Sept. 13, 1960 D. D. DUNLOP ET AL 2,952,356

CONTROL OF PARTICLE SIZE DISTRIBUTION IN FLUID COKING OF PETROLEUM OILS

Filed April 12, 1954 2 Sheets-Sheet 1

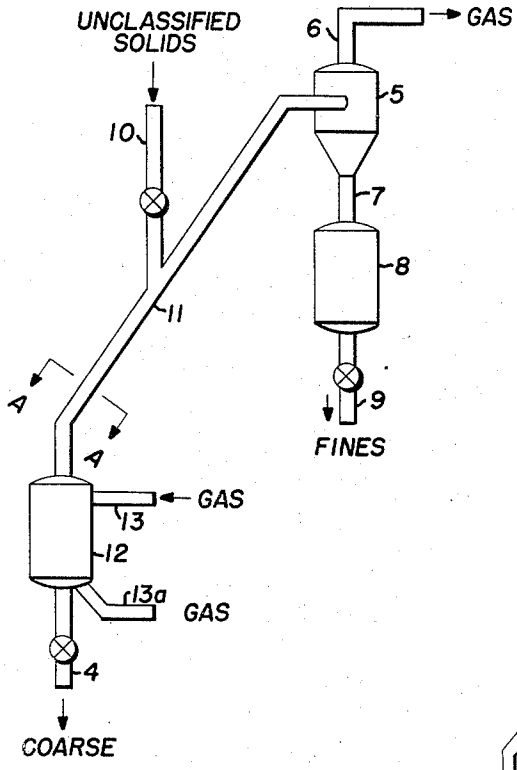


FIGURE-I

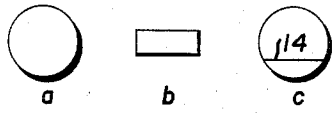


FIGURE-II

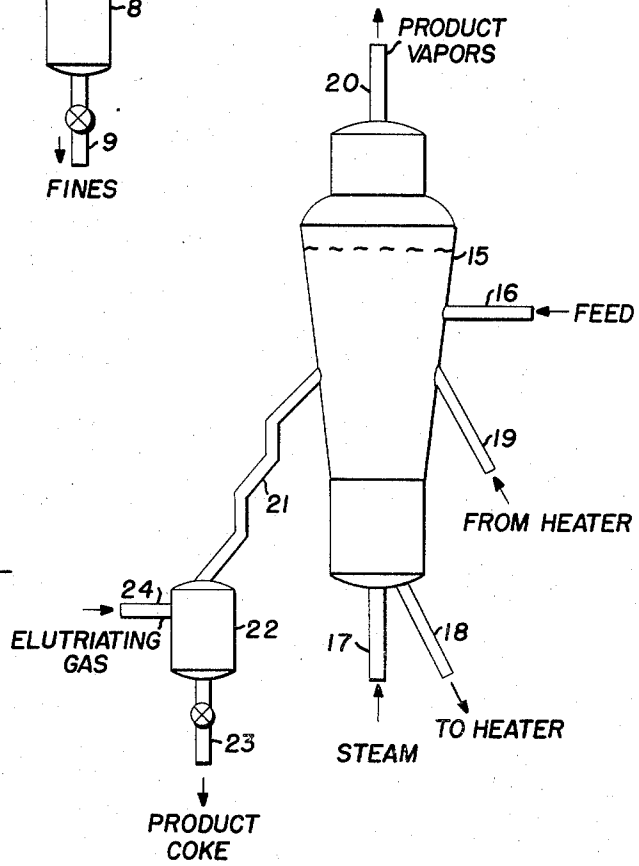


FIGURE-III

Donald D. Dunlop
Robert S. Whiteley Inventors
Werner A. Bauch

By L. Chasan Attorney

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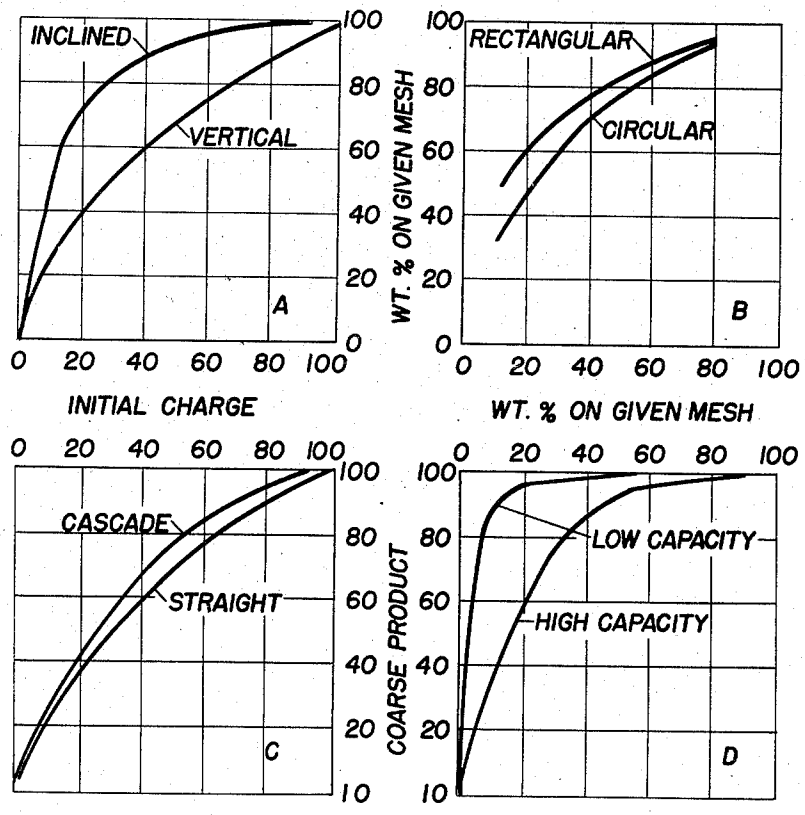


FIG.-IV

Donald D. Dunlop
Robert S. Whiteley
Werner A. Bauch
Inventors

By L. Chasan Attorney

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CONTROL OF PARTICLE SIZE DISTRIBUTION IN FLUID COKING OF PETROLEUM OILS

Donald D. Dunlop, Werner A. Bauch, and Robert S. Whiteley, Baton Rouge, La., assignors to Esso Research and Engineering Company, a corporation of Delaware

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3 Claims. (Cl. 209—136)

This invention relates to the art of elutriating solids. It pertains to a method and apparatus for classifying particulate solids according to size. An application of this invention is concerned with controlling the size distribution of finely divided heat carrying solids used in hydrocarbon oil fluid coking systems.

Recently it has been proposed to coke hydrocarbon oils such as petroleum residua by injecting them into a coking vessel containing a fluidized bed of high temperature finely divided solids, e.g. coke, sand, spent catalyst and the like. In the coking vessel, the oil undergoes pyrolysis in the fluidized bed, evolving lighter hydrocarbons and depositing carbonaceous residue on the solid particles. The necessary heat for the pyrolysis is supplied by circulating a stream of the fluidized solids through an external heater, generally a combustion zone, and back to the coking vessel. The solids, which have had carbon deposited on them during the coking, are partially combusted in the heater.

