Aug. 13, 1957 M. E. NASH 2,802,236

COOLING AND HANDLING OF PETROLEUM PITCHES

Filed July 27, 1953 2 Sheets-Sheet 1

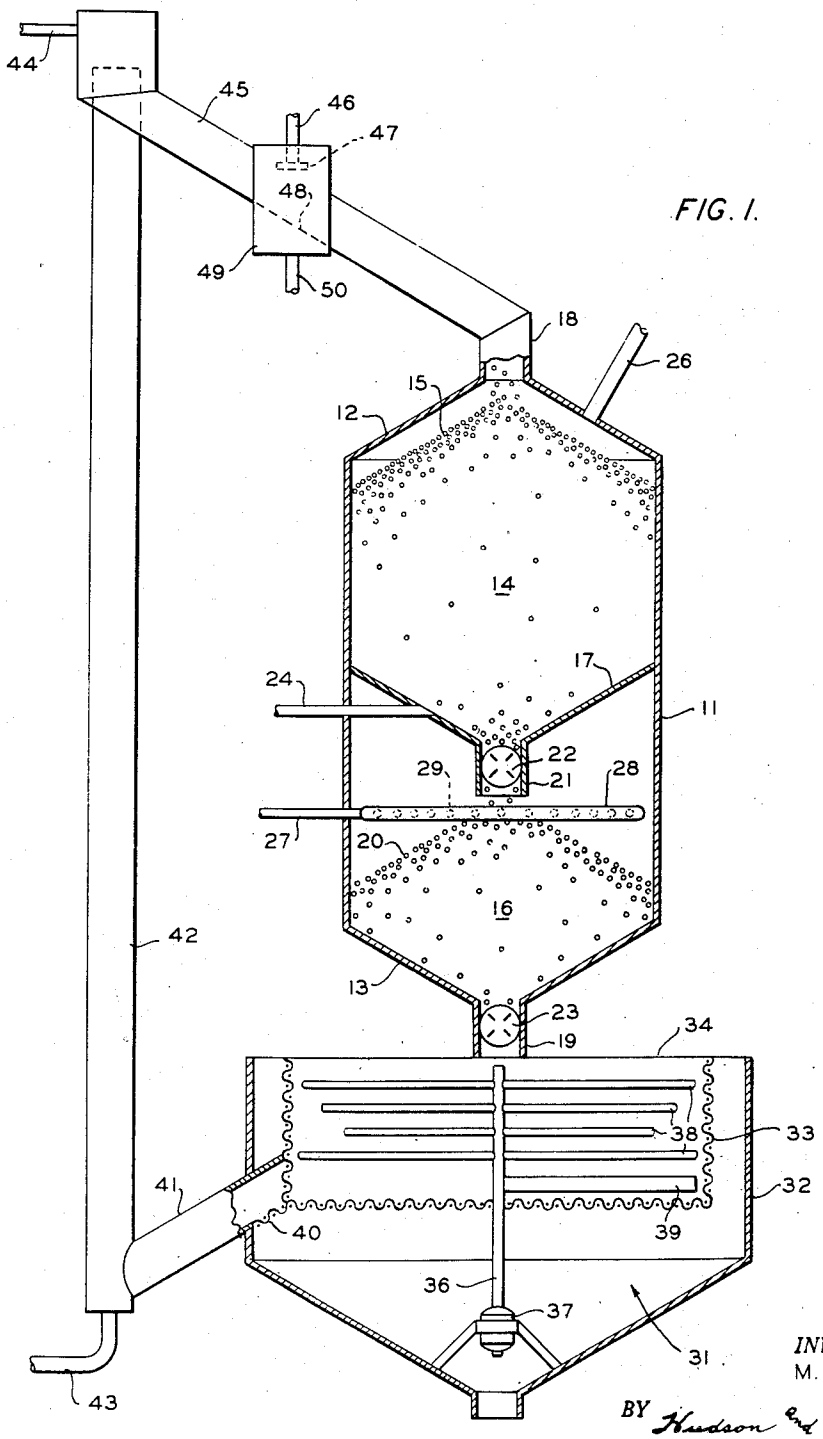


FIG. 1.

