

1

3,176,767

CONSOLIDATION OF INCOMPETENT EARTH FORMATIONS

Harry Brandt, Whittier, Barney R. Treadway, Brea, and Phillip H. Parker, Jr., San Rafael, Calif., assignors to California Research Corporation, San Francisco, Calif., a corporation of Delaware
No Drawing. Filed Aug. 22, 1962, Ser. No. 218,573
13 Claims. (Cl. 166—33)

This application is a continuation-in-part of copending application Serial No. 59,168, filed September 29, 1960, now abandoned.

The present invention relates to a method for the consolidation of incompetent earth formations, such as those adjacent to a well bore for the purpose of reducing or eliminating invasion of particulate matter into the well bore during fluid production from the formation.

A problem in the production of fluids, such as oil, gas and water, from an earth formation is the flow of fine earth particles, such as sands, silts and clays, from the surrounding incompetent formation into the well bore with the produced fluids. Particle invasion into a well bore is especially a problem when the formation comprises unconsolidated fine-grained sands and clay or silt particles, such as certain oil producing sands in California and in the Gulf Coast area. Loose earth particles get into pumping equipment, valves and pipes, causing costly erosion and often making them inoperative. Disposal of produced sand on the surface is also a problem. Further, a successful sand control method permits multiple well completions. In some oil producing fields, such as the Bay Marchand Field, in the Gulf Coast area several oil-bearing zones are present. These oil zones are located above each other and are often separated from each other by fluid-impermeable zones. A single well bore may penetrate several of the fluid-producing zones. To produce oil, gas or water from a single well bore but from several of these zones, a method has to be available that allows fluid production from all of the zones simultaneously without production of formation particles from any of the zones because equipment in the well bore necessary to produce from all of the zones usually becomes inoperative if formation particles flow into the well bore. It is estimated that several hundred existing wells of one oil-producing company alone could be completed into multiple producing zones if a satisfactory method of sand consolidation were available. A satisfactory sand consolidation method would also increase the number of new wells that could be produced economically because the new wells could be produced from multiple zones instead of from single zones. Involved in multiple zone production is a number of pipes or conduits within the casing, the length of the pipes depending on the zones to be tapped. Unconsolidated sand filling up the casing and the spaces between pipes makes it difficult, and often impossible, to move the pipes in and out of the casing as desired. Accordingly, if the sand can be consolidated within the formation, the sand would not move into the well bore and free movement of the pipes within the casing would remain unimpeded.

A number of processes have heretofore been proposed for the consolidation of incompetent oil-producing formations. A method of sand consolidation involves the steps of removing the film of water from the particles of the formation by employing a suitable agent, e.g. a surfactant, followed by coating the water-free particles with a cementing agent. A cementing agent is usually selected which is fluid when forced into the formation and then, after it is in place, sets up into a chemically unreactive oil-, gas-, and water-insoluble solid resin. If

2

desired, the fluid resin can be introduced into the formation together with the surfactant, whereby the sand particles are preferentially wetted by the cementing agent. When the formation has been permeated with the fluid resin, a flow of fluid is flushed through the formation to displace excess resin and to leave spaces or interstices between particles, thus providing a permeable mass for the flow of the production fluid, while leaving a sufficient amount of resin to bind the particles or grains of sand together.

Now in accordance with the present invention, it is proposed to consolidate an incompetent earth formation of loose earth particles adjacent to a bore-hole by a process involving the following essential steps of:

(1) Impregnating the formation with a liquid resinous mixture curable to the solid, insoluble, infusible state by treatment with a curing agent;

(2) Flushing the formation thus impregnated with an inert resin immiscible liquid to impart permeability to the treated formation, and leave a film of the resinous mixture around the earth particles; and then

(3) Contacting said film of resinous mixture surrounding the particles with a curing agent to render the film solid, insoluble, and infusible.

