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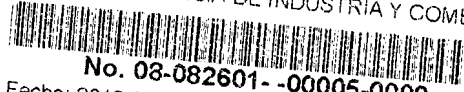
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SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO



No. 08-082601- -00005-0000

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REF: Expediente No. **08-082601**
Patente de Invención: **"EMULSIONES DE AGENTES CON
CONSOLIDACION Y METODOS
ASOCIADOS"**
Fase Nacional: **PCT/GB2007/000421**
Titular: **HALLIBURTON ENERGY SERVICES
INC.**

RESPUESTA A REQUERIMIENTO OFICIO No. 6801 NOTIFICADO POR ESTADO DE 27 DE ABRIL DE 2012.

HUMBERTO RUBIO CAMACHO, mayor de edad, domiciliado en Bogotá, D.C., identificado con la cédula de ciudadanía número 17'192.062 de Bogotá, abogado graduado portador de la Tarjeta Profesional No. 50.007 expedida por el Consejo Superior de la Judicatura, actuando en carácter de Apoderado de **HALLIBURTON ENERGY SERVICES INC.**, domiciliado en P.O. Box. 1431, Duncan, Oklahoma, Estados Unidos de America; encontrándome dentro de la debida oportunidad procesal para dar respuesta al auto proferido por su Despacho, mediante Oficio No.6801, notificado por fijación en lista 27 de abril de 2012, me permito:

Manifiestar:

1.- En el informe oficial emitido sobre la presente solicitud, se han emitido las objeciones que a continuación se resumen:

a.- De las Reivindicaciones:

- La reivindicación 1 no es clara porque utiliza términos indefinidos tales como "al menos, pluralidad", los cuales son imprecisos.

- Las Reivindicaciones 4-3, 11-12, 18-19 no son claras porque utilizan términos indefinidos tales como "aproximadamente, alrededor", los cuales son imprecisos.

- Las reivindicaciones 1, 10 y 16 no son concisas porque a pesar que estás reivindicaciones de métodos se han elaborado por separado como reivindicaciones independientes, están relacionadas de manera efectiva con la misma materia y se diferencian entre si solo en cuanto a la definición del objeto para el que se solicita protección. El solicitante debe limitar su pliego de reivindicaciones.

- La reivindicación 17 no es clara porque utiliza términos indefinidos tales como "al menos, una porción", los cuales son imprecisos.

La reivindicación 17 no es clara porque se refiere a una etapa de modificación de la superficie de la formación subterránea que es un resultado a alcanzar por la aplicación de las partículas recubiertas, no definen al compuesto y no queda definida por el resultado. Se sugiere remplazarlo por las características esenciales.

b.- Determinación del Estado de la Técnica:

Examen de patentabilidad:

Novedad:

La reivindicación 1 consiste en un método que comprende las etapas de:.....

Las características esenciales de la composición habían sido divulgadas previamente en D1.

Las reivindicaciones 3- 7, 9 no mencionan alguna característica que haga nueva a la composición de la reivindicación 1, por lo que no se considera nueva ya que D1 divulga una amina como surfactante.

La reivindicación 10 consiste en un método que comprende ciertas etapas y no es nueva, según lo divulgado en el documento D1.

Nivel Inventivo:

La reivindicación 2 consiste en que el método de la reivindicación 1 comprende además las etapas de colocar los particulados recubiertos de la emulsión de un agente de consolidación en un fluido de fracturación y colocar el fluido de facturación en una formación subterránea a una presión suficiente para crear o mejorar, al menos, una o mas facturas de la misma.

Los documentos D1 y D2 se consideran el estado de la técnica cercano a la invención definida en la reivindicación 2.

Las reivindicaciones 2, 8, 14 y 21 cumplen con el requisito de novedad pero no cumplen con el requisito de nivel inventivo.

2.- En contestación a las objeciones anteriores, manifestamos lo siguiente:

a.- De las reivindicaciones

Acompañamos a esta respuesta un nuevo juego de reivindicaciones (1-17), todas sustentadas en el contenido de la solicitud originalmente presentada, el cual no ha sido ampliado en modo alguno.

Las expresiones “al menos” “aproximadamente, alrededor” “al menos, una porción” objetadas se han eliminado de las nuevas reivindicaciones.

b.- Novedad y Nivel Inventivo:

Con las limitaciones introducidas en las nuevas reivindicaciones aquí presentadas, respetuosamente considero que la invención ha quedado claramente distinguida a partir de los documentos del estado de la técnica citados D1 y D2.

Aún más, la invención proporciona un método para incrementar la temperatura de una sección de un conducto utilizado para la producción o transmisión de hidrocarburo que no era conocido.

Un experto en el campo técnico podría incluir agentes adhesivos no acuosos, con los antecedentes que se han citado, pero con el nuevo pliego de reivindicaciones no es posible anticipar la obtención de lo reivindicado en la presente solicitud ni de los resultados que se obtiene a partir de ellos.

Un ejemplo claro de que la invención es patentable y satisface plenamente los requisitos de patentabilidad, es que una patente análoga para la misma se ha concedido para el territorio Americano, bajo el número de patente US 7624743 sin

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que se presentaras oposiciones a dicha patente.

Por todo lo anteriormente argumentado solicito que, sobre la base de las nuevas reivindicaciones que aquí se adjuntan, se proceda a la concesión de la patente colombiana correspondiente.

Teniendo en cuenta lo expuesto consideramos que la presente solicitud es novedosa tiene nivel inventivo, las nuevas reivindicaciones definen la materia que se desea proteger siendo claras, concisas y debidamente sustentadas en la descripción y no está comprendida en el estado de la técnica, conforme lo establece el Art. 14, 16 y 30 de la Decisión 486.