INVENTOR.
M. E. NASH

BY Hudson & Young

ATTORNEYS

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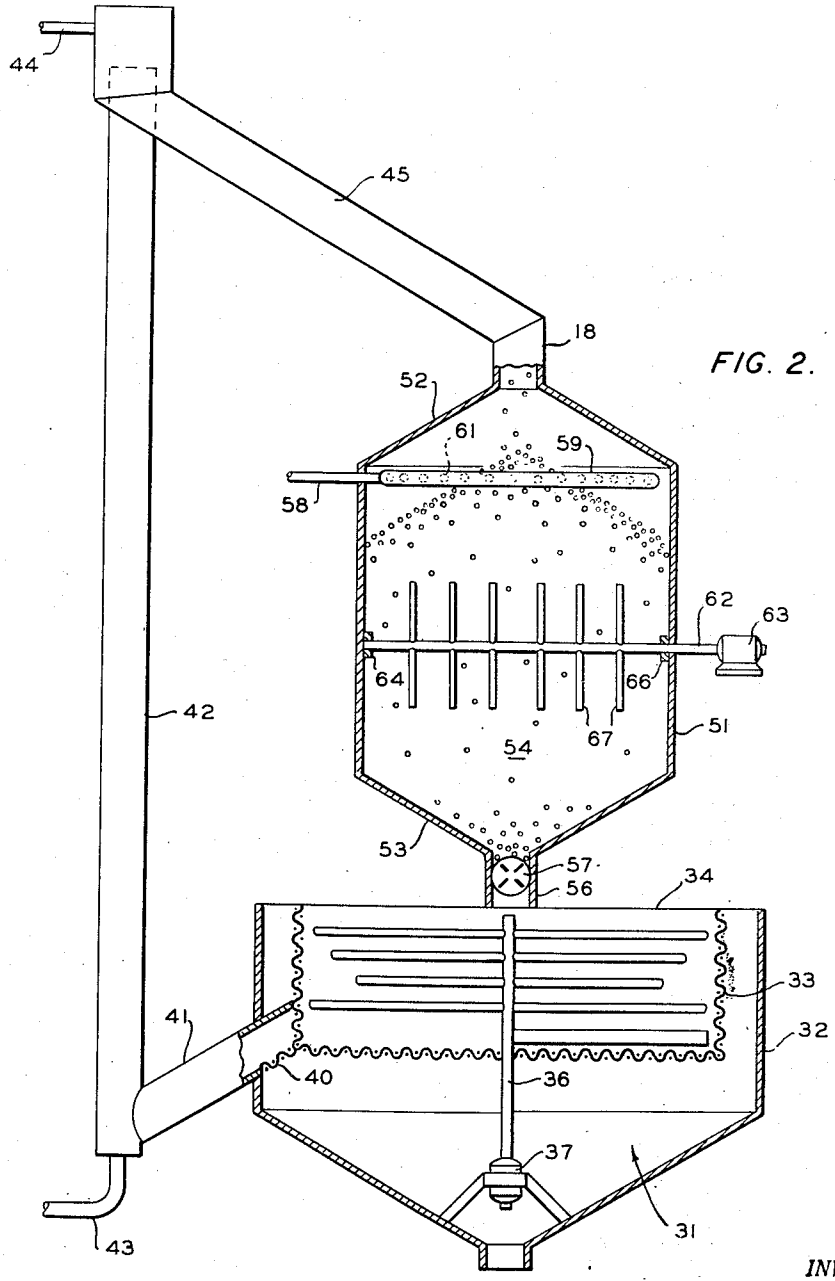


FIG. 2.

INVENTOR.
M.E.NASH

BY Hudson and Young

ATTORNEYS

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2,802,236

COOLING AND HANDLING OF PETROLEUM PITCHES

Martin E. Nash, Bartlesville, Okla., assignor to Phillips Petroleum Company, a corporation of Delaware

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16 Claims. (Cl. 18—1)

This invention relates to the cooling of liquids so as to form solid products flaky and granular in structure. In one of its specific aspects, it relates to the cooling of hot liquid petroleum pitch. In another of its specific aspects, it relates to a process for cooling hot liquid petroleum pitch and recovering solid pitch, flaky and granular in form.

