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PARAFFIN CONTROL COMPOSITION AND METHOD

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This invention relates to compositions and methods useful in controlling paraffin or paraffin-forming and similar substances, such as by reducing or preventing the deposition, formation or accumulation of paraffin on the surfaces of pipes or other apparatus containing flow passageways through which fluid containing paraffin or paraffin-forming substances is conducted.

It is anticipated that the invention will be widely applied in connection with oil and gas well operations. For example, the composition and method of the invention may be employed in treating operations performed to stimulate or increase production from an underground oil-bearing zone or formation. The treatment may reduce or prevent the deposition, formation or accumulation of paraffin in flow passageways located in the well and/or in related equipment at the surface of the ground.

Heretofore naphthalene has been used successfully by well operators in many areas to control paraffin deposition in producing wells and flow lines. The common practice has been to pour the naphthalene crystals in the annulus of the well. The naphthalene settles to the bottom and is produced back with the well production. If trouble is encountered due to paraffin in the flow line to the tank battery, naphthalene is put directly into the line by some mechanical device. It has been found that regularly timed, successive treatments may be employed to keep the paraffin from accumulating to an undesirable extent.

While generally successful results have been obtained using naphthalene, as aforesaid, frequent treatments have been necessary due to the relatively rapid rate of dissolution of the naphthalene in the oil produced from the well. Due to the relatively low melting point of naphthalene (about 176° F.), rapid dissolution thereof has been particularly noticeable in the deeper wells where higher temperatures are generally encountered.

Accordingly, one object of the present invention is to provide an improved paraffin-control composition which is relatively slowly soluble in liquid hydrocarbons, such as oil.

Another object of the invention is to provide an improved paraffin-control composition which is relatively slowly soluble at the higher temperatures, such as are generally encountered in deep oil and gas wells or the like.

Another object of the invention is to provide an improved paraffin-control composition and an improved method utilizing the same in treating oil and gas wells or the like.

Another object of the invention is to provide an improved paraffin-control composition and an improved method utilizing the same in well treating operations, the composition being particularly suited for injection into the underground strata surrounding the bore of the well.

Another object of the invention is to provide an improved well treating composition and an improved method utilizing the composition in a combined fracturing and paraffin-control treatment.

A further and more specific object of the invention is to provide an improved hydraulic fracturing fluid containing an improved paraffin-control additive and to provide an improved well treating method utilizing the same, the paraffin-control additive being relatively slowly soluble in hydrocarbon liquids, such as oil, and being particularly

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suitable for injection with the fracturing fluid through openings created or present in the fractured zone or formation of the well.

The foregoing and additional objects and advantages are attained by the invention, which involves the concept of a paraffin-control composition consisting essentially of naphthalene and a material, such as beta naphthol or anthracene or a mixture thereof, which is more slowly soluble in liquid hydrocarbons, particularly at the higher temperatures, such as commonly prevail in deep oil and gas wells.

As previously indicated, the melting point of naphthalene is about 176° F. On the other hand, the melting point of beta naphthol is about 251° F., while the melting point of anthracene is about 422° F. By varying the ratio of the naphthalene to the less soluble material, any of a wide variety of dissolution rates may be obtained and, in effect, the paraffin-control composition can be tailored to meet different conditions of use.

In one way of proceeding, the paraffin-control composition is prepared by combining melted quantities of the ingredients, such as the naphthalene and beta naphthol, and then allowing the mixture to cool and solidify. The resulting mass may then be ground or otherwise reduced to the desired particle sizes for use in fracturing fluids as a paraffin-control additive.

The proportion of the more slowly soluble material should be sufficiently large to assure that the protection provided by the paraffin-control treatment extends for a considerable period of time. Thus, at least a substantial quantity of beta naphthol or anthracene should be included. It is believed that the inclusion of about equal quantities of naphthalene and beta naphthol will produce satisfactory results in most applications. However, it is understood that the proportions are not critical and more or less of the beta naphthol may be included, if desired, although the inclusion of less than the recommended amount may result in reducing the long-term effectiveness of the protection afforded or make more frequent treatments necessary.

