

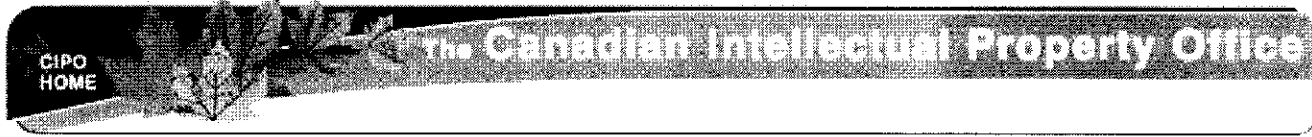


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12/20/2001 - 08:11:58

(11) CA 928072

(12) Patent:

(54) PROCESS FOR THE PREPARATION OF GASES CONTAINING HYDROGEN AND CARBON MONOXIDE BY PARTIAL OXIDATION

(54)

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(74)		
(45)		June 12, 1973
(22)		
(43)		
(52)		48/29
(51)		N/A
		No
(30)		None

N/A
Unknown

*** Note: Data on abstracts and claims is shown in the official language in which it was submitted.

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The invention relates to a process for the preparation of gases containing hydrogen and carbon monoxide by partial oxidation of a heavy hydrocarbon oil-containing liquid feed and subsequently washing of the gases so produced with water resulting in the formation of an aqueous suspension containing soot and ash particles.

The use of heavy hydrocarbons such as residual fractions as a feed for the preparation of gases containing hydrogen and carbon monoxide by means of partial oxidation offers economic advantages because the price of this feed is relatively low. Such feeds may, however, contain metal compounds which upon combustion form ash particles, which, in a finely divided form, are entrained together with soot by the gas stream. Upon washing of the said gas stream the ash particles get into the aqueous suspension. If it is desired to recover the soot from the aqueous suspension with a view of incorporating it into the feed for the partial combustion process ash particles are to be prevented from getting into the soot recovered, because in that case ash particles will accumulate in the combustion process, which may lead to the formation of metal deposits in the process equipment in places where this is undesirable.

It is also undesirable that ash particles should remain in the water phase, because in the latter case this water phase cannot be recirculated in the process for re-use. This would entail the necessity of continuously supplying fresh water, which is expensive. Furthermore, draining of contaminated water is often a source of difficulties.

The invention now indicates in what way the difficulties mentioned can be overcome.

The invention therefore relates to a process for the preparation of gases containing hydrogen and carbon monoxide by partial oxidation of a heavy hydrocarbon oil-containing liquid feed and subsequently washing of the gases so produced with water resulting in the formation



of an aqueous suspension containing soot and ash particles, which process comprises the following steps:

- 5 a) soot is recovered from the said aqueous suspension by means of agglomerating the said soot with a hydrocarbon oil as an auxiliary agent and separating the soot agglomerates formed from an aqueous phase, the hydrocarbon oil used as the auxiliary agent being lighter than the heavy hydrocarbon oil contained in the feed for preparing the gases by means of partial oxidation,
- 10 b) ash particles present in the said aqueous phase are removed therefrom by means of flotation after separation of the soot agglomerates, producing purified water,
- c) at least part of the soot agglomerates separated are incorporated into the feed for preparing the gases, and
- 15 d) at least part of the purified water is used again for washing the gases produced.

It has been found that if a hydrocarbon oil which is lighter than the one used as a feed for the partial combustion process, is applied as an auxiliary agent the soot may be agglomerated from a suspension containing not only soot, but also ash particles. Under the influence of the said auxiliary agent there is a preferential agglomeration of soot particles, only few ash particles being incorporated into the growing soot agglomerates during agglomeration.

25 The ash particles combine with other ash particles or with soot particles to a slight extent only, if at all.

The soot agglomerates can be separated from the water phase containing the ash particles in a simple way, for instance, with the aid of a sieve. They may be used for the preparation of fresh feed. In this case it may be necessary first to dry the agglomerates, particularly when the temperature of the other components of the feed is high, as through the resulting sudden evaporation of the water present in the said agglomerates foam would be formed.