In carrying out the invention, therefore, there is used a cementing agent which is the thermosetting type of resin. It is the type of resin which, after it is in place, can undergo further reaction by treatment with a curing agent, such as a catalyst or cross-linking agent, which finally sets up the resin into the solid thermoset or insoluble, infusible state. Illustrations of such resinous materials are the partially polymerized or condensed resins such as phenol formaldehyde resins, urea formaldehyde, and melamine formaldehyde resins, which are finally cured by the reaction with a basic or acidic catalyst, such as formic acid, hydrochloric acid, oxalic acid, toluene sulfonic acid, trichloroacetic acid, quaternary ammonium compounds, primary and secondary amines sodium hydroxide, sodium carbonate, potassium hydroxide, barium hydroxide and ammonia; ethylenically unsaturated alkyd resins, i.e., unsaturated polyesters, such as those derived from a polyhydric alcohol, an α,β -ethylenically unsaturated aliphatic acid, such as a maleic, a portion of which is replaced, if desired, with a phthalic acid and/or a saturated aliphatic dibasic acid, and a monomer polymerizable therewith containing the group $\text{CH}_2=\text{C}<$, in an amount, for example, of 40 to 80 percent based on it and unsaturated polyester, the whole curable to the final solid state by treatment with a catalyst, such as a peroxy catalyst; epoxy resins, obtained by condensing epichlorohydrin with a polyhydroxy compound, the condensation product being finally cured to the solid, insoluble, infusible state by means of a curing cross-linking agent, such as an amine, a dicarboxylic acid anhydride or other resins, such as urea formaldehyde, and melamine formaldehyde.

In all instances, it is important that the catalyst or cross-linking curing agent be applied to the curable resin after the latter is in place in the formation, the curing agent being applied in an independent step, as will hereafter be more fully shown.

In carrying out the process of the invention, a curable resin of a type defined above is caused to premeate the formation by any of the conventional methods. Generally, sufficient resin is injected into the formation to impregnate it to a radial distance of from a few inches to five or more feet from the well bore or in an amount of two to sixty gallons of the aforesaid mixture per vertical foot of well hole in the formation to be treated. In order to prevent fracturing of the formation, the viscosity of the treating fluid mixture is kept low to reduce the

required injection pressure. Viscosities of the order of 5 to 1000 centipoises measured at 80° F. will generally be found satisfactory, a viscosity in the range of 5 to 200 centipoises at 80° F. being preferred.

In some instances, it may be desirable to treat the incompetent formation to remove water and thus render it preferentially wettable with the resinous mixture rather than with water. For this purpose, commonly available surfactants, such as sulfonated naphthenic acids, sulfonated higher alcohols and hydrocarbons, quaternary ammonium salts, silicones, and heavy metal soaps or organic liquids, such as alcohol, can be employed. When a surfactant is used, it can be mixed directly with the resin mixture or can be mixed with oil, water or other fluids and introduced into the formation as an independent preconditioning step prior to the injection of the resin mixture. Generally, an amount of surfactant ranging from 0.01% to 1%, by weight, based on the resin mixture is satisfactory.

When the formation has been treated with the aforesaid resin to the extent indicated, a resin immiscible flushing fluid is forced through the formation to render the formation permeable, but yet in an amount to leave the particles with a film of the consolidating resin sufficient to bind the incompetent particles into an aggregate mass. Considerable latitude is possible in the injection of flush fluid ranging from immediate application, after the resin is placed, to a delay of several days. The advantage of such flexibility will be readily apparent to those engaged in completing oil wells and can be considered a salient advantage of this process. The preferred inert flushing fluid is oil, and oil of like character to that to be recovered is eminently suitable. Other examples of inert flushing fluid are water, brine, refined oil, and various organic liquids not mutually compatible with the liquid resin. An amount of flushing fluid which is at least equal to the volume of resin mixture injected, up to ten volumes of the resin mixture, will in most cases be satisfactory, the preferred amounts being 1.5 to 4 volumes of flushing fluid per volume of the original resinous mixture introduced.

The resin mixture now in place in the formation is next treated with the curing agent, which may be characterized as a catalyst or cross-linking agent, as already mentioned. The curing agent is introduced into the formation following the flushing operation. Where this curing agent is soluble in oil, it can be injected into the formation in a solution of oil, such as that used for flushing, the total volume of this latter mixture being again at least equal to the volume of original resin mixture introduced into the formation. On the other hand, where the curing agent is soluble in water, it can be introduced as an aqueous solution, such as a caustic or acid solution used, for example, in the curing of a phenol-formaldehyde resin.

The following examples illustrate the practice of the invention.

Example I

Isophthalic acid, 157 pounds, and propylene glycol, 147.5 pounds, are reacted under an inert atmosphere at 200–205° C. until the resin acid number is below 5, whereupon 110 pounds fumaric acid and any propylene glycol lost are added, and the resulting mixture reacted for 20 hours at reflux temperature, beginning at 160° C. and gradually going up to 190° C. The final resin acid number is below 50. The reaction product, after cooling, is dissolved in 504 pounds N-vinyl-2-pyrrolidone containing 0.42 pound p-benzoquinone inhibitor.