ANEXO:

- (i) Capitulo reivindicatorio.
- (ii) Declaración realizada por el Inventor

PETICIÓN:

Conforme a lo anterior, una vez atendidos los requerimientos y exigencias del examinador y al tener en cuenta el nuevo capítulo de reivindicaciones que se presenta, así como los argumentos expuesto, solicito muy respetuosamente a su Despacho, continuar el trámite de rigor y de ser el caso se hagan las correspondientes observaciones con al ánimo de hacer lo pertinente dentro del presente trámite, teniendo en cuenta que la presente solicitud cumple con las exigencias establecidas en la D486

Atentamente,



HUMBERTO RUBIO CAMACHO

C.C. 17'192.062 de Bogotá

**T.P. 50.007 Consejo Superior
de la Judicatura**

COD. 4134

REIVINDICACIONES:

1. Un método para incrementar la temperatura de una sección de un conducto
5 utilizado para la producción o transmisión de hidrocarburo, el método que comprende las etapas de:
 - (a) Formación de un tratamiento de fluido que comprende:
 - (i) Un fluido conductor que comprende agua; y
 - 10 (ii) Un primer reactivo y un segundo reactivo, en donde:
 - (1) El primer reactivo comprende una fuente de iones de amonio y el segundo reactivo comprende una fuente de iones de nitrito
 - (2) El primer y segundo reactivo están en por lo menos 5
15 molares de concentración en el fluido conductor; y
 - (3) por lo menos uno de los primeros reactivos y el segundo reactivo esta suspendido en un fluido conductor en una forma solida.
 - (b) Introducir el tratamiento del fluido en una sección del conducto
20 utilizado para la producción o transmisión de hidrocarburo.
2. El método de acuerdo con la reivindicación 1, en donde el estado sólido comprende al menos dos formas sólidas diferentes.
- 25 3. El método de la reivindicación 1, en donde la forma solida comprende un material particulado que tiene un tamaño de malla 20-40.
4. El método de la reivindicación 1, en donde el fluido conductor comprende un agente de viscosidad.
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5. El método de la reivindicación 1, en donde el fluido conductor comprende un surfactante no-iónico.
6. El método de la reivindicación 1, en donde el fluido conductor además
35 comprende: un catalizador para una reacción química exotérmica entre el primer reactivo y el segundo reactivo

7. El método de la reivindicación 6, además comprende la etapa de: retrasar la liberación del catalizador para ayudar a retrasar el comienzo de una reacción entre el primer reactivo y el segundo reactivo.
- 5 8. El método de la reivindicación 7, en donde la etapa de retraso del catalizador comprende: encapsulamiento del catalizador.
9. El método de la reivindicación 7, en donde el catalizador comprende un ácido.
- 10 10. Un método de acuerdo con la reivindicación 9, en donde el ácido es seleccionado del grupo que consiste en ácido acético y ácido cítrico y el ácido está en una concentración de 3% por peso.
- 15 11. El método de acuerdo con la reivindicación 9, que además comprende la etapa de: formación de un tratamiento de fluido con un tampón.
12. El método de acuerdo con la reivindicación 1, en donde la formación sólida comprende: tener uno de los primeros reactivos y el segundo reactivo empotrado en una matriz sólida con un material polimérico.
- 20 13. El método de acuerdo con la reivindicación 1, en donde la formación sólida comprende: tener una de las primeras sustancias reactivas y el segundo reactivo encapsulado con un material encapsulador.
- 25 14. El método de acuerdo con la reivindicación 13, en donde el material encapsulador es un material que libera un reactivo encapsulado a una temperatura superior de 49° C.
- 30 15. El método de acuerdo con la reivindicación 14, en donde el material encapsulado comprende alcohol polivinilo.
16. El método de acuerdo con la reivindicación 1, que adicionalmente comprende las etapas de: aislamiento de la sección conductora; y en donde la etapa de introducción del tratamiento del fluido en el conducto, adicionalmente comprende: la introducción en la sección aislada del conducto.
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17. El método de acuerdo con la reivindicación 1, que adicionalmente comprende la etapa de: desplazar el tratamiento del fluido de la sección del conducto.

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No. : 11/521,530
Applicant : Diptabhas Sarkar
Filed : 09/14/2006
TC/A.U. : 1792
Docket No. : 2005-IP-018796U1
Customer No. : 01224
Confirmation No. : 1544

DECLARATION OF DIPTABHAS SARKAR

01. My name is Diptabhas Sarkar. My current residence address is 3354 Rogerdale Rd., #1037, Houston, Texas 77042. I am over 21 years of age, of sound mind, and competent to make this Declaration.

02. All the statements made in this Declaration made on personal knowledge are true, or, if made on information and belief, are believed to be true.

03. I have a Doctor of Engineering Science in colloid chemistry from Columbia University, New York. A copy of my curriculum vitae and list of publications are attached.

04. I have been employed by Halliburton Energy Services, Inc., since January 6, 2004, as a Senior Research Scientist in its Research Group. Among other oilfield research activities, I have worked in its Pipeline and Process Services Group.

A. SCOPE OF REVIEW

05. In making this Declaration, I have reviewed and considered the following: (a) the above-referenced application as originally filed on September 14, 2006 (hereinafter the "Application"); (b) at least pages 16 – 29 of the Office Action dated February 6, 2009 (hereinafter the "Office Action"); and (c) at least the specific citations to the following references as discussed in this Office Action: De Souza, U.S. Patent No. 6,003,528; Characklis, U.S. Patent No. 4,561,981; McGinity, U.S. Patent No. 5,051,261; Nürnberg, U.S. Patent No. 5,382,601;

Thesing, U.S. Patent No. 6,830,105; Nimerick, U.S. Patent No. 4,669,544; Ashton, U.S. Patent No. 4,755,230; and Reddy, U.S. Patent No. 6,444,316.