35 After the soot agglomerates have been separated, the ash particles, which are still suspended in the water,

can now be removed by flotation, after which the purified water obtained can be used again for washing the gases produced. Flotation may be effected by mixing the aqueous phase with a flotation agent and by passing finely divided gas, e.g. air, through the said phase. The suspended material then passes into a layer of foam and can subsequently be removed and be used, for instance as starting material for the preparation of metals.

The extent to which the separation between soot and ash particles proceeds in the agglomeration step with the auxiliary agent can be improved in several ways. Thus, the affinity of the ash particles for an auxiliary agent can be reduced by passing air or an oxygen-containing gas in finely divided form through the suspension prior to the addition of the auxiliary agent.

Besides the hydrocarbon oil added as an auxiliary agent and serving as a binding agent, an auxiliary substance with surface-active properties which, for instance, renders the soot particles hydrophobic and oleophilic, may be added.

The affinity of the auxiliary agent for the soot also depends on its viscosity. Preferably, the viscosity of the hydrocarbon oil used as auxiliary agent during the agglomeration is lower than 1000 cS. It is very suitable if the auxiliary agent is a distillate fraction, for instance, a gas-oil or kerosine fraction. An auxiliary agent with a low viscosity wets the soot particles more preferentially and promotes the formation of agglomerates with a low content of ash particles.

A special advantage of many of the lighter distillate fractions as auxiliary agent is that the average soot agglomerates formed are lighter in weight than the suspending water and hence will have a smaller chance of coming into contact with the ash particles, which as a rule are heavier. In this case it is very suitable if the agglomeration is effected in a cylindrical contacting apparatus which has been provided with a cylindrical rotor. In the space between the cylindrical wall of the

apparatus and the rotor a centrifugal field develops in which the agglomerates accumulate near the wall of the rotor.

A suitable flotation agent is an N-alkyl-1,3-propane diamine, in which the alkyl group is derived from coconut oil, soy-bean oil or tall oil. Very good results were obtained with a compound in which the alkyl group is derived from tall oil,

The flotation can be effected continuously and in at least two steps. In the continuous embodiment a device of comparatively small size which requires little attention and can easily be automated, will suffice. By effecting the process in at least two steps, the requirements specified for the purity of the water can always be met.

10 A particularly suitable device for carrying out the flotation step are plate separators. Such separators may have tilted or tiltable plates. Plate separators may be defined as an apparatus comprising a plate assembly in which a number of corrugated or zigzag plates are interconnected in parallel in such a way that the liquid can flow between them and that the ridges and valleys of one and the same plate extend in a direction parallel to the direction in which the liquid will flow, the ridges and valleys of the various plates being vertically situated above or below the respective ridges and valleys of the other plates. Generally such a plate assembly is situated in a reservoir having an inlet for the aqueous phase to be treated and at least two outlets,
20 one for the liquid treated and one for the material separated from the said aqueous phase. Separators of the above type are for instance described in U.K. Patent Specifications Nos. 1,130,620 and 1,154,070.

The advantage of carrying out the flotation in a plate separator is that any soot particles or hydrocarbon oil used as an auxiliary agent remaining in the aqueous phase after separating therefrom the soot agglomerates formed will be removed simultaneously with the ash particles.

The invention will now be elucidated with the aid of a figure in which an embodiment of the process has been represented in a diagram.