It has recently been found that petroleum pitches having properties equal or superior to those of coal tar pitch can be produced from certain pre-selected feedstocks by a vacuum distillation process. Because of their superior properties, these pitches have found numerous industrial uses, such as in the making of coal briquettes, the manufacture of electrodes for aluminum refining, and the fabrication of certain types of pressed board products. For a detailed description of such petroleum pitches and a process for their production, reference may be had to my copending U. S. application, Serial No. 328,989, filed December 31, 1952, now Patent No. 2,768,119.

In the production of petroleum pitches, one of the many problems encountered has been that of providing a practical, efficient, and reasonably rapid method of cooling the hot liquid product. Closely associated with the problem of cooling are those of handling and storage, for it is desirable to obtain a solid pitch product, flaky and granular in form in order to promote ease of handling, storage and shipment. Since pitch becomes an excellent insulator after solidification, only small thicknesses can be rapidly cooled to form a solid product. Because of this poor thermal conductivity of pitch, it is estimated that about two years would be required to solidify a pond of liquid pitch about 20 feet deep, or still again about a five-day cooling period would be required to solidify pitch in pans or enclosures about one foot thick. If the pitch is thus allowed to cool naturally, it is apparent that a vast area would have to be set aside for the cooling operation in order to accommodate even a small volume of production. And furthermore, in order to recover an uncontaminated pitch product, an enclosed installation would be called for if a method of natural cooling of the pitch is utilized. In addition to the expensive installation involved, there is the further objection to natural cooling in that the product obtained is not of the desired flaky granular form.

There are several other cooling methods available which can be employed, but which have certain disadvantages. One of these methods involves rotating a cooled drum in a pan of liquid pitch so as to form thereon a thin film of solid pitch which is shaved from the drum as it rotates. Another somewhat similar method utilizes an endless belt mounted on a pair of pulleys, the lower portion of the belt being submerged in a pan of liquid pitch. As the belt passes through the liquid, it picks up a thin film of pitch which is subsequently cooled and removed from the belt as it passes over one of the pulleys. It is evident that the rotating drum or endless belt type methods of cooling would involve extremely

large installations which would materially increase the production costs of solid pitch. Still again liquid petroleum pitch can be cooled by water contacting methods, such as sluicing and prilling in a water fogging tower. Such latter methods, however, result in an undesirable product containing occluded and absorbed water. By employing the cooling process of this invention the disadvantages of the above enumerated methods are eliminated, and it is possible to obtain an uncontaminated solid pitch product, flaky and granular in structure.

The objects and advantages of the present invention will be attained by the various aspects of the invention.

It is an object of this invention to provide a method of cooling liquids so as to form solid products, flaky and granular in structure.

Another object is to provide a method for cooling hot liquid petroleum pitch.

A further object is to provide a method for cooling hot liquid petroleum pitch so as to obtain a solid pitch product, flaky and granular in structure.

Still other objects and advantages will become apparent to one skilled in the art from a consideration of the accompanying disclosure.

Broadly, the present invention involves the cooling of solid spheres to a temperature below the melting point of the petroleum pitch and the coating of the cooled spheres with hot liquid petroleum pitch so as to form thereon a thin film of solid pitch. Thereafter, the coated spheres are continuously agitated so as to remove the thin film of solid pitch which is recovered in flaky granular form.

For a more complete understanding of the invention, reference may be had to the drawing in which:

Figure 1 is a diagrammatic elevational view, partly in section, of one arrangement of apparatus suitable for practicing the invention, and

Figure 2 is a diagrammatic elevational view, partly in section, of another arrangement of apparatus suitable for practicing the invention.