Among the problems exposed by the development of this fluid coking process is one that apparently is peculiar to this type of operation, i.e., the problem of counteracting the particle size growth of the circulating solids in the coking system. Because more carbon is produced by the coking process than is required to be burnt to supply heat, the particulate solids, usually coke produced in the process, continue to grow by reason of the carbon deposition on them to the extent of becoming difficult to fluidize or nonfluidizable.

It has been found necessary to add to the coking system, small size particles to act as "seeds" or nuclei for the normal growth process and to maintain an optimum size distribution of the particles. The production of these seed particles necessitates expensive solids size reduction facilities. By application of the method of this invention, the cost of creating seed particles is reduced considerably.

The present invention is concerned with the control of particle size and distribution of the coke particles in a hydrocarbon oil fluid coking system, whereby the operability of the fluid coking process is improved.

An excess of coke is produced by the coking process and is removed, according to the present invention, as selected coarse particles by means of an improved elutriation system. By the method of this invention, fine particles are preferentially retained in the coking vessel thereby reducing the amount of seed coke that must be supplied to the coking system and maintaining an optimum particle size distribution, thus achieving optimum coker performance.

Broadly, however, the invention is applicable to any process wherein it is necessary to classify solids. As an example of another application, it has been suggested to use in fluid hydroforming operations for the production of gasoline an inert heat carrying material substantially heavier than the catalyst. This "shot" is circulated from a heating vessel to the vessel containing the fluidized catalyst and falls through the bed, giving up heat to the

reaction. The shot is then separated and recycled to the heater. This invention is particularly suited to separating the shot from the catalyst prior to its return to the heater.