5 In the figure 1 represents a reactor into which a feed stream 2 containing a hydrocarbon and free carbon and an oxygen stream 3 or a gas stream containing free oxygen is introduced. In addition, a stream of steam 4 can be supplied. The gas stream 5 formed in reactor 1 by partial oxidation of the feed is brought into indirect contact with a stream of water 7 in heat exchanger 6 whereby steam 8 is formed. 10 The cooled gas stream 9 is brought into direct contact with a stream of water 11 in contact device 10. Here a more deeply cooled gas stream 12 freed from soot and ash particles is obtained. An aqueous suspension of soot and ash particles 13 is brought into contact with a stream of auxiliary agent 16 in an agglomeration device 14, which is provided with a stirrer 15. From a stream 17 consisting of water with soot agglomerates and ash particles which agglomerated to a slight extent only, if at all, the soot agglomerates are separated 20 in a separating device 18 and returned via line 19 to a device 20 for the preparation of the feed stream 2. The other components required for the preparation of the feed constitute stream 21.

25 The suspension of ash particles 22 issuing from separating device 18 is subjected to flotation in the flotation devices 23 and 24 successively. A stream of flotating agent 25 is added to the suspension and a stream of air 26 to the flotation devices. The ash particles separated from the suspension are withdrawn from the flotation devices via 27. 30 A stream of purified water 11 is passed to contact device 10, any water not being used being discharged via 28.

THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE
PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

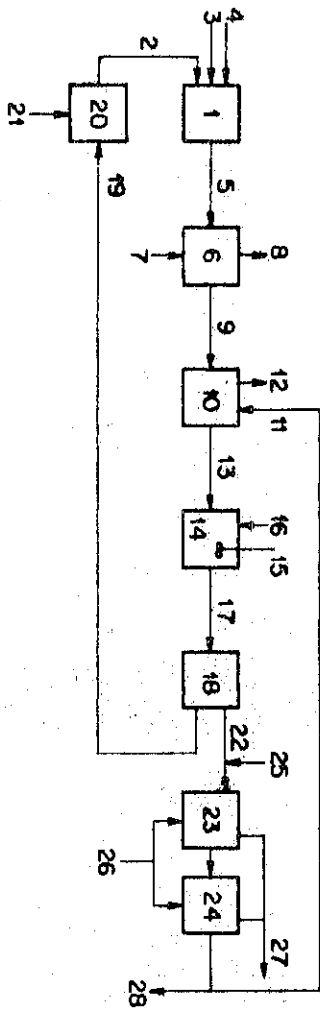
1. A process for the preparation of gases containing hydrogen and carbon monoxide by partial oxidation of a heavy hydrocarbon oil-containing liquid feed and subsequently washing of the gases so produced with water resulting in the formation of an aqueous suspension containing soot and ash particles which process comprises the following steps: (a) soot is recovered from the said aqueous suspension by means of agglomerating the said soot with a hydrocarbon oil as an auxiliary agent and separating the soot agglomerates formed from an aqueous phase, the hydrocarbon oil used as the auxiliary agent being lighter than the heavy hydrocarbon oil contained in the feed for preparing the gases by means of partial oxidation, (b) ash particles present in the said aqueous phase, are removed therefrom by means of flotation after separation of the soot agglomerates, producing purified water, (c) at least part of the soot agglomerates separated are incorporated into the feed for preparing the gases, and (d) at least part of the purified water is used again for washing the gases produced.
2. A process according to claim 1, in which the viscosity of the hydrocarbon oil used as an auxiliary agent is lower than 1000 cS.
3. A process according to claim 1, in which the hydrocarbon oil used as an auxiliary agent is a distillate fraction.
4. A process according to claim 1 in which flotation is effected by the addition of a flotation agent and passing a finely divided gas through the aqueous phase.
5. A process according to claim 4, in which the flotation agent is an N-alkyl-1,3-propane diamine, the alkyl group whereof is derived from coconut oil, soy-bean oil or tall oil.
6. A process according to any one of claims 1 to 3 in which flotation is effected continuously and in at least two steps.

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7. A process according to any one of claims 1 to 3, in which flotation is carried out in a plate separator.

8. A process according to any one of claims 1 to 3 in which prior to agglomerating the soot finely divided air or an oxygen-containing gas is passed through the aqueous suspension.





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