06. I have reviewed the following from the current Manual of Patent Examining Procedure ("MPEP"), Eighth Edition, August 2001, Latest Revision July 2008, § 716.04 (published online by the U.S. Patent and Trademark Office):

**"I. THE CLAIMED INVENTION MUST SATISFY A LONG-
FELT NEED WHICH WAS RECOGNIZED, PERSISTENT,
AND NOT SOLVED BY OTHERS**

Establishing long-felt need requires objective evidence that an art recognized problem existed in the art for a long period of time without solution. The relevance of long-felt need and the failure of others to the issue of obviousness depends on several factors. First, the need must have been a persistent one that was recognized by those of ordinary skill in the art. *In re Gershon*, 372 F.2d 535, 539, 152 USPQ 602, 605 (CCPA 1967) ("Since the alleged problem in this case was first recognized by appellants, and others apparently have not yet become aware of its existence, it goes without saying that there could not possibly be any evidence of either a long felt need in the . . . art for a solution to a problem of dubious existence or failure of others skilled in the art who unsuccessfully attempted to solve a problem of which they were not aware."); *Orthopedic Equipment Co., Inc. v. All Orthopedic Appliances, Inc.*, 707 F.2d 1376, 217 USPQ 1281 (Fed. Cir. 1983) (Although the claimed invention achieved the desirable result of reducing inventories, there was no evidence of any prior unsuccessful attempts to do so.).

Second, the long-felt need must not have been satisfied by another before the invention by applicant. *Newell Companies v. Kenney Mfg. Co.*, 864 F.2d 757, 768, 9 USPQ2d 1417, 1426 (Fed. Cir. 1988) (Although at one time there was a long-felt need for a "do-it-yourself" window shade material which was adjustable without the use of tools, a prior art product fulfilled the need by using a scored plastic material which could be torn. "[O]nce another supplied the key element, there was no long-felt need or, indeed, a problem to be solved".)

Third, the invention must in fact satisfy the long-felt need. *In re Cavanagh*, 436 F.2d 491, 168 USPQ 466 (CCPA 1971).

“II. LONG-FELT NEED IS MEASURED FROM THE DATE A PROBLEM IS IDENTIFIED AND EFFORTS ARE MADE TO SOLVE IT

Long-felt need is analyzed as of the date the problem is identified and articulated, and there is evidence of efforts to solve that problem, not as of the date of the most pertinent prior art references. *Texas Instruments Inc. v. Int'l Trade Comm'n*, 988 F.2d 1165, 1179, 26 USPQ2d 1018, 1029 (Fed. Cir. 1993).

“III. OTHER FACTORS CONTRIBUTING TO THE PRESENCE OF A LONG-FELT NEED MUST BE CONSIDERED

The failure to solve a long-felt need may be due to factors such as lack of interest or lack of appreciation of an invention's potential or marketability rather than want of technical know-how. *Scully Signal Co. v. Electronics Corp. of America*, 570 F.2d 355, 196 USPQ 657 (1st. Cir. 1977).

See also Environmental Designs, Ltd. v. Union Oil Co. of Cal., 713 F.2d 693, 698, 218 USPQ 865, 869 (Fed. Cir. 1983) (presence of legislative regulations for controlling sulfur dioxide emissions did not militate against existence of long-felt need to reduce the sulfur content in the air); *In re Tiffin*, 443 F.2d 344, 170 USPQ 88 (CCPA 1971) (fact that affidavit supporting contention of fulfillment of a long-felt need was sworn by a licensee adds to the weight to be accorded the affidavit, as long as there is a bona fide licensing agreement entered into at arm's length).

B. ESTABLISHMENT OF LONG-FELT NEED

1. Recognized Problem in the Art for a Long Period Without Solution

07. In my opinion, including based on the scope of my review, an art-recognized problem existed in the art for a long period of time without solution.

a. The Need Was Recognized by Those of Ordinary Skill in the Art

08. Paraffin wax buildup in a section of conduit used for the production or transmission of hydrocarbon is a decades-old and costly problem in the oil and gas industry. For example, the scope of this problem is briefly outlined in the “Background” section of the Application, Paragraphs 0002 – 0012, and illustrated in Figure 1.

09. Removal of the paraffin wax deposits has been attempted through three main approaches: mechanical, thermal, and chemical, as briefly outlined in the “Background” section of the Application, Paragraphs 0013 – 0019. Numerous attempts have been made to remediate paraffin wax buildup in hydrocarbon-bearing subterranean formations and in conduits for the production and transmission of hydrocarbon. Application, Paragraph 0020. *See also*, for example, the references identified in the “Background” section of the Application, Paragraphs 0021– 0033.

10. As discussed in the Application, one of the oldest attempts to solve this problem employs heated oil, so the technique is commonly referred to as “hot oiling.” A heated fluid is introduced into a production tubular or pipeline to help remove paraffin deposits. See, for example, the outline of this type of attempt in the “Background” section of the Application, Paragraphs 0016 – 0017.

11. An art-recognized problem associated with introducing a heated fluid through a conduit, such as a production tubular or especially a long pipeline, however, is that, as the heated fluid moves through the conduit, it cools to a temperature that is ineffective for the remediation of paraffin wax buildup. This can be particularly problematic in subsea pipelines because the surrounding water on the seafloor is very cold, typically about 39°F (4°C), as briefly discussed in the “Background” section of the application, Paragraphs 0010 – 0011.

i. Recognized Need for Delayed Heating

12. The prior attempts to solve the problem of paraffin wax buildup in a conduit used for the production or transmission of hydrocarbon in the oil and gas industry include using various thermochemical approaches for delayed heating. In general, these approaches attempt to carry reactant chemicals to a remote portion of the conduit, which make a highly exothermic chemical reaction to produce heat at a desired remote location in the conduit.

13. Numerous attempts to delay the exothermic reaction to produce delayed heating have been made to remediate paraffin wax buildup in hydrocarbon-bearing subterranean formations and in conduits for the production and transmission of hydrocarbon. See, for example, the references identified in the “Background” section of the Application, Paragraphs 0022 – 0024, 0026 – 0031 and 0033.

14. For example, De Souza, U.S. Patent No. 6,003,528 issued Dec. 21, 1999, teaches a method for removing paraffin in “transmission conduits which are longer than 10 and up to 50,000 meters” (up to about 30 miles). De Souza, Column 1, lines 9 – 10. De Souza recognized “the onset of the reaction of heat and nitrogen generation should be strongly delayed,” in order for the reaction to take place at the desired location in a long conduit. *See* De Souza, Column 2, lines 61 – 62.

15. Further, as discussed in Ashton, U.S. Patent No. 4,755,230 issued July 5, 1988, another prior reference recognized the need for “retarding” the reaction.