Referring to Figure 1 of the drawing, a substantially vertically disposed shell **11** is closed at its upper end by closure member **12** and at its lower end by closure member **13**. Closure member **13** is preferably funnel shaped in order to facilitate removal of solid heat transfer material from the bottom of shell **11**. Shell **11** is divided into an upper or cooling chamber **14** and a lower or liquid contact chamber **16** by means of inverted conical member **17** which is attached to the walls of the shell. Cooling chamber **14** is provided with a solid heat transfer material inlet conduit **18** while contact chamber **16** has an outlet conduit **19**. The two chambers are connected by throat **21** attached to conical member **17** and containing a flow control means such as star valve **22**, a gate valve or the like. Outlet conduit **19** is provided with a similar flow control means **23**. It is also within the contemplation of the invention to omit star valve **22** from throat **21** and effect the desired control solely by means of flow control means **23**. A fluid inlet means **24** communicates with the lower part of chamber **14** while fluid outlet means **26** is positioned in closure member **12**. The apparatus include liquid inlet means **27** connected to liquid distributor ring **28** having perforations **29** therein for the distribution of the hot liquid petroleum pitch. Distributor ring **28** is positioned within chamber **16** above the bed of heat transfer material so that the material will pass through the ring on leaving throat **21**. The perforations are spaced along the inside circumference of the ring and are also staggered along the under part of the ring.

Positioned below outlet conduit **19** is a means **31** for agitating and abrading the heat transfer material, which agitating means in turn is disposed within hopper **32**,

Agitating means **31** comprises a container **33** having sides and bottom formed of wire screening of a mesh small enough to prevent the escape of heat transfer material while at the same time allowing the particulate solid pitch to pass therethrough. Outlet conduit **19** communicates with the interior of container **33** through an opening in plate **34** which covers the top of the container. Vertical shaft **36**, operatively connected to motor **37**, is disposed within container **33** and attached to the shaft are horizontal arms or rods **38** which are illustrated as being of varying lengths. The distance between the ends of any arm and the walls of the container should be greater than the diameter of the heat transfer material and preferably not less than about two or three diameters. A horizontal arcuate member **39**, which is attached to the shaft in close proximity to the bottom of container **33**, is disposed so that its leading edge is convex.

Outlet conduit **41** leads from container **33** into the lower part of elevator **42**. That part of conduit **41** which is positioned within hopper **32** has a bottom portion **40** constructed of wire screening similar to that used with container **33**. Elevator **42** is illustrated as being a conventional gas lift elevator provided with a gas inlet **43** and a gas outlet **44**. Conduit **45** connects the upper part of elevator **42** with inlet conduit **18**.

Referring to Figure 2, which illustrates another arrangement of apparatus for practicing the invention, a substantially vertically disposed shell **51** is closed at its upper end by closure member **52** and at its lower end by closure member **53** so as to form therein liquid contact chamber **54**. Closure member **53** is preferably funnel shaped and is provided with an outlet conduit **56** containing a flow control means **57** such as a star valve. Liquid inlet means **58** is connected to liquid distributor ring **59** having perforations therein for the distribution of the hot liquid petroleum pitch. Distributor ring **59** is disposed within the upper part of chamber **54** so that the heat transfer material on entering the chamber will pass through the ring. A substantially horizontal shaft **62** operatively connected to motor **63** extends through the side of shell **51** and is supported within liquid contact chamber **54** by means of bearings **64** and **66**. Stirrer rods **67** affixed to shaft **62** at spaced intervals along its length provide means for continuously stirring the solid heat transfer material within the liquid contact chamber. While the stirring means is illustrated as comprising a single horizontal shaft with attached rods, it is within the scope of the invention to utilize more than one shaft or to dispose the shaft or shafts in other positions as, for example, vertically. Furthermore, the stirring means is not restricted to the apparatus of Figure 2, but can be used in conjunction with the apparatus of Figure 1. Agitating means **31** and gas lift elevator **42** are similar to those described in conjunction with Figure 1, and identical numerals have been used to indicate corresponding elements.