An object of this invention is to provide the art with an improved method for elutriating and classifying particulate solids. It is a specific object of this invention to decrease the seed coke requirements of a residual oil fluid coking process and to enhance a balanced particle size distribution in such a process by means of an improved solids-gas elutriation system. These and further objects will become readily apparent as the drawings, forming a part of this specification, are discussed in detail.

In the drawings,

Figure 1 illustrates diagrammatically a system adapted to achieve the objects of the present invention.

Figure 2 shows alternate sectional views taken along the line A—A of Figure 1.

Figure 3 depicts schematically a modification of the invention applied to a fluidized solids system for the coking of heavy petroleum oils.

Figure 4 graphically presents data illustrating the advantages of this invention.

Generally, the objects of this invention are attained by passing a mobilized mass of particulate solids through a narrowly confined inclined substantially unobstructed path in countercurrent flow to an ascending gasiform medium, whereby the finer particles of the mass are segregated and conveyed upwardly and the coarser particles pass downwardly. Control of the degree of elutriation is obtained by control of the degree of inclination of the path, the length of the path and the velocity of the elutriating gas. A more complete segregation of the solids is obtained by passing the solids through a series of inclined paths, separated by vertical sections which act as mixing chambers. The angle of inclination of the path is from 1° to 60° from the vertical, preferably, 30° to 60°, and it is also preferred to have a flat or plane surface on the lower side of the inclination as a surface upon which descending solids slide.

The more particular objects of this invention are achieved by applying the invention to a vessel adapted to the fluid coking of particulate solids. Specifically, a portion of the fluid solids bed within a coking vessel is withdrawn, elutriated and the fines returned to the vessel while the coarser coke from the elutriation is withdrawn as product.

It will be seen that the present invention is distinguishable from conventional elutriation or classification systems. In conventional elutriation systems, the solids are classified while greatly dispersed or diluted by the aerating medium. According to the present invention, the solids move downwardly in the dense phase while the aerating medium strips from the dense phase interface the finer particles and entrains them.

Referring now to the drawings, there is shown in Figure 1 a system for segregating solids. The solids are introduced by line **10** into an inclined conduit **11**. The particulate solids may be of varying densities but it is contemplated that the invention will be applied to the classification of solids of substantially uniform unit density. This invention is applicable to particulate solids like coke, sand, catalyst, etc. or mixtures thereof.

At the lower end of the conduit a receiver **12** or expanded settling zone is used to collect the coarser particles and line **4** is used to remove them from the receiver.

At the upper end of the conduit a solids-gas separator **5** or cyclone is used to remove the finer particles from the elutriating gas. Gas is removed overhead by line **6** and the fines are transferred by line **7** to a hopper **8** from which they are removed by line **9**.

Elutriating gas may be admitted near the base of the conduit or into hopper **12** by line **13**, as is shown. Alternately, gas may be admitted by line **13***a* to the hopper to serve to fluidize the solids therein and then may be passed upwardly through conduit **11** to serve as elutriating gas. Any suitable gas may be used, e.g. steam, air, inert gases, etc. It may be desirable in certain instances to use a gas that reacts, either chemically or physically, with the solids.

The velocity of the gas in the conduit will, of course, depend upon the size and weight of the particles being classified, the density of the gas, the angle of inclination and the degree of elutriation desired. For particles having a size less than 500 microns, the superficial velocity of the gas in the conduit may vary from 2 to 10 feet per second, preferably from 2 to 6 f./s., for classifying particulate coke at an elutriator angle of about 30° from the horizontal.

In Figure 2, alternate views of the inclined conduit are shown. Although a circular pipe, as is shown in view A, will operate satisfactorily, a rectangular pipe is preferred, shown in view B. In high pressure operations, it is advantageous to retain the strength of a circular pipe but modify it by inserting a flat plate **14** or slide in the lower portion of the pipe. This arrangement is shown in view C.

The basic principle of the present invention is that the solids being classified slide down the bottom of the inclined conduit in a dense phase, while countercurrent streams of gas strip fine particles off the top of the dense phase. This is quite analogous to a countercurrent stripper.