The successful results which are attainable using the present invention in hydraulic fracturing operations are due in part to employing additive particles which are of sufficiently small size to pass through the fracture openings into the strata surrounding the well bore but which are nonetheless of sufficiently large size to have a relatively small total surface area and, thus, a relatively slow rate of dissolution in the oil subsequently produced from the fractured zone or formation. In particular, it is believed that maximum benefits are attained where the particle sizes are large enough so that the relatively slow rate of dissolution of the paraffin-control composition, considering the prevailing temperature conditions, is not unduly increased as a result of the material being used in particulated form.

Accordingly, it is generally desirable to employ the largest size particles which can be successfully introduced into the fractured zone or formation in the required amounts to provide long-term paraffin control or protection. In actual practice, this will ordinarily involve including particles of sufficiently small size to permit entry and deposit of appreciable quantities of the paraffin-control composition in openings and crevices located in the strata at considerable distances from the bore of the well.

In considering maximum recommended particle sizes and also the ratio of large to small particle sizes to be included, it is understood that certain wells may accept a considerably larger quantity of large size particles than can be successfully introduced into the fractured strata of more typical wells. While it is anticipated that substantially all of the particles employed in a particular well

operation will be sufficiently small to pass a standard 4 mesh sieve, the quantity also capable of passing a standard 10 mesh sieve may vary from about 5% to about 95%, with the higher percentages being more common in typical well operations.

With regard to minimum recommended particle sizes, it is believed that satisfactory long-term paraffin control or protection will be obtained if not more than about 20% of the particles added to the fracturing fluid are of sufficiently small size to pass a standard 30 mesh sieve.

For general usage in typical well fracturing operations, it is believed that best results will be obtained using 10-30 mesh particles of the paraffin-control composition. It is understood, however, that not all of the particles included need fall within this recommended range. Also, experience may show that best results are obtainable in certain wells using particles of larger or smaller size.

The amount of the paraffin-control composition needed to treat a particular well will, of course, depend upon several factors, not the least of which will be the extent to which paraffin has been a problem in the area. In general, it is believed that more than about 10 to 15 pounds of the paraffin-control additive will not be needed per 100 gallons of the fracturing fluid, with the general practice being to include from about 200 to about 1,000 pounds of the additive per 10,000 gallons of fracturing fluid.

In carrying out a fracturing and treating operation using the present invention, the paraffin-control additive particles may be added to the fracturing fluid at the well site using conventional mixing equipment, such as a mechanical stirring and proportioning device. In many instances, the fracturing fluid will also contain sand or other hard solid substances to function as spacing or propping agents. If desired, the additive particles may be mixed or blended with the sand or like particles prior to the mixture thereof being added to the fracturing fluid. Where other ingredients or agents are to be included, these may be added at convenient times in accordance with standard procedures. If desired, all of the ingredients may be mixed together at the same time.

The introduction of the fracturing fluid into the well may be accomplished using conventional pumping equipment and procedures. If desired, well packers may be employed at one or both locations in the well above and below the zone or formation to be fractured. In any event, the fluid carrying the additive particles and sand, where included, is pumped or otherwise introduced into the well and thence is caused or allowed to contact a section of the underground strata. Then pressure is applied to the fluid, as by pumping, which causes pressure to be built up in the well against the strata section in contact with the fluid. The application of pressure is continued until the strata section is fractured, whereupon fluid carrying the additive particles is injected through openings extending into the strata section.

While the exact behavior of the additive solids in the well cannot be known with certainty and the invention is not limited to any particular theory of behavior thereof, it is believed that the oil-soluble particles of the paraffin-control composition are carried with the oil-insoluble sand particles into crevices and openings present or created in the fractured zone or formation. As in conventional fracturing operations, at least some of the injected solid particles become disposed between the opposing surfaces of the openings and function as spacing or propping agents to hold these surfaces apart even after the fracturing operation has been completed. However, the additive solids of the present invention also function for a considerable period of time (until eventual dissolution and removal thereof) to control paraffin and paraffin-forming substances which may be present in the well fluids produced from the fractured zone or formation.