In the operation of the apparatus of Figure 1, solid heat transfer material is introduced into cooling chamber **14** through inlet conduit **18** and takes the form of a contiguous mass or bed having an upper surface **15**. Solid spheres having a diameter from about ⅛ inch to about ⅜ inch are utilized as the heat transfer material. While it is preferred that the spheres be formed of a metal such as steel or aluminum, it is within the scope of the invention to employ spheres formed of ceramic material such as are utilized in conjunction with pebble heater apparatus. A liquid refrigerant such as propane is introduced into the lower part of cooling chamber **14** through fluid inlet means **24** in commingled relation with the solid spheres. The liquid refrigerant evaporates lowering the temperature of the spheres to a point substantially below the softening point of the petroleum pitch.

The cooled solid spheres flow downwardly through throat **21** into liquid contact chamber **16** as a contiguous mass to form a bed therein having an upper surface **20**. The rate of flow of the solid spheres into chamber **16** can

be controlled by means of star valve **22**, or alternatively star valve **22** can be omitted and the desired rate of flow obtained by regulating star valve **23** in outlet conduit **19**. Hot liquid petroleum pitch is passed into chamber **16** through liquid inlet means **27** which is connected to distributor ring **28**. Perforations **29** in distributor ring **28** are so located that the pitch leaves the distributor ring in a plurality of streams, which impinge against the spheres as they pass across surface **20**. By distributing the hot liquid pitch in this manner, the cool solid spheres are coated with a thin film of liquid pitch which upon contact immediately solidifies to form solid pitch. The thickness of this film is preferably about 1/16 to ¼ inch, although some of the solids may be coated with films of greater or less thickness, depending upon the size and nature of the solids. The coated spheres thereafter gravitate downwardly through chamber **16** and pass through outlet conduit **19** into container **33**. The rate of flow of the spheres into container **33** can be controlled by star valve **23**. When the spheres enter container **33**, they are struck by arms **38** which are slowly rotating with shaft **36**. The impact of the spheres against the sides of container **33** and against one another causes the coating of solid pitch to crack and break off from the spheres. Because of the brittleness of the thin film, only a comparatively small amount of agitation of the spheres by slow rotation of the arms is necessary in order to completely clean the spheres. Most of the flaky granular pitch falls to the bottom of container **33** and passes through the screen to hopper **32** while some passes through the side screens into the hopper. Arcuate member **39**, which turns with shaft **36** and is positioned in close proximity to the bottom screen, wipes the bottom of container **33** clean and breaks up or forces any of the larger pieces of solid pitch through the screen. The spheres leave the container through the conduit **41** which is partly screened to take care of any of the pitch which might pass into the conduit with the spheres. Arcuate member **39** also propels any of the spheres which may tend to settle in the bottom of container **33** into conduit **41**. The spheres enter the bottom of gas lift elevator **42** through conduit **41** and are lifted by means of air introduced through line **43** to the top of the elevator. Thereafter, the spheres leave the elevator through conduit **45** which connects with inlet conduit **18** through which the spheres pass into cooling chamber **14**.

It is also within the scope of the invention, utilizing the apparatus of Figure 1, to use a stream of cold air to cool the spheres within chamber **14**. In this embodiment of the invention the spheres are sprayed with water as they pass through conduit **45**. The water is sprayed upon the spheres by nozzle **47** connected to water line **46**. Excess water is removed from conduit **45** through screening **48** disposed in the bottom of conduit **45** and surrounded by container **49** having an outlet line **50** connected thereto. A stream of cold air which is passed into chamber **14** through line **24** dries and cools the downwardly moving mass of moist spheres. The air and moisture leave the chamber through outlet means **26**, and thereafter the operation of the apparatus proceeds as explained in conjunction with Figure 1. Outlet **26** may be connected to means, not shown, for producing a partial vacuum above upper surface **15** of the bed of solid heat transfer material.

In the operation of the apparatus of Figure 2, a stream of cold air is introduced into gas lift elevator **42** through line **43**. The spheres are cooled in their passage through the elevator by contact with the cold air and are then introduced into chamber **54** through inlet conduit **18** connected to elevator **42** by conduit **45**. The cool spheres are contacted with hot liquid petroleum pitch so as to form thereon a thin film of solid pitch as previously described. The coated spheres thereafter gravitate downwardly through chamber **54** and pass through outlet conduit **56**, provided with valve **57** for flow control, into container **33**. The operation of the apparatus of Figure

2 then proceeds as described in conjunction with Figure 1.