When circular conduits are used, the ratio of the coke-gas interface to the coke mass decreases with increase in pipe diameter. Hence elutriation efficiency decreases with increasing pipe diameter. Therefore, it is a preferred feature of the present invention to have a flat bottom surface in the lower portion of the inclined elutriating conduit to promote elutriation efficiency.

Also, it is preferred to use a multiple or cascade arrangement of the inclined elutriating conduits as this promotes the movement of solids within the sliding solids mass and more readily causes fines to be brought to the solids-gas interface. Other mixing devices or baffling arrangements may be used to promote this agitation of the solids mass.

The mobility of the sliding solids and the capacity of the conduit may be increased by a small amount of aeration of the solids. Thus, the bottom of the inclined conduit, or a portion thereof, may be composed of porous micro metallic metal, and aerating gas passed through it.

Figure 3 illustrates another form of the inclined elutriator of this invention used in combination with a petroleum oil fluid coking vessel.

The art of fluid coking is well known and for this reason only a brief description of the coking operation will be made. The oil to be subjected to pyrolysis is injected into a coking vessel **15** by line **16** where it contacts a fluid bed of particulate solids, e.g. coke produced in the process. The fluidity of the bed is maintained by admitting steam to the base of the vessel by line **17**. The reaction temperature of the bed is maintained at about 900–1600° F. by continuously withdrawing a portion of it by line **18** and transferring it to an external heater, e.g. combustion zone, and back, via line **19**.

The injected oil undergoes pyrolysis evolving substantial amounts of lighter hydrocarbon vapors, removed by line **20**, and depositing coke on the particulate solids.

To remove the excess coke from the process, a portion of the fluid bed is withdrawn from the vessel by line **21**, composed of a multiplicity of inclined and vertical sections having flat bottoms. The solids flow downwardly in countercurrent flow to steam. Finer particles are selectively removed from the solids by the steam and are returned to the coking vessel.

The coarse solids then enter a hopper **22** from which they are removed by line **23** as product. Steam is admitted to the hopper by line **24** in amounts sufficient to obtain the desired degree of segregation. This steam may be admitted to the base of the hopper to fluidize the solids therein before being used for elutriation.

Although the solids flow rate may vary considerably, it is preferred to introduce solids into the inclined pipe at a rate of 1000 to 5000 lb./hr.-ft.^{2} to obtain optimum performance. For coke under approximately 500 microns in size, the preferred rate is 1000 to 3000 lb./hr.-ft.^{2}. The invention is operable, however, at any rate below 1000 lb./hr.-ft.^{2}.

As an alternate, the inclined conduit may be connected to the coking vessel so as to remove solids from the dilute phase above the interface of the dense fluidized solids bed. This results in some sacrifice in throughput but a much finer separation is made.

It has been found that the sharpness of the separation increases very rapidly as the rate of solids flow decreases. However, the optimum rate is determined by economic considerations involving line size and gas rate.

Further, it has been found that it is desirable to have a length over cross-sectional height ratio for the inclined conduit of at least 2 in order to allow sufficient time for the stripping action to occur.

The following examples are given to further elucidate this invention. A 0.622″ I.D. pipe, 0.7 foot long, was connected to a vessel containing a bed of fluidized particulate solids, as in Figure 3, at a 30° angle from the vertical. Elutriation was accomplished at gas, e.g. air, velocities between 4 and 8 feet per second and about 2 lbs. of solids were withdrawn per hour. Table I illustrates the extent of the elutriation obtained:

Table I

Percent on Mesh	Original Coke Mixture	Elutriated Coke
20	2	6
48	8	42
60	16	72
80	46	94
100	62	98
200	90	100
Pan	10	0

Figure 4–A compares this data with that obtained from a vertical dispersed phase elutriator, 4.03″ I.D., 11 feet long, handing about 1000 lbs./hr.-ft.^{2} at gas velocities of 9 to 11 feet/second. It is readily apparent that inclination of the path of elutriation greatly increases the efficiency of the separation.

Figure 4–B illustrates the benefits derived from the use of an elutriator the lower portion of which is a flat surface. The data for the circular elutriator were obtained from a conduit 1″ I.D., 4½ ft. long, inclined 30° from the vertical and handling 1970#/hr.-ft.^{2} of solids at an elutriating gas velocity of 7.5 feet/second. The data for the flat-bottomed conduit were obtained from a rectangular conduit 2″ wide x ¾″ deep, 5 feet long, inclined 30° from the vertical and handling 2000#/hr.-ft.^{2} at an elutriating gas velocity of 6.5 feet/second. Laboratory tests show a 6% to 12% improvement in particle size segregation of the on 48 and on 60 mesh material when using a flat-bottomed conduit as compared to a cylindrical-bottomed vessel.