The resinous mixture as thus prepared is introduced into the formation through a piping stringer placed in the well bore or casing, the stringer being provided with two packers. One packer is positioned just above the formation to be treated, and the other packer is positioned just below this formation, following which the resinous mixture

of unsaturated polyester and copolymerizing monomer is pumped down the stringer through perforations in the casing between the two packers and into the formation. Pump pressure is applied to force the resinous mixture into the formation. A column of flushing fluid is then introduced into the stringer and is pumped into the formation. After a sufficient amount of flushing fluid has removed the excess resin from the centers of the interstices of the formation and has left a film of resin at the grain surfaces, the pumping is discontinued, and the resin is allowed to set up into an oil-insoluble, infusible solid. The final set of the resin is then effected by pressuring another column of oil with catalyst into the formation. After a reasonable set time, e.g., eight hours, the well is allowed to produce.

Example II

Unconsolidated sand of about 150 mesh is placed in a tube of the Hassler sleeve type. The permeability of the sand pack is measured by flowing oil through the pack, and is found to be 4.05 darcys. The sand is then saturated completely with water. Oil with viscosities of 113 and 12 centistokes at room temperature and 210° F., respectively, is flowed through the pack at a flow rate of about four feet per hour. The sand pack is then heated to 180° F.

A sand pack saturated with both oil and water at elevated temperature approximates, for the purpose of consolidation experiments, the conditions of the formation around an oil producing well. To consolidate the sand, five pore volumes of polyester resin are flowed through the pack, the resin being obtained from one part isophthalic acid and one part maleic anhydride, esterified to 1600 molecular weight with propylene glycol, and diluted with an equal weight of N-vinyl-2-pyrrolidone, the resin mixture containing in addition 0.05 percent by weight of diphenyl quinone for inhibition, and 0.3 percent cobalt naphthenate for cure acceleration. The polyester resin is followed by five pore volumes of oil, which in turn is followed by two pore volumes of a 6 percent by weight solution of methylethylketone peroxide used to effect final cure. The sand pack is shut in for about 16 hours and is kept at a temperature of 180° F. and at a pressure of 5000 p.s.i. At the end of this period the sand is found to be consolidated. The permeability of the consolidated sample is 1.45 darcys, which is about 35.8 percent of the original permeability. The compressive strength of the consolidated sample at ambient pressure and temperature is 3000 p.s.i.

To prepare the sample for aging tests, the sample is extracted in toluene. Sections of the sample are boiled in typical oil field brines for a period of about one month. Other sections of the consolidated sample are maintained at a temperature of 330° F. in both brine and oil for a period of about one month. In both the aging experiments the sand remains consolidated.

Example III

An oil well provided with a casing reaching into the earth a distance of 6000 feet and perforated into an incompetent formation, which is capable of producing sand along with the oil, is treated with 500 gallons of the polyester-monomer resin mixture described above, by introducing the resin into a tubing string having packers set just above and below the formation. 1500 gallons of crude oil flushing fluid is then introduced into the tubing string and an injection pressure of 500 p.s.i.g. is maintained across the formation for the eight hours required for injection and flushing. One thousand gallons of oil containing 0.5 pound per gallon of methylethylketone peroxide catalyst is then placed into the formation. The well is then shut in for eight hours to allow the resin to reach maximum strength. After the treatment, the well produces sand-free oil, with approximately 25 to 50 percent of the permeability of the formation being retained.

Example IV

A liquid phenol-formaldehyde resin is prepared as follows:

Formaldehyde (37 percent in water), 670 parts; phenol, 515 parts; and caustic (33 parts of sodium hydroxide in 33 parts of water) are mixed together and then heated to 175° F. After about 2½ hours at this temperature, hydrochloric acid (32 percent by weight), 26.2 parts, is added to the solution, thereby lowering the pH to somewhat less than 6. The resulting two-phase mixture is separated, the upper layer discarded, and the lower layer retained for sand consolidation.

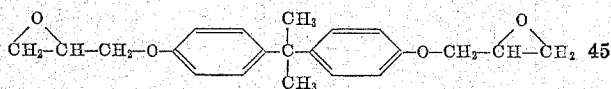
A Hassler sleeve type tube is charged, as previously described in Example II, with sand, oil and water and maintained at 180° F. To consolidate this sand, a portion of the phenol-formaldehyde resin prepared as above and amounting to 5 pore volumes is poured through the sand pack. This is first followed by an equal volume of oil of 25 centistokes (77° F.) viscosity, and then by one-half of this volume of the same oil containing HCl gas in about 10 percent concentrations. After this treatment the sand pack is shut in for 12 hours at a temperature of 180° F. and at a pressure of 5000 p.s.i. At the end of this time the sand is found to be consolidated and to have about 40 percent of its original permeability.