The following are specific examples of actual field oper-

ations illustrating how the present invention may be employed:

Example No. 1

Upon completion, following a hydraulic fracturing treatment, a well in Kansas had flowed 2,580 barrels of 38 degree A.P.I. gravity oil per day. Perforations were located in the 5½ inch well casing between 4,226 feet and 4,238 feet. After a period of about 34 months, with no further stimulation treatments, daily production from this well was 19.25 barrels of oil and 1.68 barrels of water.

The wells in this field have always produced with a high gas-to-oil ratio. Hard paraffin forms in the upper 800 feet of tubing and in surface well head connections. Also, a small amount of paraffin trouble is encountered in lead lines. The prior art practice in this field has been to treat the wells for paraffin by dumping 200 gallons of diesel oil down the annulus and then pumping it back to the tank battery. This type of treatment was necessary about every 60 days.

As a preliminary step prior to fracturing and treating the foregoing well in accordance with the present invention, additional perforations were provided in the well casing at zones lying between 4,238 and 4,243 feet, between 4,197 and 4,204 feet and between 4,210 and 4,218 feet. A bridge plug was set at 4,222 feet. Then the perforated zones were treated with 2,000 gallons of an acid-containing liquid. The load was not recovered. The bridge plug was then forced to the bottom of the well.

In the hydraulic fracturing and treating operation which followed, the materials used on this well were 25,000 gallons of fracturing oil heated to 140° F.; 23,000 pounds of 20-40 mesh sand; 2,000 pounds of 10-30 mesh sand; 1,500 pounds of about 4-20 mesh particles of naphthalene; and 500 pounds of 10-30 mesh particles of a composition in accordance with the invention containing equal amounts of naphthalene and beta naphthol. This operation proceeded as follows:

- (1) Pumped 36 barrels of lease oil, with input pressure at 500 p.s.i. and pumping rate of 21 barrels per minute.
- (2) Pumped 8,000 gallons of fracturing oil carrying 8,000 pounds of 20-40 mesh sand, with pressure at 1,100 p.s.i.
- (3) Pumped 1,000 gallons of fracturing oil carrying 1,000 pounds of 20-40 mesh sand, 500 pounds of about 4-20 mesh particles of naphthalene and 500 pounds of 10-30 mesh particles of the naphthalene-beta naphthol composition, with pressure at 1,175 p.s.i.
- (4) Pumped 4,000 gallons of fracturing oil carrying 4,000 pounds of 20-40 mesh sand.
- (5) Pumped 1,000 gallons of fracturing oil carrying 1,000 pounds of 20-40 mesh sand and 1,000 pounds of about 4-20 mesh particles of naphthalene, with pressure at 1,350 p.s.i.
- (6) Pumped 11,000 gallons of fracturing oil carrying 9,000 pounds of 20-40 mesh sand and 2,000 pounds of 10-30 mesh sand, with pressure at 1,300 p.s.i.
- (7) Pumped 147 barrels of lease crude oil, with pressure at 1,200 p.s.i.
- (8) Stopped pumping. Pressure dropped to 500 p.s.i. in 12 minutes. Well was shut-in for 14 hours.
- (9) Swabbed 113 barrels of oil over period of 11 hours. Cleaned out 8 feet of fracturing sand.
- (10) Placed well on pump with initial daily production of 75 barrels of oil. After 13 days, load was recovered and daily production was 49 barrels of oil with some 20-40 mesh sand also being pumped.
- (11) After about 45 days the pump became sanded up and was pulled for cleaning. About 5 feet of sand was removed from the well when the tubing was pulled. However, there was no paraffin in the tubing, although some soft, easily removable paraffin was present in a back pressure valve in the pumping T.

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(12) After placing well on pump again, daily production was 25 barrels of oil.

Example No. 2

A well in Texas contained a pay zone extending from 3,218 feet to 3,266 feet, with a total depth of 3,271 feet and with 5½ inch pipe extending from the surface downwardly to 3,121 feet. The well had been completed in 1941 using 120 quarts of nitroglycerine.