In the operation of the apparatuses of Figures 1 and 2, the spheres after being coated with the liquid pitch may tend to agglomerate. In order to guard against possible agglomeration and resultant bridging which would impede the flow of spheres into and through outlet conduits **19** and **56**, stirring means can be provided within liquid contact chambers **16** and **54**. As illustrated in Figure 2, shaft **62** with attached rods **67** is slowly and continuously rotated, thereby preventing the spheres from cementing together and allowing free passage of the spheres through the chamber.

While the present invention has been described specifically in relation to the cooling of hot liquid petroleum pitch, it is within the contemplation of the invention to cool other pitches, such as coal tar pitch, and other liquids so as to form solid products, flaky and granular in structure. Examples of liquids which can be cooled utilizing the process of this invention are liquid sulphur, liquid caustic soda, liquid sodium sulphide and liquid waxes.

As will be evident to those skilled in the art, various modifications of this invention can be made or followed is the light of the foregoing disclosure and discussion without departing from the spirit or scope of the invention.

I claim:

1. A process for forming granular solid products from hot liquid products which comprises coating a cooled mass of solid fragmentary heat transfer material with a hot liquid product; agitating said solid heat transfer material so as to remove therefrom the coating of solid product in granular form; and separating said solid heat transfer material from said solid product.

2. A process for forming granular petroleum pitch which comprises coating a cooled mass of solid fragmentary heat transfer material with hot liquid petroleum pitch; agitating said solid heat transfer material so as to remove therefrom the coating of solid pitch in granular form; and separating said solid heat transfer material from said solid pitch.

3. A process for forming granular petroleum pitch which comprises cooling a mass of solid fragmentary heat transfer material; thereafter spraying said solid heat transfer material with hot liquid petroleum pitch so as to form thereon a thin film of solid pitch; agitating said solid heat transfer material so as to remove therefrom said film of solid pitch in granular form; and separating said solid heat transfer material from said solid pitch.

4. A process for forming granular petroleum pitch which comprises introducing cooled solid spheres into the top of a vertically elongated chamber; contacting said spheres with a spray of hot liquid pitch; removing the spheres coated with a thin film of solid pitch from the bottom of said chamber; agitating said spheres so as to remove therefrom said thin film of pitch in granular form; separating the clean spheres from the solid pitch; passing the clean spheres into a gas lift elevator; and elevating said spheres to the top of said chamber by means of a cold stream of air, thereby cooling said spheres.

5. The process of claim 4 wherein the spheres after being contacted with hot liquid pitch are continuously and slowly stirred within said chamber so as to prevent agglomeration of the coated spheres.

6. A process for forming granular petroleum pitch which comprises introducing moist solid spheres into the upper portion of a vertically elongated cooling chamber; passing a stream of cold air into the lower part of said cooling chamber in commingled relation with said solid spheres, thereby drying and cooling same; withdrawing the air and moisture from the upper portion of said cooling chamber; gravitating the cooled solid spheres from the lower portion of said cooling chamber into a liquid contact chamber; contacting said cooled spheres with a spray of hot liquid petroleum pitch; removing the spheres coated with a thin film of solid pitch from the lower portion of said liquid contact chamber; agitating the spheres so as to remove therefrom said thin film of solid pitch in granular form; separating the clean spheres from the solid pitch; and returning the spheres to the upper portion of said cooling chamber.

7. The process of claim 6 wherein the spheres after being contacted with hot liquid pitch are continuously and slowly stirred within said liquid contact chamber so as to prevent agglomeration of the coated spheres.

8. A process for forming granular petroleum pitch which comprises introducing solid spheres into the upper portion of a vertically elongated cooling chamber; passing a liquid refrigerant into the lower part of said cooling chamber in commingled relation with said solid spheres; evaporating said liquid refrigerant, thereby cooling said solid spheres; removing the vaporized refrigerant from the upper portion of said cooling chamber; gravitating the cooled solid spheres from the lower portion of said cooling chamber into a liquid contact chamber; contacting said cooled spheres with a spray of hot liquid petroleum pitch; removing the spheres coated with a thin film of solid pitch from the lower portion of said liquid contact chamber; agitating the spheres so as to remove therefrom said thin film of solid pitch in granular form; separating the clean spheres from the solid pitch; and returning the spheres to the upper portion of said cooling chamber.