Example V

To a Hassler tube prepared as previously described in Examples II and IV, there is charged 5 pore volumes of a mixture of phenol and furfuraldehyde (28/72 weight ratio). This material is flushed with another 5 pore volumes of diesel oil. Finally, the same type of flushing oil, containing about 4 percent by weight of formic acid is charged to the tube. After this treatment, the sand pack is shut in for 18 hours at 180° F. and at a pressure of 5000 p.s.i. At the end of this time the sand is found to be consolidated to an insoluble, strong, permeable mass.

Example VI

To the same type of Hassler tube previously described, there is charged 5 pore volumes of an epoxy resin having the following formula:



The sand pack is flushed with 5 pore volumes of diesel oil. Another 2 pore volumes of this same type oil containing 4 percent by weight of dimethylaminopropylamine are charged to the core which is then shut in for 16 hours, at 180° F. and at 5000 p.s.i. This procedure results in an insoluble, strong, permeable sand core capable of passing crude oil at about 50 percent of its former volume per unit time.

We claim:

1. Process for consolidating an incompetent earth formation of loose earth particles adjacent to a borehole, which comprises impregnating the formation with a liquid resinous mixture curable to the solid, insoluble and infusible state by treatment with a curing agent, flushing the formation thus impregnated with an inert, resin immiscible liquid in proportions of 1 to 10 volumes per volume of said resinous mixture to impart permeability to the treated formation and leave a film of the resinous mix-

ture around the earth particles, then contacting said film of resinous mixture with a curing agent to render the film solid, insoluble and infusible.

2. Process according to claim 1, which includes pre-treating the loose earth particles to render them preferentially wettable by the liquid resinous mixture.

3. Process according to claim 1, wherein the liquid resinous mixture is a mixture of unsaturated polyester and ethylenically unsaturated monomer containing the group $\text{CH}_2=\text{C}<$.

4. Process according to claim 1, wherein the liquid resinous mixture is an epoxy resin.

5. Process according to claim 4, wherein the curing agent is an amine.

6. Process according to claim 1 wherein the proportions of inert liquid range from 1.5 to 4 volumes per volume of resinous mixture.

7. Process according to claim 1, wherein the liquid resinous mixture is partially polymerized phenol aldehyde resin.

8. Process according to claim 7, wherein the curing agent is formic acid.

9. Process for the cementing together in a permeable aggregate of loose earth particles contained in an incompetent formation adjacent to a borehole for the recovery of oil, which comprises impregnating the formation with a liquid resinous mixture of unsaturated polyester and an ethylenically unsaturated oil-insoluble monomer containing the grouping $\text{CH}_2=\text{C}<$ copolymerizable therewith to an insoluble, infusible resin, said mixture having a viscosity in the range of 0.05 to 10 poises at 80° F., flushing the so-treated formation with petroleum oil in proportions of 1 to 10 volumes per volume of said liquid resinous mixture, and thereafter injecting into the said formation a further quantity of petroleum oil containing 0.1 to 5 percent, by weight, based on the mixture, of a polymerization peroxy catalyst.

10. Process according to claim 9 wherein the proportions of petroleum oil range from about 1.5 to 4 volumes per volume of liquid resinous mixture.

11. Process according to claim 9, wherein the unsaturated monomer is N-vinyl-2-pyrrolidone.

12. Process according to claim 9, wherein the peroxy catalyst is methylethylketone peroxide.

13. Process according to claim 12, which includes pre-treating the formation with a surfactant to remove water, after which the formation is impregnated with the resinous mixture.

References Cited by the Examiner

UNITED STATES PATENTS

2,252,271	8/41	Mathis	166—33
2,770,306	11/56	Clark	166—33
2,815,815	12/57	Hower et al.	166—33 X
2,823,753	2/58	Henderson	166—33 X
2,987,499	6/61	Werner	260—45.4
3,047,067	7/62	Williams et al.	166—33
3,097,692	7/63	Holland et al.	166—33
3,100,527	8/63	Hilton et al.	166—33

FOREIGN PATENTS

230,393 9/60 Australia.

CHARLES E. O'CONNELL, Primary Examiner.