Before fracturing and treating in accordance with the present invention, the static fluid level of this well was 70 feet. The well was first cleaned out to a total depth using a sand pump. Then 200 pounds of naphthalene was dissolved in 2,000 gallons of kerosene and this, after being heated to 180° F., was placed in the well in an effort to dissolve any paraffin that might have precipitated out of the formation oil and formed a sheath on the bore of the open hole. After cooling, the well was bailed in an attempt to remove the clean-up material. The oil removed appeared to have a heavy concentration of paraffin. The static fluid level after this operation was 225 feet of oil.

In the fracturing and treating operation which followed, the materials used were 9,000 gallons of fracturing oil; 8,000 pounds of 10-20 mesh sand; 3,000 pounds of 10-20 mesh sand blended with 1,000 pounds of 10-30 mesh particles of a composition in accordance with the invention containing equal amounts of naphthalene and beta naphthol; 500 gallons of a 15% non-emulsifying acid solution; 4,000 pounds of 20-40 mesh sand, and 2,000 pounds of grade "A" rock salt. This operation proceeded as follows.

First stage:

- (1) Pre-flushed well using 85 barrels of lease oil.
- (2) Pumped 1,000 gallons of fracturing oil carrying 2,000 pounds of 20-40 mesh sand.
- (3) Pumped 600 gallons of fracturing oil carrying 1,200 pounds of 10-20 mesh sand blended with 400 pounds of 10-30 mesh particles of the naphthalene-beta naphthol composition.
- (4) Pumped 2,000 gallons fracturing oil carrying 4,000 pounds of 20-40 mesh sand.
- (5) Flushed well using 60 barrels of lease oil.
- (6) Pumped 1,000 gallons of fracturing oil carrying 2,000 pounds of grade "A" rock salt.
- (7) Flushed well using 20 barrels of lease oil.
- (8) Pumped 500 gallons of 15% non-emulsifying acid solution.
- (9) Flushed well using 75 barrels of lease oil.

Second stage:

- (1) Pumped 1,000 gallons of fracturing oil carrying 2,000 pounds of 20-40 mesh sand.
- (2) Pumped 1,200 gallons of fracturing oil carrying 1,800 pounds of 10-20 mesh sand blended with 600 pounds of 10-30 mesh particles of the naphthalene-beta naphthol composition.
- (3) Pumped 2,000 gallons of fracturing oil carrying 4,000 pounds of 10-20 mesh sand.
- (4) Flushed well using 100 barrels of lease oil.

Due to the pressure limitation or rating of the well casing, it was desired to maintain the input pressure as nearly as possible at 1,000 p.s.i. or less throughout the job, with the result that the average injection rate was only 12.2 barrels per minute. During the first stage treatment, the input pressure ranged between 900 and 1,000 p.s.i. The rock salt appeared to function as a plugging material which caused the input pressure to increase to 1,100 p.s.i. During the second stage treatment, the pressure was maintained at 1,000 p.s.i.

After the treatment, approximately 35 feet of salt, sand and formation detritus was found in the open hole. Between 1,000 and 1,500 pounds of this material was subsequently cleaned out of the well.

It is normal procedure in this area for wells to be

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placed on vacuum immediately after completion of fracturing treatments. On this well, the pressure after completion of the treatment rapidly dropped from 1,000 to 700 p.s.i., then gradually decreased to 200 p.s.i. over a period of 50 minutes, whereupon the remaining pressure was lost. After a period of 48 hours, the static fluid level in the well was 500 feet.

Example No. 3

A well in Texas contained a pay zone extending from 3,060 feet to total depth at 3,158 feet, with 7 inch pipe extending from the surface downwardly to the top of the pay zone.

Before fracturing and treating in accordance with the invention, this well was cleaned out using cable tools and diesel oil. This was followed by an acid treatment using 250 gallons of 15% non-emulsifying acid solution. Then the well was pre-flushed using 120 barrels of water.

