

According to the present invention a process for the removal of the carbon black particles from suspension in an aqueous liquid includes the steps of contacting the suspension with a water-immiscible auxiliary liquid, which is predominantly a hydrocarbon fraction having a boiling point or boiling range between 190°C and 400°C, at a temperature between 60°C and 95°C, maintaining the resultant mixture in a state of vigorous agitation, and separating the resulting aggregates of carbon black particles and auxiliary liquid from the aqueous phase.

Generally the aggregates formed in such processes will be 2—6 mm in diameter, although by suitable adjustment of the operating conditions, e.g., by varying the amount of auxiliary liquid and the agitation speed, it is also possible to obtain aggregates of larger or smaller dimensions.

The hydrocarbon fraction used as the auxiliary liquid should preferably have a boiling point or boiling range between 190°C and 350°C. If lighter hydrocarbon fractions are used, the vapour pressure at ordinary temperatures will be such that if the pellets are stored in open drums, there may be considerable evaporation, this being both uneconomical and involving a risk of fire or explosion. On the other hand, if a heavier hydrocarbon fraction is used which has a boiling range extending above 400°C, it is difficult to obtain shaped coherent aggregates, as irregular lumps of a buttery consistency generally result. This is particularly the case when the fraction has a high content of asphaltic materials.

Preferably, gas oil is used as the auxiliary liquid but other hydrocarbon oils obtained during the fractional distillation of crude hydrocarbon oils and having a boiling range within the specified limits are also suitable. Thus, use may also be made for example of catalytically cracked cycle oils, of certain lubricating oil fractions and of aromatic extracts. Moreover, it is not essential for the auxiliary liquid to boil entirely within the range specified, the presence of a small quantity, for example 20%, of a petroleum fraction having a boiling point outside the said range being unobjectionable in itself, except insofar as a high content of asphaltic materials interferes with the formation of the desired aggregates so that the presence of such materials in the auxiliary liquid should be avoided.

The quantity of auxiliary liquid used may lie in the range of from 0.7 to 5, and preferably in the range from 1 to 3, parts by weight of hydrocarbon per part by weight of suspended carbon black.

In the process according to the invention, the magnitude of the boiling point or boiling range of the auxiliary liquid enables the aggregates to be formed from aqueous carbon black particle suspensions at higher temperatures than in processes in which say gasoline or benzene is employed. This is particularly important when the process according to the invention is used for the production of aggregates from the hot aqueous suspensions obtained by water-washing gas which is at a high temperature and contains carbon black particles. This occurs, for example, when water washing is used to remove black particles from the gas which contains mainly hydrogen and carbon monoxide, obtained by partial combustion of hydrocarbons with oxygen or gas containing oxygen.

(Price 3s. 6d.)

In the partial combustion of hydrocarbons with oxygen or gas containing oxygen, the formation of carbon black is in general unavoidable. When the gas has to be processed further, for example, for the production of ammonia or town gas, it is necessary to remove the carbon black, and for this purpose a washing treatment with water is preferably used, the gas being cooled at the same time. The aqueous carbon black suspension obtained is therefore usually at an elevated temperature, for example a temperature in the range of 60°C to 95°C. The use of volatile auxiliary liquids is impossible at these temperatures, unless the operation is carried out under an elevated pressure, and this would entail a greater complexity of equipment. It is therefore an important advantage of the present invention that aqueous carbon black suspensions at a temperature within the range 60°C to 95°C may be contacted immediately with the auxiliary liquid without either cooling or an application of increased pressure.

The aqueous carbon black suspension should be vigorously agitated when contacting it with the auxiliary liquid. This agitation is preferably effected by means of a propeller mixer. It has been found to be preferable that the aqueous carbon black suspension should be agitated by rotation in such a way as to create a vortex in the surface of the liquid. The aggregates of carbon black particles and auxiliary liquid then form at the air/water interface and may be readily separated from the aqueous phase by decanting. When a propeller mixer is used, particularly good results are obtained if the propeller rotates at such a rate that its peripheral speed is at least 4 metres per second.