Figure 4–C illustrates that better elutriation is attained with a cascade arrangement (see Figure 3), e.g. a series of inclined sections separated by substantially vertical sections that act as mixing stages. The data for the cascade arrangement were obtained from a conduit 1″ O.D. handling 3200#/hr.-ft.^{2} of solids at a gas velocity of 7.1 ft./second. Four sections, having a total length of 3⅓ feet, were used and were inclined 30° from the vertical. The inclined portions were separated by verti-

cal sections 2 feet long. The data for the straight section were obtained from a 1" O.D. pipe, 6 feet long, handling 2620#/hr.-ft.^{2} at a gas velocity of 6.8 feet/second.

Figure 4–D illustrates the advantages obtained by operating at lower throughputs. Both curves were obtained from an elutriator inclined 30° from the vertical. The low capacity curve was obtained at a gas velocity of 5.7 feet/second and a solids rate of 156 lbs./hr.-ft.^{2}. The high capacity curve was obtained at a gas velocity of 4.7 feet/second and a solids rate of 1890 lbs./hr.-ft.^{2}. It can be seen that the sharpness of the separation increases with decreases in capacity.

It should be noted that this invention is susceptible to many variations. As examples, it is feasible to arrange inclined elutriating conduits in parallel or multiple arrangements. Or, the inclined sections may be arranged in a staggered fashion such that the material alternately flows in one direction and then the other. Thus, the previously described vertical mixing sections may be dispensed with.

Further, the angles of inclination of the inclined sections in a cascade arrangement may be varied. By having the angle of inclination greater at the top of the cascade system, the effect of surging in the system is minimized. For either a cascade or straight inclined elutriator, the cross-section area of the conduit may be varied. Thus a reduction of about 20% in the area at the bottom of the elutriating conduit helps to insure that fines will not be withdrawn with the coarse product.

Other variations of the present invention and applications of it will be apparent to those skilled in the art.

Having described the invention, what is sought to be protected by Letters Patent is set forth in the following claims.

What is claimed is:

1. A method for classifying fluid coke particulate solids of sizes under about 500 microns in diameter according to size which comprises, passing said solids at a rate in the range of 1,000 to 5,000 lbs. per hour per square foot as a series of inclined confined dense phase streams each having a defined upper surface and arranged one above the other in spaced vertical relation, said confined dense streams having an inclination in the range of 30° to 60° from the vertical and in the same direction, passing the dense phase stream from one inclined level to the next lower level through a vertical mixing zone in contact with a gas to form a gas-solids mixture passing to the next dense phase stream, introducing an elutriating gas into the outlet end of the bottom confined dense stream for passage upwardly through said series of inclined dense phase streams at a velocity in the range of 2 to 10 feet per second countercurrent to the downflowing solids to strip fine solids from the surface of said dense phase solids streams and upwardly through said vertically disposed mixing zones to form gas-solids mixtures therein, withdrawing said gas and a substantial portion of fine solids from the upper end of said series of inclined confined dense streams and removing coarse solids from the outlet end of the bottom inclined confined dense stream.

2. The method of claim 1 wherein the inclination of said series of inclined confined dense phase streams decreases from its top to bottom portions thus minimizing the effect of surging in the system.

3. An apparatus of the type described for elutriation of particulate solids by a gas which includes a series of flat bottomed unobstructed elutriating conduits inclined 30° to 60° from the vertical and each conduit having a minimum length to cross-sectional height ratio above 2, said conduits being arranged in cascade relation and inclined in the same direction, a plurality of vertically disposed pipes, the lower end of each conduit being connected to the upper end of the next adjacent conduit by one of said vertical pipes each of which acts as a mixing chamber between said conduits, said top conduit being adapted to discharge fine particulate solids entrained in said gas from its upper portion and said bottom conduit being adapted to discharge coarse particulate solids from its lower portion, means for admitting unclassified particulate solids to the upper portion of said top elutriating conduit for downward passage through said elutriating conduits and pipes and an inlet conduit for admitting said gas to the inlet of said bottom elutriating conduit for upward passage through said series of elutriating conduits and pipes.

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