In the fracturing and treating operation which followed, the materials used were 20,000 gallons of gelled water; 12,000 pounds of 20-40 mesh sand; 50,000 pounds of 10-20 mesh sand; and 2,000 pounds of 10-20 mesh sand blended with 750 lbs. of 10-30 mesh particles of a composition in accordance with the invention containing equal amounts of naphthalene and beta naphthol. This operation proceeded as follows:

- (1) Pumped 4,000 gallons of gelled water carrying 12,000 pounds of 20-40 mesh sand.
- (2) Pumped 900 gallons of gelled water carrying 1,000 pounds of 10-20 mesh sand blended with 375 pounds of 10-30 mesh particles of the naphthalene-beta naphthol composition.
- (3) Pumped 8,000 gallons of gelled water carrying 25,000 pounds of 10-20 mesh sand.
- (4) Pumped 900 gallons of gelled water carrying 1,000 pounds of 10-20 mesh sand blended with 375 pounds of 10-30 mesh particles of the naphthalene-beta naphthol composition.
- (5) Pumped 8,000 gallons of gelled water carrying 25,000 pounds of 10-20 mesh sand.
- (6) Flushed with 130 barrels of water.

While the extent of long-term protection afforded as the result of treating the foregoing and other wells with the paraffin-control composition and method of the present invention cannot be determined with certainty until passage of a considerable period of time, preliminary reports indicate that the treatments were successful. In particular, it appears that the frequency of treatments necessary to control paraffin in the wells and related equipment will be much less using the present invention than it has been heretofore using prior art compositions and methods.

While the invention has been described herein with particular reference to certain embodiments and features thereof, it is understood that these are by way of example and not by way of limitation.

What is claimed as the invention is:

1. In a method of hydraulically producing fractures in underground strata penetrated by the bore of a well, the steps of; introducing into the well a fracturing and treating fluid containing a paraffin-control additive consisting essentially of discrete particles of a composition prepared by combining naphthalene and a less readily oil-soluble material from the group consisting of beta naphthol and anthracene and mixtures thereof, the ratio of said less readily oil-soluble material to said naphthalene being sufficiently large to reduce the rate of oil-solubility of the total additive material as compared to the rate of oil-solubility of naphthalene alone; causing said fluid to contact a section of the underground strata to be fractured; applying sufficient pressure to said fluid to fracture said strata section and inject therein fluid containing at least an appreciable quantity of the discrete particles of said paraffin-control additive, and the rate of

oil-solubility of said particles being sufficiently slow that a continuing control of paraffin is maintained.

2. The method of claim 1 wherein said particles are capable of passing a standard 4-mesh sieve, with from about 5% to about 95% being capable of passing a standard 10-mesh sieve and with not more than about 20% being capable of passing a standard 30-mesh sieve.

3. The method of claim 1 wherein substantially all of said particles range in particle size from those passing a standard 10-mesh sieve to those retained on a standard 30-mesh sieve.

4. In a method of hydraulically producing fractures in underground strata penetrated by the bore of a well, the steps of; introducing into the well a fracturing and treating fluid containing a paraffin-control additive consisting essentially of discrete particles of a composition prepared by combining naphthalene and beta naphthol in the ratio of about 1 to 1; causing said fluid to contact a section of the underground strata to be fractured; applying sufficient pressure to said fluid to fracture said strata section and inject thereinto fluid containing at least an appreciable quantity of the discrete particles of said paraffin-control additive.

5. A method of treating oil and gas wells and the like for inhibiting the formation of paraffin on surfaces past which liquid hydrocarbons are conducted, comprising the step of, introducing into the well a treating composition consisting essentially of naphthalene and a less readily oil-soluble material selected from the group consisting of beta naphthol and anthracene and mixtures thereof, the ratio of said less readily oil-soluble material to said naphthalene being sufficiently large to reduce the rate of solubility of said composition in liquid hydrocarbons as compared to the rate of solubility of naphthalene alone in liquid hydrocarbons and the rate of solubility of said treating composition being sufficiently slow thereby providing a continuing paraffin inhibiting treatment.

6. The method of claim 5 wherein said particles are within the general size range which includes those passing a standard 4 mesh sieve.

7. The method of claim 5 wherein said particles are within the general size range which includes those passing a standard 4 mesh sieve, with from about 5% to about 95% being of sufficiently small size to pass a standard 10 mesh sieve.

8. The method of claim 5 wherein said particles are within the general size range which includes those passing a standard 4 mesh sieve, with not more than about 20% being of sufficiently small size to pass a standard 30 mesh sieve.