U.S. Pat. No. 4,219,083 to RICHARDSON, et al, [issued August 26, 1980] describes a process for cleaning well casing perforations by injecting an aqueous solution containing nitrogen-gas-generating reactants, an alkaline buffer *providing a reaction-retarding pH, in order to trigger a fast-driving pulse of heat and pressure. . . .*

Ashton, Column 2, lines 34 – 41 (*emphasis added*).

ii. Additional Recognized Need to Avoid Heat Spike

16. To delay the initiation of the exothermic reaction, De Souza utilized (a) an emulsion to separate the reactant chemicals until breaking of the emulsion, and (b) a copolyanhydride, which upon hydrolysis releases the acid catalyst necessary to initiate the exothermic reaction. However, when the emulsion breaks and the pH drops to 5 or below due to the release of the acid catalyst, the exothermic reaction then proceeds very quickly, which releases the vast majority of the heat from the reaction in the form of a heat spike. *See generally*, De Souza, Figures 1 – 5, which indicate a “heat spike.”

17. The use of a delayed-action catalyst for delaying the initiation (or “onset”) of such an exothermic reaction has a similar problem. Once a critical concentration of the acid catalyst is reached (at a pH of 5 or below), the exothermic reaction proceeds at an uncontrolled rate, which quickly releases the vast majority of the heat from the reaction in the form of a “heat spike.” *See, e.g.*, Ashton, Column 2, lines 34 – 41.

18. In addition to the need for delay, the problem of a “heat spike” was recognized in the art.

19. For example, Ashton, U.S. Patent No. 4,755,230 issued July 5, 1988, recognized problems associated with the generation of a heat spike, including in a discussion of prior patents to Richardson.

A later patent to RICHARDSON, et al, U.S. Pat. No. 4,399,868, employs the same reactive ingredients as were utilized in the earlier patent to generate nitrogen gas, to not only generate nitrogen gas but, by the exothermic reaction, produce heating of a solvent solution which is introduced into the production zone of a well to effect the dissolution of paraffin and similar deposits from the perforations of the production zone.

These operations are, however, complicated by the presence of a large amount of brine solution which is normally found in a producing zone of a well. *Additionally, the solvent solution is heated to temperatures on the order of 440°F. Temperatures of this order of magnitude can be tolerated in the production zone of a well, but would generate excessive stresses if employed in a hydrocarbon transmission conduit, such as a pipeline.* More importantly, in such patented processes, the well's ambient temperature was stated to be on the order of 175°F., thus preventing any dissolved paraffins from precipitating out of solution in crystalline form.

U.S. Pat. No. 4,219,083 to RICHARDSON, et al, describes a process for cleaning well casing perforations by injecting an aqueous solution containing nitrogen-gas-generating reactants, an alkaline buffer providing a reaction-retarding pH, and an acid-yielding reactant for subsequently overriding the buffer and lowering the pH in order *to trigger a fast-driving pulse of heat and pressure which causes a perforation-cleaning backsurge of fluid through the perforations. Again, this method, as disclosed, might produce undesirable stresses if applied to the cleaning of a pipeline and would not prevent the reprecipitation of the paraffins as the temperature falls, particularly in a subsea pipeline.*

Ashton, Column 2, lines 14 – 46 (*emphasis added*). Moreover, Aston states, “The maximum heat generated by the exothermic reaction is preferably on the order of about 175°F. Temperatures in excess of this figure would represent a threat to the integrity of the pipeline covering, due to expansion of the metal constituting the pipeline.” Ashton, Column 13, lines 58 – 63.

20. The Application also states this problem of a heat spike was recognized in the art. “However, this chemical reaction [between ammonium chloride and sodium nitrite] is known to proceed at a high rate, which has been difficult to control.” Application, Paragraphs 0064 – 0065.

b. The Need Was Recognized for a Long Period of Time

21. This Application was filed on September 14, 2006.

22. The need for both delay of the initiation of the reaction to provide delayed heating and to control the rate of reaction to avoid a heat spike were recognized in the art for at least two decades prior to the filing date of this Application, and probably much longer.

23. For example, De Souza recognized "the onset of the reaction of heat and nitrogen generation should be strongly delayed," in order for the reaction to take place at the desired location in a long conduit. *See* De Souza, Column 2, lines 61 – 62. De Souza was filed on December 19, 1997.

24. In addition, as discussed above, Ashton recognized both the need for delay in the initiation of the exothermic reaction and the desirability of avoiding a heat spike. Ashton, Column 2, lines 14 – 46. Ashton has a priority date of January 15, 1985, more than 20 years before the filing date of the Application.

c. The Need Was Persistent

25. As pointed out above, paraffin wax buildup in a section of conduit used for the production or transmission of hydrocarbon is a decades-old and costly problem in the oil and gas industry. Upon information and belief, the long-felt need described above that is associated with remediating paraffin wax buildup in a conduit used for the production or transmission of hydrocarbon has been persistent since it was recognized in the art.

2. The Need Was Not Satisfied by Another before the Invention

26. None of the references discussed in the Office Action dated February 6, 2009, satisfy the long-felt need to help control both the delay of the initiation of the exothermic reaction and thereafter to help control the rate of the reaction in the thermochemical treatment of a conduit for the transmission of hydrocarbon in the oil and gas industry.

27. De Souza, discussed above, does not satisfy this long-felt need to be able to help control both the initiation and then the rate of the reaction. To delay the initiation of the exothermic reaction, De Souza utilized (a) an emulsion to separate the reactant chemicals until breaking of the emulsion, and (b) a copolyanhydride, which upon hydrolysis releases the acid

catalyst necessary to initiate the exothermic reaction. However, when the emulsion breaks and the pH drops to 5 or below due to the release of the acid catalyst, the exothermic reaction then proceeds very quickly, which releases the vast majority of the heat from the reaction in the form of a heat spike. *See generally*, De Souza, Figures 1 – 5, which indicate a heat spike.

28. Ashton, also discussed above, does not satisfy this long-felt need. As stated in Ashton:

Lastly, a buffered pH modifier, such as a buffered solution of HCl, or sodium hydroxide, is added to the aqueous solution to provide a pH sufficiently low so as to retard the heat and nitrogen-generating reaction *so that no significant portion of the reaction occurs until* the conduit containing the deposits is filled with the emulsified mixture of the aqueous and solvent solutions.