9. The process of claim 8 wherein the spheres after being contacted with hot liquid pitch are continuously and slowly stirred within said liquid contact chamber so as to prevent agglomeration of the coated spheres.

10. A process for forming granular petroleum pitch which comprises introducing solid spheres into a cooling zone; cooling said spheres to a temperature substantially below the softening point of liquid petroleum pitch; gravitating the cooled spheres into a liquid contact zone; contacting said cooled spheres with a spray of hot liquid petroleum pitch so as to form thereon a thin film of solid pitch; removing the coated spheres from said contact zone; agitating said spheres so as to remove therefrom said thin film of solid pitch in granular form; separating the clean spheres from the solid pitch; and returning said clean spheres to said cooling zone.

11. Apparatus for forming solid granular products from liquids which comprises, in combination, a substantially vertically-disposed, closed shell; a liquid distribution means in the chamber formed within said shell; solid material inlet means in the upper portion of said chamber; solid material outlet means in the lower portion of said shell; means for agitating solid material disposed below said outlet means; and gas-lift means for elevating solid material from said agitating means to said solid material inlet means.

12. Apparatus for forming solid granular products from liquids which comprises, in combination, a substantially vertically-disposed, closed shell; a liquid distribution means in the chamber formed within said shell; means for stirring solid material within said chamber; solid material inlet means in the upper portion of said shell; solid material outlet means in the lower portion of said shell; means for agitating solid material disposed below said outlet means; and gas-lift means for elevating solid material from said agitating means to said solid material inlet means.

13. Apparatus for forming solid granular products from liquids which comprises, in combination, a substantially vertically-disposed, closed shell; an inverted conical member attached to the walls of said shell intermediate its ends, thereby forming an upper and a lower chamber within said shell; solid material conduit means attached to said conical member and communicating said upper chamber with said lower chamber; solid material inlet means in the upper part of said shell; fluid outlet means in the upper part of said shell; fluid inlet means in com-

munication with the lower portion of said upper chamber; liquid distribution means in the upper portion of said lower chamber; solid material outlet means in the lower part of said shell; means for agitating solid material disposed below said solid material outlet means; and gas-lift means for elevating solid material from said agitating means to said solid material inlet means.

14. Apparatus for forming solid granular products from liquids which comprises, in combination, a substantially vertically-disposed, closed shell; an inverted conical member attached to the walls of said shell intermediate its ends, thereby forming an upper and a lower chamber within said shell; solid material conduit means attached to said conical member and communicating said upper chamber with said lower chamber; solid material inlet means in the upper part of said shell; fluid outlet means in the upper part of said shell; fluid inlet means in communication with the lower portion of said upper chamber; liquid distribution means in the upper portion of said lower chamber; means for stirring solid material within said lower chamber; solid material outlet means in the lower part of said shell; means for agitating solid material disposed below said solid material outlet means; and gas lift means for elevating solid material from said agitating means to said solid material inlet means.

15. Apparatus for forming solid granular products from liquids which comprises, in combination, a substantially vertically-disposed, closed shell; an inverted conical member attached to the walls of said shell intermediate its ends, thereby forming an upper and a lower chamber within said shell; solid material conduit means attached to said conical member and communicating said upper chamber with said lower chamber; solid material inlet means in the upper part of said shell; fluid outlet means in the upper part of said shell; fluid inlet means in communication with the lower portion of said upper chamber; liquid distribution means in the upper portion of said lower chamber; solid material outlet means in the lower part of said shell; means for agitating solid material disposed below said solid material outlet means; gas-lift means for elevating solid material, said means having its lower end connected to said agitating means; a solid material conduit connected between the upper end of said gas-lift means and said solid material inlet means; and means for spraying solid material with water disposed in said solid material conduit.

16. The apparatus of claim 15 in which a means for stirring solid material is disposed within said lower chamber.

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