Processes according to the present invention are set out below by way of example.

EXAMPLE I.

The aqueous carbon black suspension used in this example was obtained by washing a gas which consisted mainly of hydrogen and carbon monoxide and had been prepared by partial combustion of a light hydrocarbon fraction, with sufficient water for the resultant aqueous carbon black suspension to contain approximately 20 grams of carbon black per litre.

In a propeller mixer at a temperature of between 80°C and 90°C the aqueous carbon black suspension was contacted with a straight-run gas oil having a boiling range of 204°C—332°C and a specific gravity (at 20°C) of 0.823. The quantity of gas oil used was approximately 1.2—1.5 grams per gram of carbon black. The propeller mixer rotated in such a way that its peripheral speed was 4 metres per sec., a vortex being formed in the surface of the liquid.

Aggregates of carbon black particles and oil were formed having a diameter of 2—4 mm and a water content of 5—8% by weight. They were drawn off continuously from the propeller mixer, together with water freed from most of the finely dispersed carbon black and subsequently separated from the aqueous phase by means of a coarse screen. The resulting aqueous phase was substantially free from carbon black particles, the carbon black content being as low as approximately 0.001% by weight.

Similar results were obtained using an aromatic extract obtained by an Edeleanu process having a boiling range of 210°C—260°C.

EXAMPLE II.

The aqueous carbon black suspension used in this example was obtained by water-washing a gas prepared by partial combustion of a heavy fuel oil.

In a propeller mixer at a temperature of 80°C to 90°C, the aqueous carbon black suspension containing approximately 20 grams of carbon black per litre was contacted with a straight-run gas oil having a boiling range of 204°C—332°C. The speed of the propeller was such as to form a vortex in the surface of the liquid, a peripheral speed of approximately 4 metres per sec. being sufficient for this purpose.

The aggregates of carbon black particles and oil formed which had an average diameter of 2—5 mm and a water content of 5—10% by weight, could be readily separated from the aqueous phase in a manner similar to that described in Example I. The quantity of gas oil used was 2.2—3 grams per gram of carbon black.

Similar results were obtained using a light catalytically cracked cycle oil having a boiling range of 210—300°C, used in a quantity of 2.8—3.2 grams per gram of carbon black.

The aggregates of carbon black particles and oil formed by processes according to the present invention are coherent and may be handled easily as compared with powdered carbon black. They may be used as a fuel, for example in a boiler, or may be processed to make use of the carbon black they contain.

WHAT WE CLAIM IS:—

1. A process for the separation of carbon black particles from suspension in an aqueous liquid which includes the steps of contacting the suspension with a water-immiscible auxiliary liquid which is predominantly a hydrocarbon fraction having a boiling point or boiling range between 190°C and 400°C at a temperature between 60 and 95°C, maintaining the resultant mixture in a state of vigorous agitation, and separating the resulting aggregates of carbon black particles and auxiliary liquid from the

In the partial combustion of hydrocarbons with oxygen or gas containing oxygen, the formation of carbon black is in general unavoidable. When the gas has to be processed further, for example, for the production of ammonia or town gas, it is necessary to remove the carbon black, and for this purpose a washing treatment with water is preferably used, the gas being cooled at the same time. The aqueous carbon black suspension obtained is therefore usually at an elevated temperature, for example a temperature in the range of 60°C to 95°C. The use of volatile auxiliary liquids is impossible at these temperatures, unless the operation is carried out under an elevated pressure, and this would entail a greater complexity of equipment. It is therefore an important advantage of the present invention that aqueous carbon black suspensions at a temperature within the range 60°C to 95°C may be contacted immediately with the auxiliary liquid without either cooling or an application of increased pressure.

The aqueous carbon black suspension should be vigorously agitated when contacting it with the auxiliary liquid. This agitation is preferably effected by means of a propeller mixer. It has been found to be preferable that the aqueous carbon black suspension should be agitated by rotation in such a way as to create a vortex in the surface of the liquid. The aggregates of carbon black particles and auxiliary liquid then form at the air/water interface and may be readily separated from the aqueous phase by decanting. When a propeller mixer is used, particularly good results are obtained if the propeller rotates at such a rate that its peripheral speed is at least 4 metres per second.