... When the heat and nitrogen-producing reaction is completed, the temperature of the solution contained in the pipeline falls rapidly to the ambient temperature which, in the case of a subsea pipeline, is on the order of 40°F.

Ashton, Column 2, lines 8 – 14 and 26 – 30 (*emphasis added*). Based on my understanding of this language as a chemist and the rest of the disclosure, Ashton is directed to controlling the critical concentration of the catalyst (acidic pH) through a buffering capacity to delay the initiation of the exothermic reaction, but does not control the rate of reaction after the onset to avoid a heat spike. For example:

The amount of, for example, buffered acid added to the tank as a pH modifier is largely dependent upon the distance of the major deposit of paraffin precipitate in the pipeline from the access connection through which the cleaning solution is introduced. . . . A computerized calculation has been developed to indicate the pH value required *to delay the substantial initiation of the chemical reaction between the sodium nitrite and the ammonium nitrate until the inserted solutions have had time to fill up the isolated pipeline section 1 and surround the precipitate layer P*. While this calculation can be performed by anyone ordinarily skilled in this art, the details of the calculation are set forth below in the interest of completeness of disclosure. In such calculation and the working examples below, the phrase "N-SITU" has been employed to represent the aqueous solution of the reactants *which react exothermically to produce nitrogen gas and a heat spike*. N-SITU is a Trademark for products and services associated therewith of the Assignee.

In order to locate the heat spike in the pipeline generated by the injection of an N-SITU and paraffin solvent mixture, it is necessary to determine the physical properties of the mixture.

Ashton, Column 5, line 66 – Column 6, line 25, referring to Figure 1 (*emphasis added*). Further, Ashton later states:

From these calculations, the pH value of the aqueous reactant solution to produce the heat spike at the location of the paraffin precipitate P can be determined.

It should be noted that the buffered control of the pH value of the intermixed reacting solutions does not effect the total amount of heat generated nor the total amount of nitrogen gas liberated. This is determined solely by the relative proportions of the reacting components contained within the emulsified aqueous solutions. The pH value is selected at a sufficiently high level *to delay* the speed of the reaction so that no significant part of the reaction occurs *until* the fluid has filled up the isolated section of pipeline and is in contact with the precipitated layer P of paraffin.

Ashton, Column 10, line 62 – Column 11, line 7, referring to Figure 1 (*emphasis added*). Again, based on my understanding of this language and the rest of the disclosure, Ashton is directed to controlling the critical concentration of the catalyst (acidic pH) through a buffering capacity to delay the initiation of the exothermic reaction, but does not control the rate of reaction to avoid a heat spike. On the contrary, Ashton describes locating and producing “the heat spike at the location of the paraffin precipitate.” Ashton, Column 2, lines 8 – 14 and 26 – 30; Ashton, Column 5, line 66 – Column 6, line 25; and Ashton, Column 10, line 62 – Column 11, line 7, referring to Figure 1. Once the reaction begins, the total amount of heat energy produced is limited solely by the relative proportions of the reacting components. The rate of the reaction is uncontrolled. Ashton does not satisfy the long-felt need.

29. Characklis, U.S. Patent No. 4,561,981, issued December 31, 1985, discloses: “A method for controlling, preventing, or removing fouling deposits comprising microencapsulating fouling control chemicals in a slow release capsule. The microcapsules are applied to the substrate to be treated, where they stick to the substrate and slowly release the active treating agents.” Characklis, Abstract. Characklis does not satisfy the long-felt need to help control both the initiation of the exothermic reaction and the rate of the reaction after the onset for paraffin removal in a conduit used in the transmission of hydrocarbon in the oil and gas industry. In fact, Characklis teaches away from a solution to the long-felt need:

By applying these fouling control chemicals in the form of a microcapsule, the chemical is transported to the reaction sites where it will be most effective without its decay or removal by undesirable side reactions in the fluid phase.

When the capsule absorbs to (or absorbs in) the fouling deposit, *the chemical will be released in the fouling deposit. In this matter, very little residual treating chemical will become reentrained in the bulk fluid phase* where it will be either consumed by undesirable side reactions or transported to the environment.

This method of dealing with fouling will result in significantly lower requirements of a given chemical treating agent to solve a given fouling problem, as it eliminates the wasteful side reactions which occur when the chemical is merely dumped into the fluid milieu. . . .

For each application, the capsules must be engineered to maximize their sticking efficiency to the specific deposit. . . .

Characklis, Column 2, Lines 40–56 (*emphasis added*).

30. Reddy, U.S. Patent No. 6,444,316 issued September 3, 2002 discloses: “Encapsulated water soluble chemicals for use in controlled time release applications” Reddy, Abstract. Reddy discloses an “improved method of cementing a pipe string in a well bore” and an “improved method of dissolving drilling fluid filter cake and the like in well bores penetrating subterranean producing formations” Reddy, Column 2, lines 25 – 26 and lines 43 – 45, respectively. However, Reddy does not satisfy the long-felt need to help control both the initiation of the exothermic reaction and the rate of the reaction after the onset for paraffin removal in a conduit used in the transmission of hydrocarbon in the oil and gas industry. Further, as disclosed in Reddy, other types of encapsulation were known before Reddy. *See* Reddy, Column 8, line 29 – Column 10, line 17. In addition, other types of encapsulation of various chemicals were known, as shown by Characklis dating back to at least 1985.

31. Nimerick, U.S. Patent No. 4,669,544 issued June 2, 1987 discloses a method of contacting a surface with a sulphur trioxide containing liquid to react with the paraffin, and convert at least a portion of it to a water-dispersible material. Nimerick does not satisfy the long-felt need to help control both the initiation of the exothermic reaction and the rate of the reaction after the onset for paraffin removal in a conduit used in the transmission of hydrocarbon in the oil and gas industry.