9. The method of claim 5 wherein substantially all of said particles are within the size range which includes those passing a standard 10 mesh sieve but retained on a standard 30 mesh sieve.

10. A method of treating oil and gas wells and the like for inhibiting the formation of paraffin on surfaces past which liquid hydrocarbons are conducted, comprising the steps of; adding to a carrying liquid a treating composition consisting essentially of naphthalene and a less readily oil-soluble material selected from the group consisting of beta naphthol and anthracene and mixtures thereof, the ratio of said less readily oil-soluble material to said naphthalene being sufficiently large to reduce the rate of solubility of said composition in liquid hydrocarbons as compared to the rate of solubility of naphthalene alone in liquid hydrocarbons, said carrying liquid being substantially ineffective on the rate of solubility of said treating composition; introducing said carrying liquid into the well; and depositing said treating composition at a desired location in the well, and the rate of solubility of said treating composition being sufficiently slow thereby providing a continuing paraffin inhibiting treatment.

11. A method of treating oil and gas wells and the like

for inhibiting the formation of paraffin on surfaces past which liquid hydrocarbons are conducted, comprising the step of, introducing into the well a treating composition consisting essentially of naphthalene and beta naphthol in about equal amounts, and the rate of solubility of said treating composition being sufficiently slow thereby providing a continuing paraffin inhibiting treatment.

12. A method of treating oil and gas wells and the like for inhibiting the formation of paraffin on surfaces past which liquid hydrocarbons are conducted, comprising the steps of; adding to a carrying liquid a treating composition consisting essentially of naphthalene and beta naphthol in about equal amounts, said carrying liquid being substantially ineffective on the rate of solubility of said treating compositions; introducing said carrying liquid into the well; and depositing said treating composition at a desired location in the well, and the rate of solubility of said treating composition being sufficiently slow thereby providing a continuing paraffin inhibiting treatment.

13. A composition for controlling the formation of paraffin on surfaces past which liquid hydrocarbons containing such paraffin are conducted, consisting essentially of a mixture of naphthalene and beta naphthol, said composition being slowly soluble in liquid hydrocarbons, and the ratio of said beta naphthol to said naphthalene being sufficiently large to reduce the rate of solubility of the composition in liquid hydrocarbons as compared to the rate of solubility of naphthalene alone in liquid hydrocarbons.

14. A composition for controlling the formation of paraffin on surfaces past which liquid hydrocarbons containing such paraffin are conducted, consisting essentially of about equal amounts of naphthalene and beta naphthol and said composition being slowly soluble in liquid hydrocarbons.

15. A fracturing and treating fluid for use in oil and gas wells and the like consisting essentially of a carrying liquid, a quantity of oil-insoluble particles as spacing and propping agents, and a paraffin-control additive consisting essentially of particles of a material prepared by combining naphthalene and less readily oil-soluble beta naphthol, said carrying liquid being substantially ineffective on the rate of solubility of said paraffin-control additive, the ratio of said beta naphthol to said naphthalene being sufficiently large to reduce the rate of oil-solubility of the total additive material as compared to the rate of oil-solubility of naphthalene alone, and the rate of oil solubility of said paraffin-control additive being sufficiently slow that a continuing control of paraffin is maintained.

16. A fracturing and treating fluid as defined in claim 15 wherein substantially all of the particles of said naphthalene-beta naphthol material range in size from those passing a standard 10 mesh sieve to those retained on a standard 30 mesh sieve.

17. A fracturing and treating fluid for use in oil and gas wells and the like, consisting essentially of, a carrying liquid, a quantity of oil-insoluble particles as spacing and propping agents, and a paraffin-control additive consisting essentially of particles of a material prepared by combining naphthalene and less readily oil-soluble beta naphthol in a ratio of about 1 to 1, said carrying liquid being substantially ineffective on the rate of solubility of said paraffin-control additive.

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Paul L. Skolaut et al.

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 4, line 75, for "value" read -- valve --; column 5, line 13, for "naphtalene" read -- naphthalene --.

Signed and sealed this 15th day of January 1963.

(SEAL)

Attest:

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UNITED STATES PATENT OFFICE
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