Processes according to the present invention are set out below by way of example.

EXAMPLE I.

The aqueous carbon black suspension used in this example was obtained by washing a gas which consisted mainly of hydrogen and carbon monoxide and had been prepared by partial combustion of a light hydrocarbon fraction, with sufficient water for the resultant aqueous carbon black suspension to contain approximately 20 grams of carbon black per litre.

In a propeller mixer at a temperature of between 80°C and 90°C the aqueous carbon black suspension was contacted with a straight-run gas oil having a boiling range of 204°C—332°C and a specific gravity (at 20°C) of 0.823. The quantity of gas oil used was approximately 1.2—1.5 grams per gram of carbon black. The propeller mixer rotated in such a way that its peripheral speed was 4 metres per sec., a vortex being formed in the surface of the liquid.

Aggregates of carbon black particles and oil were formed having a diameter of 2—4 mm and a water content of 5—8% by weight. They were drawn off continuously from the propeller mixer, together with water freed from most of the finely dispersed carbon black and subsequently separated from the aqueous phase by means of a coarse screen. The resulting aqueous phase was substantially free from carbon black particles, the carbon black content being as low as approximately 0.001% by weight.

Similar results were obtained using an aromatic extract obtained by an Edeleanu process having a boiling range of 210°C—260°C.

EXAMPLE II.

The aqueous carbon black suspension used in this example was obtained by water-washing a gas prepared by partial combustion of a heavy fuel oil.

In a propeller mixer at a temperature of 80°C to 90°C, the aqueous carbon black suspension containing approximately 20 grams of carbon black per litre was contacted with a straight-run gas oil having a boiling range of 204°C—332°C. The speed of the propeller was such as to form a vortex in the surface of the liquid, a peripheral speed of approximately 4 metres per sec. being sufficient for this purpose.

The aggregates of carbon black particles and oil formed which had an average diameter of 2—5 mm and a water content of 5—10% by weight, could be readily separated from the aqueous phase in a manner similar to that described in Example I. The quantity of gas oil used was 2.2—3 grams per gram of carbon black.

Similar results were obtained using a light catalytically cracked cycle oil having a boiling range of 210—300°C, used in a quantity of 2.8—3.2 grams per gram of carbon black.

The aggregates of carbon black particles and oil formed by processes according to the present invention are coherent and may be handled easily as compared with powdered carbon black. They may be used as a fuel, for example in a boiler, or may be processed to make use of the carbon black they contain.

WHAT WE CLAIM IS:—

1. A process for the separation of carbon black particles from suspension in an aqueous liquid which includes the steps of contacting the suspension with a water-immiscible auxiliary liquid which is predominantly a hydrocarbon fraction having a boiling point or boiling range between 190°C and 400°C at a temperature between 60 and 95°C, maintaining the resultant mixture in a state of vigorous agitation, and separating the resulting aggregates of carbon black particles and auxiliary liquid from the

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aqueous phase.

2. A process as claimed in claim 1 in which the auxiliary liquid is a gas oil.

3. A process as claimed in either claim 1 or claim 2, in which 0.7—5 parts by weight of auxiliary liquid are used per part by weight of carbon black particles in the suspension.

4. A process as claimed in claim 3 in which 1—3 parts by weight of auxiliary liquid are used per part by weight of carbon black particles in the suspension.

5. A process as claimed in any one of the preceding claims in which the vigorous agitation is effected by a propeller mixer, which is operated to create a vortex in the surface of the liquid within it.

6. A process as claimed in claim 5, in which the peripheral speed at the extremity of the propeller is at least 4 metres per second.

7. A process for the removal of carbon black particles from suspension in an aqueous liquid substantially as hereinbefore described in the examples.

8. Aggregates of carbon black particles and auxiliary liquid whenever obtained by a process as claimed in any one of the preceding claims.

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