32. Thesing, U.S. Patent No. 6,830,105 issued December 14, 2004, provides a method “relate[d] to proppant flowback control (also known as proppant back production control).” Thesing, Column 1, lines 8 – 9. Thesing does not satisfy the long-felt need to help control both the initiation of the exothermic reaction and the rate of the reaction after the onset for paraffin removal in a conduit used in the transmission of hydrocarbon in the oil and gas industry.

33. Nürnberg, U.S. Patent No. 5,382,601 issued January 17, 1995 and McGinity, U.S. Patent No. 5,051,261 issued September 24, 1991 are directed to the field of pharmaceutical dosage compositions. Neither of these references satisfies the long-felt need to help control both the initiation of the exothermic reaction and the rate of the reaction after the onset for paraffin removal in a conduit used in the transmission of hydrocarbon in the oil and gas industry.

3. The Invention In Fact Satisfies the Long-Felt Need

34. Claim 1 is being currently amended to clarify that which is regarded as the invention, as supported by the description in the Application, including, for example, in Paragraph 0042. The amended claim provides:

“1. A method for increasing the temperature of a section of conduit used for the production or transmission of hydrocarbon, the method comprising the steps of:

- (a) forming a treatment fluid comprising:
 - (i) a carrier fluid;
 - (ii) a first reactant and a second reactant, wherein:
 - (1) the first reactant and second reactant are selected for being capable of reacting together in an exothermic chemical reaction;
 - (2) the first and second reactant are in at least sufficient concentrations in the carrier fluid to generate a theoretical heat of reaction of at least 1,000 kJ/liter of the treatment fluid; and
 - (3) at least some of at least one of the first reactant and second reactant is suspended in the carrier fluid in a solid form that is adapted to help control the initial release of the reactant into the carrier fluid and the rate of the release of the reactant into the carrier fluid; and
- (b) introducing the treatment fluid into a section of conduit used for the production or transmission of hydrocarbon.

35. The Application provides several examples of how a solid form can be employed to help control the initiation of the exothermic reaction and the rate of the reaction in a section of conduit used in the production or transmission of hydrocarbon. Application, Paragraphs 0064 – 0129.

36. The invention satisfies the long-felt need to help control both the initiation of the exothermic reaction and the rate of the reaction for paraffin removal in a conduit used in the transmission of hydrocarbon in the oil and gas industry. For example, the Application provides:

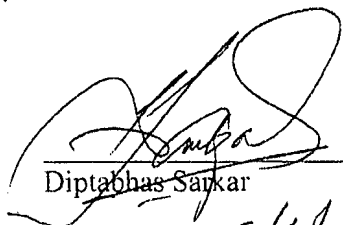
The solid form is adapted to help control the initial release of the reagent into solution and/or the rate of release of the reagent into solution. This helps control the initiation and/or the rate of reaction between the reactants, respectively, which helps control when the heat of reaction is released. As described in the detailed description of the invention, several different examples of techniques are provided for having at least one of the reactants in a solid form, which are adapted to control the mixing of the reactants in a carrier fluid and the generation of the heat of reaction.

Application, Paragraph 0042 (*emphasis added*). Also, for example, the Application, Paragraph 0087, it states: "[t]he purpose is to have the heat of reaction released, whether at an even rate or in stages, so that a major spike in the release of heat energy is avoided, but that substantially all of the heat energy is released within a matter of hours."

C. CONCLUSION

37. In my opinion, the invention as currently claimed in the Application satisfies a long-felt need not solved by others and the invention as claimed is not obvious in view of the cited references, separately or in any combination.

38. I hereby declare that all statements made herein of my own knowledge are true, that all statements made on information and belief are believed to be true, and further that these statements are made with the knowledge that willful false statements and the like are punishable by fine or imprisonment, or both, under U.S.C. Title 18 §1001, and that such willful false statements may jeopardize the validity of the application, and patent issuing thereon, or any patent to which this verified statement is directed.



Diptabhas Sarkar
6/5/09

Date

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EXPERIENCE

Halliburton

Senior Research Scientist

Houston, TX

January 2004 – Present

Pipelines and Process Services Group

- Product Development Lead for chemical technology related to Flow Assurance
 - Developed formulations for delayed heat generation for paraffin removal in seabed pipelines; Commercialization currently under way
 - Developed customer presentations, educated field development personnel and supported business development process around this technology platform.
 - Filed three patent applications protecting this technology offering.
 - Collaborated with Princeton University on understanding paraffin deposition in cold surfaces. Key role included knowledge exchange to facilitate transfer of academic knowledge to field operations.
 - Managed PPS group's presence in the Center for Hydrates Research, premier industry-university research consortium dedicated towards hydrates related flow assurance problems at Colorado School of Mines.
 - Participated in the Rice University based Brine Chemistry Consortium– dedicated towards understanding scale related flow assurance problems; Taught key field personnel in the use of scale prediction software. Knowledge developed applied to water services project in Halliburton.
- Serving as the chemicals lead in a virtual team as special support for field operations across the globe
 - Developing “greener” versions of existing chemicals portfolio for use in European Union
 - Identifying key components in current formulations. Replacing hazardous ones with new greener alternatives to develop “best of class” technology offering.
 - Developing application notes for field operations personnel.
- Developing non biocide based water disinfection systems for hydrotesting applications
- Feasibility study to develop FPSO based water treatment system for pipeline hydrotesting applications.

Stimwatch Group

- Developed key thermal tracer based on technology developed for flow assurance activity for distributed temperature sensing applications.
 - Led application in two jobs and successfully implemented the technology in field; Generated revenue of \$300K in 2007.
 - Published SPE paper in 2008 describing technical and commercial success
 - Created and delivered training sessions on new technology to four business development managers and three on-site groups for product growth.

Technology Ventures Group

- Selected to manage Halliburton's research efforts in the prestigious Advanced Energy Consortium (AEC).
 - Vision of AEC is to facilitate pre-competitive research in use of nanotechnology to improve hydrocarbon production.
 - Manage technical inputs to and from five research projects, bringing in key industry inputs to facilitate research activity.
 - Focus on bringing knowledge back to industry to develop competitive offering.
- Halliburton water services project
 - Developed screening methodology for evaluation of multiple water treatment technologies for Marcellus, Woodford and Bakken shale formations.
 - Managed third party technology development program with Palo Alto Research Center (PARC). Worked on identifying key parameters and technology transfer between Halliburton and PARC
 - Led "Voice of Customer" interviews to understand technical needs and design attributes of appropriate research and development projects for improved water treatment services.
- Analyzed intellectual property (IP) landscape including 'freedom to operate' assessments for initiation of large scale R&D projects.
- Performed technical due diligence study and supported negotiations for licensing of technology in diverse sectors of upstream oilfield services.

Production Enhancement Group

- Research scientist on a project to understand of fluid loss as applied to drilling, cementing and fracturing.

- Researched novel chemistries and filtering mechanisms in controlling the behavior of filter cakes
 - Collaborated with Harvard University on “Casing While Drilling” project. Studied penetration of polyacrylamide gel balls in microfluidic devices. Studies led to two patents.
 - Research led to development of novel fluid loss control agents.
- Project management leader for Industry-University research project with an University in India
 - Initiated Halliburton’s first research project in Asia; Pilot project aimed at identifying human resource potential and technical capability w.r.t energy services research
 - Defined research program and budget; Established effective working relationship

Columbia University
Post Doctoral Fellow

New York, NY
May 2002 – December 2003

- Worked for IUCRC (Industry University Co-Operative Research Center for advanced Studies in Novel Surfactants) in Columbia University on industrial projects
 - Worked closely with the senior scientists from industry on key projects involving use of Surface Plasmon Resonance Technology. Research led to multiple presentations at Conferences.
 - Developed routes for selective dispersion and/or flocculation of nanosized colloidal systems (aqueous slurries of mineral oxides) of varying solids loading; technique was used to synthesize of core-shell nanocomposites.
- Supported submission of research proposals to the NSF and NIH on development of sensor networks for toxin detection.
- Supervised junior graduate students on their dissertation work

Opticology Inc.
Independent Scientific Consultant

New York, NY
May 1999 – January 2004

- Scientific lead of an interdisciplinary venture team to develop healthcare equipment, based on sensor platform developed during doctoral work.

- Worked on developing instrument for dry-eye quantification; Secured \$100k funding under NIH's Small Business Innovation Research (SBIR) program.
 - Received one patent for research activities.

EDUCATION

Columbia University, Henry Krumb School of Mines, New York, NY
Doctor of Engineering Science, August 1998 – May 2002
 Major: Colloid and Interfacial Chemistry

- Thesis Title: Interfacial Dynamics of Macromolecules using Surface Plasmon Resonance Spectroscopy
 - Independently built instrument to monitor dynamic interfacial behavior of polymers and surfactants under external perturbation at short time scales
 - Systems studied include polymers and proteins, along with their interactions with various surfactants of like and opposite charge
- Published 6 papers in peer reviewed journals
- Recipient of Best Teaching Assistant Award, 1999
- Received grant of \$100,000 from NSF, in 2nd year of graduate studies

Indian Institute of Technology, Department of Chemistry, Kanpur, India
Master of Science (M.Sc.), August 1996 – May 1998
 Major: Inorganic Chemistry; Minor: Physical Chemistry

- Cumulative Performance Index (CPI) 9.2/10; Graduated in top 1% of class

Jadavpur University, Department of Chemistry, Calcutta, India
Bachelor of Science (B.Sc.), August 1993 – May 1996
 Major: Chemistry; Minor: Physics, Mathematics

- Graduated in top 5% of class; Student volunteer for 82nd Indian Science Congress

ACTIVITIES AND RECOGNITION

- 2009** Selected to evaluate American Chemical Society, Petroleum research fund proposal
- 2009** Selected to evaluate Society of Petroleum Engineers research paper
- 2009** Patent Recognition award by Opticology Inc.
- 2008** Patent Recognition Award at Halliburton
- 2007** Patent Recognition Award at Halliburton
- 2007** Patent Recognition Award at Halliburton
- 2001 – 2002** Awarded Graduate Research Assistantship at Columbia University
- 2001** Awarded Teaching Assistantship for Solids and Hazardous Waste Management at Columbia University
- 2000** Awarded Teaching Assistantship for Introduction to Colloid Chemistry at Columbia University
- 2000** Awarded Extraordinary Teaching Assistant Award at Columbia University
- 2000** Won funding of \$100,000 as a second year graduate student at Columbia University, for proposal submitted to National Science Foundation under the National Nanotechnology Initiative.
- 1998 – 2000** Awarded Graduate Research Assistantship at Columbia University
- 1998 – 2002** Evaluated National Science foundation proposals as Graduate student
- 1998 – 2002** Evaluated manuscript submission to the following journals, as a graduate student: Langmuir, Journal of Colloid and Interfacial Science, Colloids and Surfaces A

PUBLICATIONS

Patents

Patent Numbers (USPTO application numbers where applicable) have been provided. Please note there were 8 technical patent filings. Other patents are multiple applications in foreign jurisdictions.

- 1) **Sarkar, Diptabhas**, Arrington, Stephen T., Powell, Ronald J., Robb, Ian D., Todd, Bradley L.,
“Methods and compositions for thermally treating a conduit used for hydrocarbon production or
transmission to help remove paraffin wax build up”, US20080066784 A1, **2008**
- 2) **Sarkar, Diptabhas**, Arrington, Stephen T., Powell, Ronald J., Robb, Ian D., Todd, Bradley L.,
“Methods and compositions for thermally treating a conduit used for hydrocarbon production or
transmission to help remove paraffin wax build up”, US20080069961 A1, **2008**
- 3) **Sarkar, Diptabhas**, Arrington, Stephen T., Powell, Ronald J., Robb, Ian D., Todd, Bradley L.,
“Methods and compositions for thermally treating a conduit used for hydrocarbon production or
transmission to help remove paraffin wax build up”, US20080067108 A1, **2008**
- 4) Robb, Ian D., Saini, Rajesh K., **Sarkar, Diptabhas**, Todd, Bradley L., Griffin, James M.,
“Stimuli-degradable gels”, US7306040 B1, **2007**
- 5) Robb, Ian D., Saini, Rajesh K., **Sarkar, Diptabhas**, Todd, Bradley L., Griffin, James M.,
“Stimuli-degradable gels”, US20070281870 A1, **2007**
- 6) Robb, Ian D., Saini, Rajesh K., **Sarkar, Diptabhas**, Todd, Bradley L., Griffin, James M.,
“Stimuli-degradable gels”, US20070277981 A1, **2007**
- 7) **Sarkar, Diptabhas**, Robb, Ian D., Todd, Bradley L., “Treatment fluids and methods using
zeolite and a delayed release acid for treating a subterranean formation” US20070123433 A1,
2007

- 8) **Sarkar, Diptabhas**, Arrington, Stephen T., Powell, Ronald J., Robb, Ian D., Todd, Bradley L.,
“Methods and compositions for thermally treating a conduit used for hydrocarbon production or
transmission to help remove paraffin wax build up”, EP2061949 A1, **2008**
- 9) **Sarkar, Diptabhas**, Robb, Ian D., Todd, Bradley L., “Treatment fluids and methods using
zeolite and a delayed release acid for treating a subterranean formation”, EP1969082 A1, **2008**
- 10) Cappel, Anthony P. Bennett, Gregory, Gilbard, Jeffrey P., Gavaris, Paul T., **Sarkar, Diptabhas**,
“Device for measuring concentrations of constituents of tear sample”, US20090005660 A1, **2009**
- 11) Cappel, Anthony P. Bennett, Gregory, Gilbard, Jeffrey P., Gavaris, Paul T., **Sarkar, Diptabhas**,
“Device for measuring concentrations of constituents of tear sample”, WO2009032297 A2, **2009**
- 12) **Sarkar, Diptabhas**, Arrington, Stephen T., Powell, Ronald J., Robb, Ian D., Todd, Bradley L.,
“Methods and compositions for thermally treating a conduit used for hydrocarbon production or
transmission to help remove paraffin wax build up”, WO2008032067 A1, **2008**
- 13) **Sarkar, Diptabhas**, Robb, Ian D., Todd, Bradley L., “Treatment fluids and methods using
zeolite and a delayed release acid for treating a subterranean formation”, WO2007063315 A1,
2008

Peer Reviewed Journals

- 1) Dilip Kumar Chakravorty, Rozi Ghosh, Rupendranath Banerjee and **Diptabhas Sarkar***;
“Kinetics of ligand exchange between 2-thenoyltrifluoroacetone and zirconium-lactate solution
in weakly acidic lactate buffers”; *Polyhedron*, 28(7), 1315-1319, **2009**
- 2) **Diptabhas Sarkar**, P. Somasundaran, “Conformational Dynamics of Poly(acrylic acid). A Study
Using Surface Plasmon Resonance Spectroscopy” *Langmuir*, 20(11); 4657-4664, **2004**

- 3) **Diptabhas Sarkar**, P. Somasundaran, “Polymer surfactant kinetics using surface plasmon resonance spectroscopy dodecyltrimethylammonium chloride/polyacrylic acid system “ *Journal of Colloid and Interface Science*, 261(1), 197 – 205, **2003**
- 4) **Diptabhas Sarkar**; P. Somasundaran; “Overcoming Contamination in Surface Plasmon Resonance Spectroscopy” *Langmuir*, 18(22), 8271-8277, **2002**
- 5) P. Somasundaran, T. Chen, **Diptabhas Sarkar**,” A novel processing scheme for core – shell nano composites using controlled polymer adsorption”, *Materials Research Innovations*, 2(6), 325 – 327, **1999**
- 6) P. Somasundaran, S. Simpson, I. Ivanov, R. K. Jain, **Diptabhas Sarkar**; “Thinning and Rupture of Aqueous Surfactant Films on Silica” *Langmuir*, 15, 7658 – 7661, **1999**

Book Chapters

- 1) P. Somasundaran, T. Chen, **Diptabhas Sarkar**, “Core-shell Nanocomposites—New Materials for Functional Fillers”, *Functional Fillers and Nanoscale Minerals*, Ed. Keller, Herpfer, Moudgil, 77 – 83, **2003**
- 2) **Diptabhas Sarkar**, “Interfacial dynamics of macromolecules using surface plasmon resonance spectroscopy”, *Doctoral Dissertation*, **2002**

Invited Conference Papers and Other Conference Proceedings

- 1) Jose Sierra, Jiten Kaura, Dan Gualtieri, Gerard Glasbergen, **Diptabhas Sarkar**, and David Johnson, "DTS Monitoring Data of Hydraulic Fracturing: Experiences and Lessons Learned", SPE 116182, SPE Annual Technical Conference and Exhibition, Denver, Colorado, USA, 21–24 September 2008
- 2) Somasundaran P, Liu F, **Sarkar Diptabhas**, Gryte C. C.; "Cleansing and Release by Novel Nanogel VCarriers", *J Cosmet Sci.*, Mar-Apr 55(2); 222-3, 2004
- 3) **Diptabhas Sarkar**, P. Somasundaran, ; "Dynamics of adsorption of polymers with different loop size and their unfolding with surfactants studied using surface plasmon resonance spectroscopy" *Proc. of the 225th ACS National Meeting*, March 23-27, New Orleans, LA, 2003
- 4) P. Somasundaran, Z. Pan, **Diptabhas. Sarkar**; *Proc. of Mineral Processing Technology*, Goa, India, 2003
- 5) P. Somasundaran, T. Chen, **Diptabhas Sarkar**; *Society of Mineral Engineers Annual Meeting, Symposium Proc.*, 2003
- 6) **Diptabhas Sarkar**, P. Somasundaran, ; *Proc. of the 224th. American Chemical Society National Meeting*, Boston, August 18-22, 2002
- 7) P. Somasundaran, F. Liu, **Diptabhas Sarkar** ; *Proc. of International Symposium on Nanomaterials and Technology*, Beijing, China, July 2-5, 2001
- 8) P. Somasundaran, T. Chen, **Diptabhas Sarkar**, S. Shrotri; *Proc. of the American Soc. for Composites, Technical Conference 16th*, 646-651, 2001